JPL Publication 90-30

Proceedings of the First Workshop on Containerless Experimentation in Microgravity

January 17–19, 1990

E. H. Trinh
Editor

May 1, 1990

NASA
National Aeronautics and
Space Administration

Jet Propulsion Laboratory
California Institute of Technology
Pasadena, California

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This publication was prepared by the Jet Propulsion Laboratory, California Institute of Technology, under a contract with the National Aeronautics and Space Administration.

ABSTRACT

The First Containerless Experimentation in Microgravity Workshop was held January 17–19 in Pasadena, California. The workshop organizers' principal goals were first to provide scientists from academia and from industrial and government research institutions an opportunity to acquaint themselves with the past, current, and future scientific investigations carried out in the Containerless Science programs of the Microgravity Science and Applications Division of the National Aeronautics and Space Administration as well as the European and the Japanese Space Agencies. The second goal was to assess the ongoing technological development program for low gravity containerless experimentation instruments. The third goal was to obtain recommendations concerning rigorous but feasible new scientific and technological initiatives for space experiments using noncontact sample positioning and diagnostics techniques. The specific output of the workshop is an initial set of recommendations for the development of the technical capabilities of future space-based instrumentation. The concensus among the workshop attendees is that there seems to be a strong support for experimental investigations in microgravity and that this support would be strengthened by closer interaction between the user community and the flight equipment developers. Such a cooperation would help ensure that priorities are assigned to scientific returns.

ACKNOWLEDGMENTS

I would like to thank Professors Robert Bayuzick and William Johnson for providing the scientific leadership of this workshop in their capacity as technical program co-chairmen. I am also grateful to Professor John Perepezko for chairing the pre-workshop panel meeting, which determined the organization and thrust of the workshop. I thank all the following workshop panel members for their valuable technical contribution: Professor Hal Brody, Dr. Ared Cezairliyan, Dr. Daniel Elleman, Dr. Edwin Ethridge, Dr. Robert Hauge, Dr. William Hofmeister, Dr. Marc Lee, Dr. Paul Nordine, Professor Taylor Wang, Professor Michael Weinberg, and Dr. George Nielsen.

This workshop was funded by the Microgravity Science and Applications Division of the National Aeronautics and Space Administration. Special thanks goes to Dr. Marc Lee, the Program Scientist, and Mr. Larry Spencer, the Program Manager, for their continued support of this endeavor.

I also want to express my gratitude to Ms. Pat McLane and Mr. Andrew Morrison for their imaginative and skillful handling of the organization and logistics required for this workshop.

SUMMARY OF PROCEEDINGS CONTENTS

The workshop format was determined by a committee of scientists who participated in the current ground-based and flight programs during a preworkshop meeting organized by the Jet Propulsion Laboratory. A decision was made to emphasize open scientific discussions yet still allow the presentation of short technical papers during splinter sessions that were divided according to the following discipline groups:

- Thermophysical properties of materials and very high temperature chemistry
- Containerless materials processing in microgravity. (Benchmark materials development, quiescent undercooled melts nucleation studies, and exploratory investigations of protein and other novel crystals growth.)
- Fluid dynamics and interfacial phenomena.

The workshop program is included in Appendix C and shows the various workshop sessions, beginning with invited speakers overview presentations on the first day, splinters sessions presentations and discussions on the second day, and a summary plenary session on the third day. This proceedings contains the abstracts and some of the presentation materials from most of the papers presented during the overview and splinter sessions. A summary of the discussions and recommendations from the splinter sessions is also included.

The workshop was attended by approximately eighty scientists and engineers (see Appendix B for attendance list). Also included is a summary describing past, current, and future experimental instrumentation. A survey form was sent to all potential attendees prior to the workshop. This form was used to gather information on the potential experimental capabilities requirements of individual scientists and is included in Appendix A.

TABLE OF CONTENTS

EXECUTIVE SUMMARY

The Containerless Experimentation in Microgravity Workshop hosted by the Jet Propulsion Laboratory (JPL) convened January 17–19, 1990 in Pasadena, California. Three basic issues were addressed by the attendees during the two and a half days of technical presentations and discussions. The first issue dealt with the scientific justification for carrying out long-duration containerless experiments in low gravity. The second topic addressed the specific scientific requirements to be levied upon the currently offered flight equipment. The third area centered around the future needs for scientific experimental facilities that would be used in microgravity.

For the purpose of this workshop, the various relevant technical disciplines involved in containerless experimentation were divided by topic into three basic groups:

- Very high-temperature chemistry and thermophysical properties
- Materials processing in low gravity (fundamental studies and benchmark materials development)
- Fluid physics and interfacial phenomena

Attendees agreed that in all three areas there was a strong justification for pursuing specific experimental investigations. The following requirements for studies in materials science and fluid physics can be accomplished in the microgravity environment:

- Larger samples observation for a longer period of time
- Reduced convection/sedimentation
- High-temperature processing of nonconducting melts
- Free liquid surfaces and elimination of boundary effects
- More accurate measurement results due to long-term observation
- Quiescence of melt, reduction of effects of external forces
- Potential for optimal vibration isolation
- Potential for elimination of melt shape distortion
- Control of processing environment/contamination reduction

Potentially fruitful scientific investigations were identified in the areas of high-temperature thermophysical properties measurement, deep undercooling of melts and metastable solid phase formation, melt purification, rheology of surfaces, thermocapillary phenomena, nonlinear dynamics of free drops, phase transitions and metastable liquid phases properties, convection-free transport phenomena, and, finally, novel processing techniques.

A general need to increase the versatility of currently offered flight instrumentation in terms of high-temperature capability (2000 to 3000 K) as well as of the diagnostics (property measurement and environment control and monitoring) was expressed by the majority of the attendees. The currently available Drop Physics Module (DPM) was found to be adequate as a first iteration for studies in fluid physics, but the need for more versatile diagnostic instrumentation was clearly noted. The temperature range of operation of the DPM was not satisfactory to most of the materials scientists in attendance. The capabilities of the electromagnetic positioner Tempus were also found in need of improvement in terms of sample temperature measurement and of optical access to the specimen under investigation. Some of the needed improvements were found to be in the specific areas of the control of the environment and of the heating and cooling rates (sample quenching), the flexibility to accommodate samples of different properties, the capability for processing a larger number of samples, the availability of a versatile optical diagnostic capability, and the capability to measure a wide range of thermophysical properties.

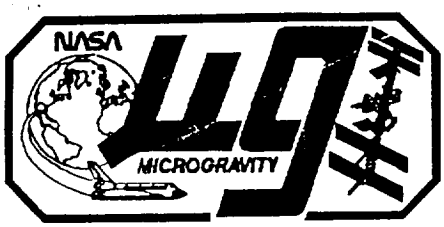
Future requirements for containerless experimentation in microgravity must begin with the nurturing and maintenance of a strong ground-based research program that will support the development and evaluation of flight equipment. The development of various specific facilities was requested, but a recurrent theme was the need for the development and implementation of noncontact thermophysical properties measurement techniques for both ambient as well as high-temperature applications. High-temperature processing facilities for refractory metals, alloys, semiconductors, and nonconducting materials were placed as high priority items. Finally, a majority of the scientists in attendance held the view that an alternate carrier capability should be developed either for long-duration, low perturbation unmanned facility or for frequent-flight low-cost microgravity experiments of shorter duration.

There seemed to exist a strong support for experimental investigations in microgravity, and it was believed that this support would be strengthened by closer interaction between the user community and the flight equipment developers. Such a cooperation would help ensure that the relevant capabilities are included in the flight instrumentation, and that the appropriate priorities are assigned to scientific returns.

OVERVIEW SESSIONS

Invited Speakers Papers

N91-21332



Office of Space Science and Applications
Microgravity Science and Applications Division

Charge of

the Containerless Experimentation in Microgravity

Workshop

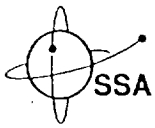
Pasadena Hilton

Pasadena, California

January 17-19, 1990

Mark C. Lee
NASA Headquarters

90-1-12-WHW



Containerless Experimentation in Microgravity

1. Elimination/Reduction of Surface Contamination
   - Adequate Earth-based technology

2. Reduction of dynamic nucleation
   - Paucity of reliable data

90-1-12-1-WHW

Containerless Experimentation
in Microgravity

Objectives

- To delineate scientific justification for the U.S. Containerless Experimentation Program in Microgravity for the next decade and beyond

- To guide NASA to define the next generation of containerless experimentation instruments in microgravity

90-1-12-2-WHW

Containerless Experimentation in Microgravity

Pre-Workshop Panel Meeting

Held at Caltech on August 16, 1989

Chairman: Professor John Perepezko

Members:

Prof. R. Bayuzick	Vanderbilt University
Prof. H. Brody	University of Pittsburgh
Dr. A. Cezairliyan	NIST
Dr. D. Elleman	JPL
Dr. E. Ethridge	MSFC
Dr. R. Hauge	Rice University
Dr. W. Hofmeister	Vanderbilt University
Prof. W. Johnson	Caltech
Dr. M. Lee	NASA Headquarters
Dr. P. Nordine	CPI
Dr. E. Trinh	JPL
Prof. T. Wang	Vanderbilt University
Dr. M. Weinberg	University of Arizona

90-1-12-3-WHW

Objectives of
Pre-Workshop Panel Meeting

1. To recommend to full workshop pertinent science and technology areas for discussion

2. To organize and structure full workshop

3. To take ownership of the full workshop

90-1-12-4-WHW

Containerless Experimentation
in Microgravity

Recommendations for Discussion from Pre-Workshop Panel

1. Fluid dynamics (surface tension/thermocapillary at T < 200 °C)
2. Thermophysical properties (diffusion at extremely high temperatures, viscosity and surface tension)
3. Benchmark materials
4. Very high temperature chemistry for nonconducting materials
5. Quiescent undercooled melt nucleation study
6. Exploratory growth of protein and other novel crystals
7. Diffusional interactions of gas-particle dispersion
8. Development/verification of processing modeling

90-1-12-5-WH-W

Containerless Experimentation
in Microgravity

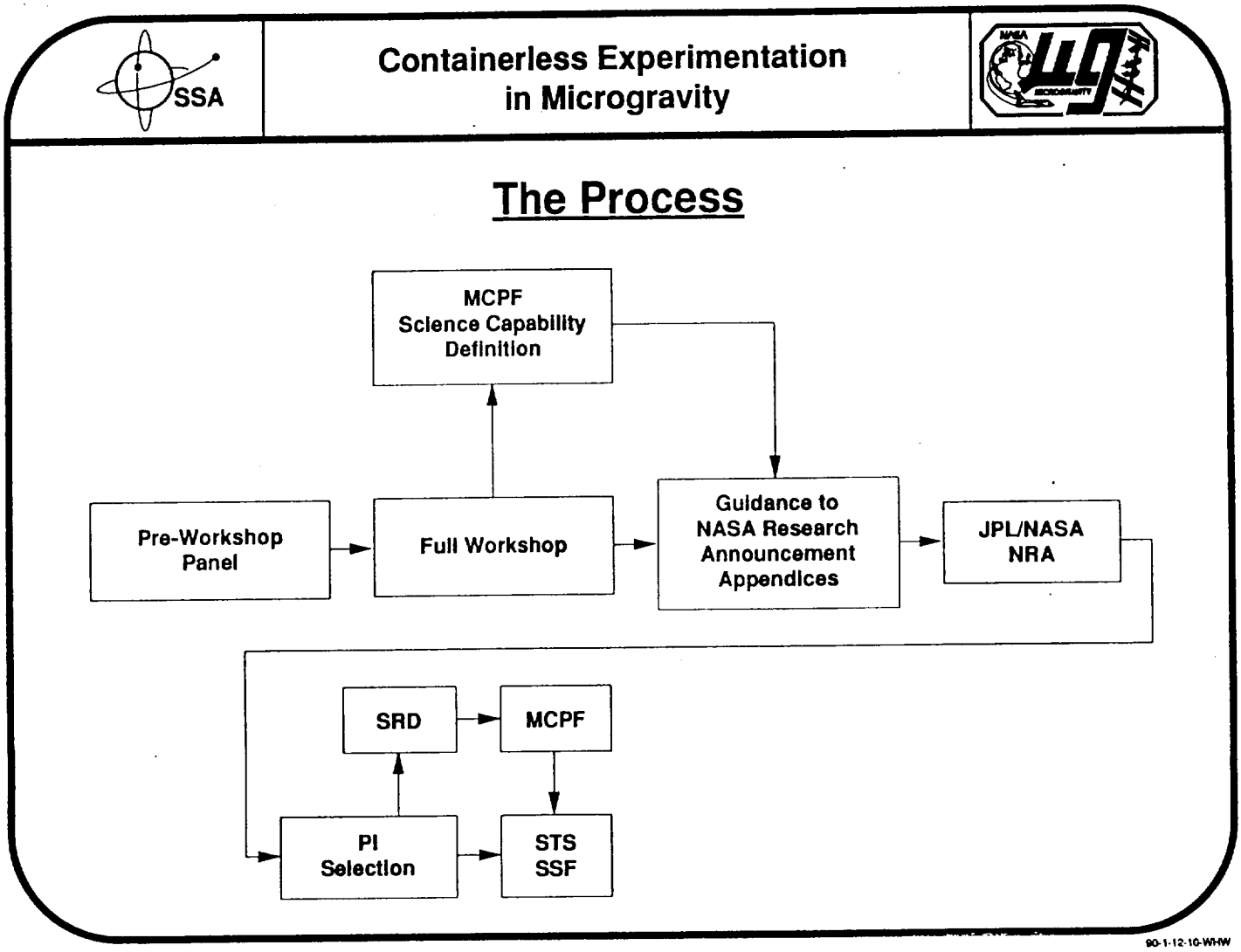
Ten Suggested Questions to be Addressed by the Workshop and Splinter Sessions

1. Is the removal of surface contamination alone enough to justify containerless experimentation in microgravity?
2. If not, then what are the other primary scientific justifications for performing containerless experimentation in microgravity?
3. What is the sensible way to acquire data for the purpose of verifying science justifications not currently available?
4. What should future containerless flight instruments look like if they are developed to meet those scientific justifications?
5. Does NASA need to develop a next generation electromagnetic manipulator?

90-1-12-12-WHW

Ten Suggested Questions (continued)

6. Does NASA need a high temperature acoustic program?
7. Is there any advantage to electrostatic positioning for space applications? Is it useful for melt undercooling study? Is it useful for low temperature protein crystallization applications?
8. Is there any need for a heavy-ion beam positioning scheme in space?
9. Can containerless manipulator capability be better achieved through a hybrid system such as acoustic-electromagnetic or acoustic-electrostatic?
10. How much investment is reasonable for the NASA containerless program? What percentage of the budget is adequate to cover high risk and, if successful, high yield areas?


SSA
Containerless Experimentation
in Microgravity
The Process
MCPF
Science Capability
Definition
Pre-Workshop
Panel
Full Workshop
Guidance to
NASA Research
Announcement
Appendices
JPL/NASA
NRA
SRD
MCPF
PI
Selection
STS
SSF
90-1-12-1G-WHW

Multiuser Hardware
"The Double NRA Approach"

NRA Selections ▼

PI Funding Starts ▼

minimum 2 years

definition studies with approved proposals

▼ Release 2nd NRA

Advantages: Multiuser HW better defined in 2nd announcement

All Science community has an equal chance for flight opportunities

ISSUE: Time Required for Double NRA

MCPF	√
Materials Science	X
Fluids	√
Combustion	√
Fundamental Science	TBD
PCG	√

90-1-12-7-WHW

Containerless Experimentation
in Microgravity

NRA and AO Phasing

	Center Submission					
	90	91	92	93	94	95
1. Combustion		√			√	
2. PCG		√			√	
3. Containerless		√			√	
4. Materials Science		√			√	
5. Fluids			√			√
6. Biotechnology		√	√		√	
7. Fundamental Science				pending		

90-1-12-9-WHW

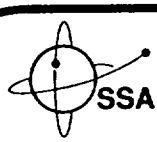
Containerless Experimentation
in Microgravity

Products of Workshop

1. Information to guide JPL/NASA in putting together a Containerless NRA to be released in FY90

2. Information to guide JPL/NASA in defining a Modular Containerless Processing Facility (MCPF) for Space Station Freedom

90-1-12-6-WHW



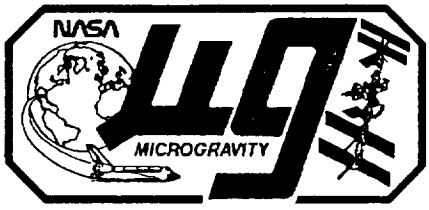
Containerless Experimentation
in Microgravity

The Challenge

Containerless experimentation in microgravity must be based on sound scientific justification. As NASA and this nation's investment in this area increases, it is even more critical to do so. Without strong scientific justification, it is increasingly difficult for NASA to maintain the current level of effort needed for the Space Station era in the face of mounting criticism voiced by the scientific community at large.

The challenge of this workshop is to provide this scientific justification, and to guide NASA in developing the next generation of flight instruments.

90-1-12-11-WHW



NASA Headquarters
Office of Space Science and Applications
Microgravity Science and Applications Division

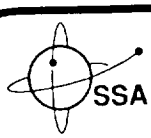
Status and Outlook of the Microgravity Science and Applications Program at NASA

Presentation to

Containerless Experimentation in Microgravity Workshop

Larry Spencer
January 17, 1990

9001-008-01CW 01/10/90

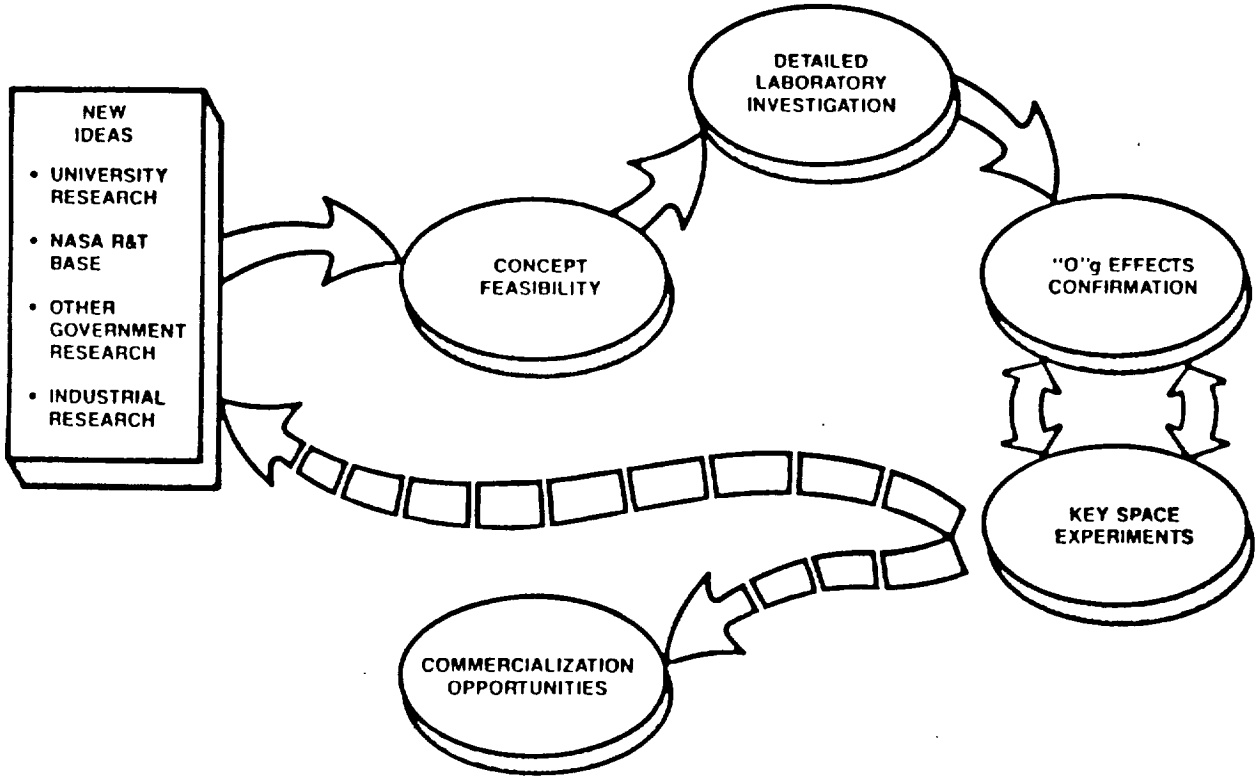


NASA Microgravity Program Goals

- Develop comprehensive research program in fundamental sciences, materials science, and biotechnology
- Develop understanding of gravity-dependent physical phenomena as basis of reliable predictive capability for processing operations/ technological issues in Earth/non-Earth environments
- Foster growth of an interdisciplinary research community
- Encourage international cooperation
- Explore new materials and processes relevant to basic research and commercial applications
- Develop permanently manned, multi-facility national microgravity laboratory in low-Earth orbit
- Promote industrial application of space research

9001-008-02CW 01/10/90

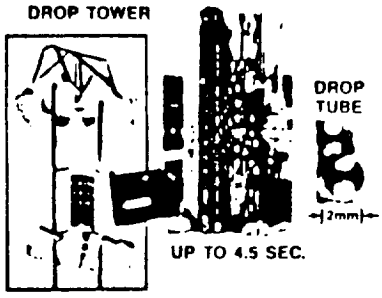
THE APPROACH



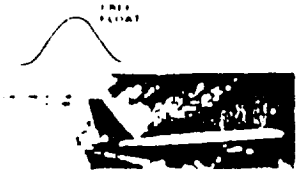
MICROGRAVITY SCIENCE AND APPLICATIONS EXPERIMENT CAPABILITY

FREE FALL FACILITIES

DROP TOWER

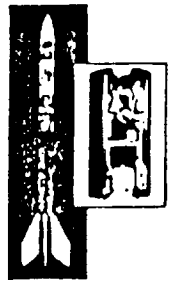


AIRCRAFT



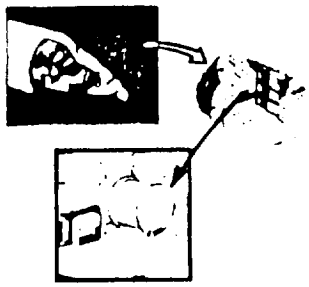
UP TO 60 SEC.

SOUNDING ROCKETS



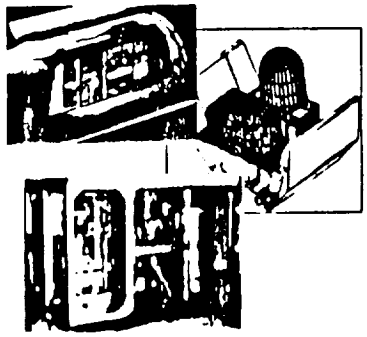
UP TO 6 MIN.

ORBITER MIDDECK



UP TO 14 DAYS

SHUTTLE-SPACELAB



UP TO 14 DAYS

SPACE STATION

CONTINUOUS

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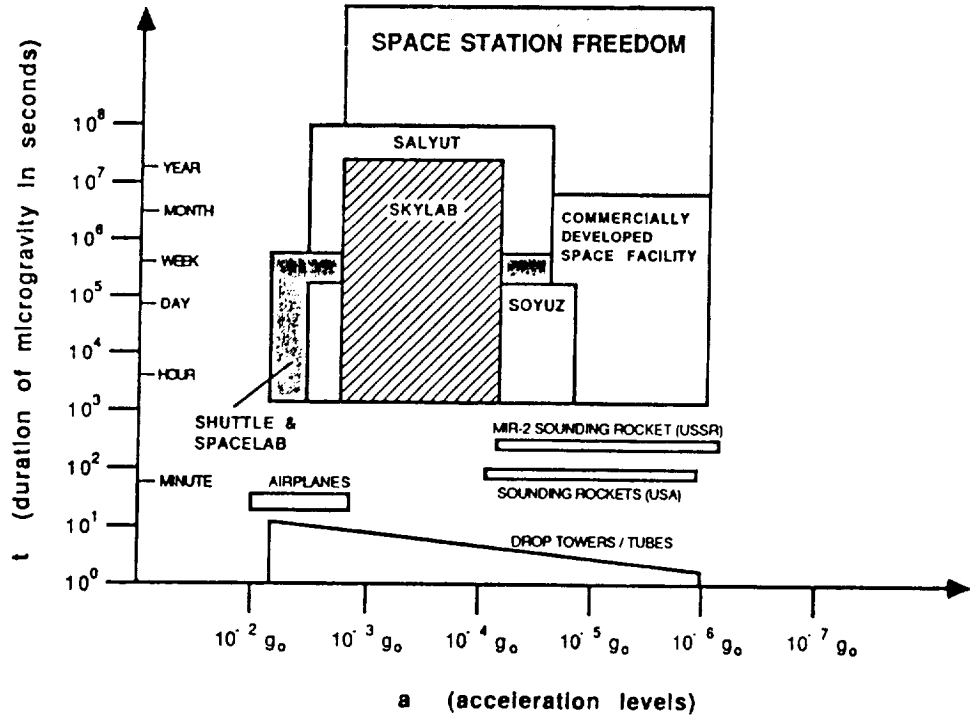
NASA

Microgravity Science and Applications Characteristic Duration and Acceleration Levels

MSAD



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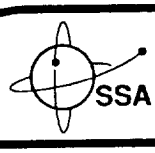


Microgravity Science and Applications Program

Fundamental Science

- Fluid Physics
- Combustion Science
- Critical Phenomena
- Relativity Theory

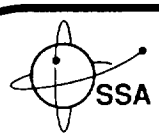
Materials Science

- Electronic Materials
- Metals and Alloys
- Glasses and Ceramics

Biotechnology

- Cell Physiology
- Cell Differentiation
- Protein Crystal Growth
- Biological Separations

9001-008-03CW 01/10/90



Announcements Outlook

Release Date	Proposals Due	Announcement
9 Nov 89	31 Dec 89	ESA AO for Materials and Fluid Science Experiments: IML-2
26 Dec 89	26 Mar 90	NASA NRA for Microgravity Combustion Science: Research and Flight Opportunities
FY90 *	TBD	Protein Crystal Growth Announcement
FY90 *	TBD	Solidification Research Announcement
FY90 - 91 *	TBD	Containerless Research Announcement
FY91 *	TBD	Fluids Research Announcement
FY91 *	TBD	Foreign Hardware IML-3 Announcement
FY92 *	TBD	Fundamental Phenomenal/Critical Point Research Announcement

*** Dates identified are tentative pending budget availability**

9001-008-13CW 01/10/90

NASA

OFFICE OF SPACE SCIENCE AND APPLICATIONS

Flight Systems Division

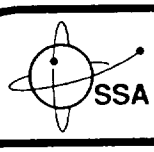
OSSA

INTERNATIONAL MICROGRAVITY LABORATORY (IML) -1

PAYLOAD COMPLEMENT

EXPMT No.	OV LOC	EXPERIMENT / FACILITY TITLE	ACRONYM	HQ CODE SPONSOR	EXPERIMENT / FACILITY DEVELOPER
2	SPACELAB RACK	FLUIDS EXPERIMENT SYSTEM	FES	EN	MSFC
3		VAPOR CRYSTAL GROWTH SYSTEM	VCGS	EN	MSFC
4		MERCURIC IODIDE CRYSTAL GROWTH	MICG	EN	CNES
19		CRITICAL POINT FACILITY	CPF	EN	ESTEC
13		ORGANIC CRYSTAL GROWTH FACILITY	OCGF	EN	NASDA
17		SPACE ACCELERATION MEASUREMENTS SYSTEM	SAMS	EN	LaRC
10		MICROGRAVITY VESTIBULAR INVESTIGATIONS	MVI	EB	JSC
16		RADIATION MONITORING CONTAINER/DOSIMETER	RMCD	EB	NASDA
15		MENTAL WORKLOAD AND PERFORMANCE EVAL.	MWPE	EB	JSC
14		BIOSTACK	BSK	EB	DLR
...		IMAX	IMAX	MC	JSC
6		GRAVITATIONAL PLANT PHYSIOLOGY FACILITY	GPPF	EB	ARC
7		BIORACK SYSTEMS	BR	EB	ESA/ESTEC
5	SMIDEX / MIDDECK	PROTEIN CRYSTAL GROWTH	PCG	EN	MSFC
18		CRYOSTAT	CRY	EN	DLR
8		SPACE PHYSIOLOGY EXPERIMENTS	SPE	EB	CSA

IML-1-C EM 11/89



First United States Microgravity Payload (USMP-1)

Payload Complement

No.	Experiment/Facility Title	NASA HQs Sponsor	Developer
1	Lambda Point Experiment	Code EN	JPL
2	MEPHISTO	CNES	CNES
3	Advanced Automated Directional Solidification Furnace (AADSF)	Code EN	MSFC
4	Space Acceleration Measurement System (SAMS)	Code EN	LeRC

9001-008-14CW 01/10/90

First United States Microgravity Laboratory (USML-1)

Baseline Payload Complement

No.	Experiment/Facility Title	NASA HQs Sponsor	Developer
1	Crystal Growth Furnace (CGF)	Code EN	MSFC
2	Crystals, Monomers, Deposition and Separation Facility (CMDSF)	Code C	UAH CCDS
3	Drop Physics Module (DPM)	Code EN	JPL
4	Surface Tension Driven Convection Experiment (STDCE)	Code EN	LeRC
5	Glovebox (GBX)	Code EN	TBD
6	Space Acceleration Measurement System (SAMS)	Code EN	LeRC
7	Solid Surface Combustion Experiment (SSCE)	Code EN	LeRC
8	Zeolite Crystal Growth (ZCG)	Code C	Battelle CCDS
9	Protein Crystal Growth (PCG) (3 R/IM's)	Code C	MSFC
10	Generic Bioprocessing Apparatus	Code C	Bioreserve
11	Solution Crystal Growth (SCG)	Code C	Battelle CCDS
12	Astroculture (ASC)	Code C	Wisconsin CCDS

9001-008-15CW 01/10/90

Second United States Microgravity Payload (USMP-2)

Payload Complement

No.	Experiment/Facility Title	NASA HQs Sponsor	Developer
1	Critical Fluid Light Scattering Experiment	Code EN	LeRC
2	Isothermal Dendritic Growth Experiment	Code EN	LeRC
3	MEPHISTO	CNES	CNES
4	Advanced Automated Directional Solidification Furnace (AADSF)	Code EN	MSFC
5	Space Acceleration Measurement System (SAMS)	Code EN	LeRC

9001-006-16CW 01/10/90

NASA

OFFICE OF SPACE SCIENCE AND APPLICATIONS

Flight Systems Division

SSA

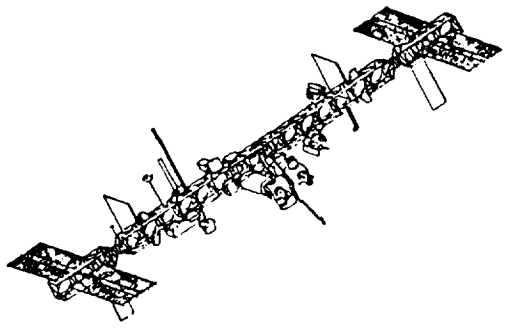
INTERNATIONAL MICROGRAVITY LABORATORY (IML) -2

CANDIDATE PAYLOAD COMPLEMENT

EXPMT No.	OV LOC	EXPERIMENT / FACILITY TITLE	ACRONYM	HQ CODE SPONSOR	EXPERIMENT / FACILITY DEVELOPER
	SPACELAB RACK	BIORACK (W/O CLR/FZR)	BR	EB	ESTEC/NASA JSC
		AQUATIC ANIMAL ENVIRONMENTAL UNIT	AAEU	EB	NASDA
		PERFORMANCE WORKSTATION	PWS	EB	NASA JSC
		VESTIBULAR & SENSORI-MOTOR EXPERIMENT	VSE	EB	CNES
		SLOW ROTATING CENTRIFUGE WITH MICROSCOPE	NIZEMI	EB	DLR
		REAL-TIME RADIATION MONITORING DEVICE	RRMD	EB	NASDA
		BACK PAIN IN ASTRONAUTS	BPA	EB	CSA
		BIOSTACK	BSK	EB	DLR
		VIBRATION ISOLATION BOX EXPERIMENT SYSTEM	VIBES	EN	NASDA
		ELECTROMAGNETIC CONTAINERLESS PROCESSING FAC.	TEMPUS	EN	DLR
		BUBBLE, DROP & PARTICLE UNIT	BDPU	EN	ESTEC
		APPLIED RESEARCH ON SEPARATION METHODS USING SPACE ELECTROPHORESIS	RAMSES	EN	CNES
		FREE FLOW ELECTROPHORESIS & THERMO-ELECTRIC INCUBAT.	FFEU/TEI-HT	EN	NASDA
		QUASI-STEADY ACCELERATION MEASUREMENT	QSAM	EN	DLR
		ADVANCED GRADIENT HEATING FACILITY	AGHF	EN	ESTEC
		LARGE ISOTHERMAL FURNACE	LIF	EN	NASDA
	SPACELAB AISLE	CANADIAN MINI-SLED	CMS	EB	CSA
		LOWER BODY NEGATIVE PRESSURE DEVICE	LBNPD	EB	NASA JSC
		DOUBLE RACK ADAPTOR PLATE	DRAP	EB	NASA JSC
		EDOMP EXERCISER	---	EB	NASA JSC
		SPACE ACCELERATION MEASUREMENT SYSTEM	SAMS	EN	NASA LeRC
	SMIDEX/ MIDDECK	SLEEP MONITORING EXPERIMENT	SME	EB	NASA JSC
		ADVANCED PROTEIN CRYSTALIZATION FACILITY	APCF	EN	ESA

IML-2-C EM 11/89

1989 Highlights
Advanced Programs



- **Space Station:**
  - Joint Science Utilization Study Support
  - May 1989: Modular Combustion Facility Assessment Workshop
  - June 1989: Space Station Furnace Facility One Year Conceptual Design Study awarded to Teledyne Brown Engineering
  - August 1989: Deployment dates for multi-user facilities rephased
  - November 1989: Request out to all MSAD investigators to provide model experiment scenarios for Space Station
  - December 1989: Microgravity Requirement addressed at combined Level I/Level II Space Station Control Board meeting at Reston, Virginia
- **Human Exploration Initiative**
  - Preliminary Program Plan developed for Microgravity Science and Applications in response to call for 90-day NASA report to Vice-President Quayle

9001-008-17CW 01/10/90

Microgravity Science and Applications Plans for Space Station

- **Six multi-user experimental facilities planned for Space Station Freedom**
  - Advanced Protein Crystal Growth Facility
  - Space Station Furnace Facility
  - Modular Containerless Processing Facility
  - Fluid Physics/Dynamics Facility
  - Modular Combustion Facility
  - Biotechnology Facility

9001-006-18CW 01/10/90

Microgravity Science and Applications Evolution Strategy

- **Initial Strategy: Deploy six facilities prior to SSF Assembly Complete**
- **Current Strategy: Rephased developments in order to resolve issues with:**
  - Phasing of Space Station
  - Budget and schedule incompatibilities
  - Technical capability constraints
- **Rephasing allows MSA Program time to:**
  - Enhance research base
  - Strengthen project management base
  - Gain more on-orbit experience

9001-008-19CW 01/10/90

Human Exploration Initiative

- **Basic approach**

1990's		2001 - 2010		Beyond 2010
Space Station Freedom	⇨	Lunar Outpost	⇨	Mars Exploration
Lunar Orbiter	⇨	Mars Robots		Lunar Operations

- **Long-range exploration goal is Mars**
- **Moon is justified on its merits, as well as a stepping stone toward Mars**
- **90-day study will develop a baseline option and analyze impact of variations on milestones and program scope**
- **Baseline and options will be approved by NASA Administrator**

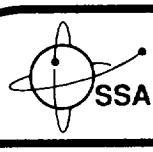
9001-008-22CW 01/10/90

Human Exploration Initiative
MSAD Program Strategy

MSAD's Role in the Human Exploration Initiative

- **Determine influence of gravity and other extraterrestrial environments on fundamental processes/phenomena. Emphasis on:**
  - Processes/phenomena significantly altered or affected by gravity variations and other unique attributes of the extraterrestrial environment
  - Processes/phenomena whose understanding under extraterrestrial conditions will benefit planned HEI activities
- **Support basic research activities which can clearly benefit from exploiting the unique attributes of the lunar environment**

9001-008-23CW 01/10/90

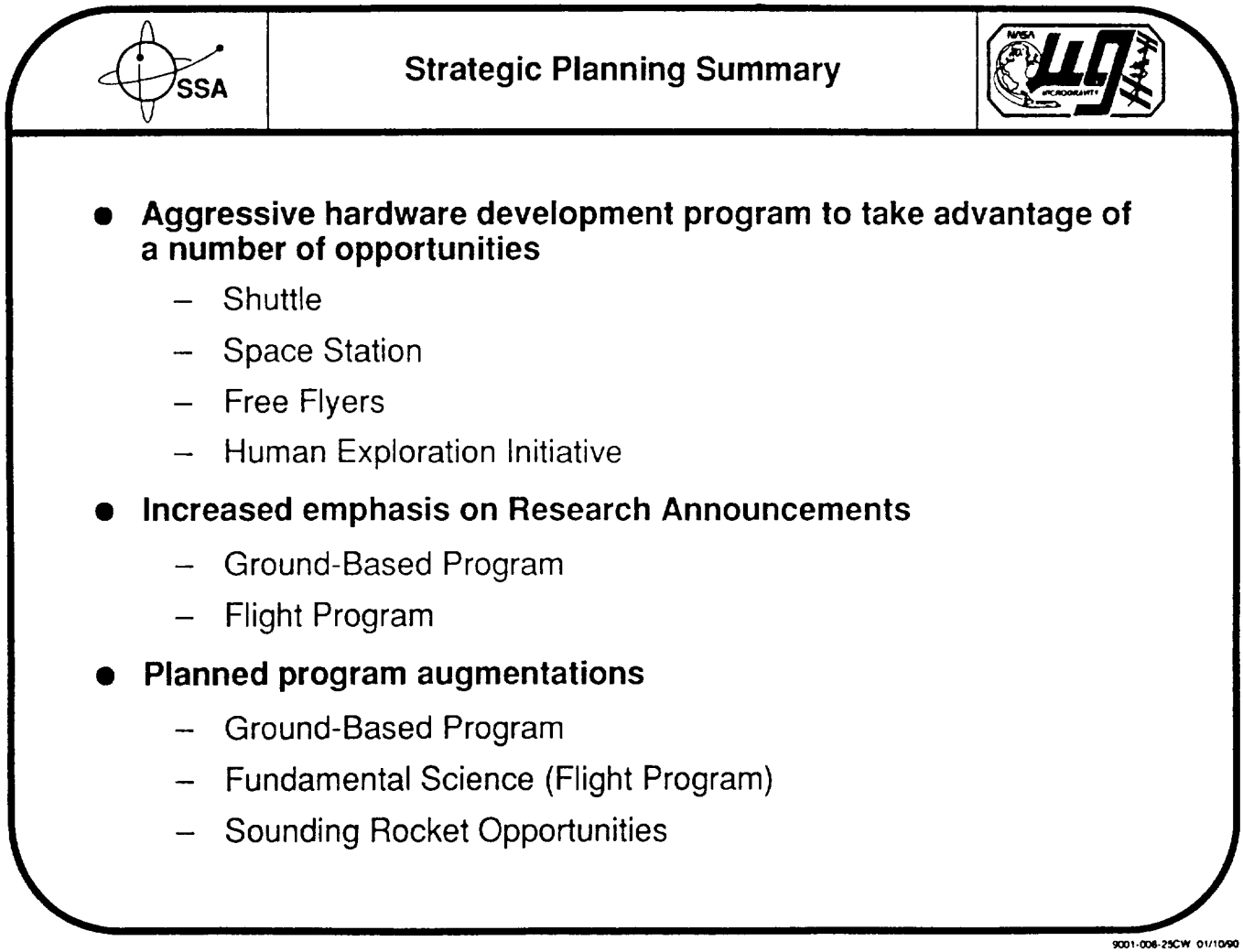


Human Exploration Initiative

Initiative Research Areas

- **Fluid Dynamics and Transport Phenomena**
  - Multiphase flow
  - Phase change heat transfer
  - Fluids management
- **Mechanics of Granular Media**
  - Soil mechanics
  - Rheology
- **Combustion**
  - Fire safety
  - Power
- **Materials Processing**
  - Resource utilization/chemical processes
  - Materials manufacturing

9001-006-24CW 01/10/90


SSA
Strategic Planning Summary
NASA
Aggressive hardware development program to take advantage of a number of opportunities
– Shuttle
– Space Station
– Free Flyers
– Human Exploration Initiative
Increased emphasis on Research Announcements
– Ground-Based Program
– Flight Program
Planned program augmentations
– Ground-Based Program
– Fundamental Science (Flight Program)
– Sounding Rocket Opportunities
9001-008-25CW 01/10/90

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY WORKSHOP

GROUND-BASED AND MICROGRAVITY CONTAINERLESS POSITIONING TECHNOLOGIES AND FACILITIES

MARTIN BARMATZ

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

JANUARY 17, 1990

PRESENTATION OUTLINE

- CONTAINERLESS EXPERIMENTS
- UNIQUENESS OF MICROGRAVITY
- MICROGRAVITY EXPERIMENTS
- CONTAINERLESS PROGRAM DEVELOPMENT
  - SCIENTIST PARTICIPATION
- ACCOMMODATING MICROGRAVITY EXPERIMENTS
  - POSITIONING APPROACHES
  - SPACE FLIGHT FACILITIES
  - GROUND-BASED FACILITIES AND TECHNOLOGY DEVELOPMENT
  - SPACE STATION FACILITIES

CONTAINERLESS EXPERIMENTS

- SAMPLE IN FREE SUSPENSION IN A FLUID OR VACUUM
- SINGLE OR MULTI-PHASED MATERIAL (LIQUID, SOLID, PHASE TRANSFORMATION)
- NON-CONTACT MEASUREMENT TECHNIQUES
- MINIMUM SAMPLE PERTURBATION (STABILITY)
- HIGH PURITY ENVIRONMENT - MINIMIZES CRUCIBLE CONTAMINATION
- EXTREME ENVIRONMENTS (HIGH TEMPERATURE, PRESSURE, OR VACUUM)

UNIQUENESS OF MICROGRAVITY

- GRAVITY PERTURBS PHENOMENA (CONVECTION, SEDIMENTATION, SMALL FORCES OVERWHELMED BY GRAVITATIONAL ACCELERATION)

- EXPERIMENT NOT OPTIMUM IN GRAVITY FIELD (LEVITATION OF GLASSES, CERAMICS)

- HIGH INTENSITY LEVITATION FIELDS MASK OR PERTURB PHENOMENA - (SHAPE DEFORMATION, LARGE EDDY CURRENT OR CHARGE DENSITY EFFECTS, DYNAMIC NUCLEATION)

- LONG DURATION QUIESCENT ENVIRONMENT (REDUCED FLUID FLOWS)

- EXCELLENT VIBRATION ISOLATION POSSIBLE (CRYSTAL GROWTH - PROTEIN CRYSTALS)

MICROGRAVITY EXPERIMENTS

- REQUIRES EXTENSIVE GROUND-BASED EXPERIMENTATION
- CONSIDERATION OF PAST FLIGHT EXPERIENCE, UNDERSTANDING OF CURRENT CAPABILITIES, AND LONG RANGE PLANNING
- STRONG INTERACTION BETWEEN CURRENT AND FUTURE SCIENTISTS AND FACILITY BUILDER
- LONG LEAD TIME FOR DEVELOPMENT - SHORT PERFORMANCE TIME
- EXPENSIVE - GENERALLY ACCOMMODATED BY MULTI-USER FACILITY
- COMPLEX TO PERFORM - REMOTE OR AUTOMATED OPERATIONS AND RESTRICTED ACCESS - SAFETY CONCERNS
- EVOLUTIONARY PROCESS : FIRST - SIMPLE EXPERIMENTS, LATER - MORE PRECISE EXPERIMENTS WITH ADVANCED CAPABILITIES AS EQUIPMENT MATURES
- UNIQUE ENVIRONMENT - ORBITAL LABORATORY IS A VALUABLE RESOURCE

CONTAINERLESS PROGRAM DEVELOPMENT

SCIENTIST PARTICIPATION

- EVALUATE PRESENT SCIENTIFIC CAPABILITIES OF FACILITIES AND SUGGEST ENHANCEMENTS
- PROPOSE NOVEL EXPERIMENTS TO MATCH CURRENT FACILITIES
- DOCUMENT CURRENT AND POTENTIAL NEW SCIENCE REQUIREMENTS
- PARTICIPATE IN GROUND BASED RESEARCH AND DEVELOPMENT PROGRAM (PROPOSALS, AO, NRA)
- **RESPOND TO SURVEY ON CONTAINERLESS EXPERIMENTATION SCIENCE CAPABILITIES**

ACCOMMODATING MICROGRAVITY EXPERIMENTS

POSITIONING APPROACHES

- DEVELOPMENT OF DIFFERENT POSITIONING APPROACHES
  - ACOUSTIC - HIGH INTENSITY SOUND FIELDS
    FLUID MEDIUM, ANY MATERIAL,
    STATIC METHOD
  - ELECTROMAGNETIC - INDUCED EDDY CURRENTS
    FLUID OR VACUUM MEDIA,
    CONDUCTING MATERIAL,
    STATIC METHOD
  - ELECTROSTATIC - FORCES BETWEEN OPPOSITE
    CHARGES, FLUID OR VACUUM
    MEDIA, SURFACE CHARGES,
    DYNAMIC METHOD
  - GAS FILM - PRESSURE DROP ASSOCIATED WITH
    THIN LAYER OF FLOWING GAS
  - FREE FLOAT - NO EXTERNAL FORCES

EARLY NASA SPACE FLIGHTS

- ACOUSTIC CONTAINERLESS EXPERIMENTAL SYSTEM - **ACES** (STS 11, 1984)

- DROP DYNAMICS MODULE - **DDM** (SPACE LAB 3, 1985)

- TRIPLE AXIS ACOUSTIC LEVITATOR - **3AAL** (STS 24, 1986)

- ELECTROMAGNETIC LEVITATOR - **EML** (STS 24, 1986)

- SINGLE AXIS ACOUSTIC LEVITATOR - **SAAL** (STS 7, 1983 - STS 61A, 1985)

EARLY NASA SPACE FLIGHTS

LESSONS LEARNED

- LOW BUDGET, HIGH RISK FLIGHTS PROVIDE SPARSE SCIENTIFIC RETURNS

- TOTALLY AUTOMATED POSITIONERS REQUIRE MORE THAN ONE SHORT DURATION (2 HOUR) FLIGHT TO BECOME OPERATIONAL

- ASTRONAUT OPERATED POSITIONERS INCREASE PROBABILITY OF SUCCESS

- PRE-FLIGHT IMPROVEMENTS
  - IMPROVED GROUND-BASED CALIBRATION
  - PRECURSOR FLIGHT EXPERIMENTS (KC-135, ROCKET FLIGHTS)
  - SCIENCE EVALUATION OF POSITIONERS

ACCOMMODATING MICROGRAVITY EXPERIMENTS

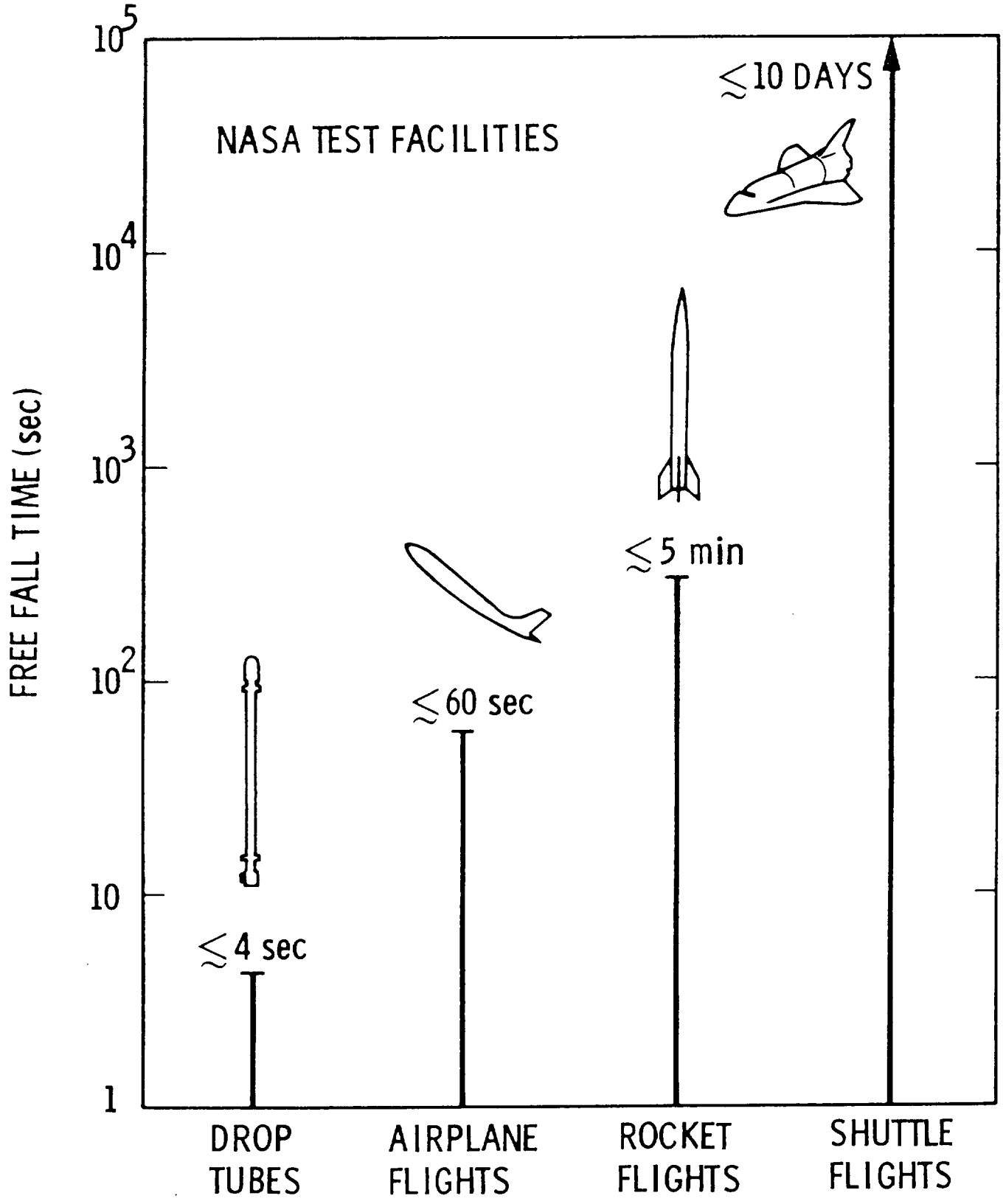
GROUND-BASED FACILITIES

- INDIVIDUAL INVESTIGATORS FACILITIES (ELECTROMAGNETIC, ACOUSTIC, ELECTROSTATIC, AERODYNAMIC, GAS FILM, ...)

- DROP TUBE FACILITIES (NASA LRC, MSFC)

- NASA KC-135 / ESA CARAVELLE FACILITIES

- ROCKET FLIGHTS (TEXUS, PRIVATE COMPANIES)


NASA TEST FACILITIES
FREE FALL TIME (sec)
10^5
10^4
10^3
10^2
10
1
≲10 DAYS
≲5 min
≲60 sec
≲4 sec
DROP TUBES
AIRPLANE FLIGHTS
ROCKET FLIGHTS
SHUTTLE FLIGHTS

ACCOMMODATING MICROGRAVITY EXPERIMENTS

SPACE FLIGHT FACILITIES

- PAST FLIGHT EQUIPMENT (SAAL, ACES, DDM, 3AAL, EML)

- CURRENT FACILITIES

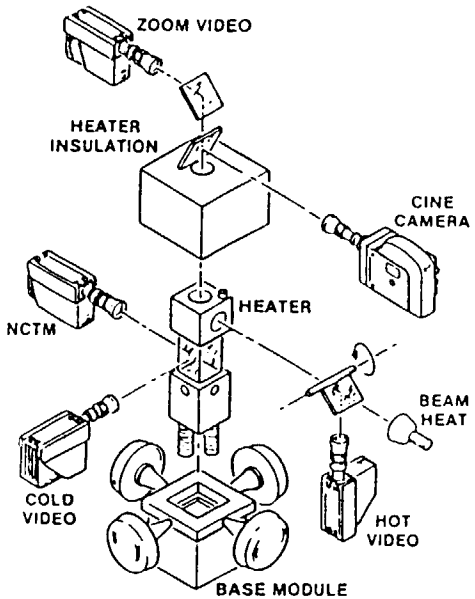
  - DROP PHYSICS MODULE - **DPM** (NASA JPL ACOUSTIC POSITIONER)

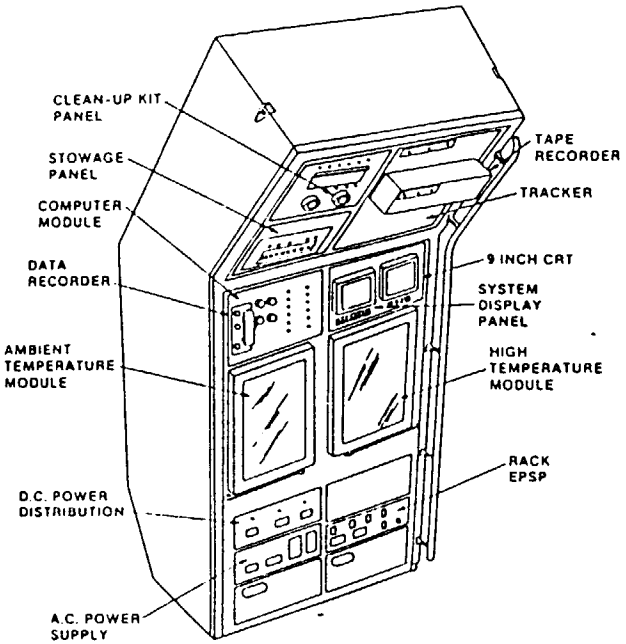
    - UNITED STATES MICROGRAVITY LABORATORY - **USML** SERIES **USML-1** (1992), **USML-2** (1994)

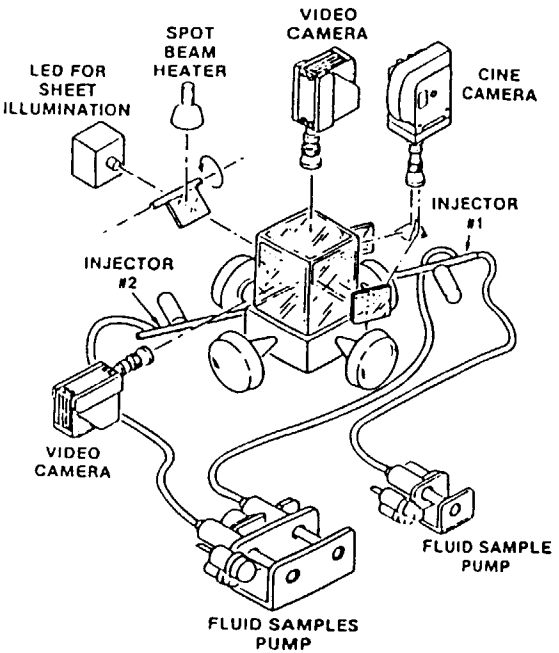
  - TEMPUS (GERMAN ELECTROMAGNETIC POSITIONER)

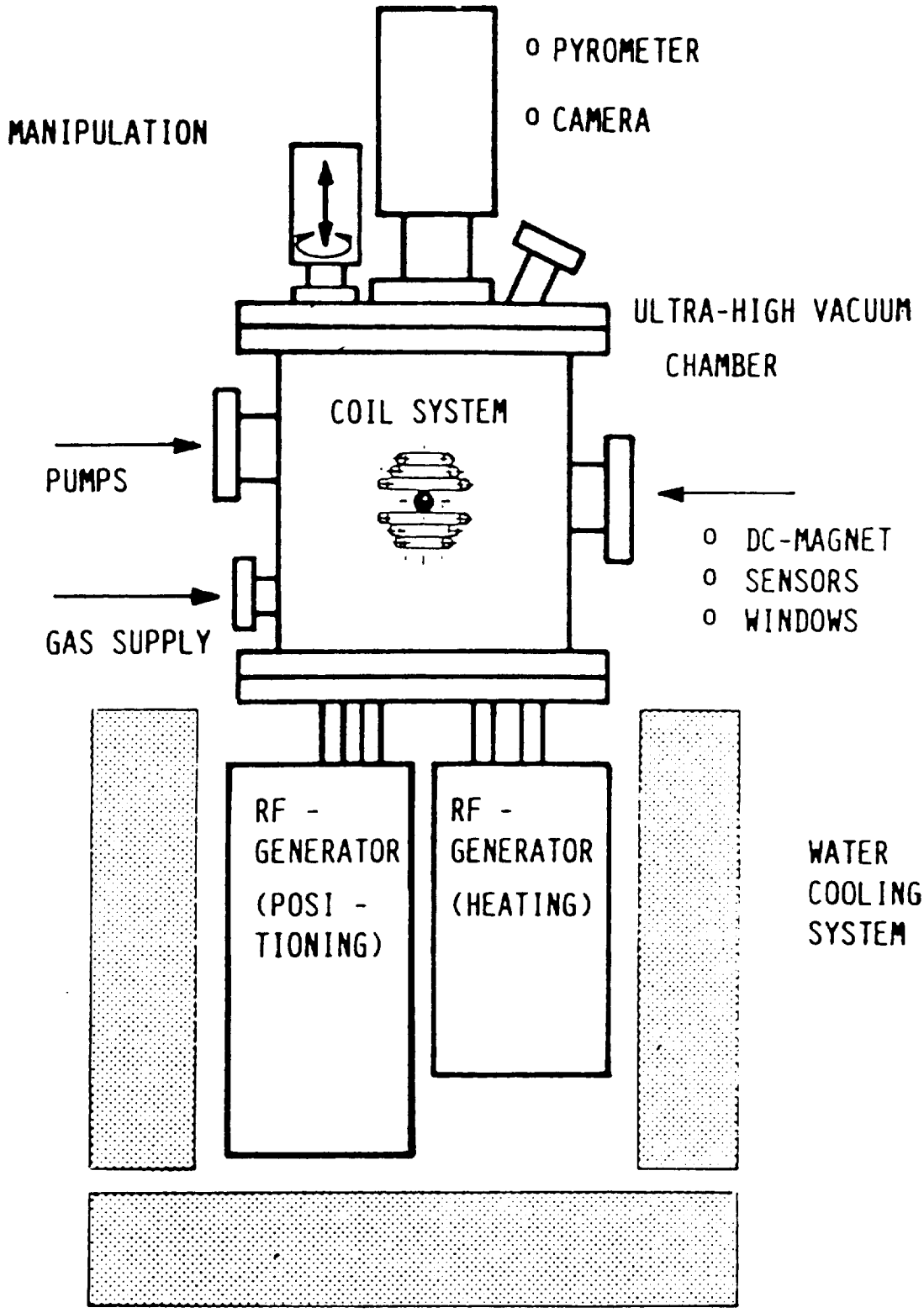
    - INTERNATIONAL MICROGRAVITY LABORATORY (**IML-2**) (LATE 1992)

- INTERNATIONAL COOPERATION (AMERICAN, EUROPEAN, AND JAPANESE PROGRAMS)


ZOOM VIDEO
HEATER INSULATION
CINE CAMERA
HEATER
NCTM
BEAM HEAT
COLD VIDEO
HOT VIDEO
BASE MODULE


CLEAN-UP KIT PANEL
STOWAGE PANEL
COMPUTER MODULE
DATA RECORDER
AMBIENT TEMPERATURE MODULE
D.C. POWER DISTRIBUTION
A.C. POWER SUPPLY
TAPE RECORDER
TRACKER
9 INCH CRT
SYSTEM DISPLAY PANEL
HIGH TEMPERATURE MODULE
RACK EPSP


LED FOR SHEET ILLUMINATION
SPOT BEAM HEATER
VIDEO CAMERA
CINE CAMERA
INJECTOR #1
INJECTOR #2
VIDEO CAMERA
FLUID SAMPLE PUMP
FLUID SAMPLES PUMP


o PYROMETER
o CAMERA
MANIPULATION
ULTRA-HIGH VACUUM
CHAMBER
COIL SYSTEM
PUMPS
o DC-MAGNET
o SENSORS
o WINDOWS
GAS SUPPLY
RF -
GENERATOR
(POSI -
TIONING)
RF -
GENERATOR
(HEATING)
WATER
COOLING
SYSTEM

ACCOMMODATING MICROGRAVITY EXPERIMENTS TECHNOLOGY DEVELOPMENT

- POTENTIAL FUTURE FACILITIES (DEVELOPING TECHNOLOGIES)
  - ACOUSTIC LEVITATION FURNACES
    - ISOTHERMAL, BEAM HEATING
  - MODULAR ELECTROMAGNETIC LEVITATOR
  - STABILIZED ELECTROMAGNETIC LEVITATOR
  - ELECTROSTATIC TETRAHEDRAL POSITIONERS
  - GAS LAYER LEVITATOR
  - MICROWAVE/ACOUSTIC HYBRID POSITIONER
- NON-CONTACT PARAMETER MEASUREMENTS
  - TEMPERATURE

ACCOMMODATING MICROGRAVITY EXPERIMENTS

SPACE STATION FREEDOM

- MODULAR CONTAINERLESS PROCESSING FACILITY - **MCPF**
  - FACILITY CONCEPT FOR SPACE STATION
  - SCIENCE COMMUNITY INPUT NEEDED
- MULTIPLE POSITIONING MODULES
  - ACOUSTIC
  - ELECTROMAGNETIC
  - ELECTROSTATIC
  - HYBRIDS

Modular Containerless Processing Facility (MCPF)

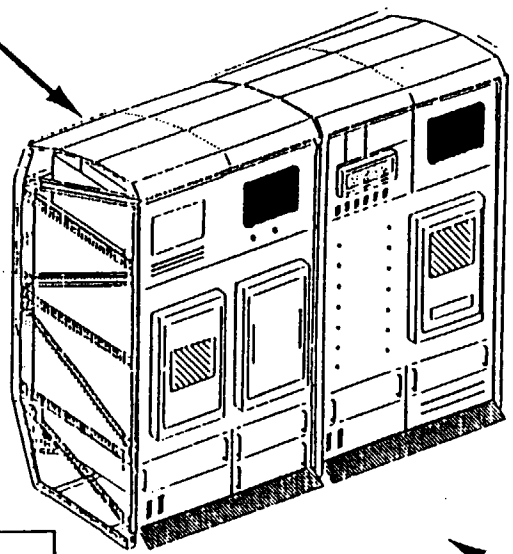
Provides 4 kinds of experimental facilities on Space Station Freedom

ACOUSTIC POSITIONING FACILITY

Fundamental Fluid Physics
Interfacial Phenomena
Interparticle Dynamics
Geophysical Fluid Dynamics
Glasses and Ceramics Processing
Phase Transition Phenomena

ELECTROSTATIC POSITIONING FACILITY

Protein Crystal Growth
High Temperature/
high vacuum chemistry
Charged fluid/particle dynamics
High temperature materials



ELECTROMAGNETIC POSITIONING FACILITY

Metals and Alloys Processing
Very High Temperature Chemistry
Metastable Structures Formation
Homogeneous Nucleation
Advanced Materials Structures

EXOBIOLOGY FACILITY

Aerosol Physics
Fractals Dynamics
Interparticle Dynamics
Atmospheric Chemistry

RLGec320

N91-21334

VERY HIGH TEMPERATURE CHEMISTRY

Science Justification for Containerless Experimentation In Space

By

William H. Hofmeister, Vanderbilt University
Paul Nordine, Containerless Processing, Inc.

Following is a summary justification for application of containerless processing in space to high temperature science. Low earth orbit offers a gravitational environment that allows samples to be positioned in an experimental apparatus by very small forces. Well controlled experiments become possible on reactive materials at high temperatures in a reasonably quiescent state and without container contamination. This provides an opportunity to advance the science of high temperature chemistry that can only be realized with a commitment by NASA to provide advanced facilities for in-space containerless study of materials at very high temperature.

The laws of thermodynamics provide a fundamental scientific motivation for efforts to advance high temperature science; heat engine efficiency increases with operating temperature. Consequently, the search for higher performance and fuel efficiency in automotive and gas turbine technology center on advances in high temperature materials. Experience also shows a connection between the useful and scientifically interesting properties of materials and their melting points. The hardest and strongest materials, superconductors with high transition temperatures, most semiconductors, refractory materials, high performance coatings and several excellent infrared optical materials melt at very high temperatures. The bronze, iron, steel, nuclear and silicon eras demonstrate that human progress is closely related to advances in high temperature materials technology. Finally, aerospace technology itself requires lighter, stronger, higher melting, and more oxidation-resistant materials. For these reasons it is important that opportunities to advance high temperature science be developed.

Thermodynamics also explains why containerless technology is needed in this effort. Solutions have larger entropies than their separated components, so the extent of container contamination of materials increases with temperature. Containerless processing in space converts this fact from a problem to a wide ranging opportunity for scientific progress.

Examples

Comments on several selected areas of high temperature science are given below in which containerless processing in space has scientifically interesting and unique applications.

Liquids - An improved understanding of high temperature liquids is not only of scientific interest, but is also necessary to advance the art of high temperature processing. Novel experiments to measure the optical, thermal, mechanical, and other properties of liquids in containerless, space-based experiments would be feasible.

Thermal properties - Unusual variations of heat capacity with temperature have been determined for some liquid metals. Since the temperatures were measured by optical pyrometry with the assumption of temperature independent emissivity, it is possible that emissivity variations contribute to the apparent heat capacity effects. Continued Earth-based progress in this area is possible by making optical property measurements on electromagnetically (EM) levitated liquid metals. However, EM levitation is at best difficult,

complicated by stirring and sample oscillations, and applicable materials of high electrical conductivity. In space, measurements on a much wider range of liquids including poor conductors would be possible. Measurement precision would be advanced by the enhanced stability of melts positioned in microgravity.

Phase relations - Understanding the melting and solidification behavior of high temperature materials is essential to alloy and process design. Phase diagram determination by electromagnetic levitation techniques is well known. Acoustic positioning furnaces, with or without beam heating, will allow non-contact study of phase behavior to be extended to a wide range of materials. This is particularly important in complex systems that form many condensed phases such as the ceramic superconductor materials. The stability of any one phase in such systems is necessarily small relative to an equal composition mixture of other phases, and often sensitive to the impurities that result from processing in containers.

Intrinsic properties of solids - The synthesis of materials with improved homogeneity and purity, controlled chemical composition, and reduced mechanical flaws is needed to determine basic chemical-structural-property relationships. An improved understanding of optical and mechanical properties would have particular value to composite materials and fiber optic applications.

Purification - The environment in the wake of the shuttle or a wake shield can provide an extremely high vacuum unobtainable on earth. In addition, the effective pumping capacity of samples behind a shield is nearly infinite. This offers an opportunity to study the vacuum purification of materials at a level heretofore unachievable.

Non-equilibrium materials - Materials exhibit an increased solubility for their components at high temperatures, i.e., extended homogeneity ranges. Compositions of materials can thus be made that are retained as metastable materials when cooled to lower temperatures.

Nucleation and undercooling - The kinetics of nucleation processes provide another fundamental basis for interest in containerless processing. Solid containers induce heterogeneous nucleation from supersaturated or undercooled melts and also reduce the rate and uniformity with which melt cooling can be achieved. This greatly limits conditions under which homogeneous nucleation phenomena can be studied and non-equilibrium materials prepared. Supercooled liquids can be formed and their properties and process kinetics investigated. Some information has been obtained from studies of supercooled liquid metals by EM levitation and drop tubes. The versatility, control of cooling rate, applicability to poor electrical conductors, increased scale, ability to carry out repeated measurements on the same specimen, and other qualities that can be achieved by containerless experiments in space promise major advances in non-equilibrium materials.

Novel processing techniques - Certain processing techniques will benefit from the reduced gravity environment of space such as the manipulation of melts into useful shapes by aerodynamic, acoustic or electromagnetic forces. Conventional containerless processing could be applied to larger sizes and a wider range of materials. Indeed, the basic research expended in this area might yield as-yet unenvisioned processing opportunities.

Conclusion

The laws of thermodynamics motivate efforts to advance high temperature science and explain why this is difficult in experiments that use containers. The low gravity environment of space allows versatile equipment for containerless experiments at very high temperatures to be developed. Much scientific and technological progress will be possible if such facilities are developed.

N91-21335

NASA-SPONSORED CONTAINERLESS PROCESSING EXPERIMENTS

Prepared for

The Workshop on Containerless Processing in Microgravity

by
William H. Hofmeister

OUTLINE

GROUND BASED EXPERIMENTS

- DROP TUBE STATUS
  - PYROMETRY
  - SOLIDIFICATION VELOCITY
- EM LEVITATION
  - FACILITIES
  - EXPERIMENTS
- AERODYNAMIC LEVITATION
- ACOUSTIC LEVITATION

PURIFICATION

IML-2 MISSION

COMPARISON OF CONTAINERLESS PROCESSING TECHNIQUES FOR BULK SAMPLES

	MSFC TUBE	UHV TUBE	EM in LAB	TEMPUS
Environmental purity	fair	excellent	excellent	good
Vacuum (torr)	10^{-5}-10^{-6}	10^{-8}-10^{-10}	10^{-9}-10^{-10}	10^{-9}
Experimental duration	4.6 sec	3.1 sec	unlimited	flight timeline limited
Quiescence	good	good	large agitation	small agitation
Temp. measurement	will improve with drop tube pyrometry		excellent	potentially excellent
Temp. control	radiation cooling rate is slowest, can be varied with gas pressure		good	best
Isothermality	poor	poor	okay	okay
Position control	free fall		stability problems	excellent
Accessability	good	presently only French facility	excellent	poor

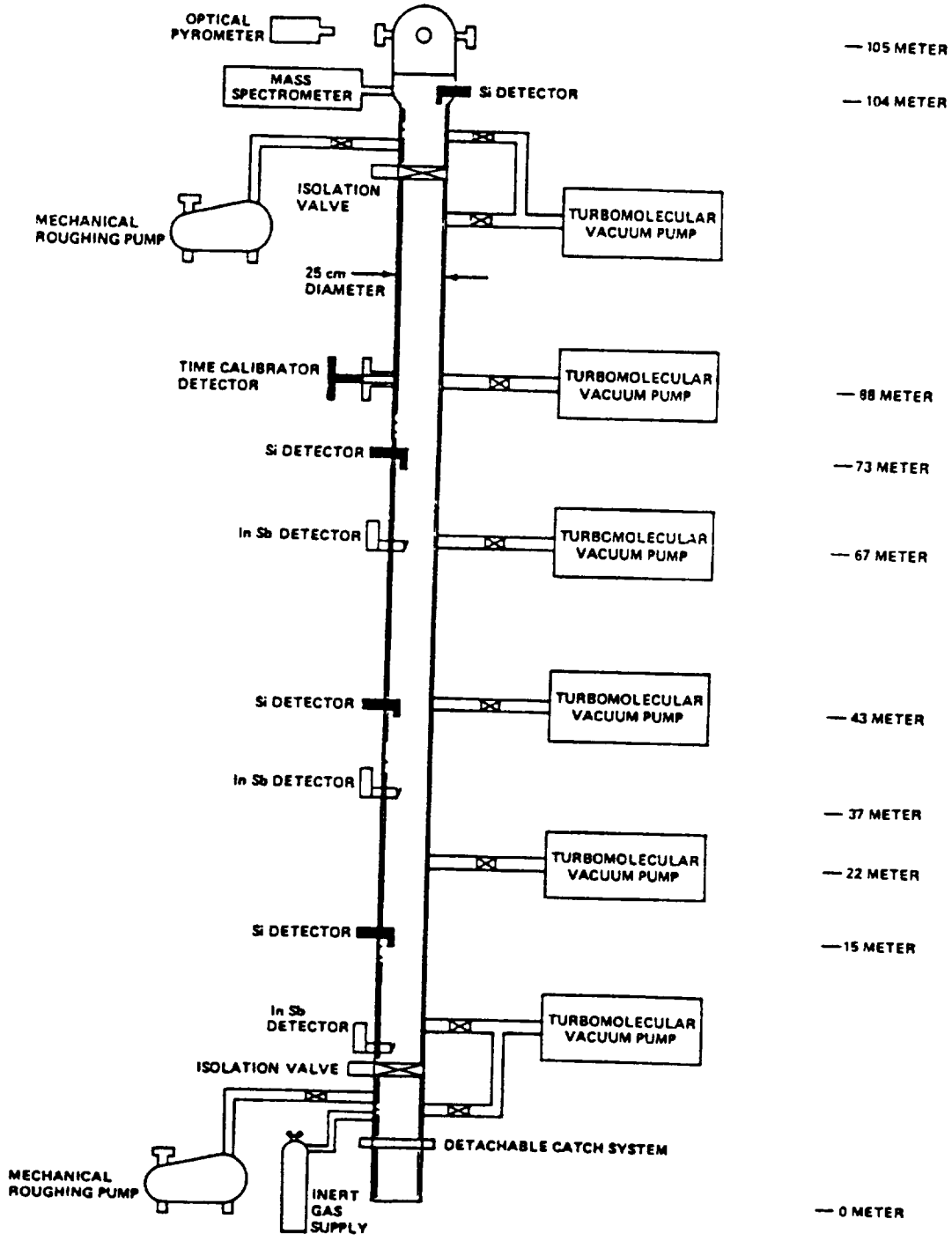
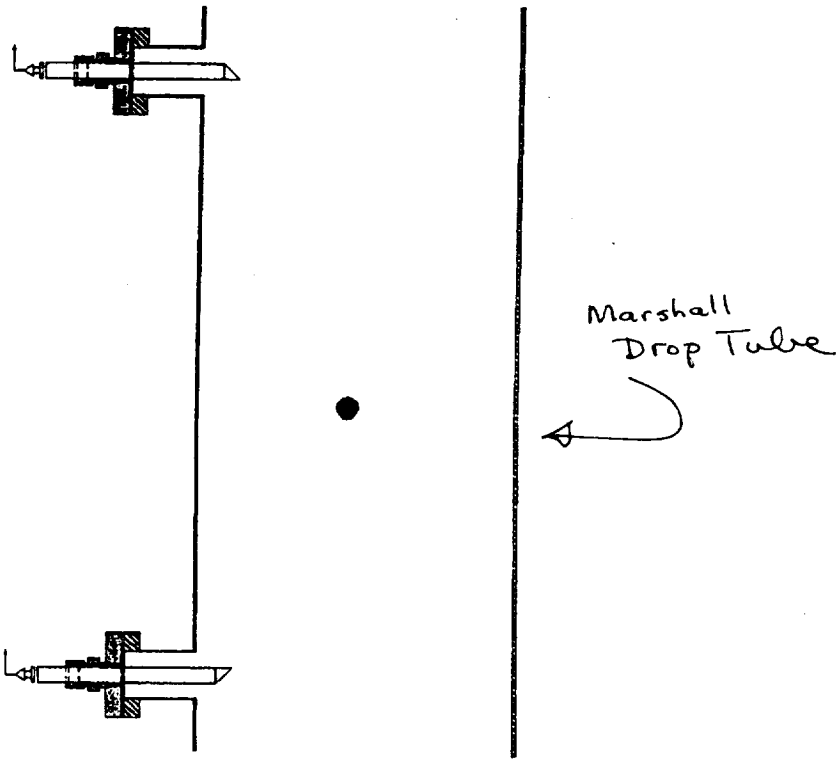
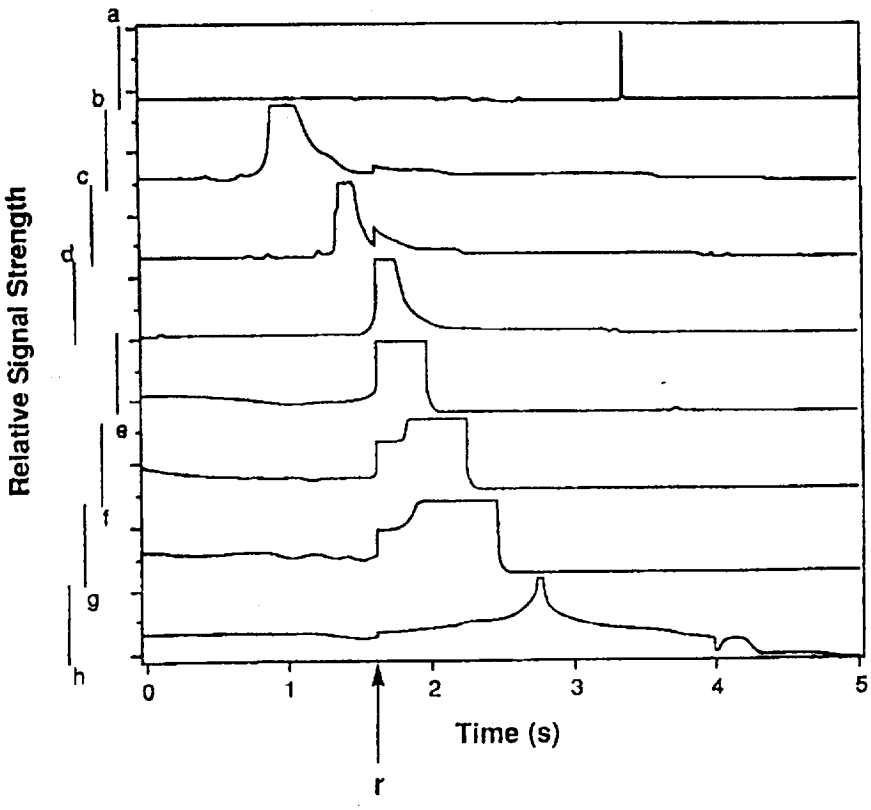


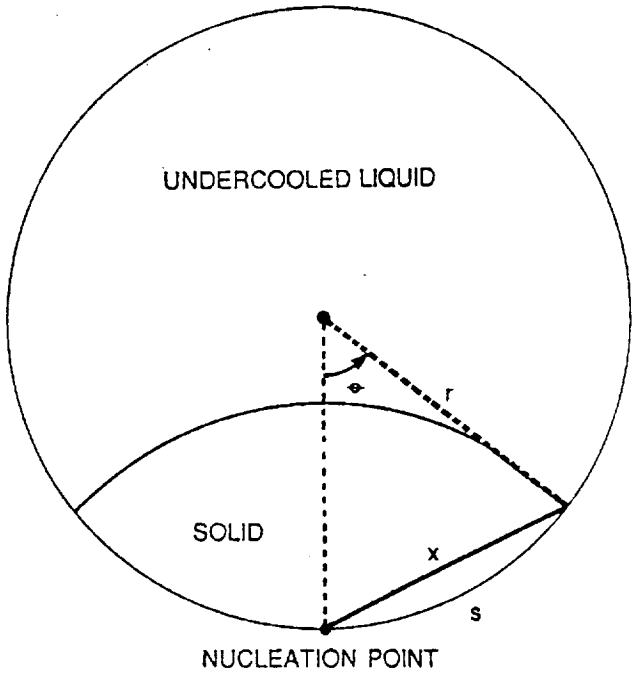
Figure 1. Schematic of the 105-meter drop tube at Marshall Space Flight Center.

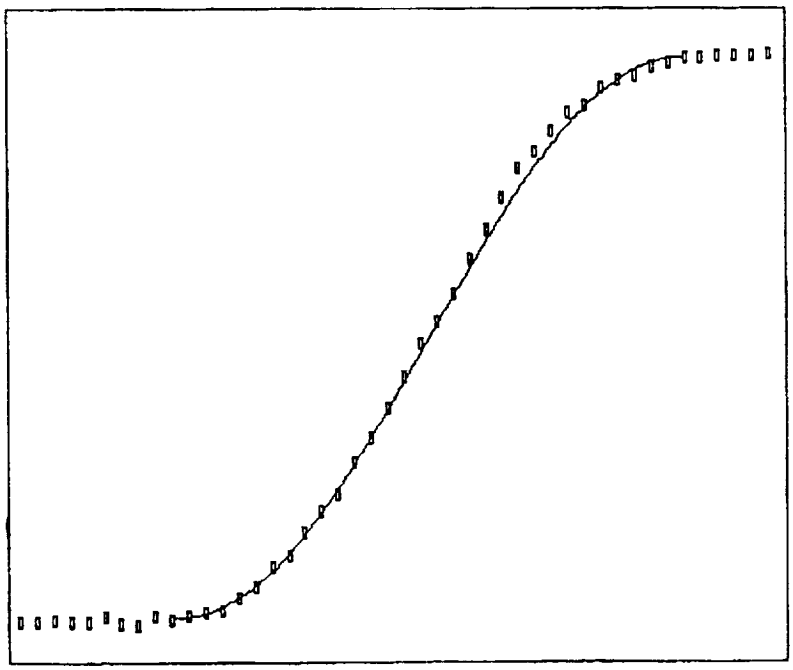




Legend:

a = Timer Detector on level 5.
b, c, d = Down-Looking Detectors on levels 14, 13, 12.
e, f, g = Up-Looking Detectors on levels 11, 10, 9.
h = Up & Down-Looking Logarithmic Amplifier/Detector on level 8.
r = Recalescence Event





MEASURED SOLIDIFICATION VELOCITIES IN MSFC DROP TUBE

PURE Nb	Nb-Pt (Primary Nb)	Nb-Pt (Primary Nb_3Pt)
20 m/s	15 m/s	1 m/s

Arc Cast

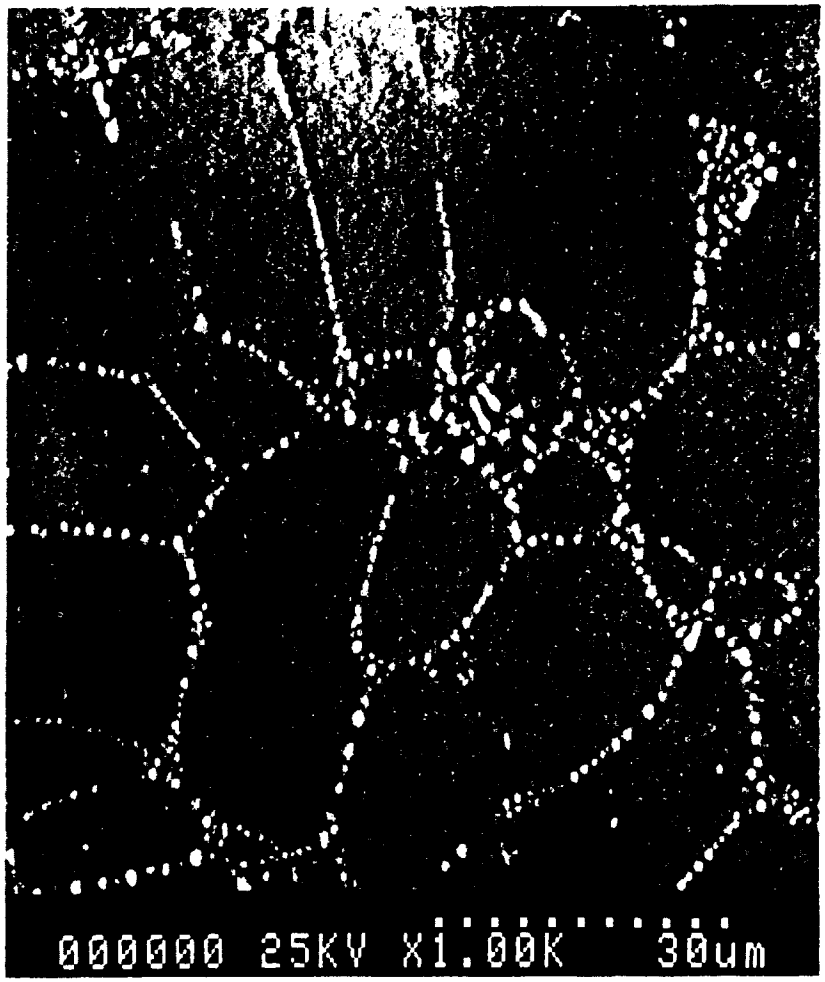


FIGURE 1. SEM OF THE ARC CAST ALLOY SHOWS MASSIVE SEGREGATION OF CERIUM AT THE PRIOR BETA TITANIUM GRAIN BOUNDARIES.

Ti - Ce Phase Diagram

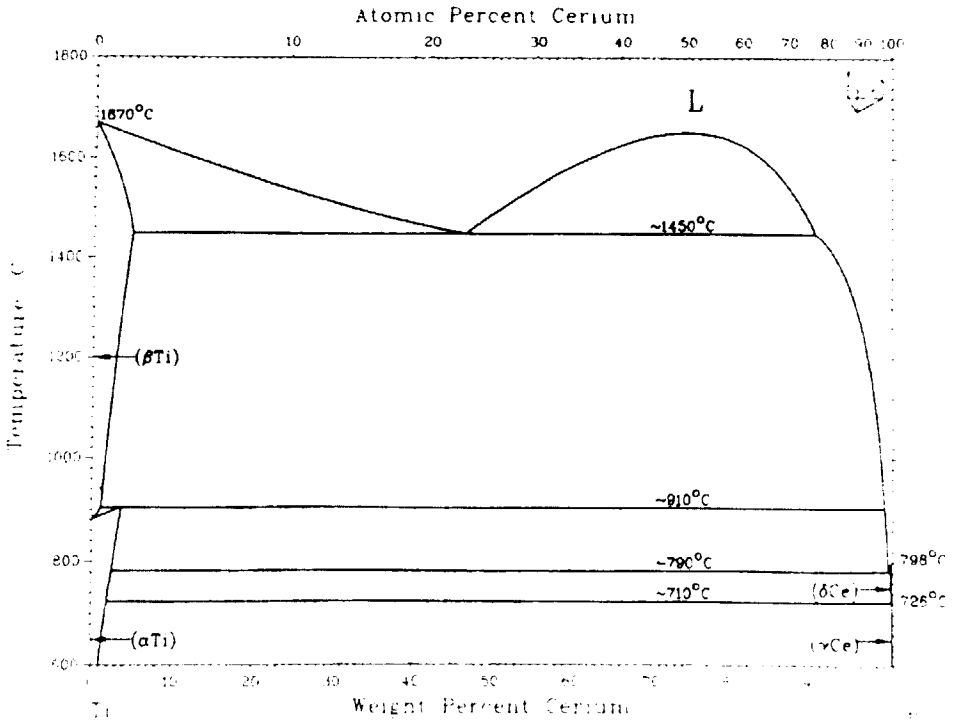


FIGURE 2. THE TITANIUM-CERIUM BINARY EQUILIBRIUM PHASE DIAGRAM REVEALS A MONOTECTIC IMMISCIBILITY GAP IN THIS SYSTEM.

Drop Tube - Gas Cooled

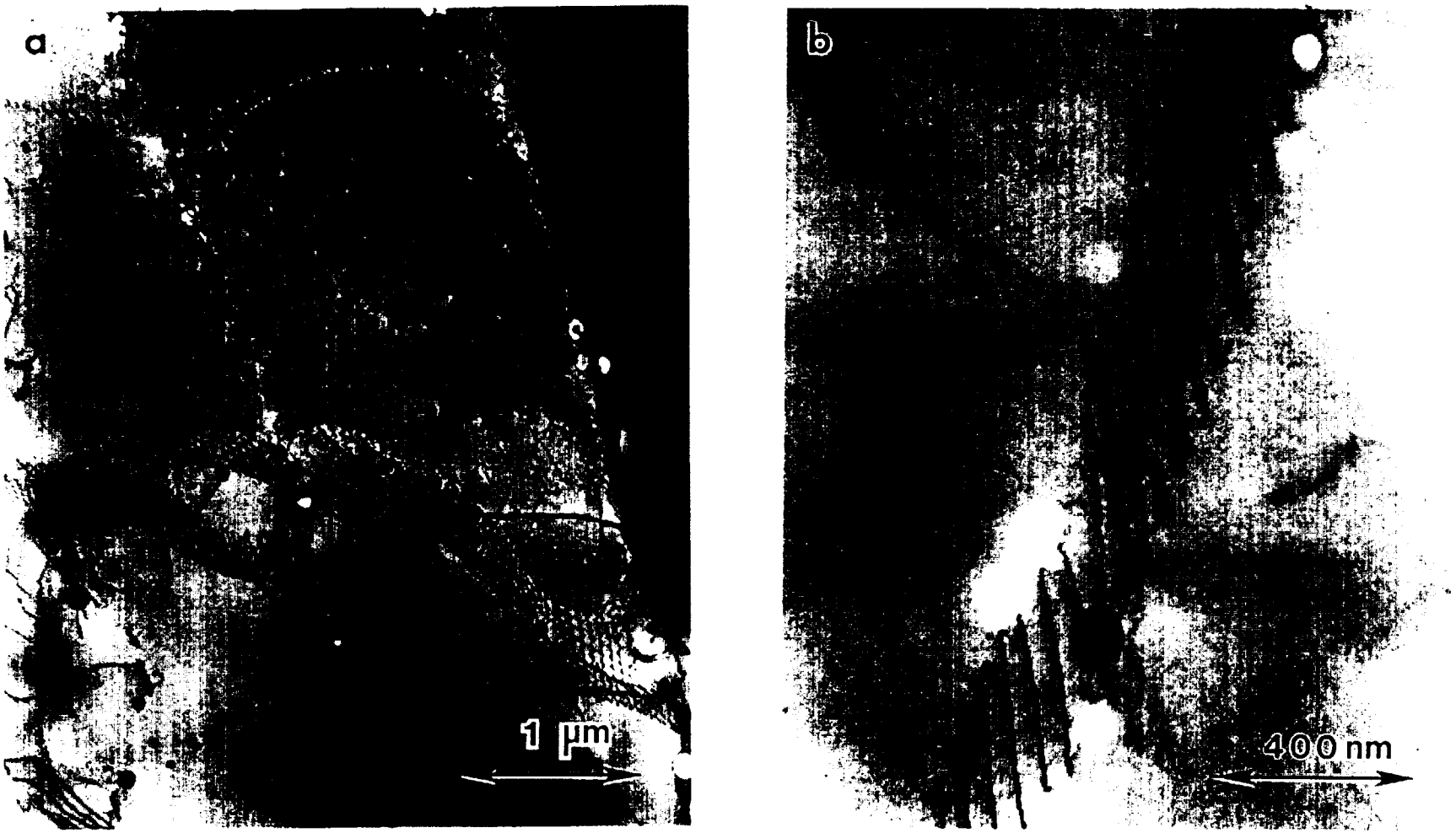


FIGURE 3. THE BRIGHT FIELD TEM IMAGES IN (a) AND (b) OF THE MICROSTRUCTURE FROM THE GAS COOLED DROP TUBE SAMPLES REVEAL AN ALPHA HEXAGONAL TITANIUM MATRIX WITH NUMEROUS LOW ANGLE GRAIN BOUNDARIES THAT ARE DECORATED WITH CERIUM PRECIPITATES. WITHIN THE ALPHA TITANIUM MATRIX, PRECIPITATION OF CERIUM IS ABSENT.

Undercooled and Rapidly Solidified

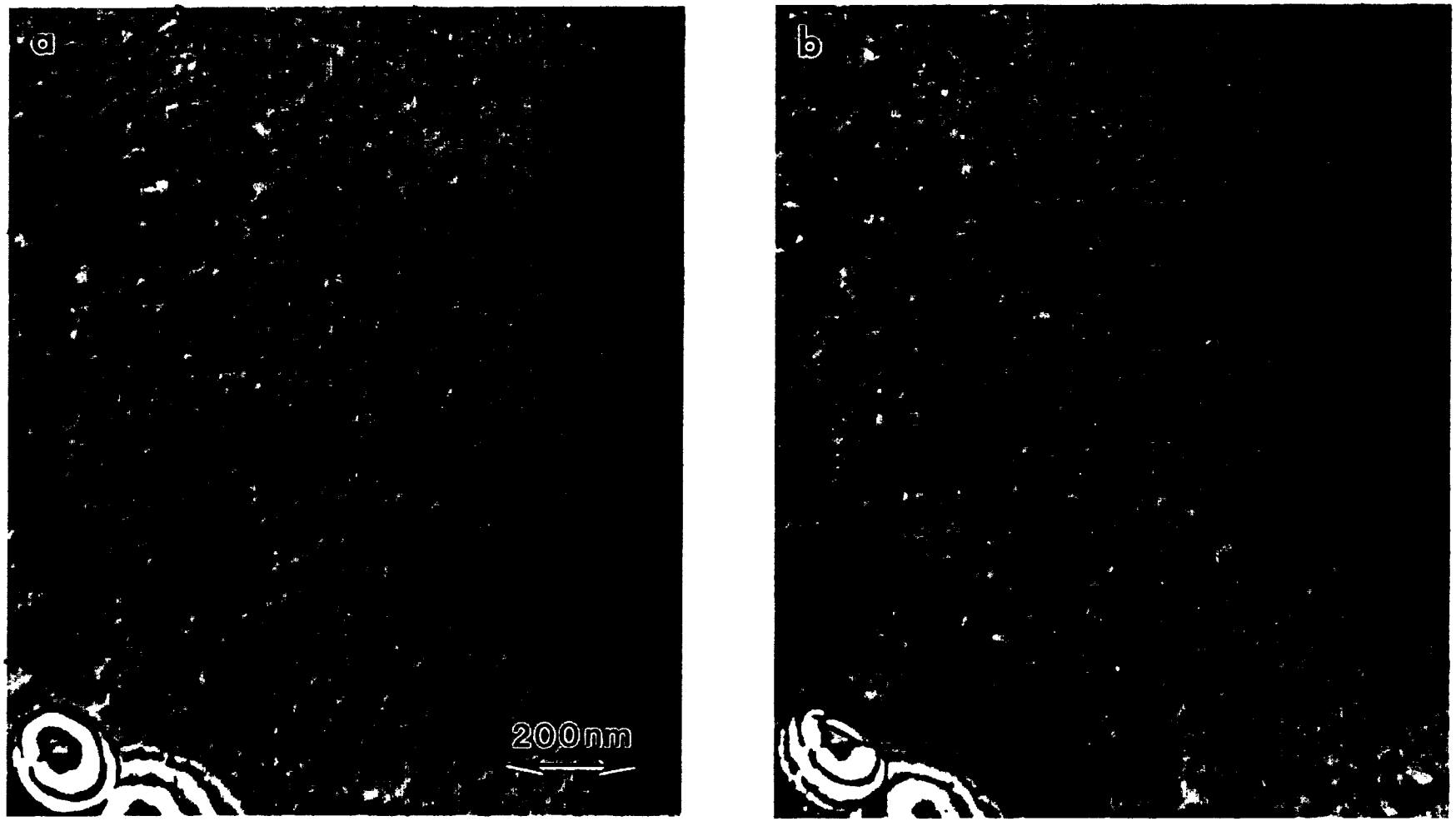
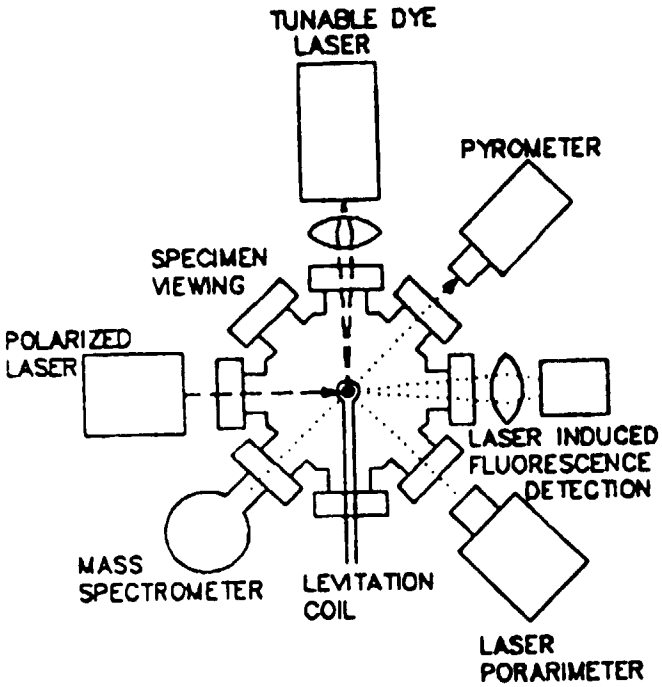
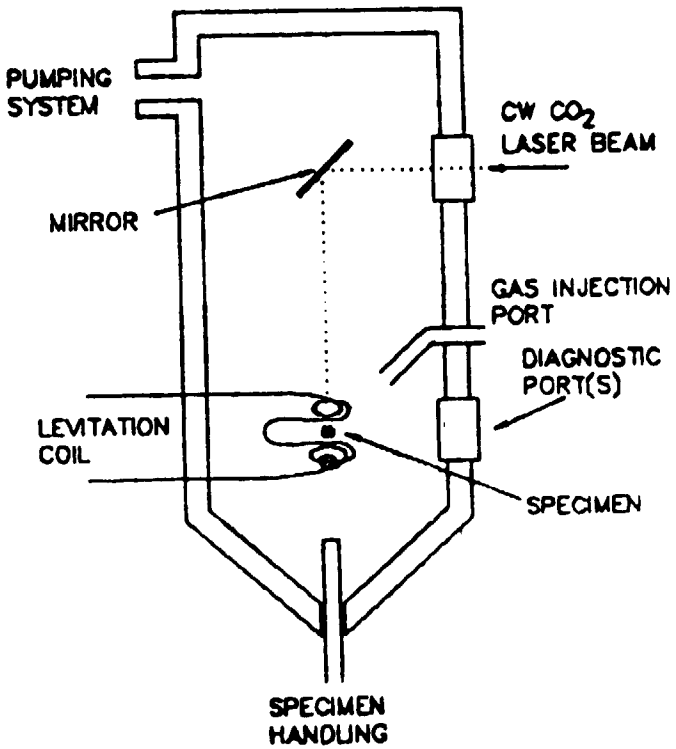


FIGURE 4. TEM EXAMINATION OF THE UNDERCOOLED AND RAPIDLY SOLIDIFIED MICROSTRUCTURE REVEALS A FINE DISTRIBUTION OF PARTICLES IN THE ALPHA TITANIUM MATRIX. THE PARTICLES, WHICH ARE 5 TO 50 nm IN SIZE, APPEAR TO BE RANDOMLY DISTRIBUTED IN (a). HOWEVER, TILTING THE SAME REGION OF THE SAMPLE IN (b) INDICATES THAT THE PARTICLES HAVE FORMED IN LAYERS DURING THE BETA TO ALPHA TRANSFORMATION.

SUMMARY OF CONTAINERLESS PROCESSING FACILITIES AT INTERSONICS

* Electromagnetic, acoustic and aerodynamic levitation
* Laser beam and arc lamp heating systems
* State of the art non-contact temperature and optical property measurement facilities
* Non-intrusive diagnostic techniques with LIF and mass spectrometer
* Controlled atmosphere processing
* Gas quenching
* Proven microgravity processing technology

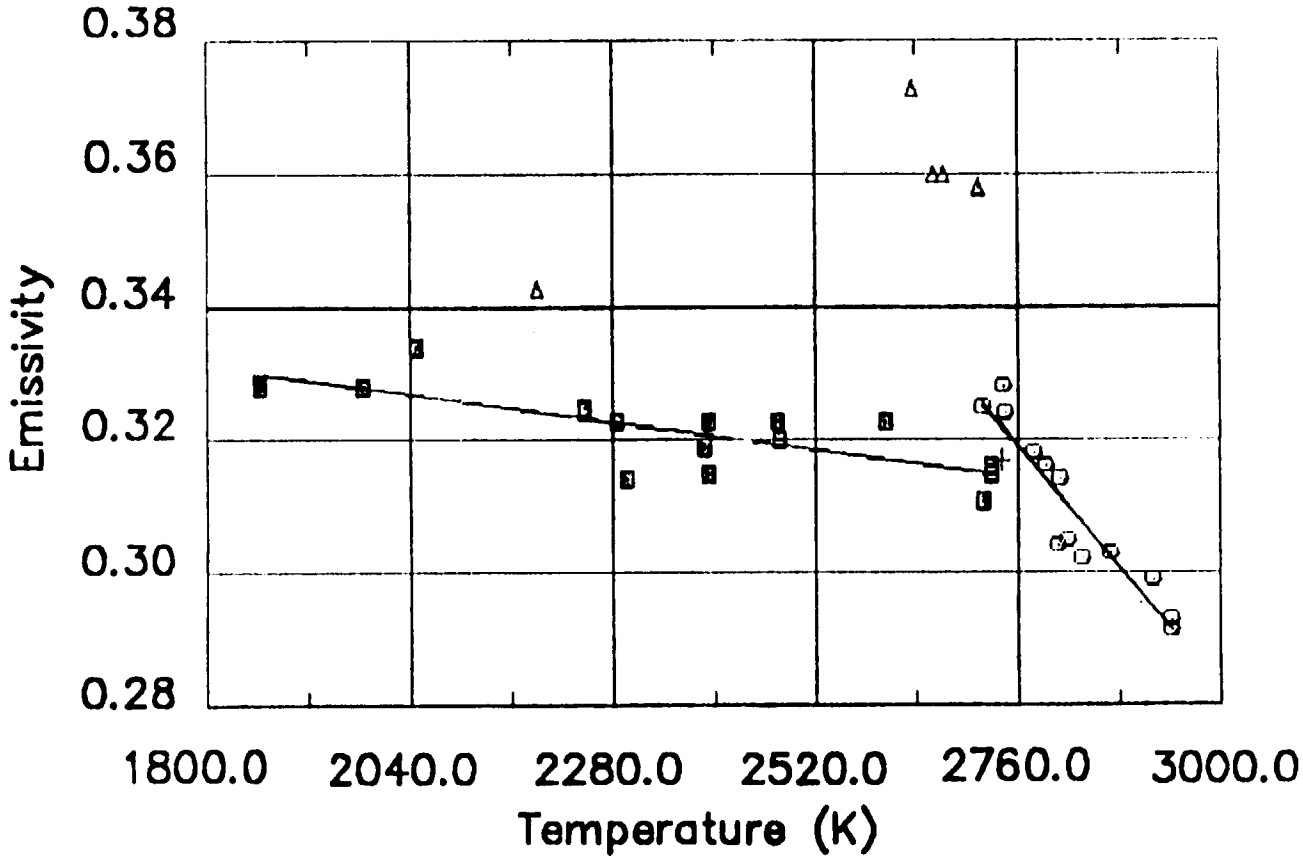




INTERSONICS GROUND-BASED ELECTROMAGNETIC LEVITATOR

INTERSONICS
INCORPORATED

The normal spectral emissivity of Niobium as a function of temperature at 0.6328 μm.



○ – Liquid Niobium ■ – Solid Niobium ▲ – Solid Niobium before clean up
+ – Values at Tm and 0.65 μm (D. W. Bonnell)

D3324.DWG

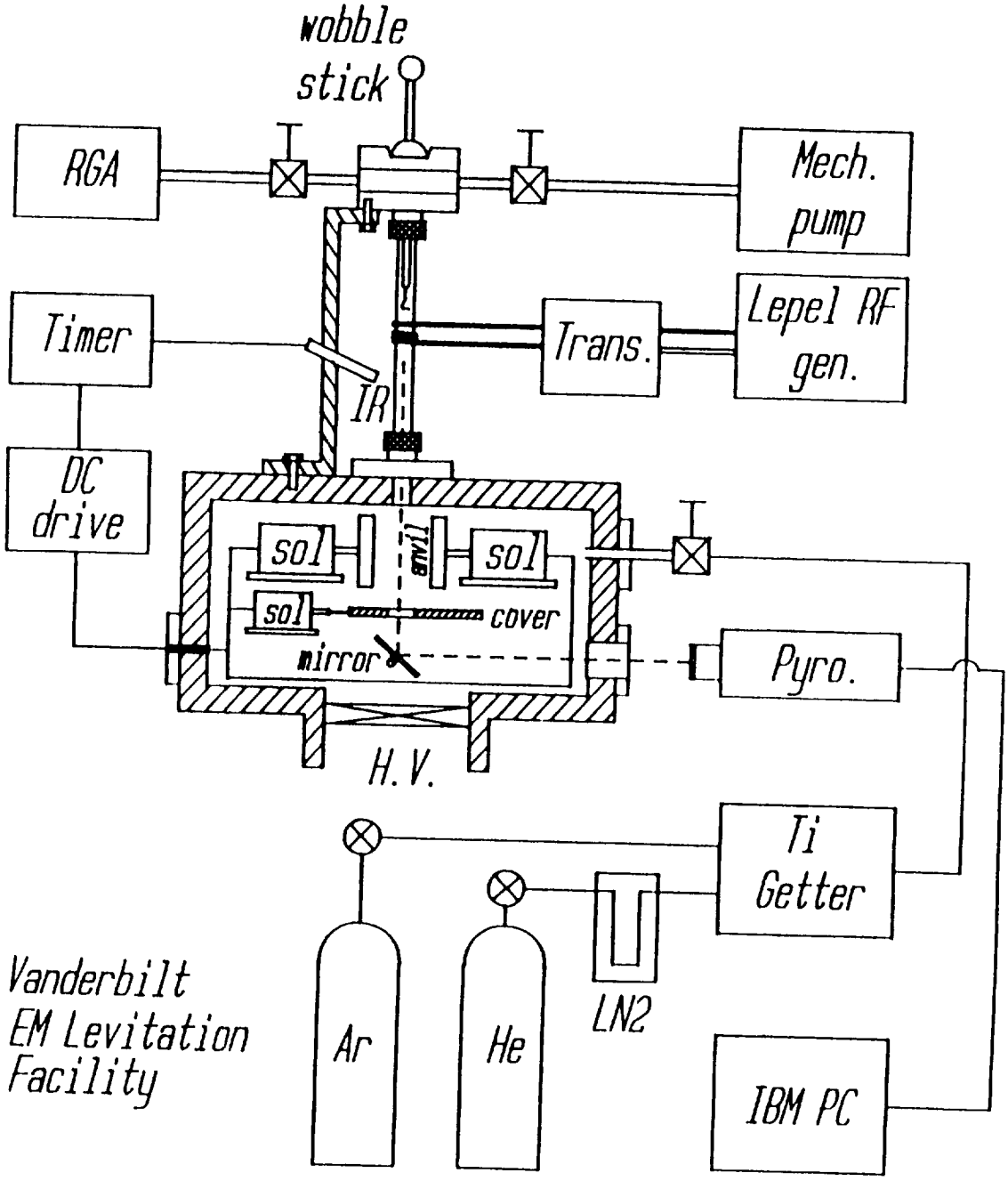
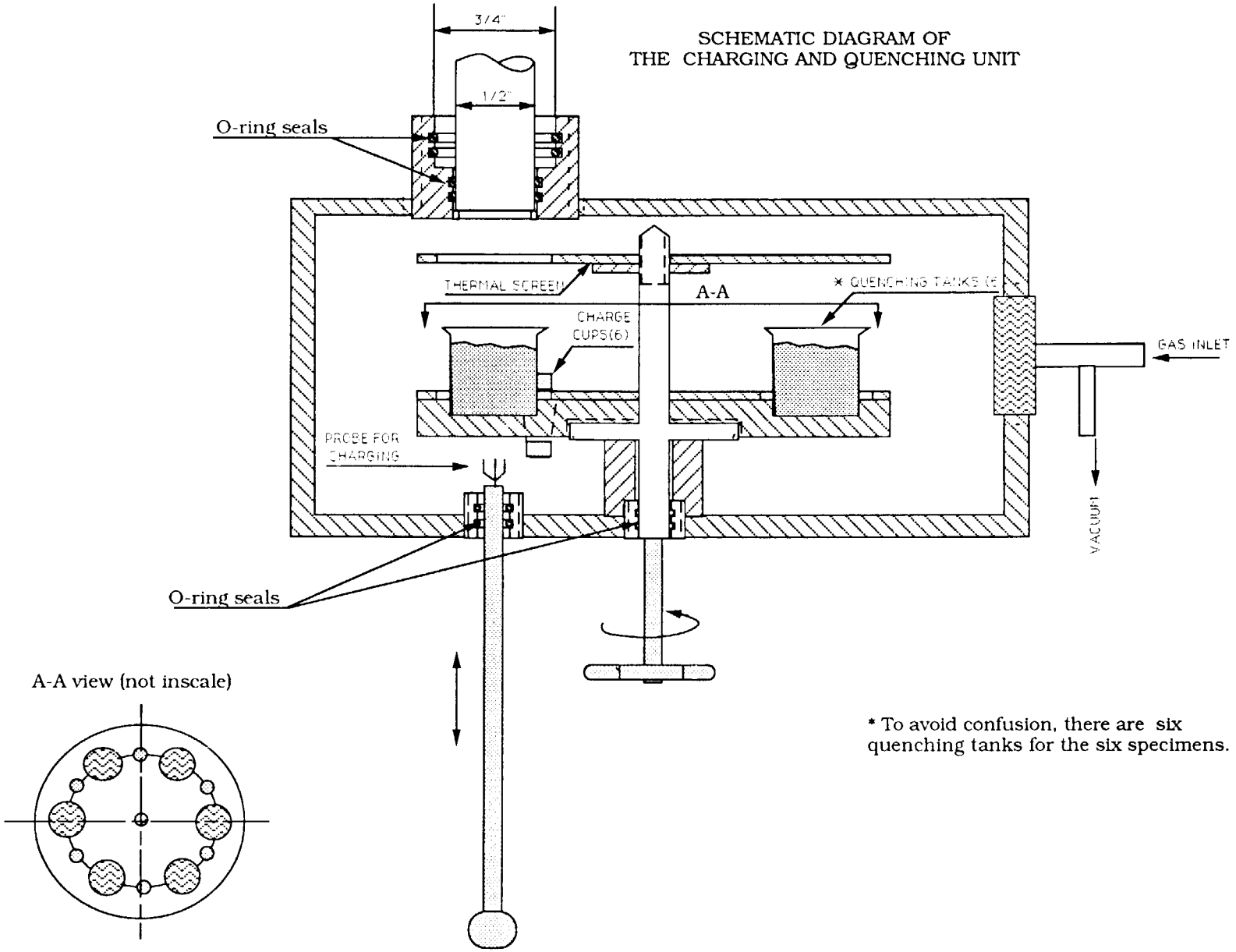
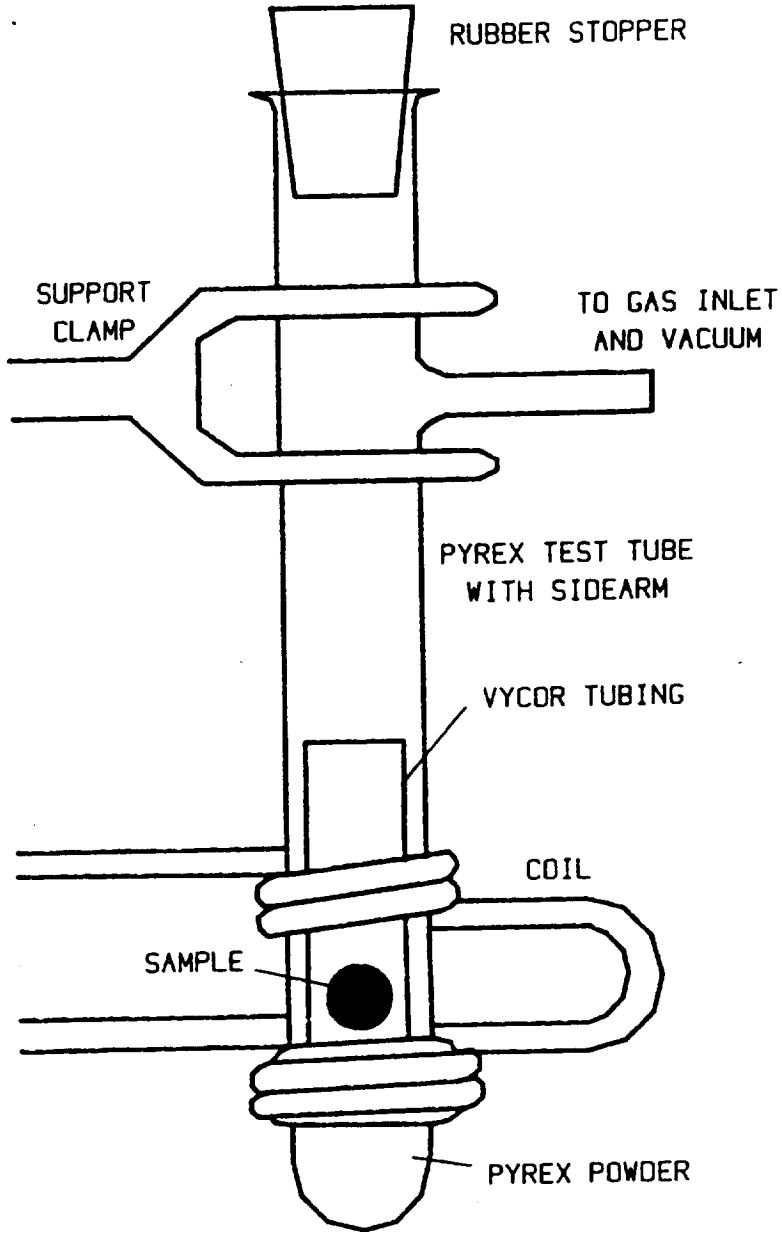


Figure 5. Schematic of the Electromagnetic Levitation Facility at Vanderbilt University.


SCHEMATIC DIAGRAM OF
THE CHARGING AND QUENCHING UNIT
3/4"
1/2"
O-ring seals
THERMAL SCREEN
A-A
* QUENCHING TANKS (6
CHARGE
CUPS(6)
GAS INLET
PROBE FOR
CHARGING
VACUUM
O-ring seals
A-A view (not inscale)
* To avoid confusion, there are six
quenching tanks for the six specimens.


RUBBER STOPPER
SUPPORT CLAMP
TO GAS INLET AND VACUUM
PYREX TEST TUBE WITH SIDEARM
VYCOR TUBING
COIL
SAMPLE
PYREX POWDER

SUMMARY OF RESULTS FOR EM LEVITATION OF Nb 16 TO 22 At.% Si ALLOYS

EUTECTIC RANGE

Metallic glass formation observed only on superheated and splatted samples.

Undercooled and splatted samples had extremely fine regular and irregular eutectic microstructures.

Undercooled gas quenched samples solidified with microstructures identical to drop tube counterparts.

Nb_3Si RANGE

Nb_3Si growth directly from the liquid for a wide composition range in splatted samples.

The depth to which this solidification path is followed depends on the previous bulk undercooling.

Undercooled and gas quenched samples tend to solidify with primary Nb_5Si_3 and a metastable α-Nb + Nb_5Si_3 eutectic. The peritectic Nb_3Si does not form.

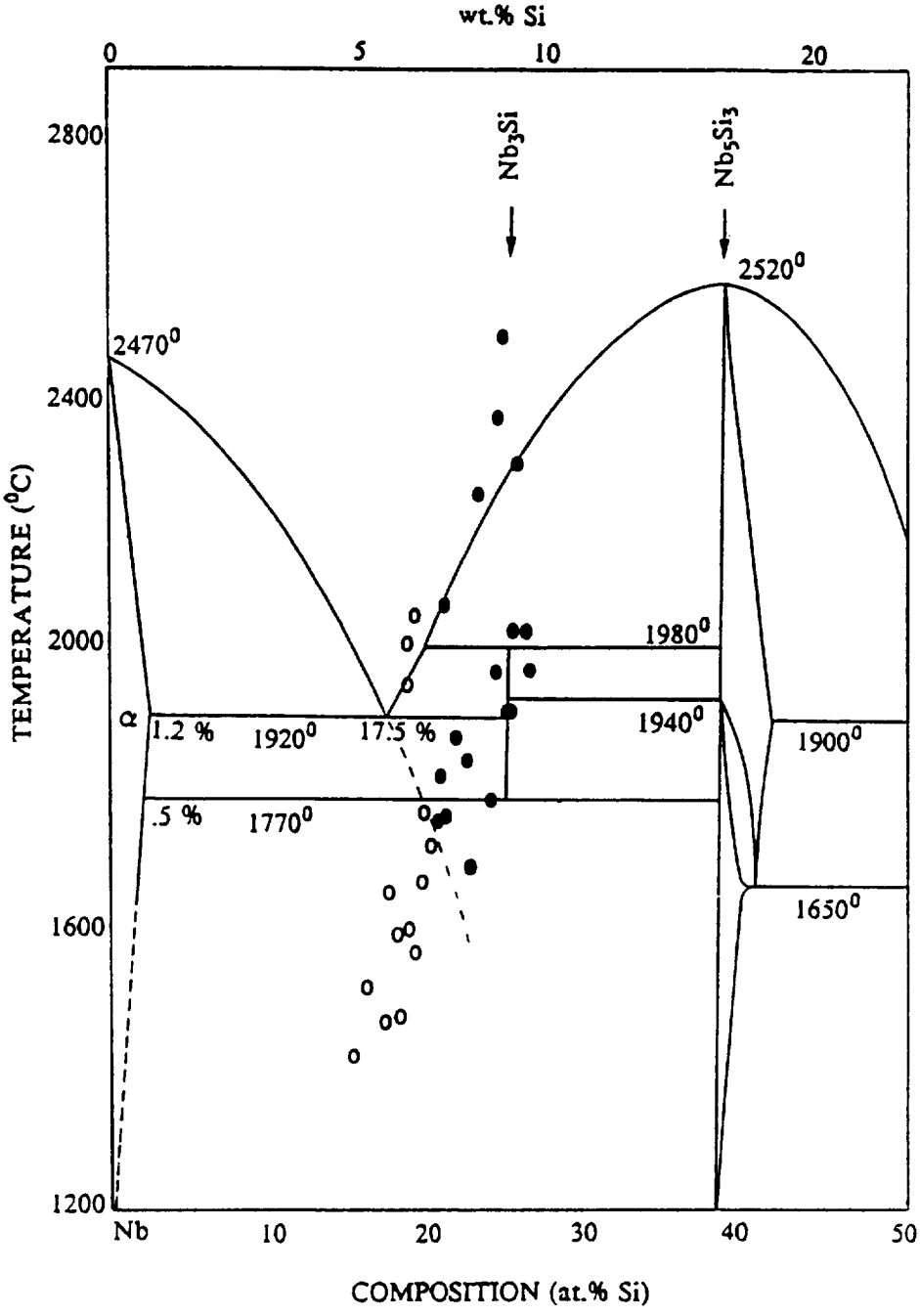
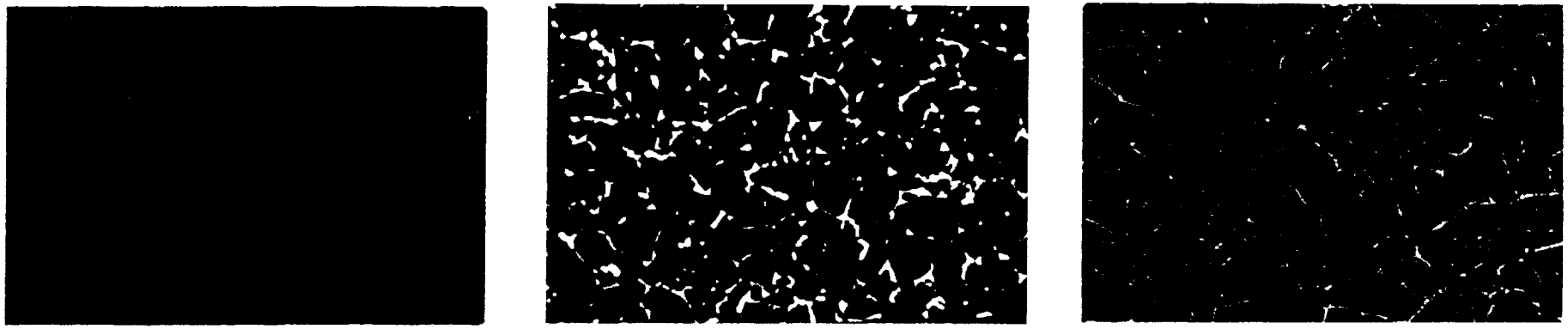
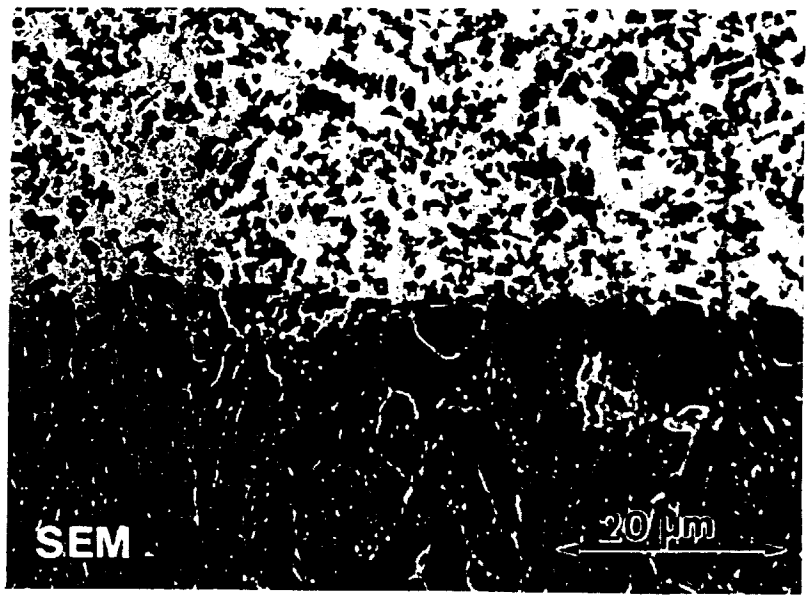


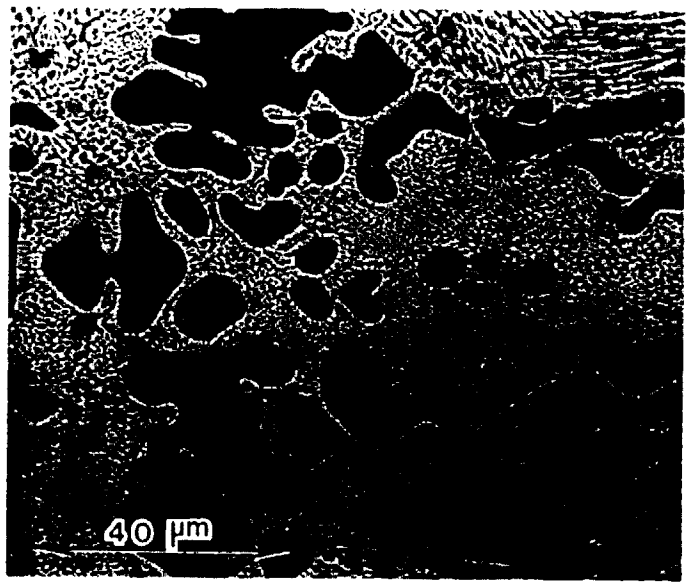
Figure 6. Nb-Si phase diagram. Empty symbols on the phase diagram represent undercooling prior to splatting and composition of samples that solidified without primary phases. Solid symbols represent those samples that solidified with primary phases.

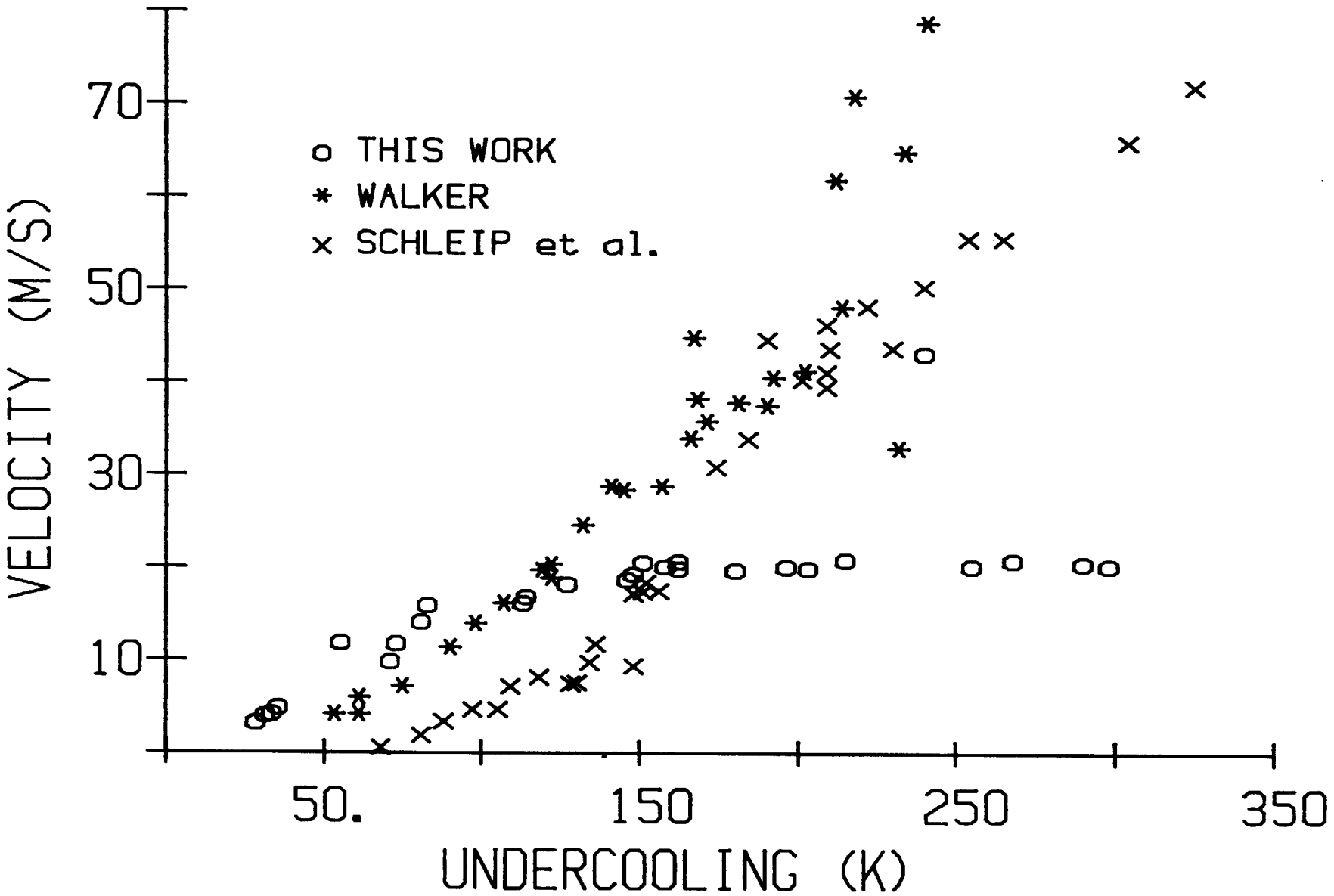
Nb 16 TO 20 At.% Si

Nb 22 TO 27 At.% Si








o THIS WORK
* WALKER
x SCHLEIP et al.
VELOCITY (M/S)
70
50
30
10
50.
150
250
350
UNDERCOOLING (K)

N91-21336

Containerless Processing Projects
Center for the Space Processing of Engineering Materials
Vanderbilt University, Nashville, Tennessee

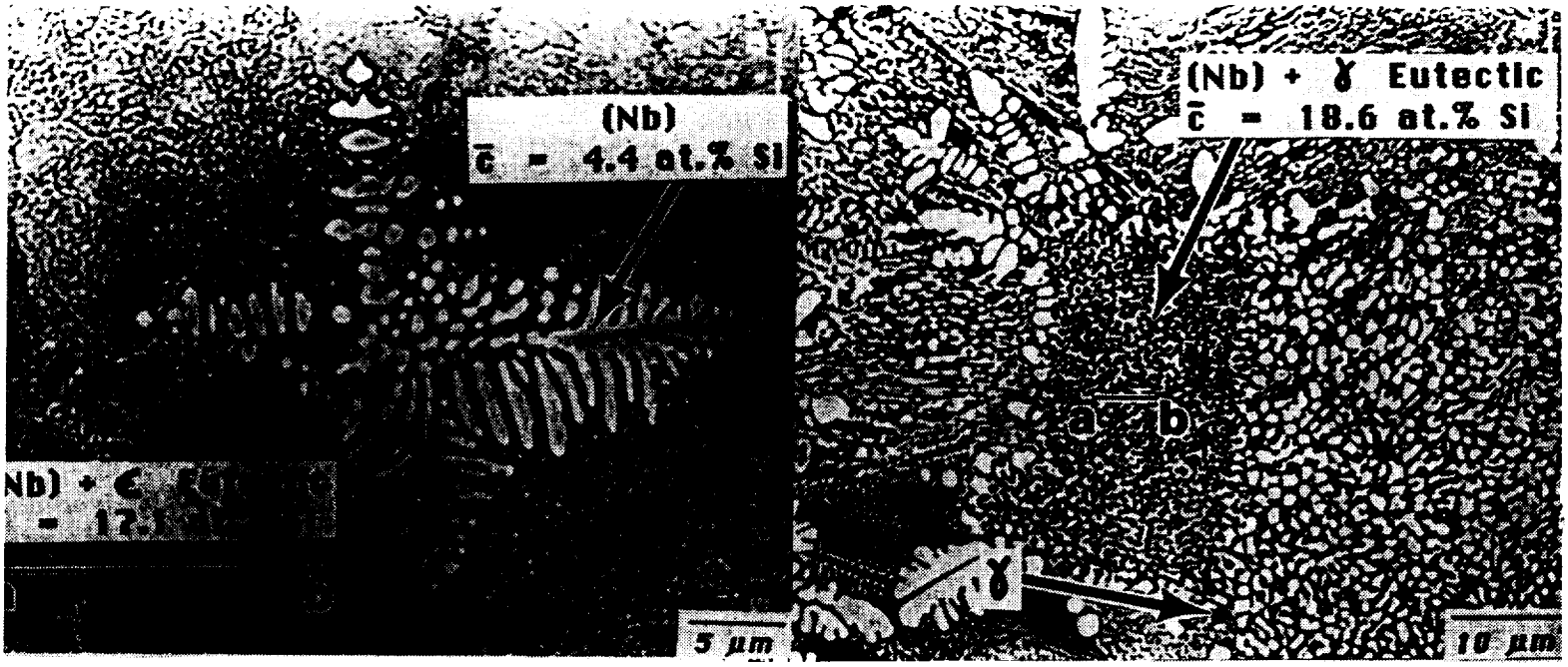
Alloy Investigations

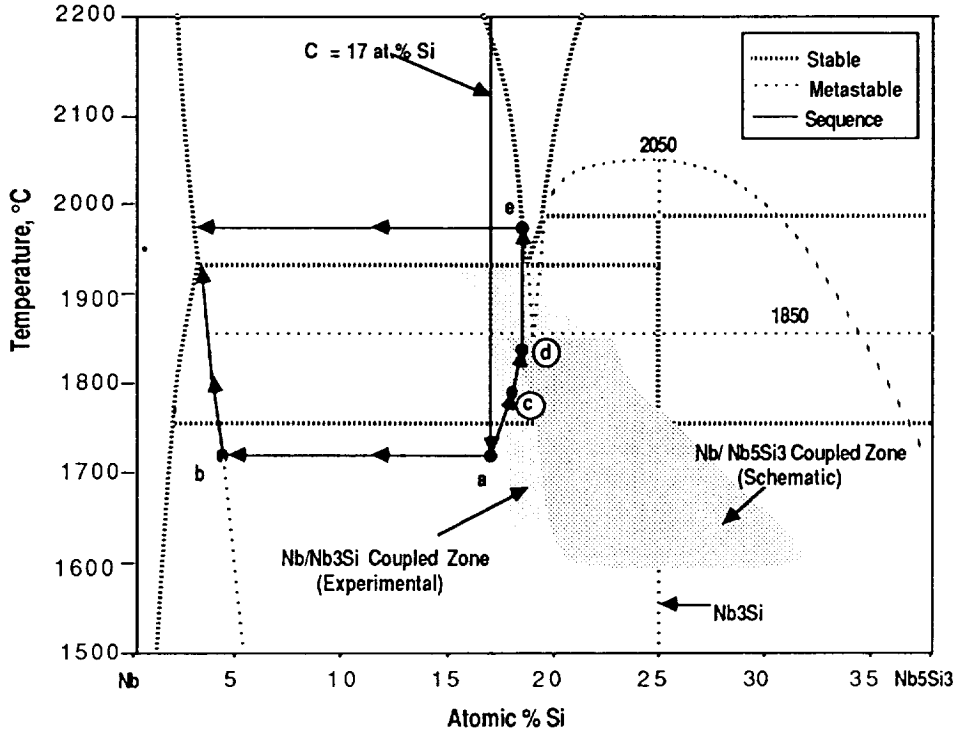
- Ti-Al
- Ti-rare earth
- Nb-Ti
- Nb-Hf
- Nb-Si

Purification

- Nb
- Ir
- Ru

Development of Aerodynamic
Levitation For Liquids





Advantages of Glass Coating "Better reproducibility of achievement of high undercoolings"

1. Nearly independent of the surrounding gas atmosphere.

2. Nearly independent of cooling rate.

3. In ground-based work using viscous borosilicate glass coatings on nickel- and iron-based alloys, undercoolings above 300 K have been easily achieved.

4. Note that the undercoolings attained in ground-based experiments with glass coatings are equivalent to those attained under ultra-high vacuum without coatings.

Because:

(Crystalline inclusions and surface convection promote heterogeneous nucleation.)

1. Coatings prevent the formation of inclusions (e.g., oxides) due to reactions with the gas phase on the metal surface.

2. If such reactions occur, the softened glass will prevent the reaction products from forming crystalline inclusions.

3. Softened glass has a scavenging effect on inclusions within the metal specimen.

4. Glass coatings reduce surface convection due to lower surface tension and to increased viscous drag.

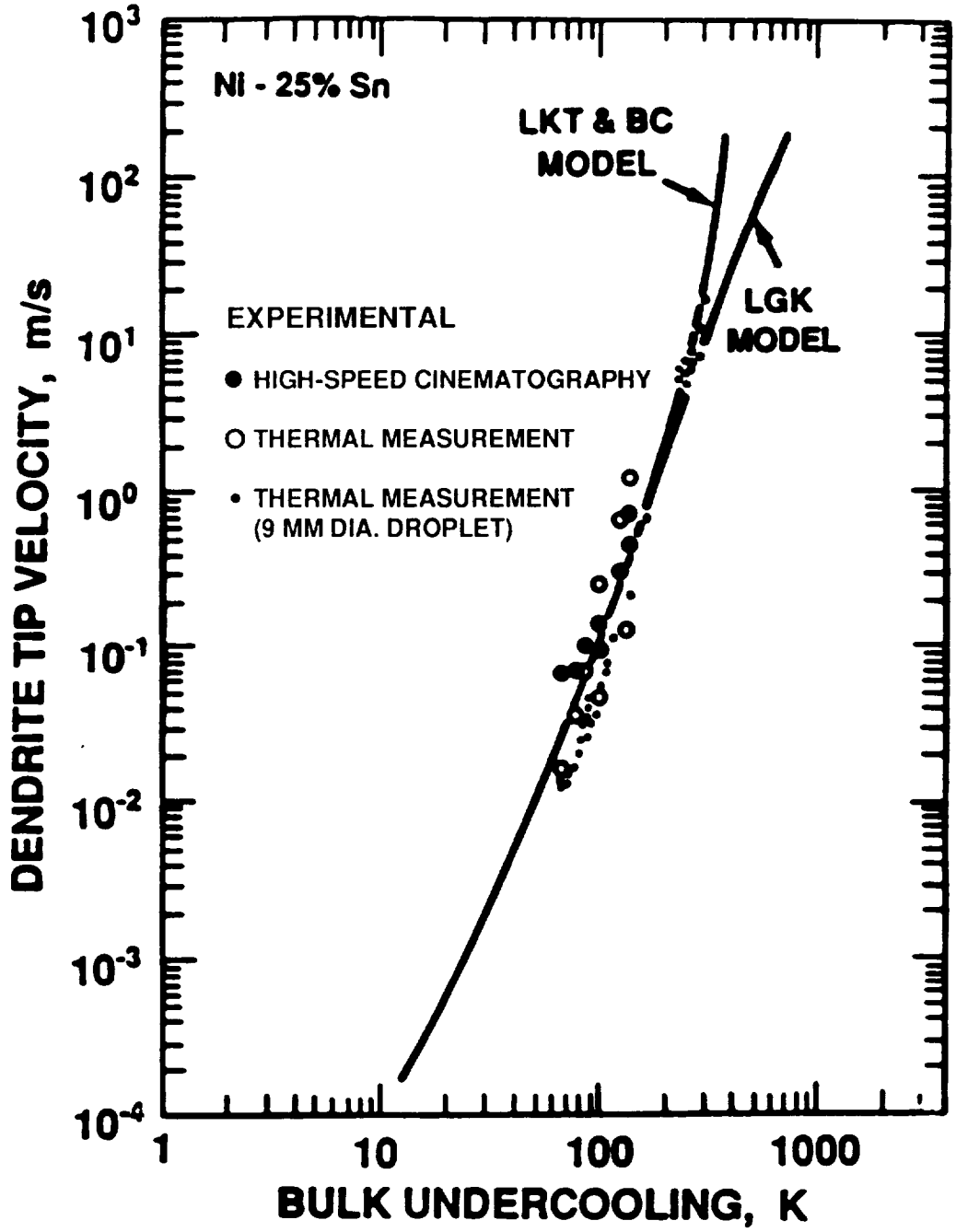


Figure 7. Dendrite tip velocity versus bulk undercooling for Ni-25 wt % Sn alloy. Experimental results and calculated curves based on the models developed by Lipton, Kurz, and Trivedi [35], Boettinger and Coriell [34] (LKT-BC), and by Lipton, Glicksman, and Kurz [33] (LGK).

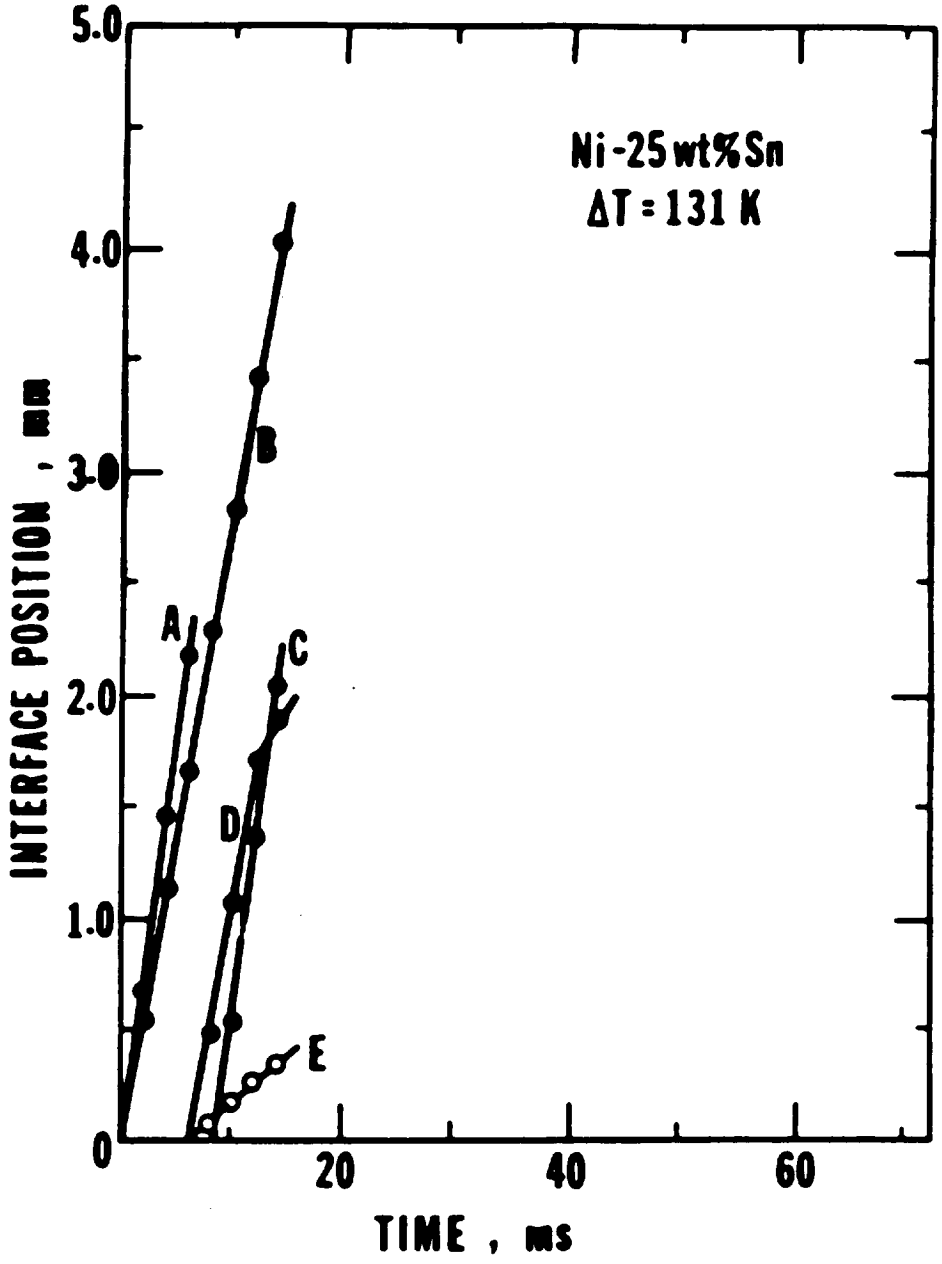


Figure 8. Plot of interface position as a function of time for the growth fronts in Fig. 1. The results for the four dendrites A, B, C, and D fall on straight lines, indicating that growth occurred at constant velocity (steady state).

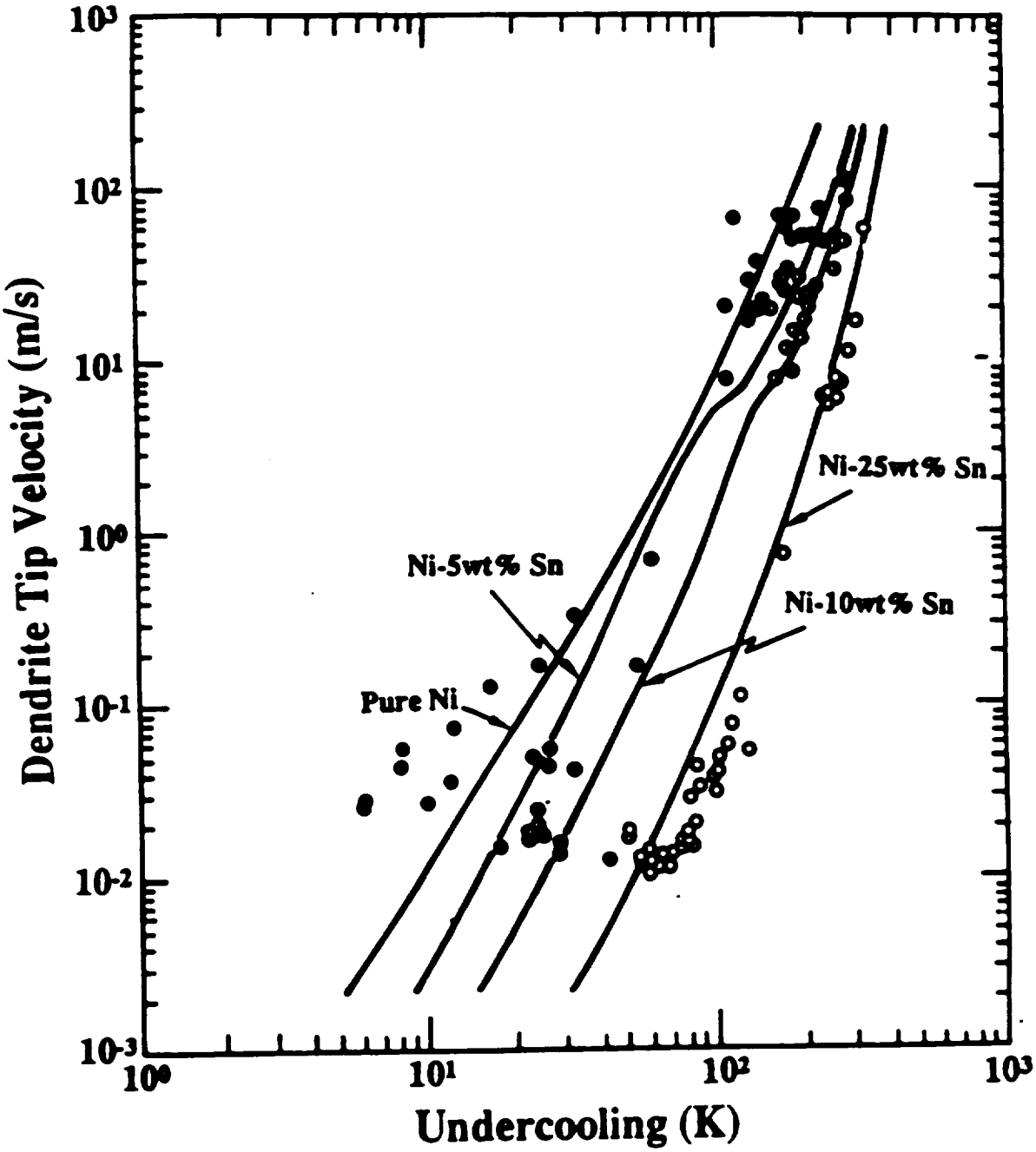


Figure 9. Log-log plot of dendrite tip velocity versus undercooling for pure Ni, Ni-5 wt% Sn, Ni-10 wt% Sn.

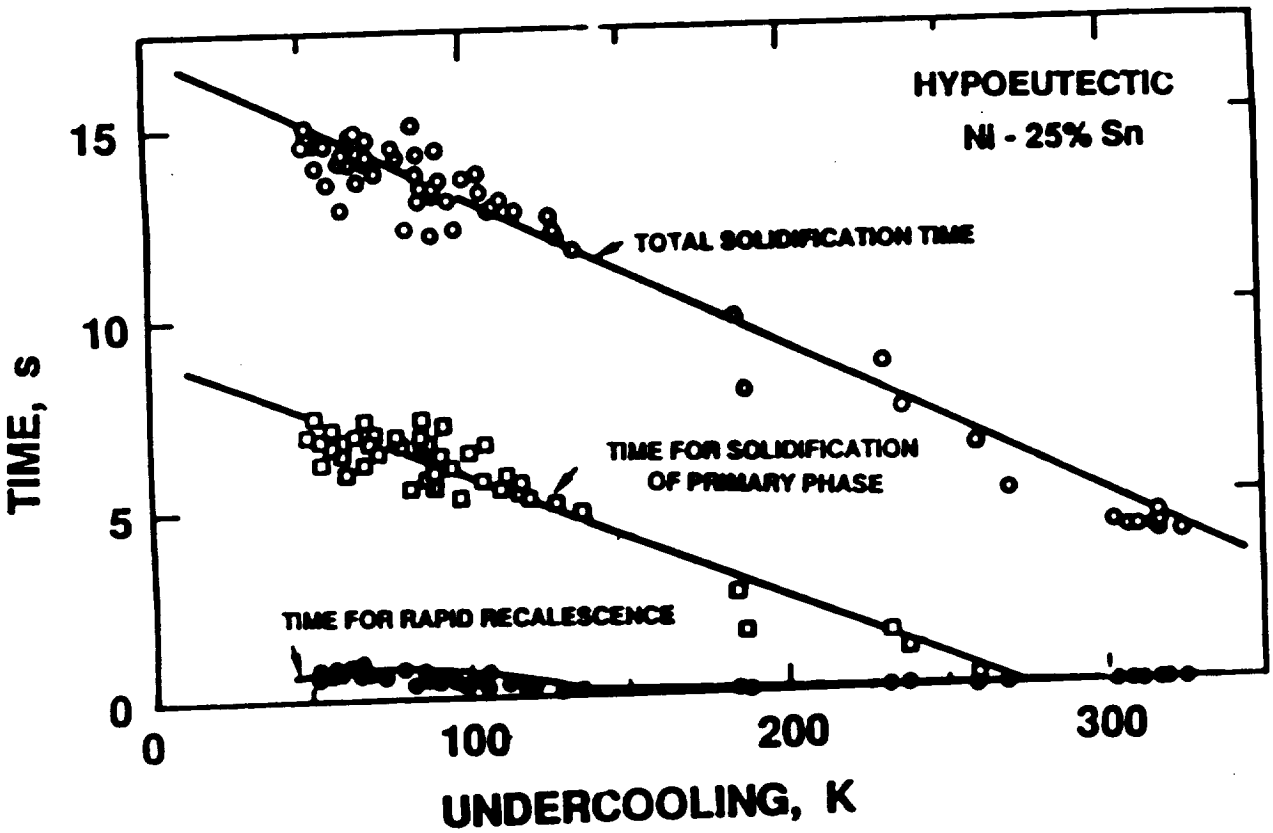


Figure 10. Plot of experimental results for solidification time, primary phase solidification time, and recalescence time versus initial undercoooling for Ni-25 wt% Sn alloy specimens.

PHYSICAL ACOUSTICS RESEARCH

- PRIMARY RESEARCH OBJECTIVE: Study fundamental acoustics of the DROP PHYSICS MODULE
- Develop Advanced Chamber Systems for Acoustic and Sample Characterization
- Study Sample Interaction With Acoustic Field
- Study Shaping of Liquid Drops by Acoustic Forces
- Study the acoustic torque as a function of sample position and shape
- Study Levitated Sample Stability and the Effects of Feedback Systems

PHYSICAL ACOUSTICS RESEARCH

- PRIMARY RESEARCH OBJECTIVE:
  Study fundamental acoustics of the DROP PHYSICS MODULE

- Develop Advanced Chamber Systems for Acoustic and Sample Characterization
  - Study optimization of chamber acoustic power
  - Study chamber - sound source coupling
  - Study different chamber dimensions
  - Study different sound source orientations

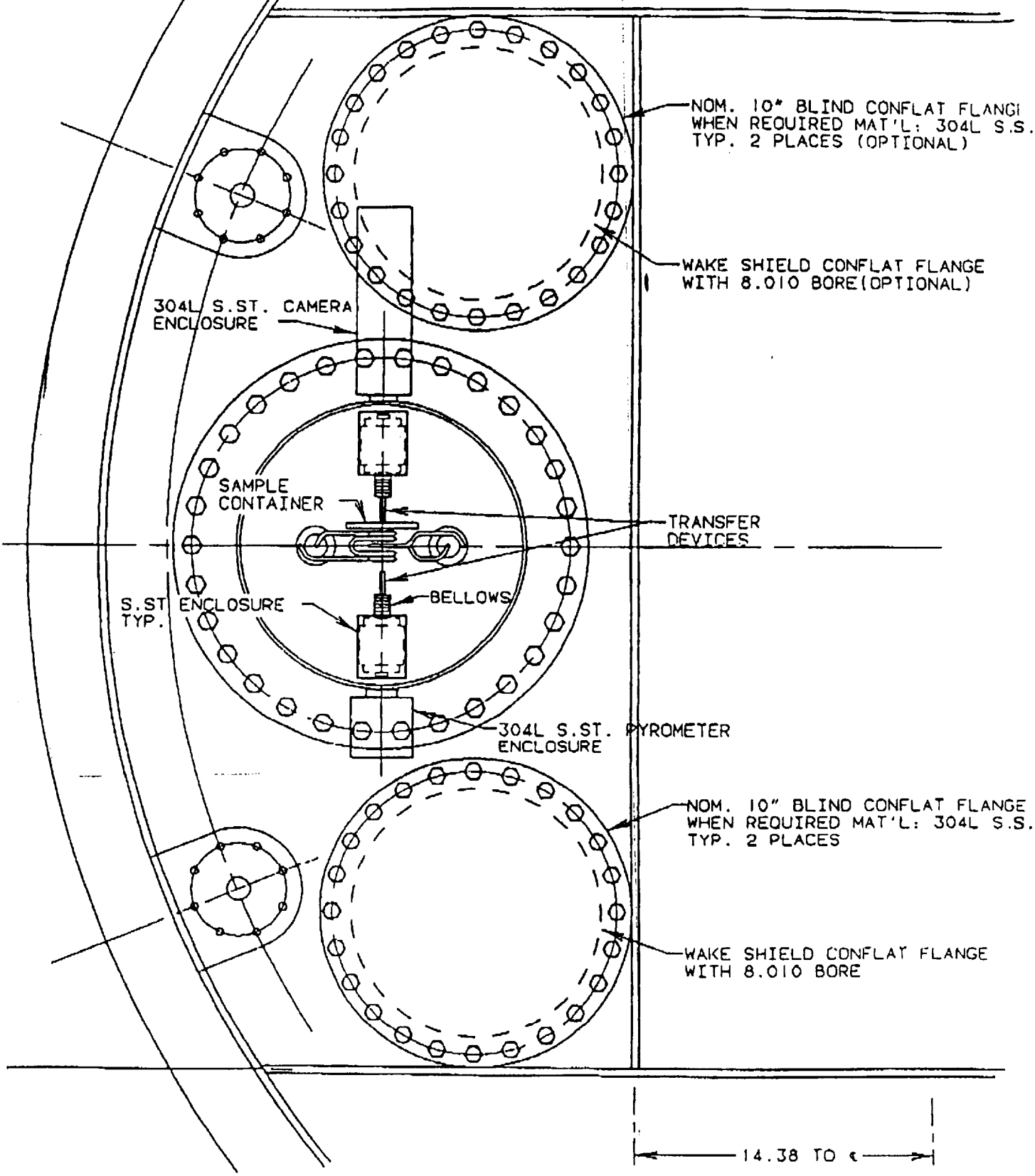
- Study Sample Interaction With Acoustic Field
  - Study sample scattering effects on higher harmonics as a function of:
    - Sample size
    - Sample shape
    - Sample position

- Study Shaping of Liquid Drops by Acoustic Forces
  - Study sample shape as a function of sample size, position, and acoustic field intensity
  - Study acoustic force and pressure profiles for various normal mode resonances

- Study the acoustic torque as a function of sample position and shape

- Study Levitated Sample Stability and the Effects of Feedback Systems
  - Intrinsic instabilities caused by: frequency, amplitude, temperature drifts
  - Extrinsic instabilities caused by: frequency, amplitude, phase modulations of driver signals

Wake Shield /ORNL Levitator



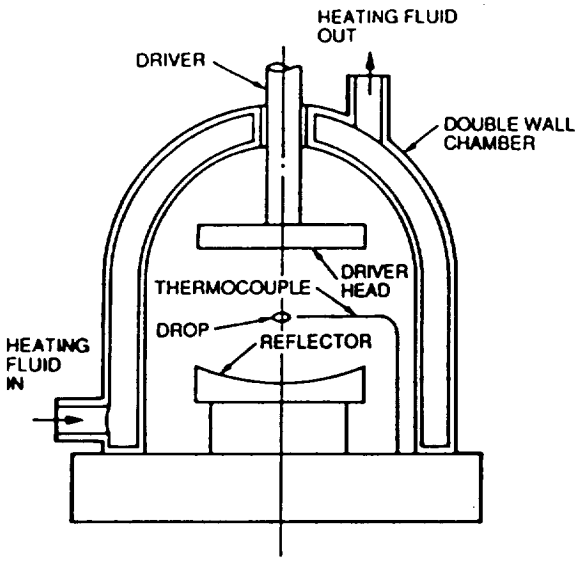


Fig. 1. The heart of a single axis acoustic levitation system.

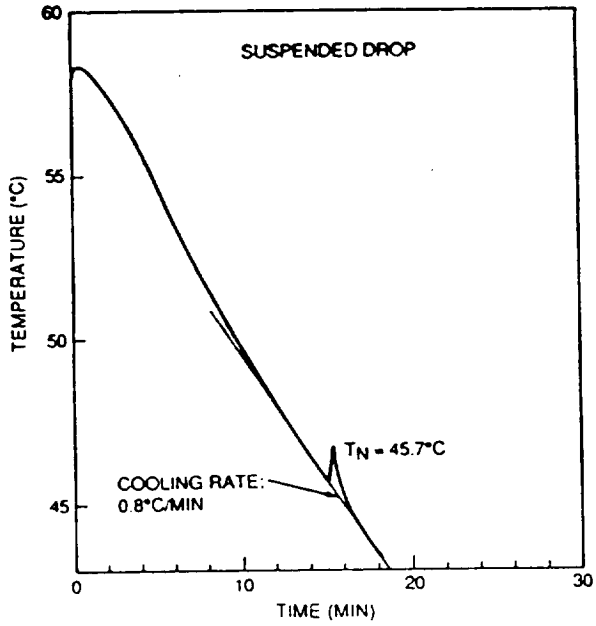


Fig. 3. A cooling curve of a levitated succinonitrile drop.

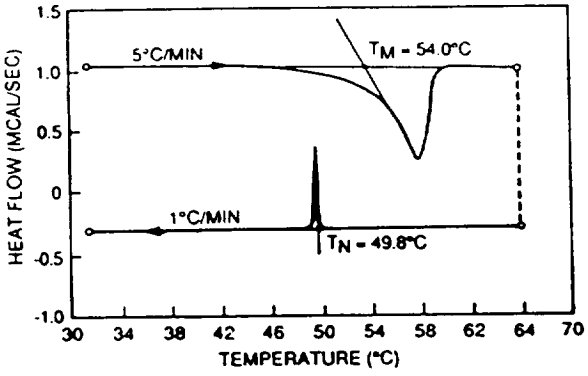


Fig. 2. A DSC thermogram showing melting and solidification of succinonitrile.

INVESTIGATORS ON TEMPUS FOR IML-2 MISSION

Effects on Nucleation by Containerless Processing in Low Gravity - R.J. Bayuzick and W.H. Hofmeister, Vanderbilt University; M.B. Robinson, George C. Marshall Space Flight Center, NASA

Viscosity and Surface Tension of Undercooled Melts - I. Egry, DLR Institute of Space Simulation

Alloy Undercooling Experiments - M.C. Flemings, Massachusetts Institute of Technology; H.D. Brody, University of Pittsburgh

Non-Equilibrium Solidification of Largely Undercooled Melts - D.M. Herlach, DLR Institute of Space Simulation

Metallic Glass Research in Space - W.L. Johnson and H.J. Fecht, California Institute of Technology; M.C. Lee, NASA Headquarters

Measurement of the Viscosity of the Undercooled Melts Under the Conditions of Microgravity - J. Szekely, Massachusetts Institute of Technology

EXPERIMENTAL OUTLINE

- Niobium and zirconium -- pure and ultrahigh pure

- Five processing conditions:
  - high vacuum electromagnetic levitation on earth
  - ultrahigh vacuum electromagnetic levitation on earth
  - high vacuum drop tube
  - ultrahigh vacuum drop tube
  - ultrahigh vacuum electromagnetic processing in LEO

- 100 undercooling experiments planned for each case to obtain histograms of nucleation frequency as a function of temperature
  - maximum undercooling
  - most probable nucleation temperature
  - dispersion in nucleation temperature

PROTOCOL

Insert individual specimen into position for heating

Heat specimen until fully molten and soak for about 30 seconds

At about 2570°C for Nb
At about 1955°C for Zr

Cool specimen until solidification occurs - cooling rate to be determined - most likely to be the natural cooling rate with no power input

Monitor thermal history and brightness history

Monitor vacuum/environment history

Monitor power history - both sets of coils

Repeat for approximately 100 cycles

Place specimen in individual container to retain identity

Repeat entire process for remaining three specimens

Entire set consists of two Nb specimens and two Zr specimens - a total of four specimens

Alloy Undercooling Experiment

OBJECTIVES:

To study the rapid solidification after undercooling of melted metal spheres levitated in microgravity and the resulting microstructures.

To obtain a semi-quantitative understanding of the effect of gravity on the containerless solidification of small diameter metal alloy spheres.

APPROACH:

Melt glass-coated spheres of nickel-tin and iron-nickel alloys in low-gravity by levitation melting.

Cause undercooling by heat withdrawal after power cutoff.

Obtain thermal history (cooling and recalescence) by pyrometry.

Observe solidification behavior in situ during recalescence by cinematography.

Perform metallographic studies on processed specimens.

Perform ground-based experiments for comparison with microgravity experiments.

Preliminary Science Requirements

TEMPUS is nearly ideal for MIT
alloy undercooling experiments.

Specific Concerns

Temperature Measurement
0.4-2 μm due to glass coating
Separate output for each detector
Careful calibration

Sample Rotation and Stability
Minimum possible for Fe-B, Fe-P

Solidification Front Recording
Video: Maximum Possible for Fe-Ni and Ni-Sn
500 fields per second > required for Fe-B, Fe-P

Sample Capture
Quenching, e.g. liquid metal.

IMMEDIATE OBJECTIVES

Solidification of highly undercooled levitation melted alloys.

Solidification of highly undercooled alloys in microgravity.

ULTIMATE OBJECTIVES

Achieve hypercooling.

Rapid solidification of bulk material.

Fundamental understanding of rapid solidification.

ULTIMATE OBJECTIVES

HYPERCOOLING

$$\Delta T = \Delta H / C_p + (T_L - T_S)$$

ΔT (K)	Alloy
500	Ni - 25 wt% Sn
450	Ni - 1 wt% Sn
375	Fe - 25 wt% Ni
440	Ni - 10 wt% Cu

Practical Implications

If hypercooling can be achieved in microgravity:

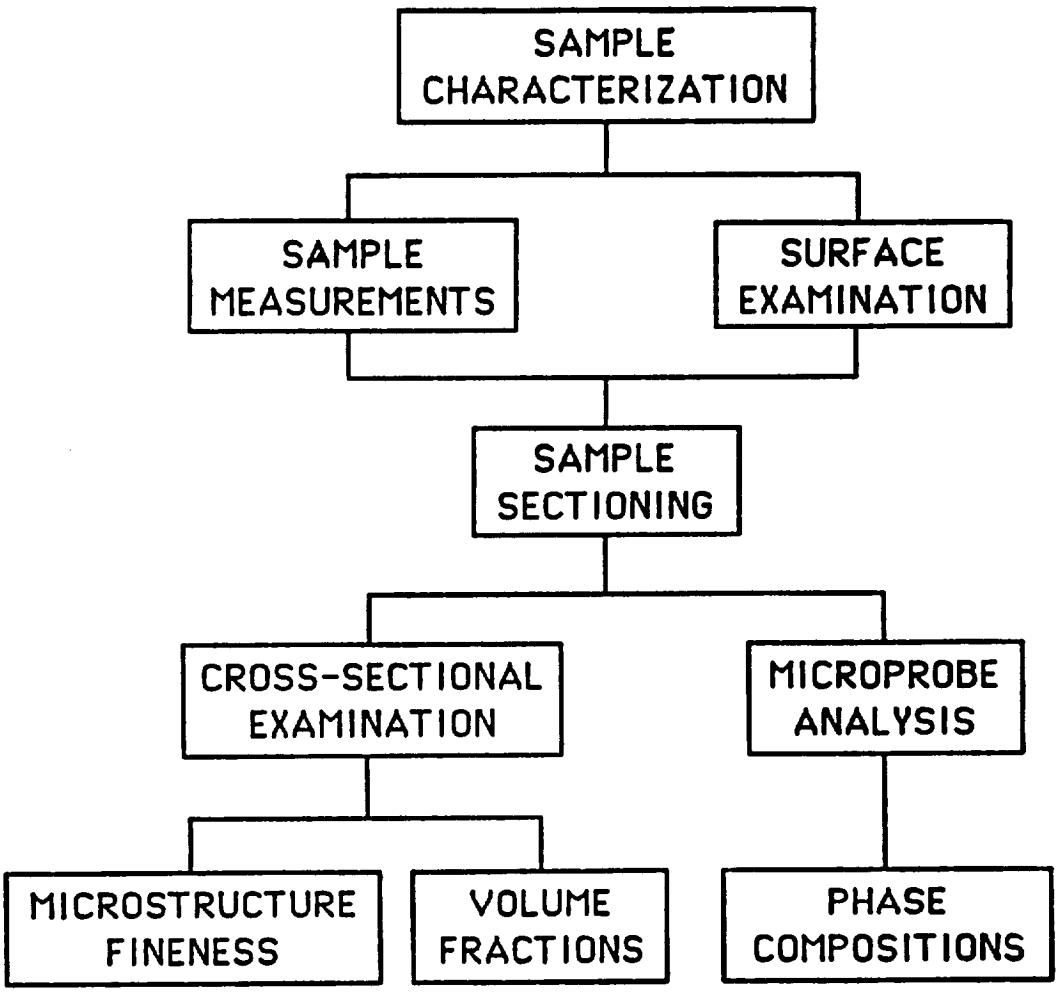
No upper size limit on fully homogeneous and metastable RSP materials as exists at 1 G.

Topics of scientific interest:

Growth rates achieved are thermally controlled (solute trapping).

Morphology of the solidification front (absolute stability).

Convection effects in rapid solidification.


SAMPLE CHARACTERIZATION
SAMPLE MEASUREMENTS
SURFACE EXAMINATION
SAMPLE SECTIONING
CROSS-SECTIONAL EXAMINATION
MICROPROBE ANALYSIS
MICROSTRUCTURE FINENESS
VOLUME FRACTIONS
PHASE COMPOSITIONS

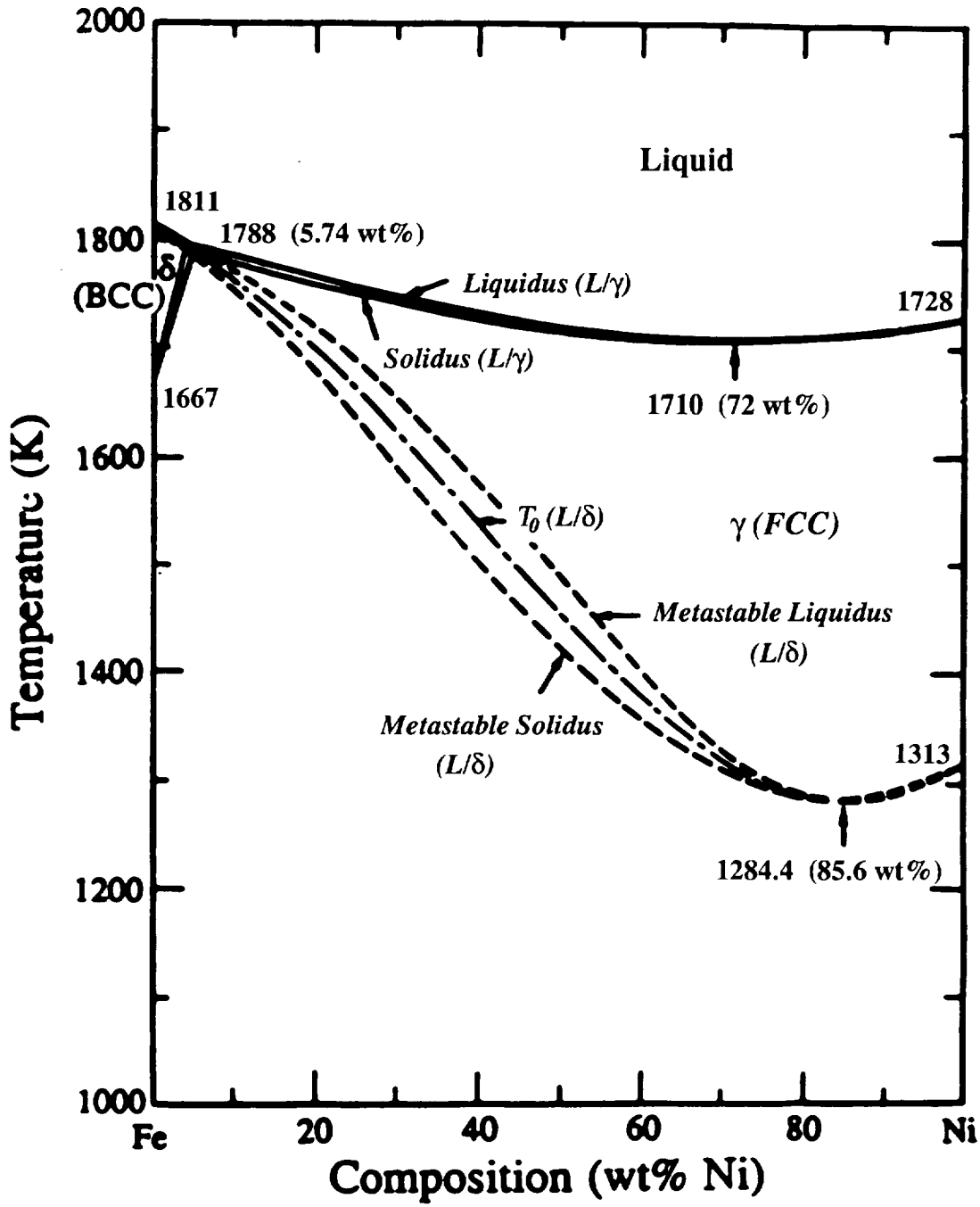
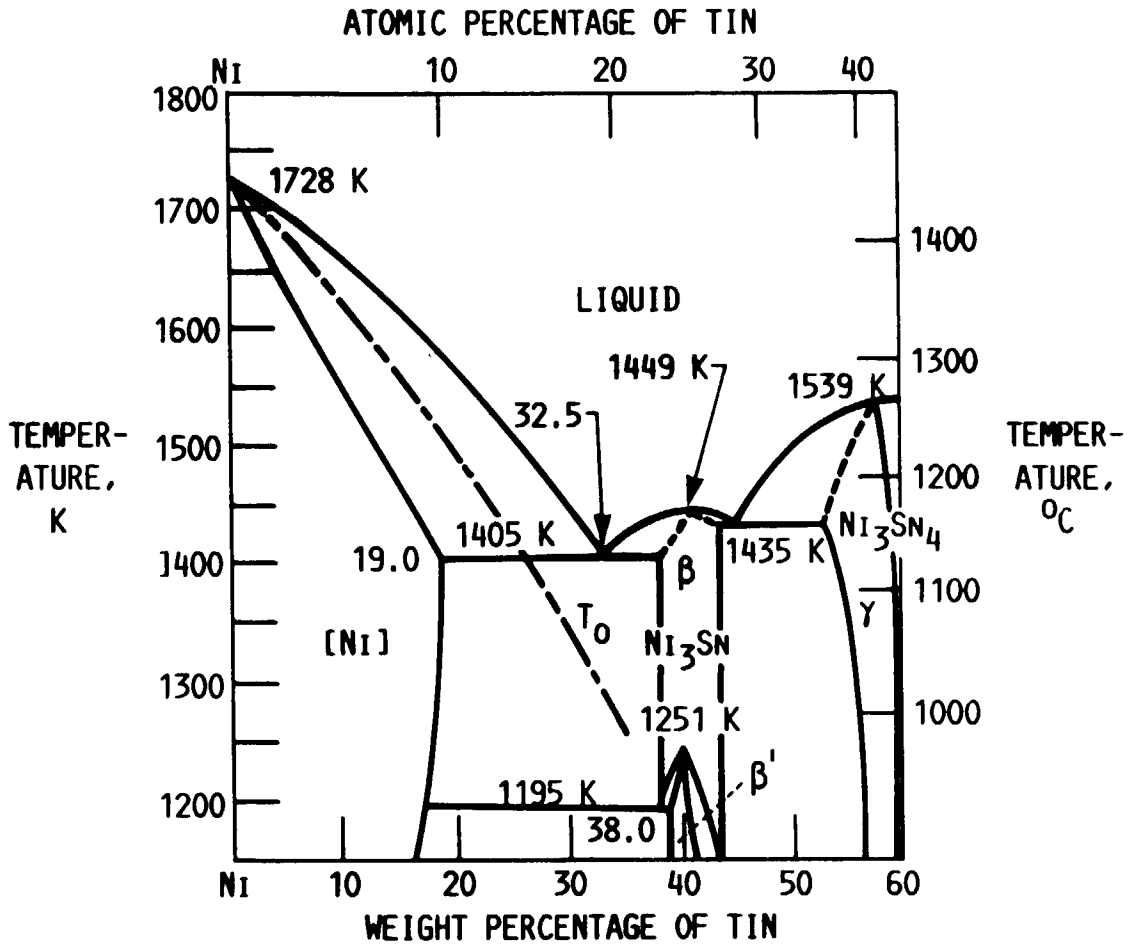


Figure 10. The Fe-Ni phase diagram, with the metastable extensions of the liquidus, solidus, and "T_0" lines for the BCC δ phase.

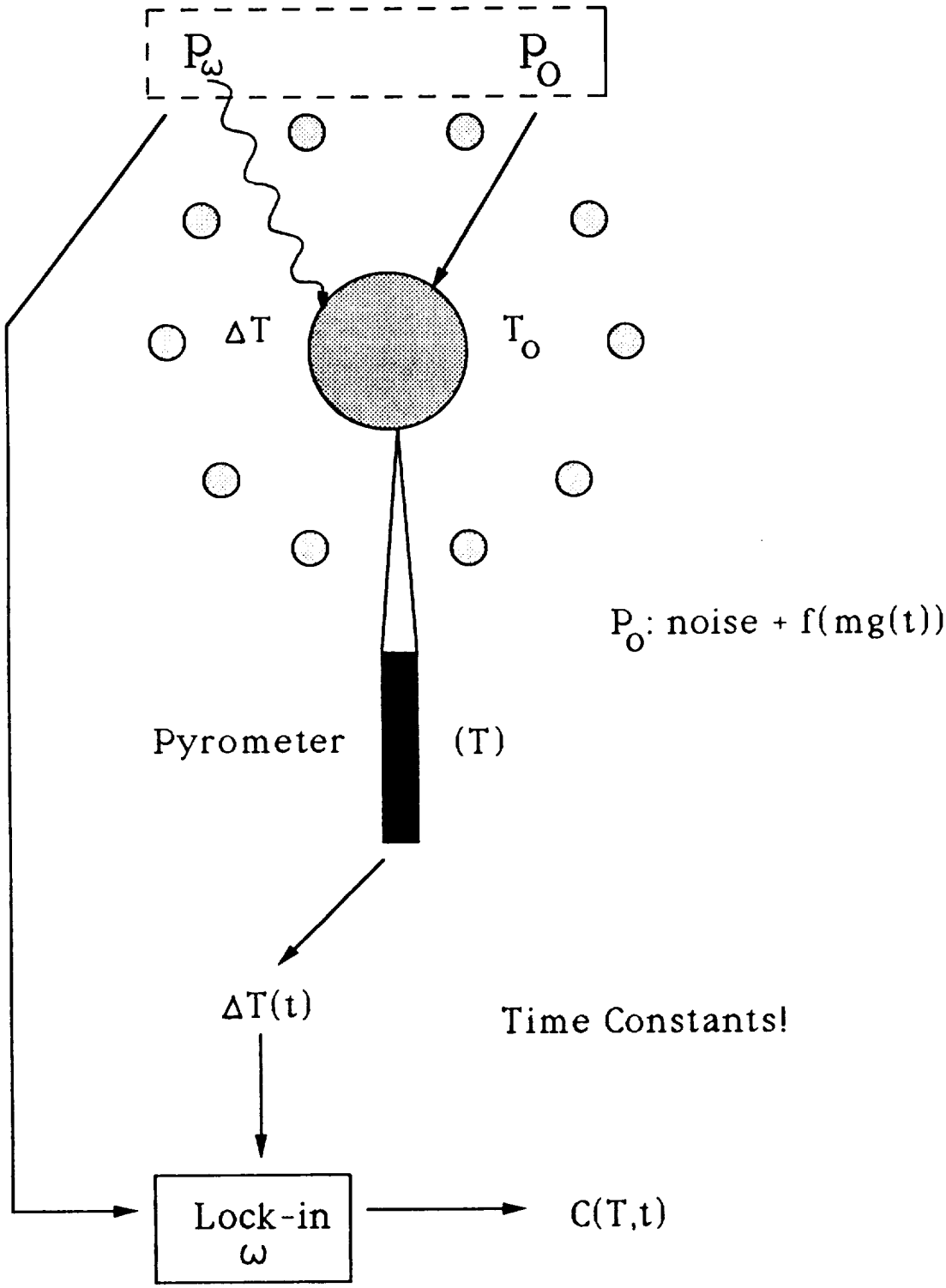
EML - ALLOY UNDERCOOLING EXPERIMENT

MATERIAL

NI-32.5 WT% SN EUTECTIC M.P.
1405 K SPHERE 8.7 MM DIAMETER
WITH 0.1 MM THICK GLASS COATING



CD-86-23107


P_ω
P_o
ΔT
T_o
P_o: noise + f(mg(t))
Pyrometer
(T)
$\Delta T(t)$
Time Constants!
Lock-in
ω
C(T,t)

N91-21337

CONTAINERLESS PROCESSING IN THE EUROPEAN MICROGRAVITY PROGRAMME

by P.Behrmann
ESA-ESTEC
NL-Noordwijk

Summary

Acoustic levitation:
Acoustic levitation has been pursued for more than a decade with the prime objective of processing undercooled melts in space. Three generations of furnaces were developed and tested in sounding rocket experiments. Reasonable levitation was obtained, but some residual instabilities in times of high thermal transients need to be eliminated. The high temperature acoustic levitator is currently pending further development after a n announcement of opportunity to European scientists.

As a spinoff the capabilities of an ambient temperature acoustic levitator in crystal growth experimentation, particularly for protein crystal growth, are being evaluated in a breadboard model.

Electrostatic levitation:
electrostatic levitation has been developed in parallel with the acoustic levitator with similar applications in mind. The system tested utilised a tetrahetral electrode configuration with uncharged samples. Sounding rocket tests of this system failed due to malfunction of the image acquisition system. Due to the residual sample accelerations inherent in the positioning of uncharged samples further development of electrostatic levitators has been put on hold, pending the identification of users with specific needs for this technique.

Electromagnetic levitation
This very promising levitation technique is developed in Europe mainly under the German national programme. The ESA involvement in electromagnetic levitation is concentrated on accommodation studies for the (European) Containerless Processing Laboratory for the Space Station Freedom.

Gas Film Levitation
Gas Film Levitation is planned to form the second major element of the Containerless Processing Laboratory next to the Electromagnetic Levitator. The gas film technique is based on the processing of samples confined by porous walls. Air flow trough the walls creates air cushions which inhibit wall contact. This technique is considered particularly promising for glasses and offers unique opportunities in the processing of non-spherical samples and sample manipulation.

A series of contracts is intended to foster ground based research with this technique, advance the high temperature levitation technology, provide low temperature levitation testing in parabolic flights (under French funding), and perform advance studies for space facilities.

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CONTAINERLESS PROCESSING IN THE EUROPEAN MICROGRAVITY PROGRAMME

P. BEHRMANN

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ACOUSTIC LEVITATION

HISTORICALLY THE FIRST LEVITATOR DEVELOPED UNDER A EUROPEAN PROGRAMME (TECHNOLOGICAL RESEARCH PROGRAMME)

AIM:
LEVITATION OF LIQUID METALS FOR SUPERCOOLING EXPERIMENTS

1. GENERATION: RESONANT CAVITY LEVITATOR WITH ACTIVE CONTROL OF PROCESSING GAS COMPOSITION TO ADJUST THE ACOUSTIC WAVELENGTH DURING TEMPERATURE CHANGES

INITIAL TESTS UNDER MICROGRAVITY FAILED DUE TO MALFUNCTION OF PERIPHERAL EQUIPMENT. IT WAS DECIDED TO DISCONTINUE THE DEVELOPMENT TO AVOID THE COMPLEX GAS CONTROL

2. GENERATION: HALF-OPEN SINGLE-AXIS LEVITATOR WITH FIXED ACOUSTIC POWER

A SOUNDING ROCKET TEST OF THIS LEVITATOR FAILED DUE TO CATASTROPHIC ENHANCEMENT (POSITIVE FEEDBACK) OF TRANSVERSE SAMPLE OSCILLATION

3. GENERATION: HALF-OPEN SINGLE AXIS LEVITATOR WITH ACTIVE MODULATION OF ACOUSTIC POSITIONING POWER AS A FUNCTION OF THE SAMPLE VELOCITY VECTOR

A SOUNDING ROCKET TEST WAS PARTIALLY SUCCESSFUL, GIVING STABLE LEVITATION AT HIGH (NEAR-CONSTANT) TEMPERATURES, WHILE THE SAMPLE DESTABILISED DURING FAST HEAT-UP AND COOL-DOWN. THIS IS EXPLAINED BY DESTRUCTIVE INTERFERENCE BETWEEN THE STABILISING BESSEL-MODE AND INSUFFICIENTLY DAMPED LINEAR MODE WAVES. THIS PROBLEM APPEARS SOLVABLE BY PROPER ABSORBER DESIGN.

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ACOUSTIC LEVITATOR CONTINUED

STATUS:
THE TECHNOLOGY DEVELOPMENT OF THE ESTEC HIGH TEMPERATURE ACOUSTIC LEVITATOR IS CONSIDERED COMPLETE. FURTHER OPTIMISATION MAY BE PERFORMED AS PART OF SCIENTIFIC UTILISATION. AN ANNOUNCEMENT OF OPPORTUNITY HAS BEEN MADE WITHIN THE ESA SOUNDING ROCKET PROGRAMME.

THE MAIN APPLICATION OF ACOUSTIC LEVITATION IS SEEN IN FLUID SCIENCE APPLICATIONS. ESA IS CURRENTLY NOT SPONSORING HARDWARE DEVELOPMENTS IN THIS AREA IN ORDER NOT TO DUPLICATE EFFORTS BY OUR PARTNERS.

THE CURRENT ESA ACTIVITIES IN ACOUSTIC LEVITATION ARE CONCENTRATED ON CRYSTAL GROWTH FROM THE SOLUTION OF LEVITATED DROPLETS. A BREADBOARD IS UNDER CONSTRUCTION TO STUDY PROCESS KINETICS.

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ELECTROSTATIC LEVITATION

ELECTROSTATIC LEVITATION HAS BEEN DEVELOPED IN PARALLEL TO ACOUSTIC LEVITATION FOR ONE DECADE. TESTING OF THE LEVITATOR UNDER MICROGRAVITY COULD ONLY BE PERFORMED AFTER COMPLETION OF THE ACOUSTIC LEVITATOR DEVELOPMENT DUE TO RESTRICTED FUNDS AND SOUNDING ROCKET FLIGHT OPPORTUNITIES.

THE CONFIGURATION TESTED CONSISTS OF FOUR PLATINUM ELECTRODES IN A TETRAHEDRAL ARRANGEMENT. THE SAMPLE POSITION IS MONITORED BY TWO CCD'S

GROUND TESTING OF THE ELECTROSTATIC LEVITATOR WAS LIMITED TO SUSPENDED SAMPLES DUE TO THE LIMITED TIME-RESOLUTION OF CCD CAMERAS (SAMPLE OSCILLATIONS ABOVE 5 HZ COULD NOT BE RESOLVED IN REAL-TIME)

FLIGHT TESTS FAILED DUE TO MISALIGNMENT OF ONE CAMERA. UNFORTUNATELY FLIGHT DATA DID NOT ALLOW RELIABLE CALCULATIONS OF POSITIONING FORCES FROM SAMPLE ACCELERATIONS

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GENERAL PROBLEMS WITH ELECTROSTATIC LEVITATION

ELECTROSTATIC LEVITATION DOES NOT POSSESS A SAMPLE EQUILIBRIUM POSITION CONSEQUENTLY ANY PURE ELECTROSTATIC LEVITATOR WILL OPERATE BY "KICKING THE SAMPLE ABOUT" IN A SPACE THE MINIMUM DIMENSIONS OF WHICH ARE DEFINED BY ELECTRODE CONFIGURATION AND THE SENSITIVITY OF THE POSITION DETECTION.

SINCE THERE IS LITTLE OR NO DAMPING THE RESULTING SAMPLE ACCELERATIONS CAN BE QUITE SUBSTANTIAL AND CAN EXCEED THE AVERAGE MICROGRAVITY LEVEL OF THE ENVIRONMENT BY ORDERS OF MAGNITUDE

AS A RESULT ELECTROSTATIC LEVITATION SHOULD BE UTILISED PREFERABLY IN COMBINATION WITH OTHER LEVITATION TECHNIQUES

IN EUROPE'S MICROGRAVITY PROGRAMMES ELECTROSTATIC LEVITATION IS PUT ON HOLD, PENDING THE IDENTIFICATION OF SCIENTIFIC EXPERIMENTS IN NEED OF THIS SPECIFIC LEVITATION TECHNIQUE.

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ELECTROMAGNETIC LEVITATION

IN EUROPE THE DEVELOPMENT OF ELECTROMAGNETIC LEVITATION IS SPEARHEADED BY THE GERMAN NATIONAL PROGRAMMES. DETAILS OF THIS VERY POWERFUL DEVELOPMENT ARE PRESENTED ELSEWHERE IN THIS WORKSHOP AND SHALL NOT BE REPEATED HERE.

THE ESA INVOLVEMENT IN ELECTROMAGNETIC LEVITATION IS CURRENTLY LIMITED TO ACCOMMODATION STUDIES FOR THE SPACE STATION FREEDOM (CONTAINERLESS PROCESSING LABORATORY).

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GAS FILM LEVITATION

GAS FILM LEVITATION IS A FAIRLY NEW CONCEPT DEVELOPED IN GRENOBLE/FRANCE BY THE GROUP OF DR POTARD AND DR FAVIER. THE MAIN PROJECT ENGINEER IS DR GRANIER.

THE CONCEPT IS BASED ON THE BLOWING OF GAS THROUGH POROUS "CONTAINERS". CONDENSED MATERIAL APPROACHING THE CONTAINER WALLS IS REPELLED BY THE PRESSURE OF THE GAS FILM BUILDING UP BETWEEN SAMPLE AND WALL.

THE MAXIMUM AIR FLOW IS DEFINED BY THE PERMEABILITY OF THE WALL, THUS ALMOST INDEPENDENT OF SAMPLE POSITION. THE GAS FLOWS REQUIRED ARE FAIRLY LOW (A FEW STD L/MIN)

ADVANTAGES:
- NO ACTIVE CONTROL OF LEVITATION PROCESS REQUIRED
- ALL MATERIALS WITH ACCEPTABLE VAPOUR PRESSURES CAN BE LEVITATED
- REASONABLY HIGH LEVITATION FORCES
- "EASY" MANIPULATION OF LEVITATED SAMPLES
- LEVITATION OF NON-SPHERICAL SHAPES (LONG CYLINDERS) IS POSSIBLE

DISADVANTAGES:
- VERY LIMITED ACCESS FOR SAMPLE DIAGNOSTICS
- GAS COMPRESSION (CLOSED LOOP) REQUIRED FOR MANNED SPACE FLIGHT

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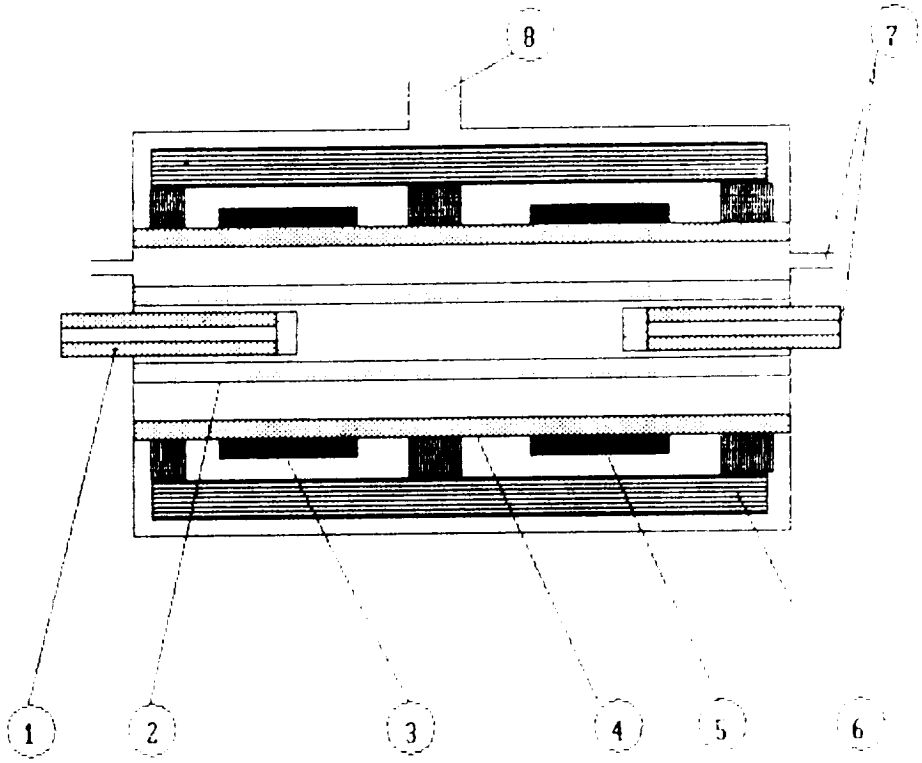
SCHEMATICS OF GAS FILM LEVITATOR



1 - Pistons
2 - Porous tube
3 - Heater : high temperature zone
4 - Leaktight tube
5 - Heater : low temperature zone
6 - Superinsulation
7 - Gas inlet
8 - Vacuum line

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CURRENT DEVELOPMENT PROGRAMME FOR GAS FILM LEVITATION

TECHNOLOGY DEVELOPMENTS:

- A LOW TEMPERATURE EXPERIMENT MODULE FOR FLUID DYNAMICS INVESTIGATIONS IN PARABOLIC FLIGHTS HAS BEEN BUILT UNDER FRENCH FUNDING. A FIRST FLIGHT CAMPAIGN IS SCHEDULE FOR THIS WINTER/SPRING
- A HIGH-TEMPERATURE BREADBOARD FUNDED BY ESA IS UNDER CONSTRUCTION FOR THE PROCESSING OF OXIDE GLASSES MAIN AIMS ARE TO VERIFY THE THERMAL CHARACTERISTICS OF THE LEVITATOR AND SAMPLE MANIPULATION ASPECTS.

SCIENTIFIC STUDIES:

- GAS FILM LEVITATION OF "BUTTON"-SHAPED SAMPLES OF MAINLY HALIDE GLASSES ARE UNDER WAY BOTH UNDER FRENCH AND ESA FUNDING, TO PREPARE THE SCIENTIFIC BASIS FOR FUTURE SPACE EXPERIMENTS. FIRST POSITIVE RESULTS WILL BE PUBLISHED SHORTLY

MICROGRAVITY APPLICATION STUDIES:

- DEFINITION STUDIES (PRE-PHASE A AND PHASE A) FOR A CONTAINERLESS PROCESSING LABORATORY OF THE SPACE STATION FREEDOM

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THE CONTAINERLESS PROCESSING LABORATORY FOR SPACE STATION FREEDOM

FIRST STUDY:
- REVIEW OF SCIENTIFIC RESEARCH FOR FIELDS THAT COULD BENEFIT FROM CONTAINERLESS PROCESSING
- REVIEW OF LEVITATION TECHNIQUES
- SELECTION OF FOUR PRIORITY CANDIDATES OF LABORATORY ELEMENTS
- FIRST-CUT DESIGNS OF THESE ELEMENTS

SECOND STUDY:
FOR THE SECOND STUDY THE ACCOMMODATION ENVELOPE OF THE LABORATORY WAS REDUCED TO A DOUBLE-RACK. TWO ELEMENTS WERE RETAINED:
- THE ELECTROMAGNETIC LEVITATOR FOR THE PROCESSING OF METALS
- THE GAS FILM LEVITATOR FOR THE PROCESSING OF OXIDE AND FLUORIDE GLASSES

THE MAIN ELEMENTS OF THE STUDY INCLUDED:
- A DETAILED DESIGN OF EACH LEVITATOR
- A DEFINITION OF SYSTEM INTERFACES
- A BUDGET ASSESSMENT
- ASSESSMENT OF STORAGE REQUIREMENTS
- PLANNING OF SERVICING OPERATIONS

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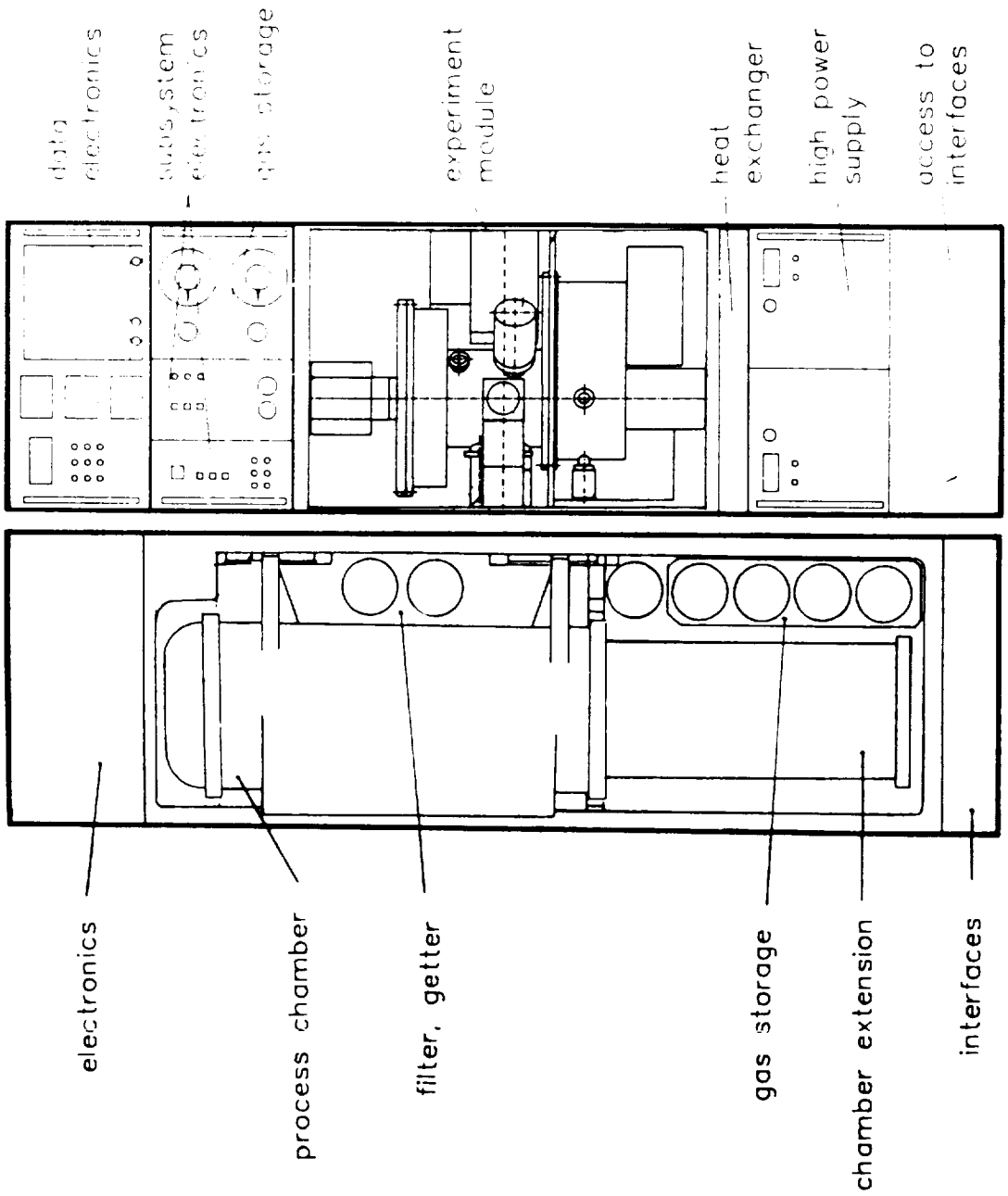

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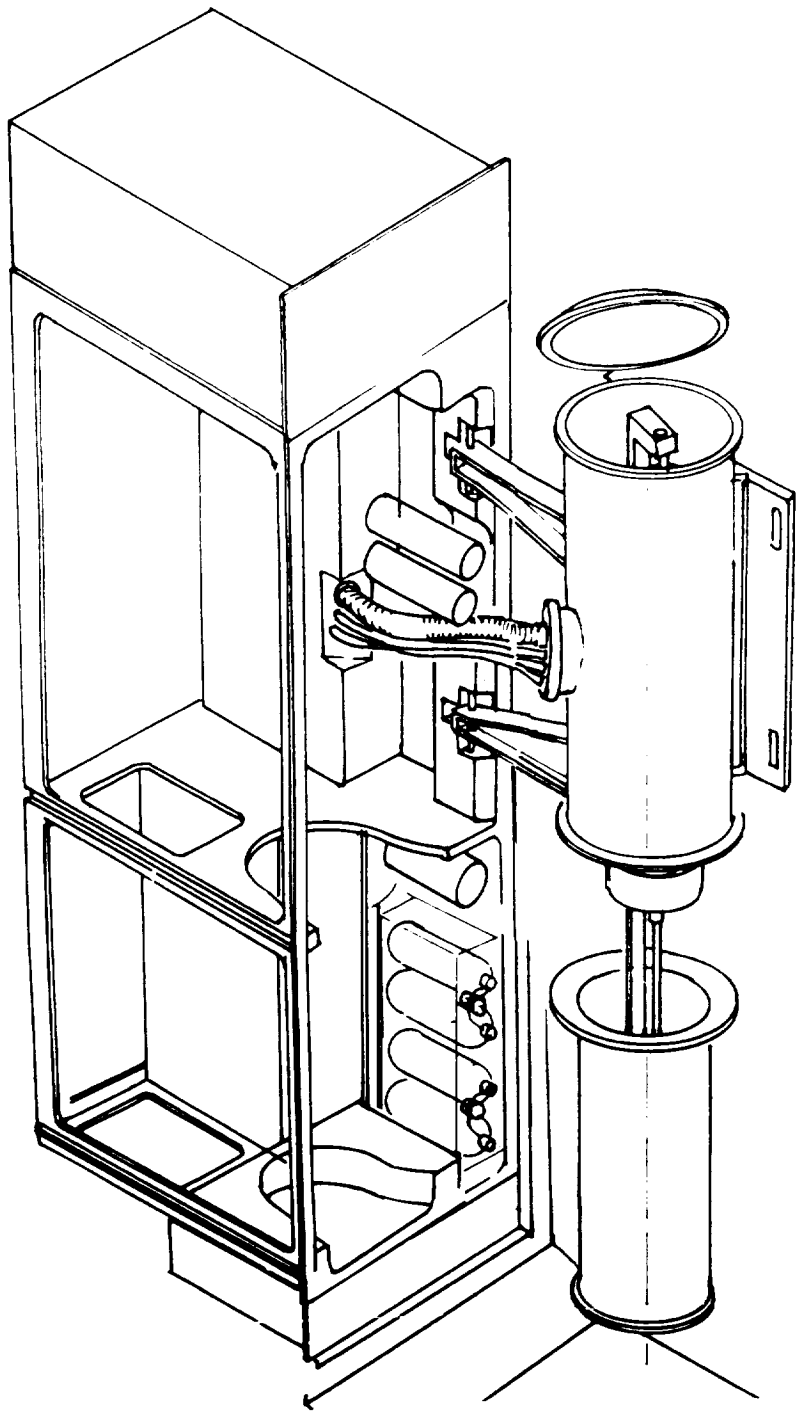


GAS FILM LEVITATOR MODULE

REFERENCES

/1/ J. GRANIER and M. DANIEL,"Study of a gas film positioning system":Midterm report,ESA/ESTEC contract n° 6962/86/NL/JG(SC),August 5,1987.

/2/ J.GRANIER and C.POTARD,"Containerless Processing and Molding Materials by the Gas Film Technique:Early Demonstration and Modelling." Proc.6th European Symposium on material science under microgravity conditions, Bordeaux,France,2-5 Dec.1986 (ESA SP-256,Feb.87,p 421)

/3/ A.RIALHE,J.GRANIER and C.POTARD,"Crystallization of a laser glass prepared by containerless processing using the gas film technique." Proc. Expermat'87:Int. Conf on Materials with exceptional properties,Bordeaux,France,24-27 Nov.1987

/4/ C.POTARD and P.DUSSERRE,"Contactless positioning, manipulation and shaping of liquids by gas bearings for microgravity application." Proc.25th COSPAR,Graz,Austria, 26 June-7 July 1984.

The Japanese Containerless Experiments

National Aerospace Laboratory
Hisao Azuma

1. Drop Dynamics Research in NAL

a) Acoustic Levitation

* FMPT related activity

Liquid drop experiment by a tri-axis acoustic levitator in the Japanese First Material Processing Test(FMPT) is to be conducted on SL-J in June 1991.

Objective of the experiment

- -Stable positioning of a liquid drop
- -Rotation of a drop
- -Deformation of a liquid drop
- -Stability of a liquid membrane

Experiments on the Earth

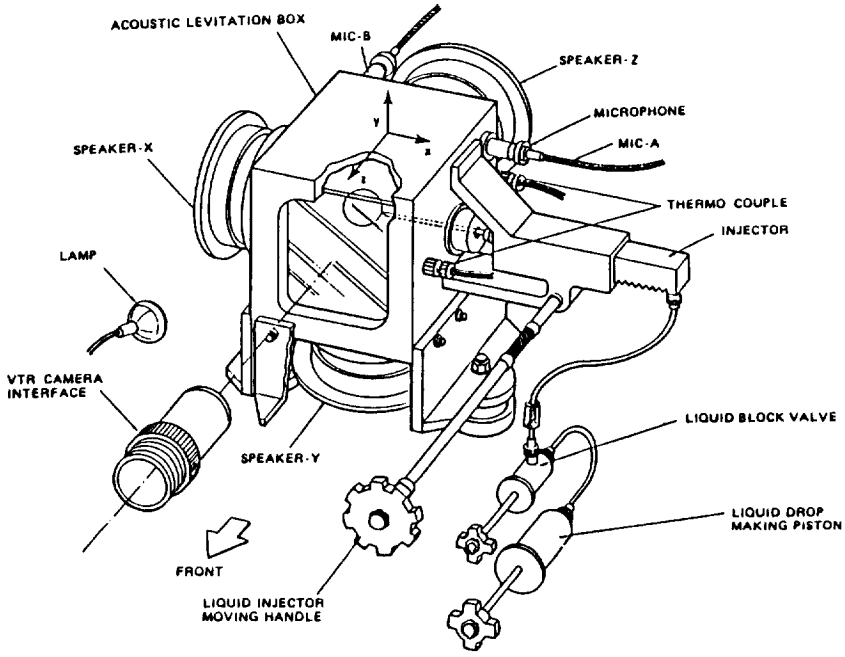
- -Levitation and rotation of light weight samples(styrofoam spheres)
- -Deformation of a drop and formation of a liquid film

Parabolic flight test

- -Separation of a drop from an injection needle in acoustic chamber
- -Determination of experimental parameters to position a drop in low gravity

Liquid drop experiment facility

Levitation box dimension	100W*100H*110D
Acoustic pressure	141-148 dB
Speaker input power	10 Wmax
Frequency	1400-1700 Hz
Drop size	10,19,23 mm dia.



* High levitating force levitator
  -Levitation of large sized liquid drop and membrane on the earth
  -High ambient pressure

b)Large amplitude drop oscillation

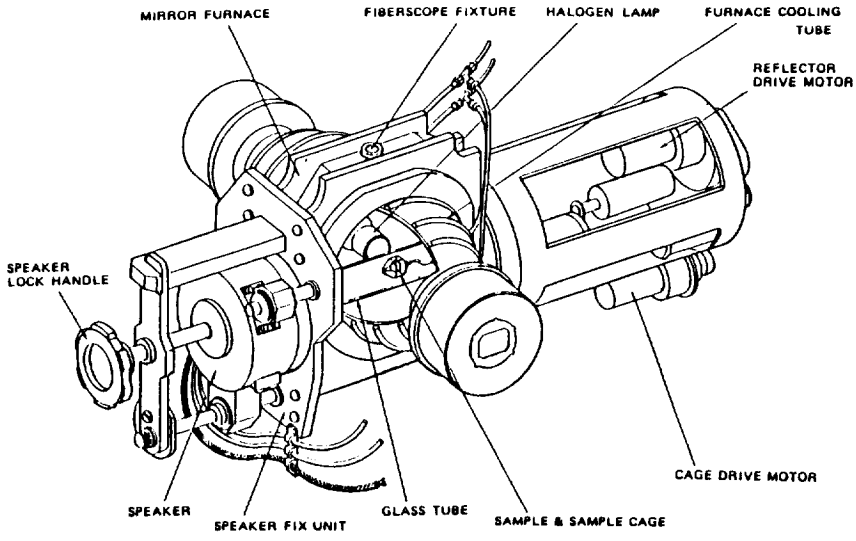
Realization of three-dimensional spherical large amplitude oscillation, tetrahedron-tetrahedron, hexahedron-octahedron, dodecahedron-icosahedron by using drop tower. The oscillations were caused with surface tension variation by applying alternating current voltage.

2. Optical Materials Processing in an Acoustic Levitation Furnace in Industrial Research Institute,Osaka

"Preparation of Optical Materials used in Non-visible region" to be conducted in the FMPT

-$65C_aO$-$25G_{a2}O_3$-$10G_eO_2$(near infrared transmitted oxide glass) was chosen
-1400 °C and platinum cage for preheating is needed

Parabolic flight test
-Levitation of heated sample was made sure



3. Electrostatic Levitator Development by Melco and IHI

a)Mitubishi Electric Corporation

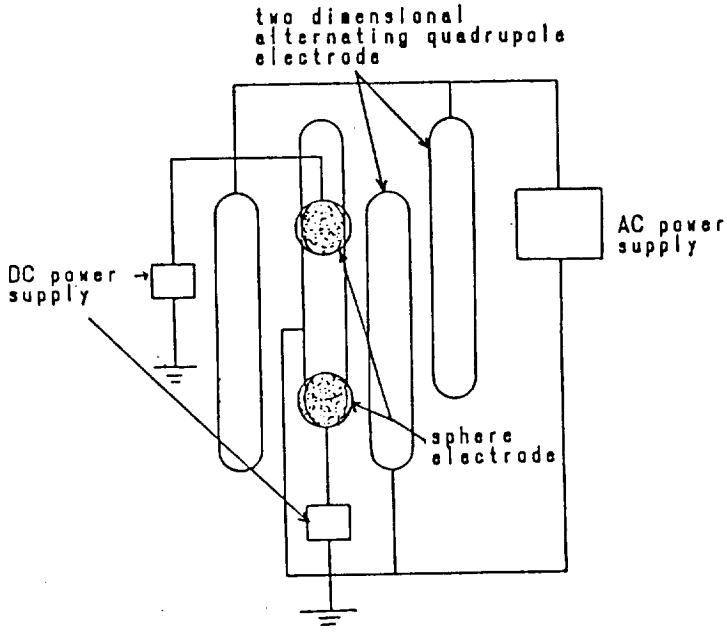
Development status
- -Levitation and rotation of 0.1g platinum coated glass shell by a double ring type levitator
- -Position data of 120Hz
- -Levitation of 50g solid (metal and glass) will be tried soon by parabolic flight

Aimed performance goal
- -Disturbance given to a sample should be less than 10^{-6}g
- -Sample should be heated up to 2500 °C (3kw AC power)
- -Sample size should be larger than 20mm dia.

An important technology-Microwave Discharge Lamp
- -High temperature in whole sphere of arbitrary size
- -Choice of arbitrary gas inside the lamp to get desired wave length light

b)Ishikawajima-Harima Heavy Industries Co.,Ltd.

Development status
- -Levitation of solid sample(4mm in dia. 1.5mg) by a levitator with quadrupole electrodes and a couple of spherical electrodes



Configiation of two dimensional alternating quadrupole levitation apparatus

THERMOPHYSICAL PROPERTIES OF SOLIDS AND LIQUIDS (MAINLY METALS AND ALLOYS)

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WHY THERMOPHYSICAL PROPERTIES ARE NEEDED

(a) ENGINEERING DESIGN PARAMETERS

- Turbine blade alloys
- Ti forgings
- Al alloys
- Composites

(b) MATERIALS PROCESSING

- On Earth: Solid state combustion synthesis:
  $Ti + C = TiC + Energy$

- In Microgravity environments:

1. On Moon

   $MO_x + F_2 \rightarrow MF_y + O_2$

   $MF_y(l) \rightarrow M + F_2$

   Breathe O_2 ; Recycle F_2

2. Space Station

3. Shuttle / Satellites

4. Wake Shield experiments

TYPES OF MATERIALS

REFRACTORY METALS

ALLOYS-SUPERALLOYS Inconel
TiAlx, NiAlx, etc...

GRAPHITE

BINARY CARBIDES

SiC, B_4C , Al_4C_3
TiC, VC_x, ...
ZrC, NbC_x , MoC_x
HfC, TaC_x, WC_x
ThC_x, UC_x, ...

BINARY SILICIDES $MoSi_2$, WSi_2, etc...

BINARY BORIDES TiB_2, ZrB_2, TaB_x, ...

BINARY NITRIDES

Si_3N_4, BN, AlN
TiN_x, VN_x
ZrN_x. etc...

TERNARY COMPOSITIONS: METAL OXYNITRIDES
METAL OXYBORIDES
METAL OXYCARBIDES, etc...

CURRENT STATE OF KNOWLEDGE

CLASS OF MATERIALS	SOLIDS	LIQUIDS	GASES
Metals	Good	Good	Excellent*
Alloys	Good	Fair	Good
Graphite	Good	Poor	Fair
Carbides	Good	Poor	Good
Silleides	Fair	Poor	Fair
Borides	Fair	Poor	Fair
Oxides	Good	Fair	Good
Nitrides	Fair	Poor	Fair

*EXCEPT FOR LASER ZAP OR EXPLODING WIRES

- TERNARY AND MORE COMPLICATED SYSTEMS ARE NOT WELL STUDIED.

- THEORIES ARE NOT EVEN ADEQUATE FOR METALS, VERY PRIMITIVE FOR LIQUID ALLOYS AND REFRACTORY COMPOUNDS.

PROPERTIES NEEDED

Cp(T)

$(H_t - H_{298})$

ΔH_{fusion}

Phase Diagram

$\varepsilon(\lambda,T)$

$\varepsilon_{total}(T)$

(Calorimetry, Pyrometry)

$\rho(T)$

CTE(T) for solids and liquids

ΔV_{fusion}

Supercooling, Nucleation, and Crystallization

(Fast Photography of weighed drops)

Surface Tension as f(T)

Viscosity as f(T)

Melting and Freezing

(Fast Photography of oscillating droplets)

Thermal Conductivity as f(T)

Thermal Diffusivity as f(T)

Resistivity as f(T)

Magnetic Properties as f(T)

***Laser flash heating**

ELECTROMAGNETIC LEVITATION IS VERSATILE AND PROVIDES RAPID HEATING FOR GOOD CONDUCTORS IN VACUUM OR IN SELECTED ATMOSPHERES

- Convenient for good conductors: metals, alloys, carbides, bordies, etc.
- Heat C or SiC but not levitate
- ZrO_2, HfO_2, UO_2, etc. can be heated inductively after pre-heating
- Al_2O_3, SiO_2, NaCl, etc. neither heat nor levitate

ACOUSTIC LEVITATION } POOR ELEC. CONDUCTORS, LOW VP'S

GAS JET LEVITATION } POOR ELEC. CONDUCTORS, LOW VP'S, POSSIBLE CONTAMINATION

MICROGRAVITY ENVIRONMENT - EVERYTHING LEVITATES

MICROGRAVITY ENVIRONMENT - RADIATIVE, LASER OR INDUCTION HEATING

LIMITATIONS OF ELECTROMAGNETIC LEVITATION

1. Must be good conductor.

2. Must have adequate surface tension.

3. Must have low VP.

UNSOLVED PROBLEMS IN DETERMINING THERMOPHYSICAL PROPERTIES OF LIQUID METALS/ALLOYS AT HIGH TEMPERATURES

- Contamination
  - Apparatus
  - Atmospheres
- Calibration Standards
  - Precision ($\pm$ 0.5%)
  - Accuracy
- Reliable T(t) and Standards for T > 2000 K
- Pre-Melting/Post-Melting Phenomena
- Clusters in Liquids?
- Are There Defects in Liquids?
- Super-Cooling; Amorphous Phases; Crystallization
- Electronic Effects: Is ∂ a f(T)?
- Limits on T_{max} by VP
- Vaporization Losses as f(T, t, Metal)
- Lack of a Comprehensive Theory for Liquid Metals/Alloys: R(T); Cp(T); ε(T); Hall Effect

SPECIAL NEW TECHNIQUES

- Pulsed Laser Heating & EM Levitation
- Polarized Laser Pyrometry Yields $\varepsilon(\lambda, T, t)$ and True T as f(t)
- High-Speed Photography of Levitated and Falling Drops
- Hybrid Levitators (EM, Acoustic, Gas Jet)

GOALS OF LEVITATION STUDIES

THERMODYNAMIC PROPERTIES

$C_p^s(T)$, ΔH_{fusion}, $C_p^l(T)$

PHYSICAL PROPERTIES

Density as f(T)
Thermal expansivity
Emissivities as f(T)
Surface tension as f(T)
Viscosity as f(T)

QUESTIONS

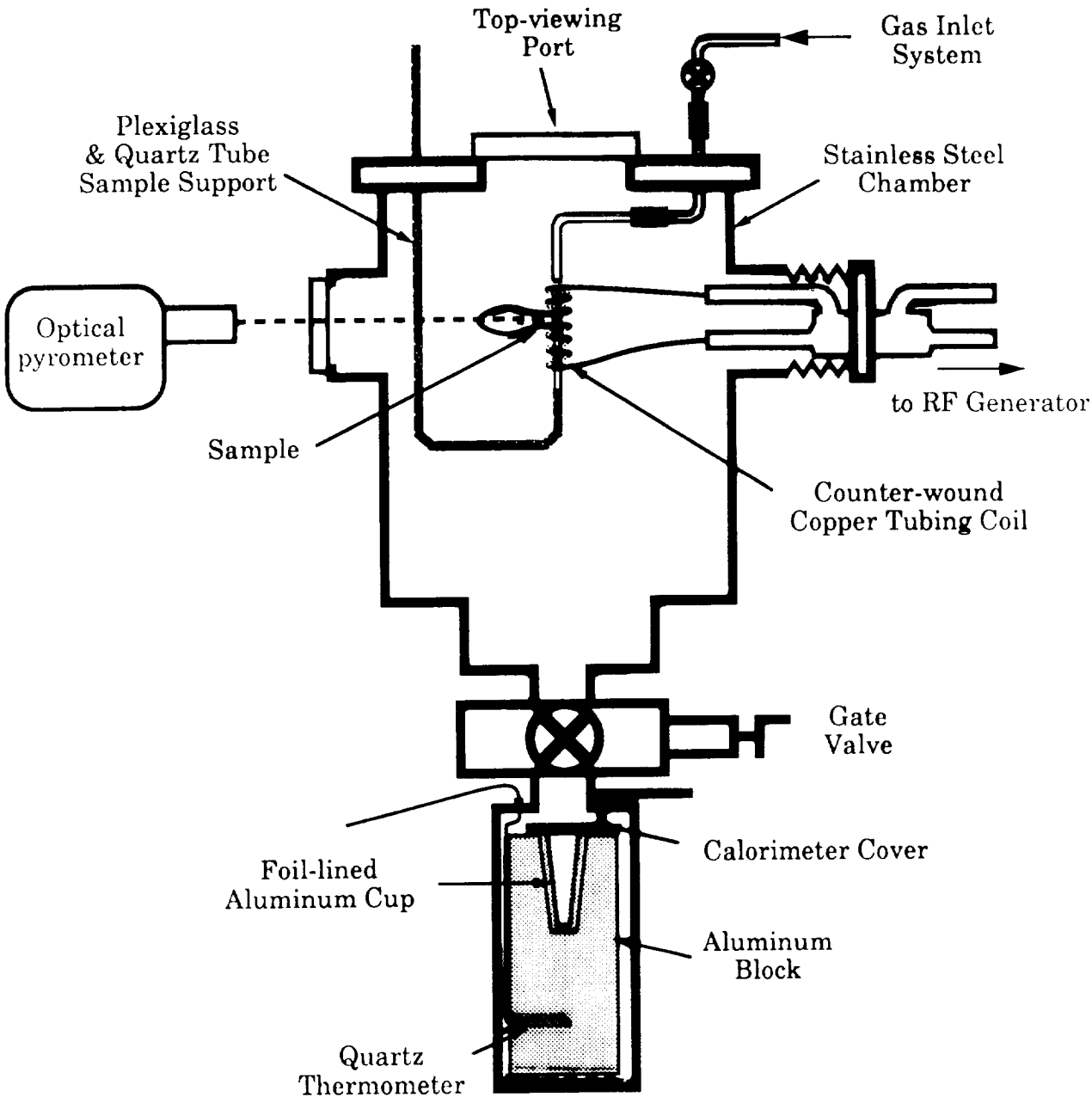
Is C_p for liquid metals:

a. Greater than, equal to, or less than C_p for the solid?

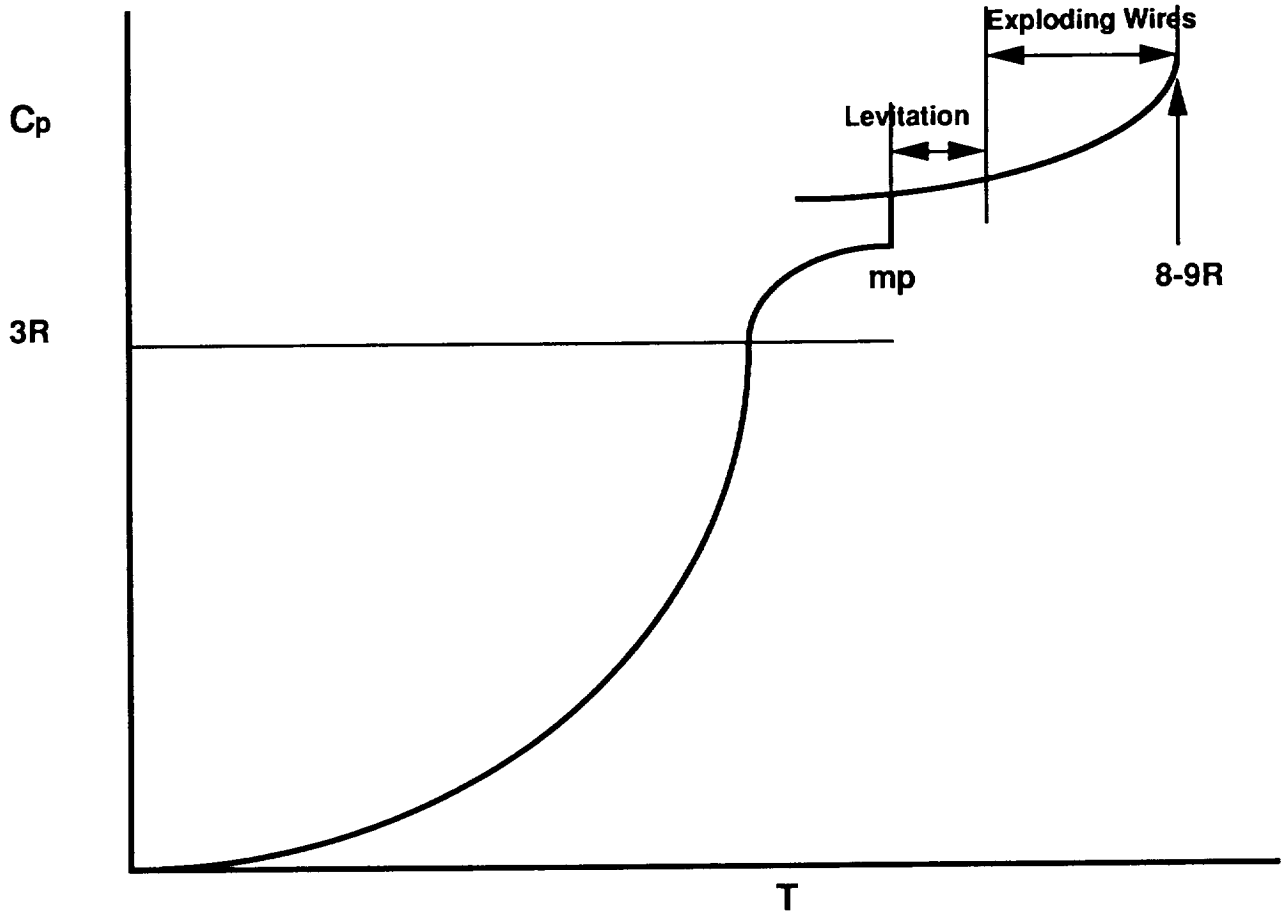
b. Increasing, constant, or decreasing with increasing T?

c. Appreciably higher at 5000 K than at 3000 K for liquid Mo ?

d. Approximately 3R, 5R, 6R,... for liquids at high T ?


Top-viewing Port
Gas Inlet System
Plexiglass & Quartz Tube Sample Support
Stainless Steel Chamber
Optical pyrometer
to RF Generator
Sample
Counter-wound Copper Tubing Coil
Gate Valve
Calorimeter Cover
Foil-lined Aluminum Cup
Aluminum Block
Quartz Thermometer

N. N.


Exploding Wires
Levitation
Cp
mp
8-9R
3R
T

RECENT LEVITATION STUDIES AT RICE UNIVERSITY

LIQUID SILVER (1281 K < T < 1549 K)

$(H_T - H_{298}) = 32.644\ T - 2944.9$ **J/gram.atom**

$C_p^l = 32.64 \pm 2.06$ **J/Gram.atom K**

$\Delta H_{fusion} = 10916 \pm 435$ **J/Gram.atom**

$\varepsilon_{650\ nm} = 0.11 \pm 0.10$

LIQUID GALLIUM (587 < T < 1630 K)

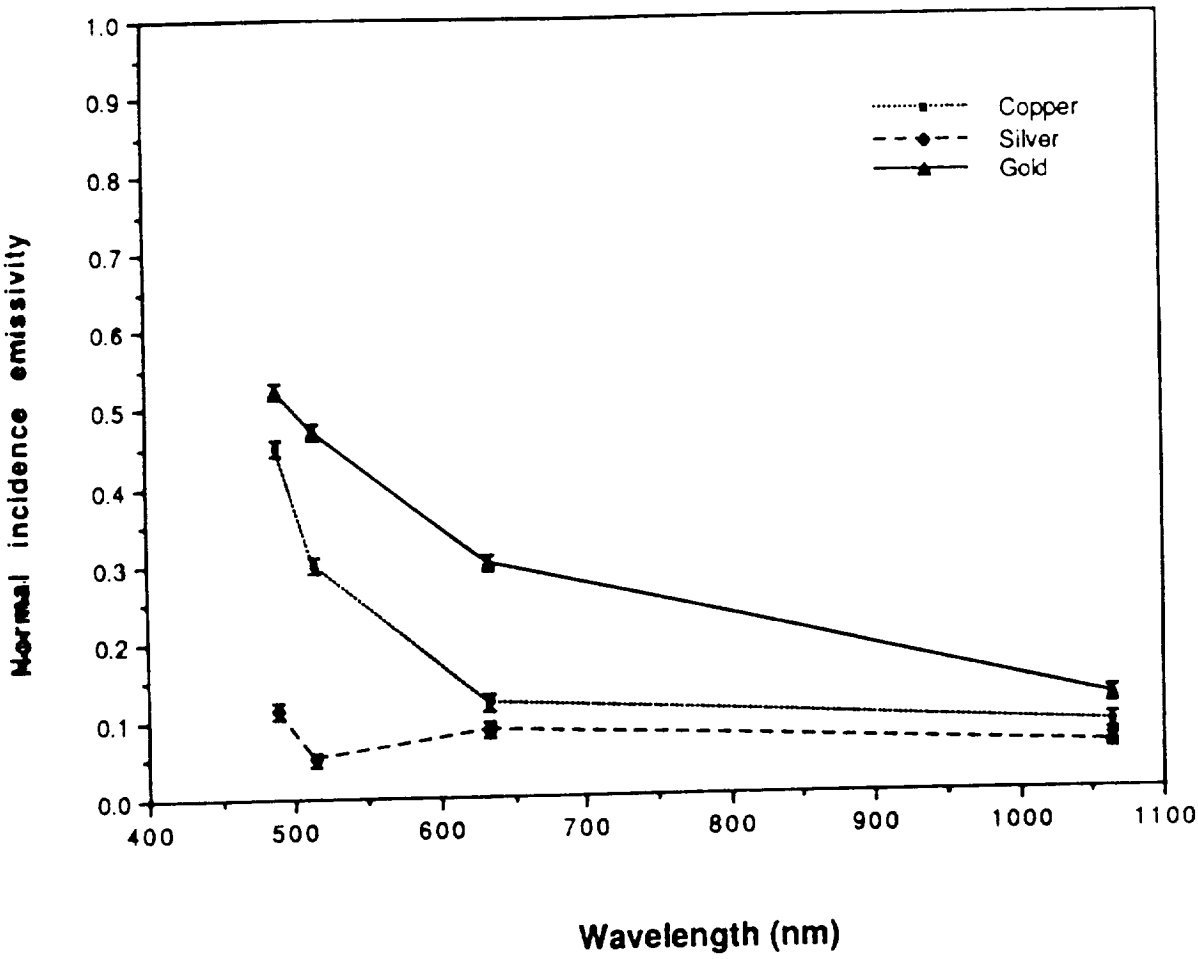
$(H_T - H_{298}) = 26.460\ T - 7677.0$ **J/gram.atom**

$C_p^l = 26.46 \pm 0.71$ **J/Gram.atom K**

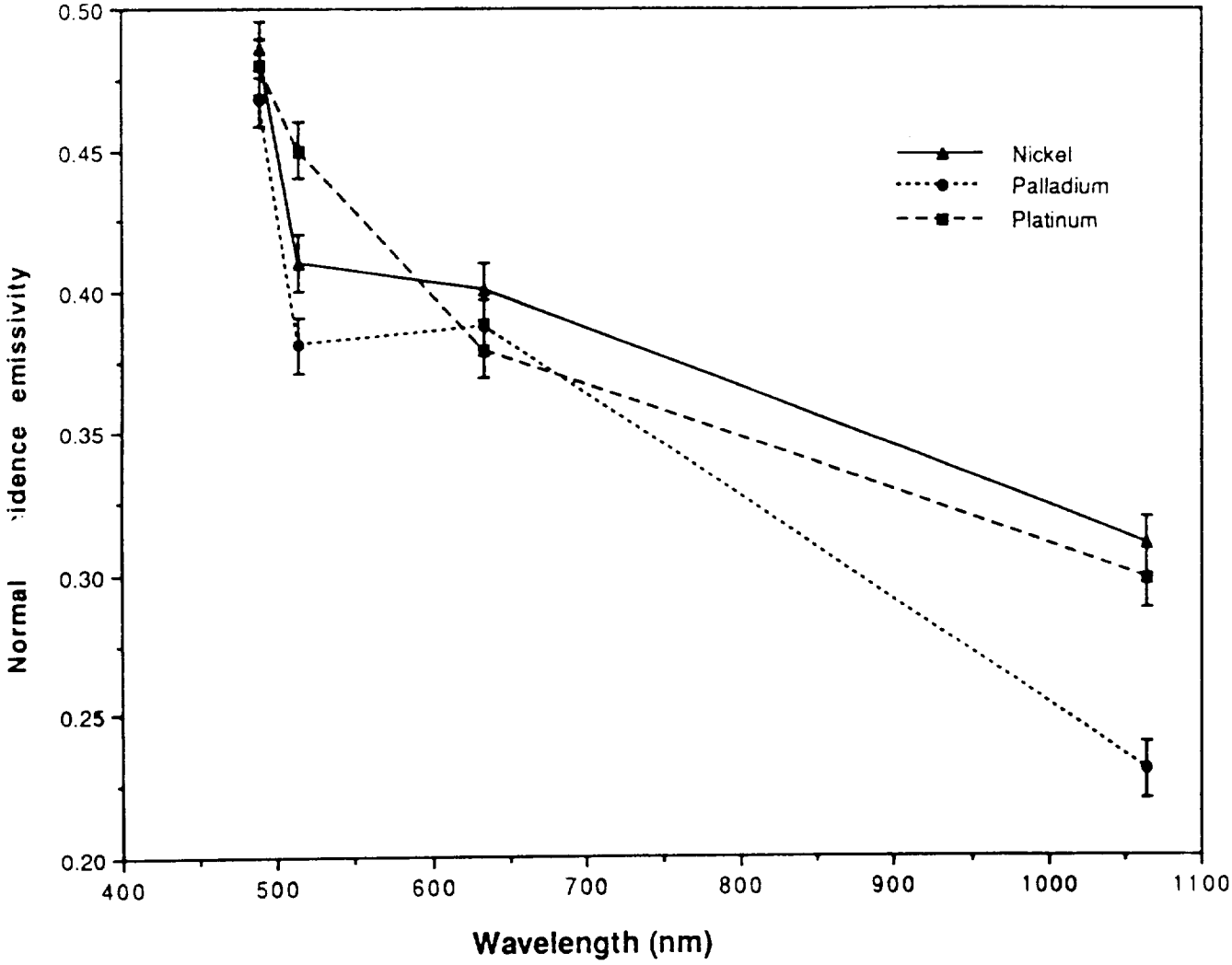
$\varepsilon_{645\ nm} = 0.14 \pm 0.10$

Also Studies of:

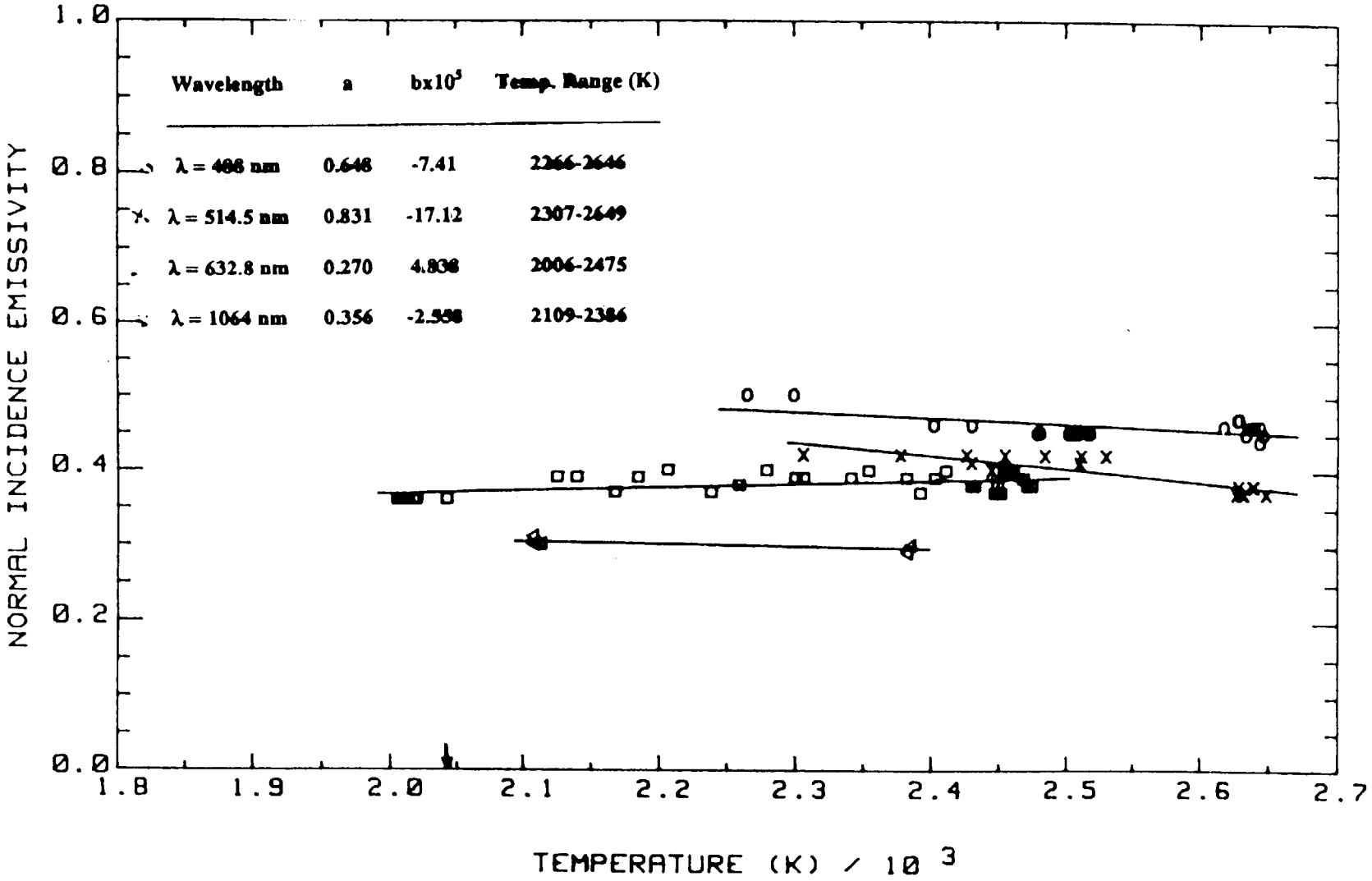
TUNGSTEN, BRASS ALLOYS, SUPERALLOYS



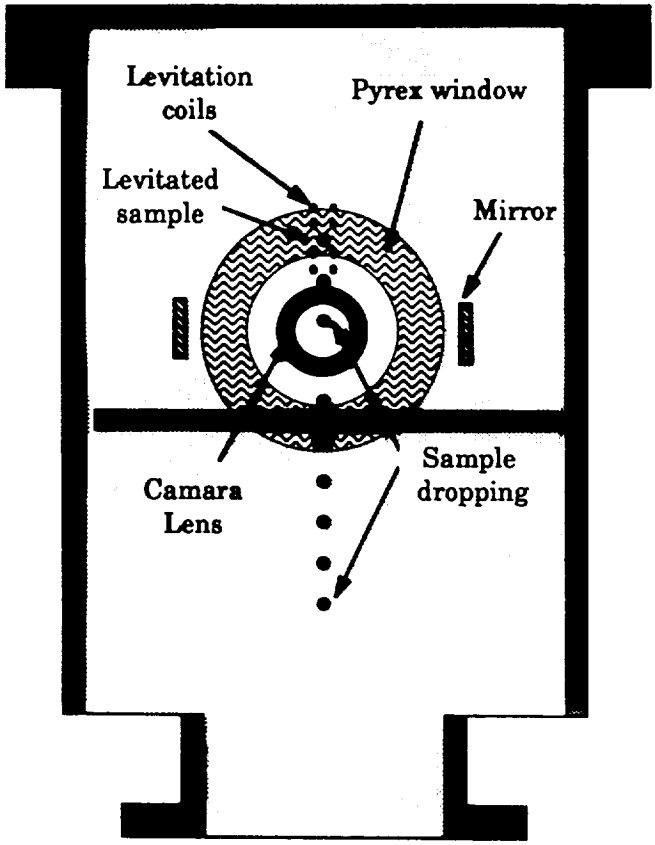
SPECTRAL EMISSIVITIES OF LIQUID METALS AS A FUNCTION OF WAVELENGTH

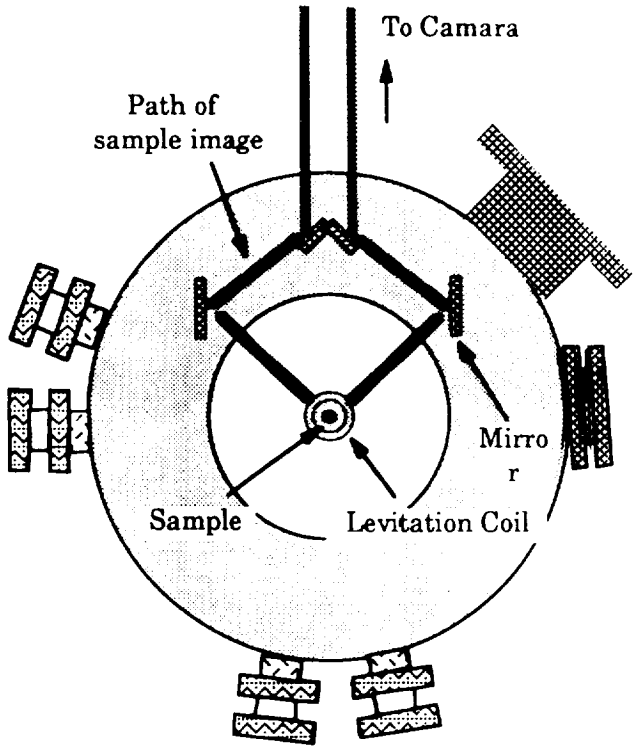


SPECTRAL EMISSIVITIES OF LIQUID METALS AS A FUNCTION OF WAVELENGTH



SPECTRAL EMISSIVITIES OF PT (liquid) AS A FUNCTION OF TEMPERATURE FOR VARIOUS WAVELENGTHS


Levitation coils
Pyrex window
Levitated sample
Mirror
Camara Lens
Sample dropping


To Camara
Path of sample image
Mirro r
Sample
Levitation Coil

N91-21339

Containerless Processing of Undercooled Melts

J. H. Perepezko
Department of Materials Science and Engineering
University of Wisconsin-Madison

All practical solidification processes involve some level of melt undercooling. Usually in bulk liquids crystallization is initiated at a heterogeneous nucleation site at low undercooling. The realization of appreciable levels of liquid undercooling requires some control over the kinetics of crystal nucleation. One level of control is available in containerless processing where the capability to melt and solidify samples without a container removes a major source of impurities and heterogeneous nucleation sites which can be effective in promoting large undercooling and in studying other potential nucleants. This is a critical advantage offered by containerless processing. On earth containerless processing may be achieved for small samples by electromagnetic, acoustic or electrostatic levitation or by dropping small molten droplets through a long evacuated tube and allowing them to solidify during free-fall. Although ground based methods are useful, they have limitations. Levitation approaches induce melt disturbances which may influence nucleation and limit the attainable undercooling. During drop tube processing it is difficult to obtain a continuous and accurate measurement of sample temperature during free-fall. Moreover, all ground based methods are restricted to small samples. The microgravity environment offers a unique opportunity for containerless processing of large liquid samples with negligible melt disturbance and continuous temperature measurement throughout processing. One use of this approach is to allow for physical property measurements that are not accessible by usual techniques. Perhaps the most important potential for microgravity containerless processing lies in the significant structural control to develop distinct microstructures and metastable phases during solidification at high undercooling. In order to realize this potential ground based experiments are required to establish the key processing parameters such as melt superheat, fluxing, thermal cycling and sample atmosphere that control undercooling and nucleation of different structures. A ground base experimental experience is also crucial to the development of a suitable microgravity experimental facility and to the interpretation of microgravity test results.

Containerless Processing of Undercooled Melts

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UNDERCOOLING

. WHAT IS IT?

Tm - T

. HOW DOES IT DEVELOP?

Crystallization rate limitations

. WHY IS IT IMPORTANT?

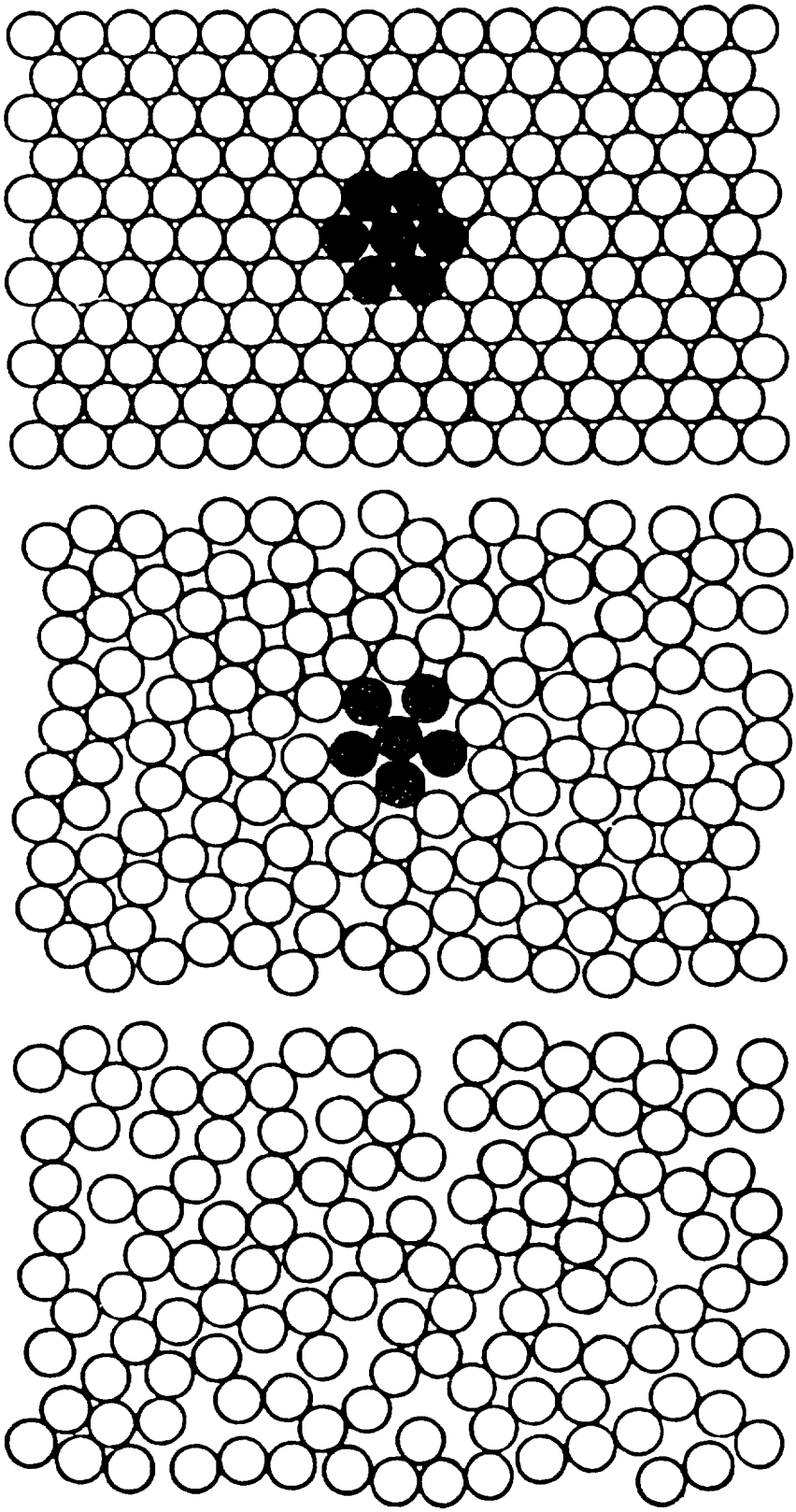
Controls initial solidification path

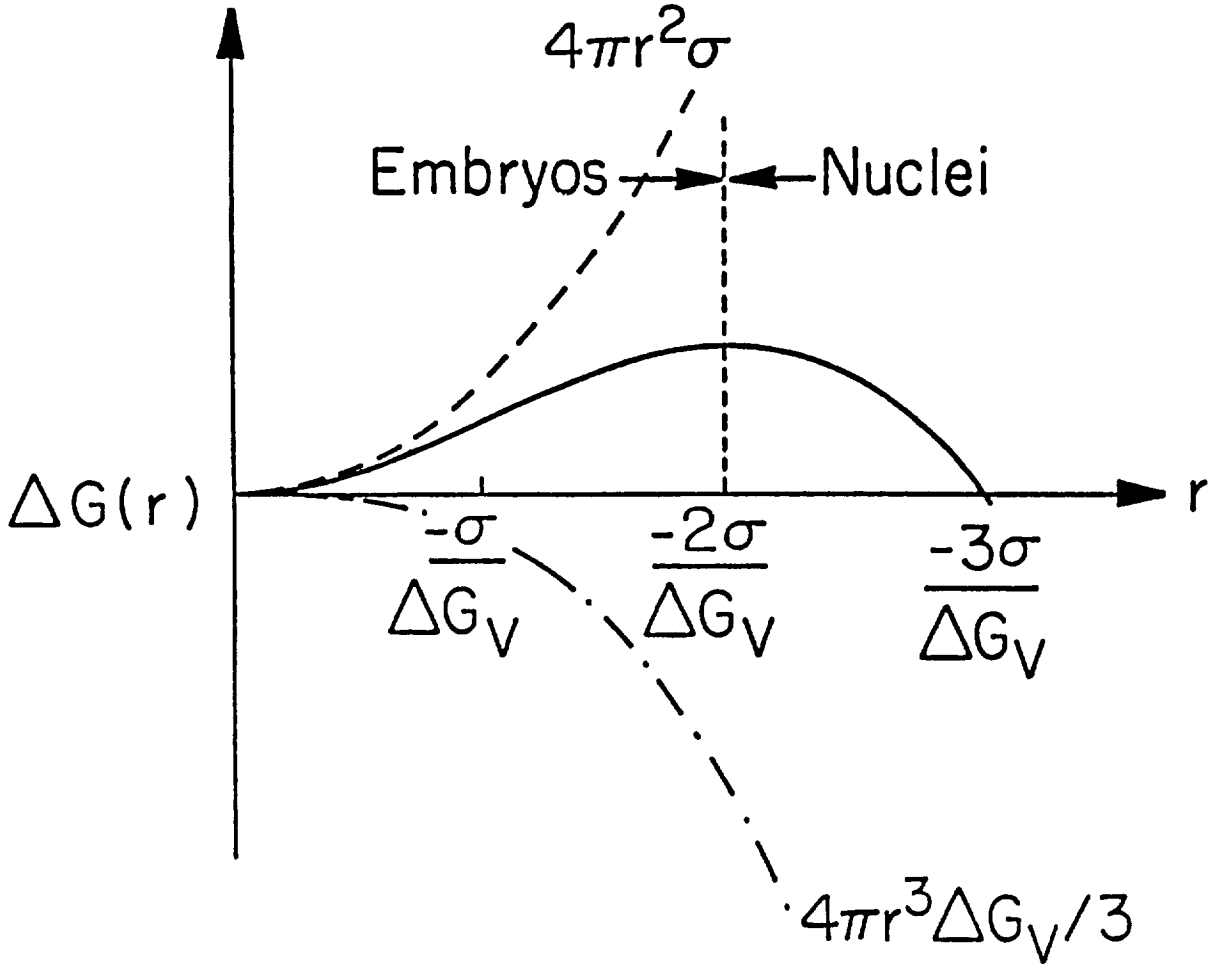
UNDERCOOLED MELTS

* General Kinetic and Thermodynamic Features of Metastability and Undercooling

* Containerless Processing - Experiment and Analysis
  - Kinetic Studies
  - New Structures
  - Process Control

* Open Issues




$4\pi r^2\sigma$
Embryos
Nuclei
$\Delta G(r)$
r
$\frac{-\sigma}{\Delta G_V}$
$\frac{-2\sigma}{\Delta G_V}$
$\frac{-3\sigma}{\Delta G_V}$
$4\pi r^3\Delta G_V/3$

DIFFICULTIES IN ACHIEVING EQUILIBRIUM

* Equilibrium diagram gives stable equilibrium

* Metastable equilibrium results when a system is subjected to constraints
  - Coherency
  - Stable phase missing or metastable phase present
  - Sluggish kinetics

* Metastable equilibrium is a reversible equilibrium

* Metastable equilibrium information can be useful in phase diagram evaluation
  - Test thermodynamic models

* Other non-equilibrium information
  - T_o curve

To Optimize Undercooling...

1. High degree of particle size refinement and narrow size distribution

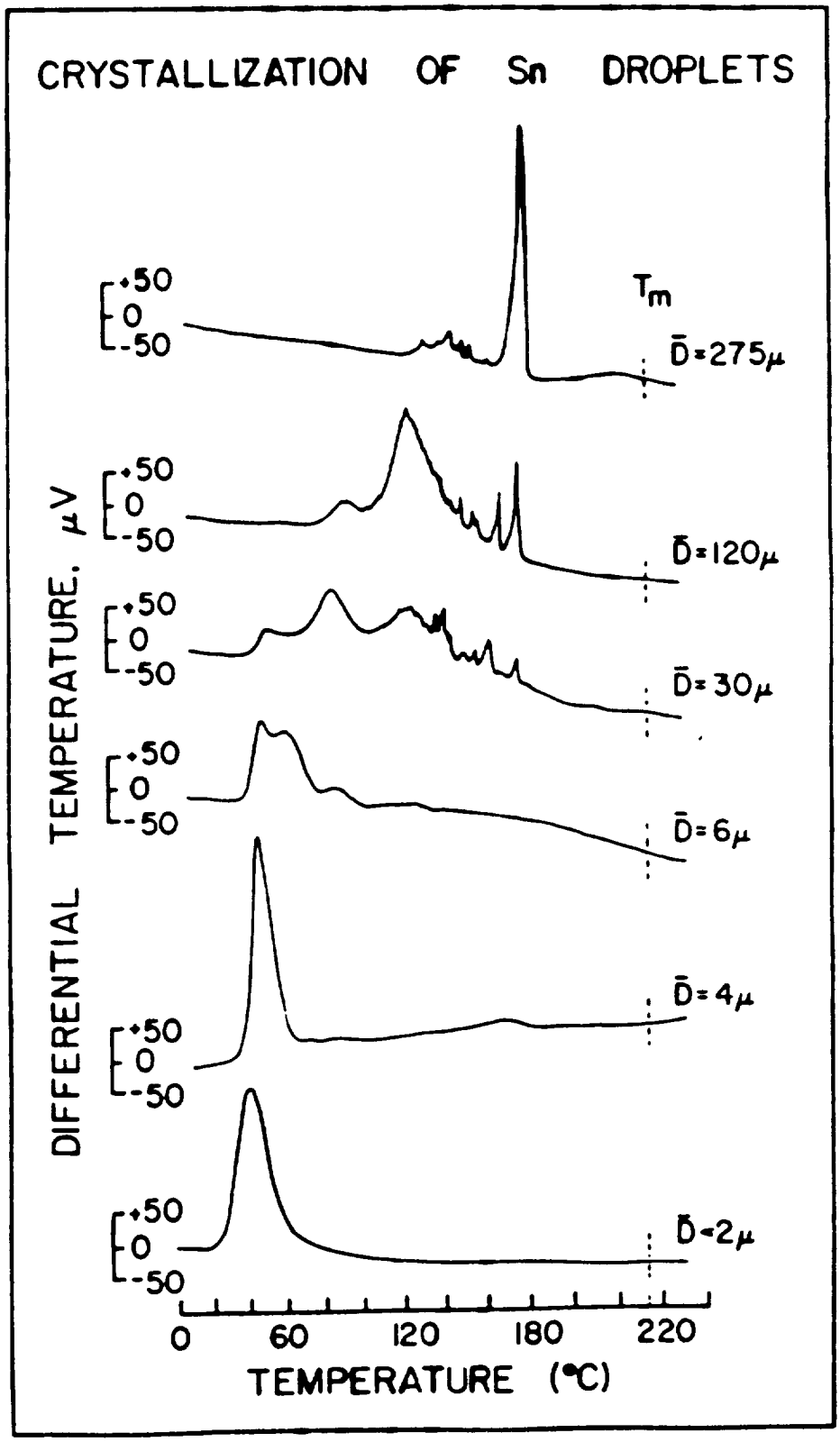
2. Non-catalytic surface coating
   -wet liquid

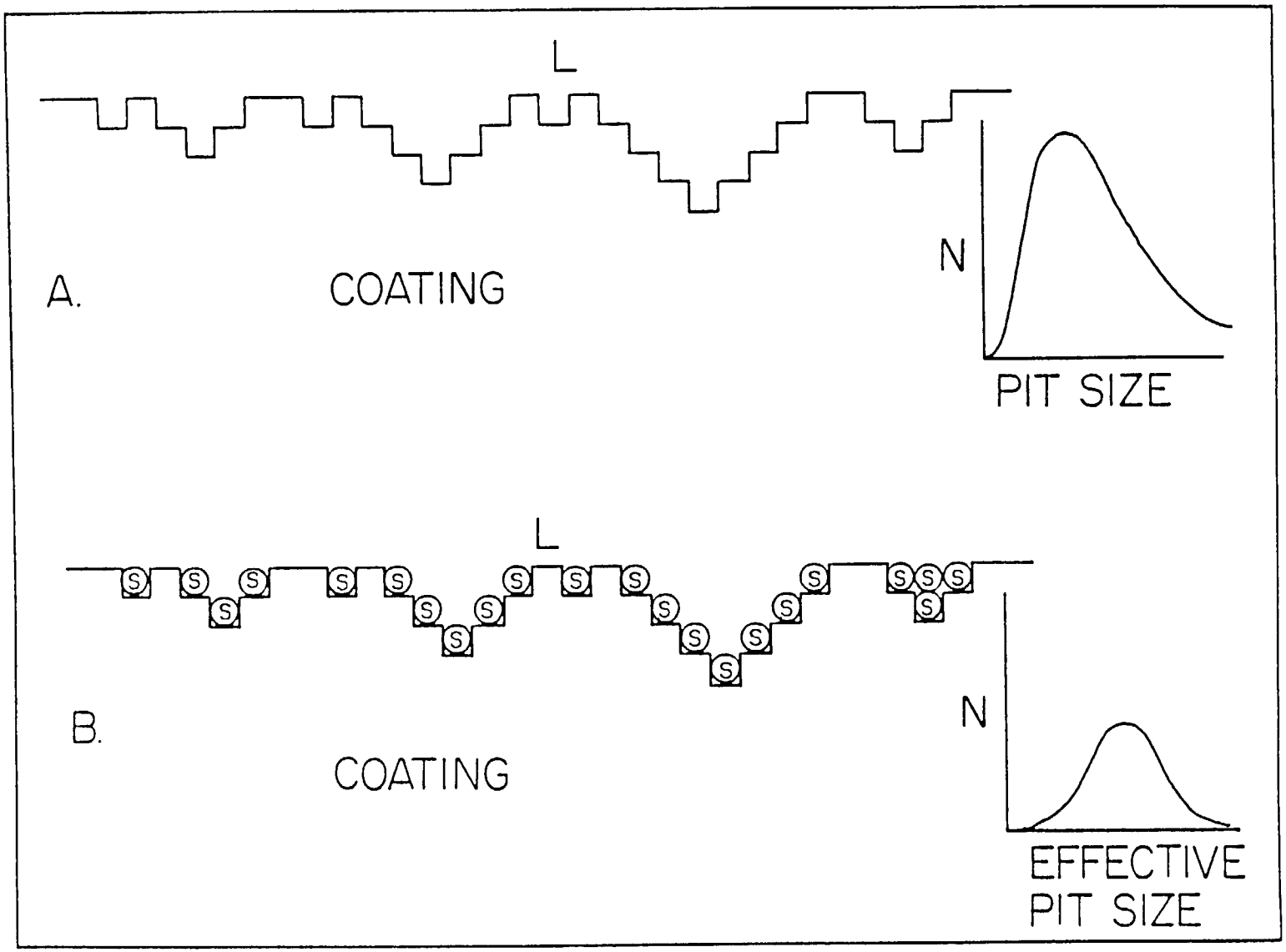
3. Uniform surface coating

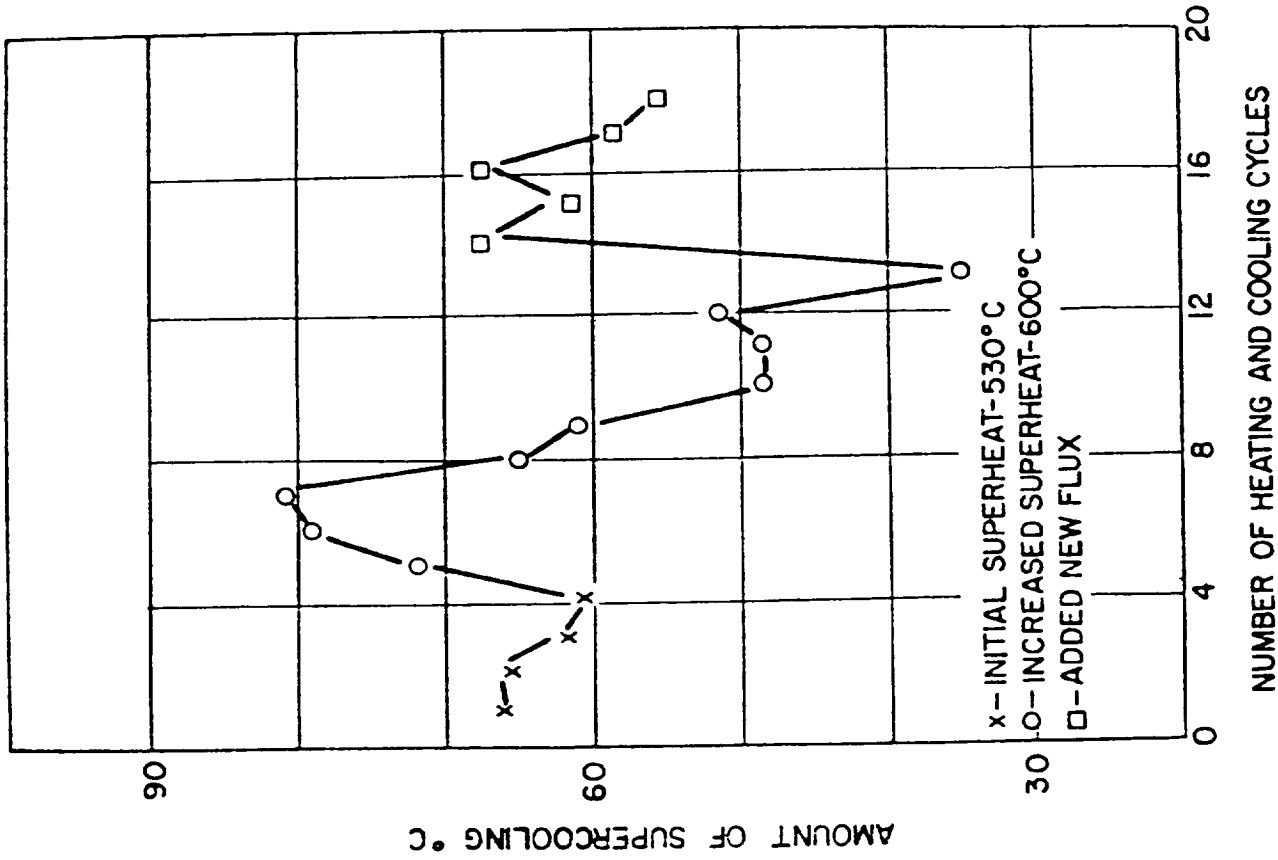
4. Purity: specific effects

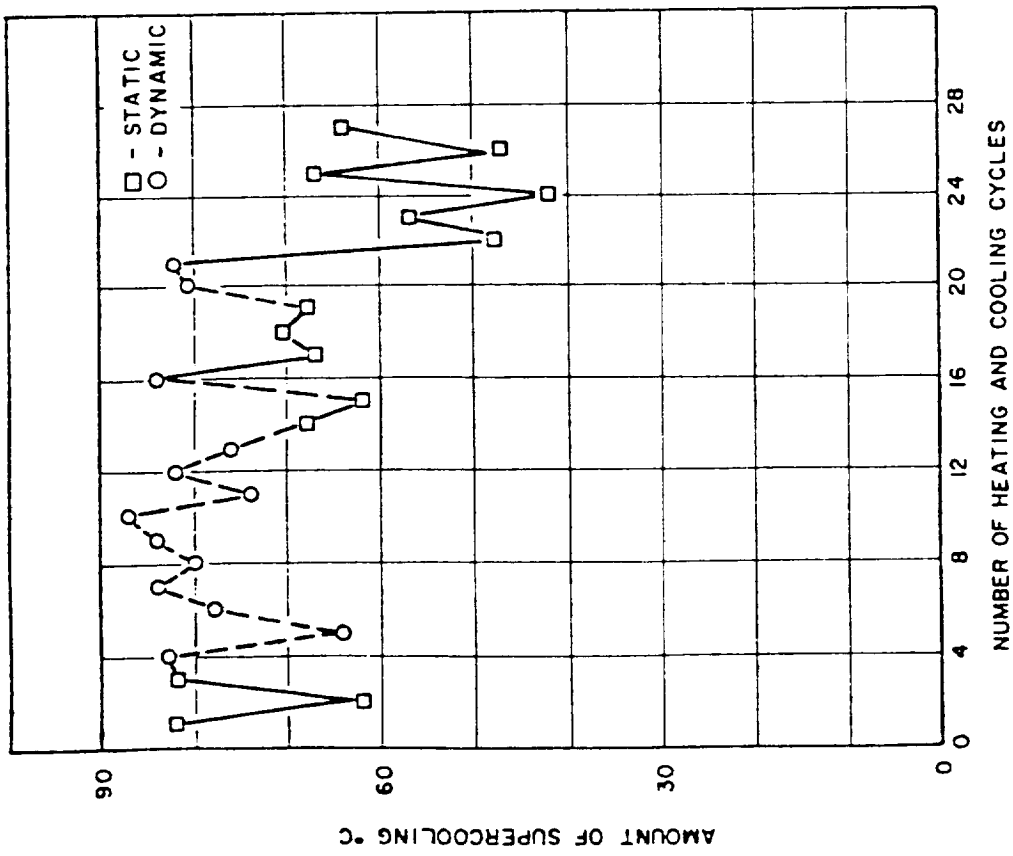
5. Perserverance

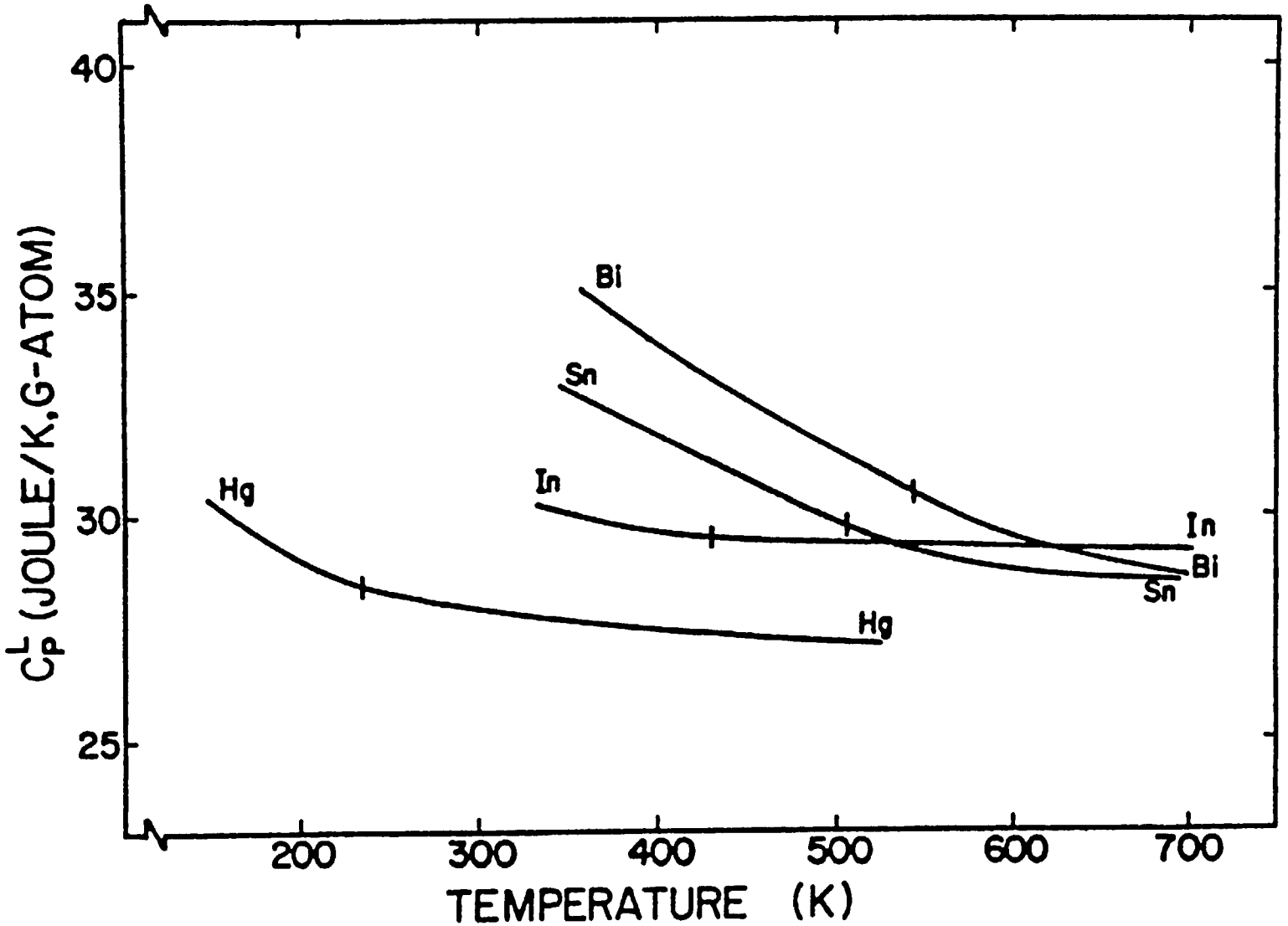
6. Increased cooling rate is often useful

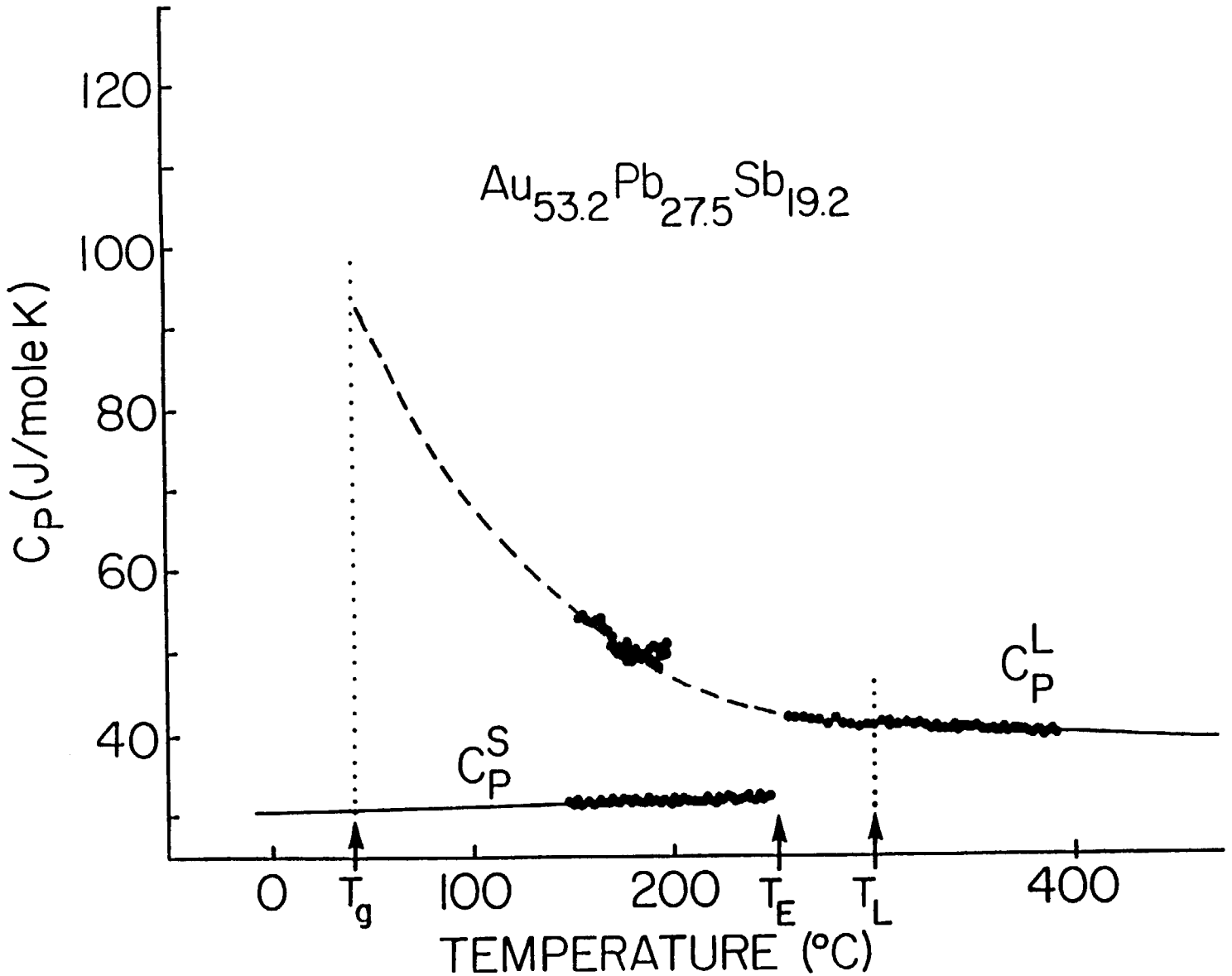

CRYSTALLIZATION OF Sn DROPLETS
DIFFERENTIAL TEMPERATURE, μV
+50
0
-50
T_m
$\bar{D}$=275μ
$\bar{D}$=120μ
$\bar{D}$=30μ
$\bar{D}$=6μ
$\bar{D}$=4μ
$\bar{D}$<2μ
0
60
120
180
220
TEMPERATURE (°C)

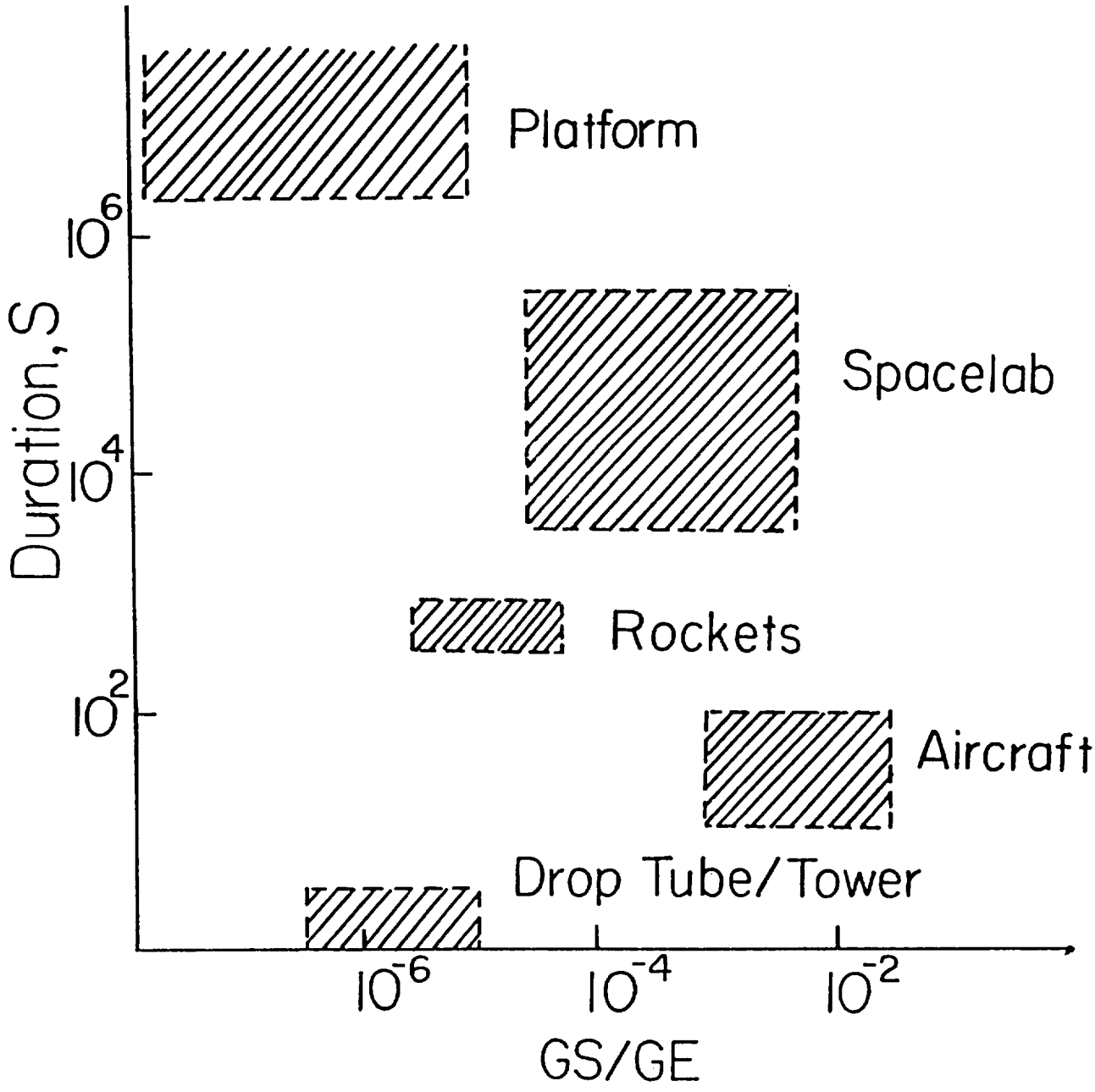

L
A.
COATING
N
PIT SIZE
L
B.
COATING
N
EFFECTIVE
PIT SIZE


AMOUNT OF SUPERCOOLING °C
90
60
30
0
4
8
12
16
20
NUMBER OF HEATING AND COOLING CYCLES
x – INITIAL SUPERHEAT-530°C
O – INCREASED SUPERHEAT-600°C
□ – ADDED NEW FLUX

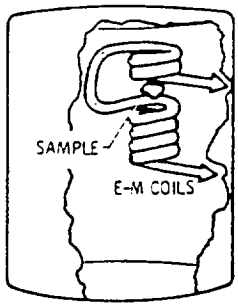

AMOUNT OF SUPERCOOLING °C
90
60
30
0
4
8
12
16
20
24
28
NUMBER OF HEATING AND COOLING CYCLES
□ - STATIC
O - DYNAMIC


40
35
30
25
C_p^L (JOULE/K,G-ATOM)
Bi
Sn
In
Hg
In
Bi
Sn
Hg
200
300
400
500
600
700
TEMPERATURE (K)

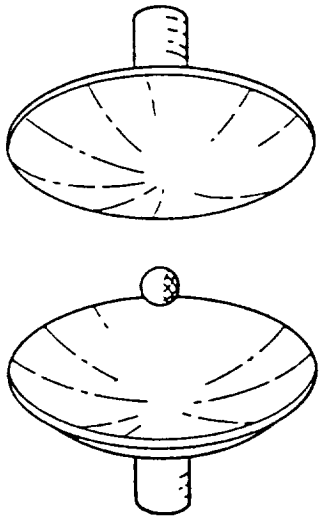

$Au_{53.2}Pb_{27.5}Sb_{19.2}$
120
100
80
60
40
C_P(J/mole K)
C_P^S
C_P^L
0
T_g
100
200
T_E
T_L
400
TEMPERATURE (°C)


Platform
Spacelab
Rockets
Aircraft
Drop Tube/Tower
Duration, S
10^6
10^4
10^2
10^{-6}
10^{-4}
10^{-2}
GS/GE

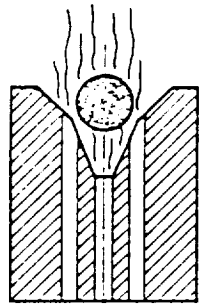
B-5. CONTAINERLESS PROCESSING TECHNIQUES



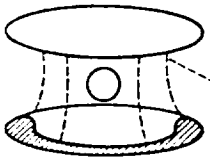
(A) ELECTROMAGNETIC



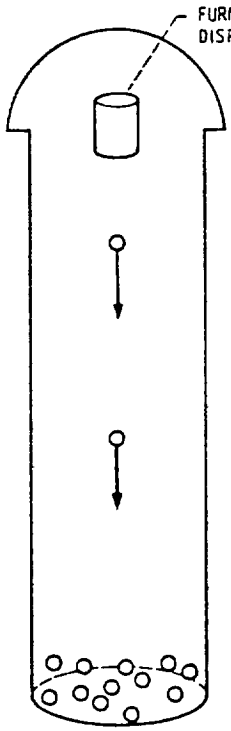
(B) ACOUSTIC



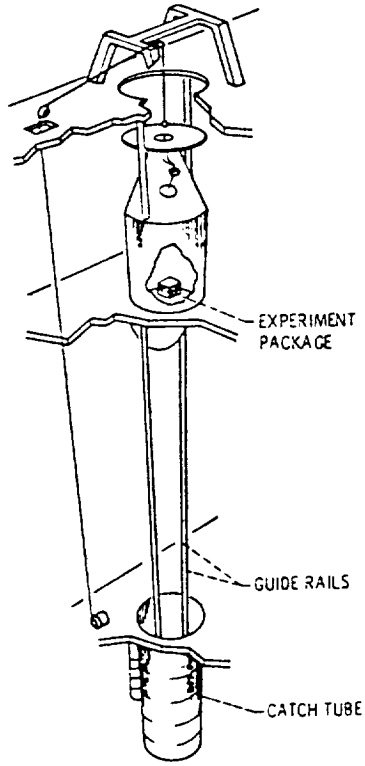
(C) AERODYNAMIC



(D) ELECTROSTATIC



(E) DROP TUBE



(F) DROP TOWER

CD-85-16835

-Containerless Processing Potential

* Minimize Reactivity
* Avoid Crucible Induced Nucleation,

but...

* Possibility of Ultrapure Melts

(especially surface)

* Avoid Vibration Effects

-Microgravity and Containerless Processing

* Large Sample Possible
* Vacuum
* Sample Positioning
* Minimize Fluid Flow Effects

-Undercooled Melts

* Refined Microstructure
* Suppressed Segregation
* New Phases - Metastable (glass)
* Control Phase Selection

- Containerless Processing/Solidification of Undercooled Melts

* Identify and control key processing variables (superheat, sample size, purity, atmosphere, cooling rate)

* Heat flow analysis

* Develop temperature measurement

* Examination of sample surface and relation to undercooling

* Solidification kinetics

---

* Sample size scale-up

* Influence of melt vibration and flow

Containerless Processing and Microgravity

1) The microgravity environment of space allows for containerless processing of extended duration.

2) Microgravity processing can allow for the study of the under-cooling and solidification response in pure, uncoated samples.

3) With extended duration treatment the influence of controlled coating reactions on undercooling response may be evaluated for application ot the development of upscaled sample volumes.

4) The experience from ground-based containerless processing work will provide the background that is needed to interpret, evaluate and maximize solidification structure control during microgravity treatment.

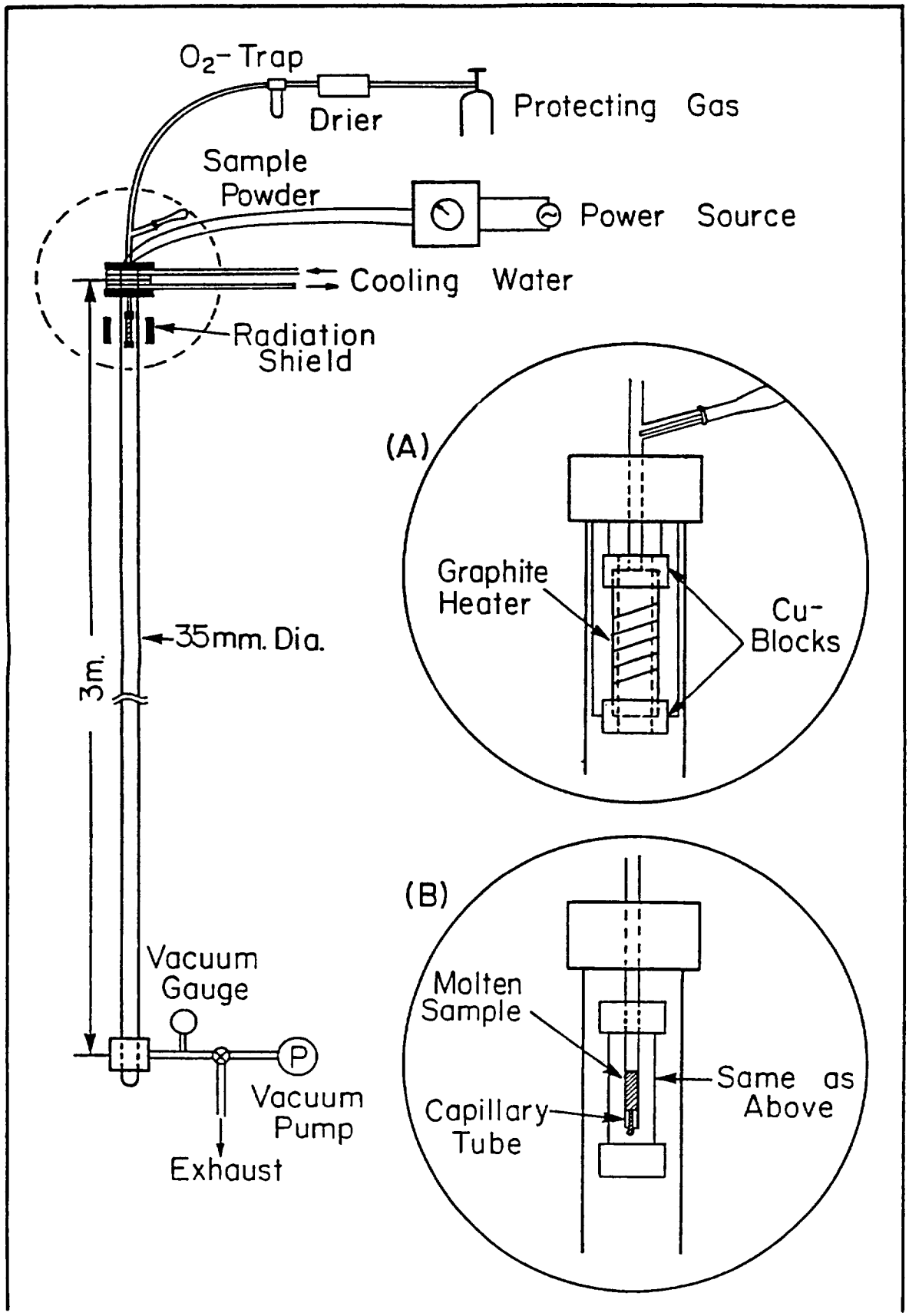
OBJECTIVES:

Develop and Test Concepts for Microgravity Experiments

* **Containerless Processing**

* **Solidification of Undercooled Melts**

* **Microstructural Evolution**


O_2- Trap
Drier
Protecting Gas
Sample Powder
Power Source
Cooling Water
Radiation Shield
35mm. Dia.
3m.
Vacuum Gauge
P
Vacuum Pump
Exhaust
(A)
Graphite Heater
Cu-Blocks
(B)
Molten Sample
Same as Above
Capillary Tube

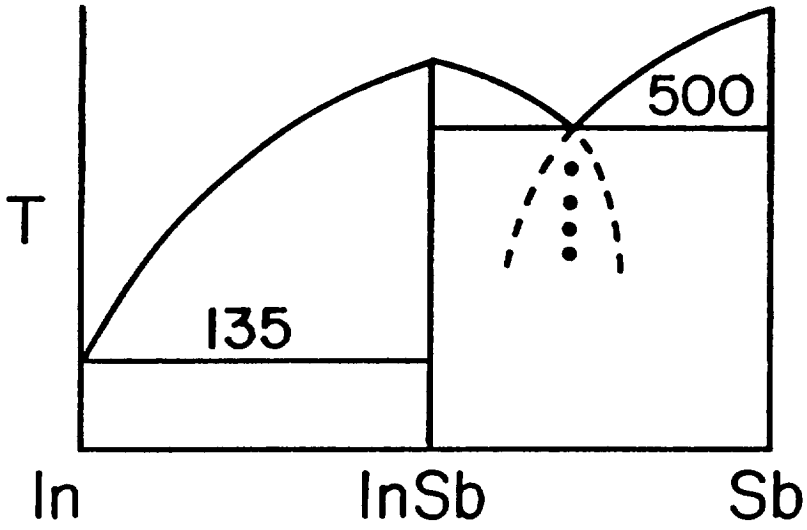
Evaluation of Liquid Undercooling

1) microstructural scale
   a) eutectic alloy

2) metastable phase formation
   a) amorphous solid
   b) Fe-Ni: bcc vs. fcc

3) heat flow analysis with
   experimental verification

Eutectic Solidification



$\Delta T = k_1 \lambda v + k_2/\lambda$ (Reg. Growth: $\Delta T_k \simeq 0$)

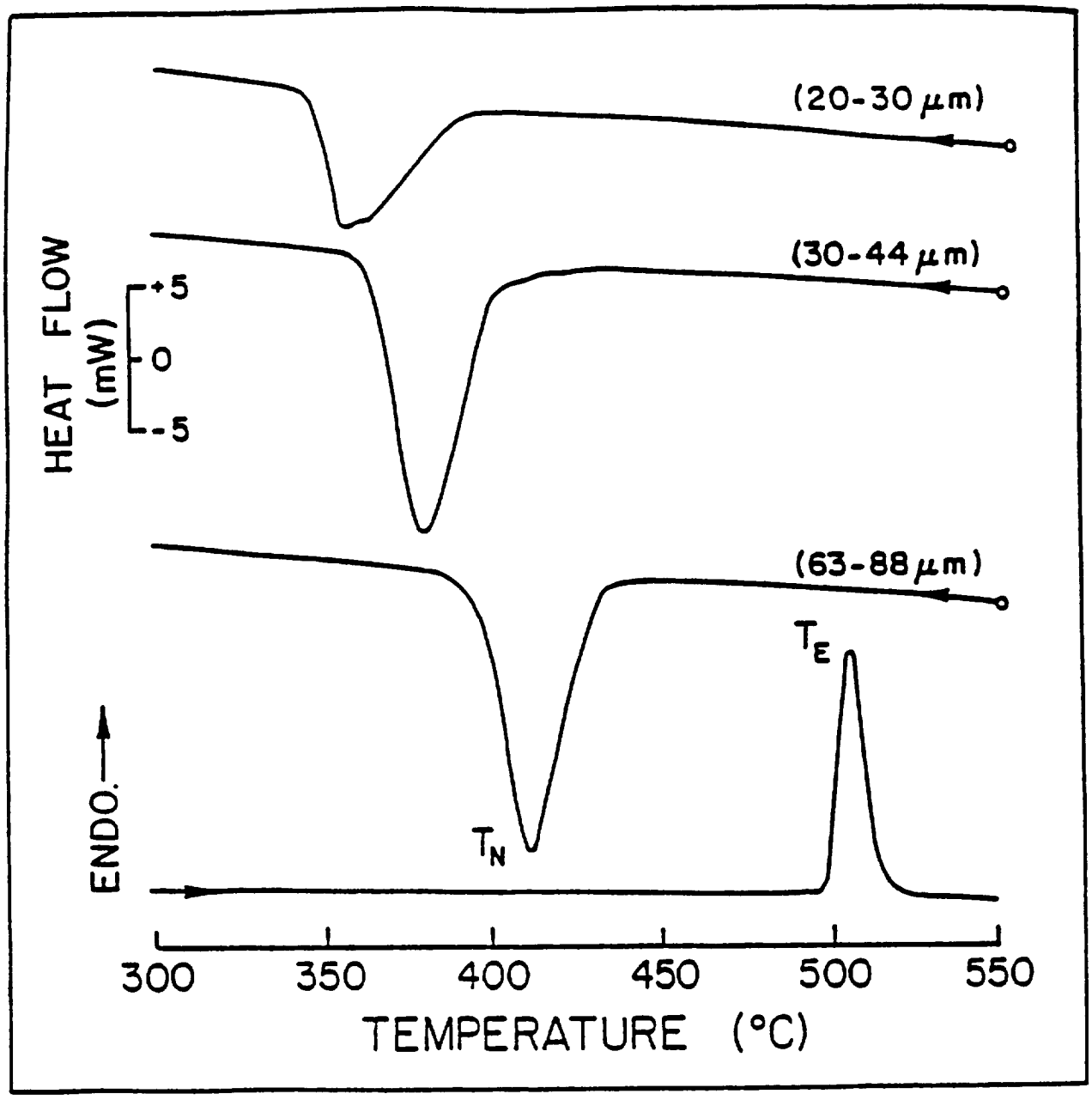
$\lambda^2 v = C_1$; $T^2/v = C_2$; $\Delta T \lambda = C_3$

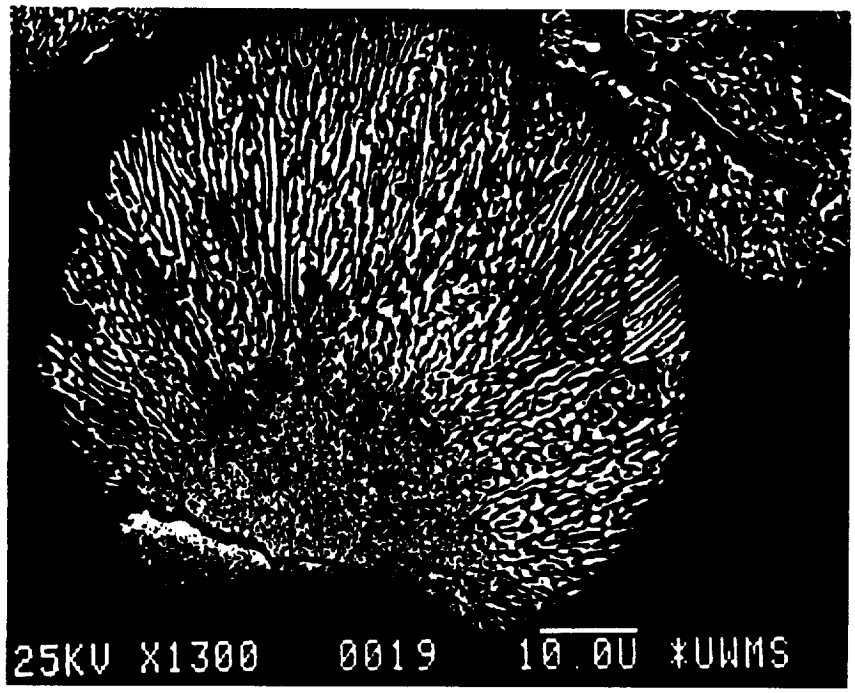
For InSb-Sb (f-f, $T_k \neq 0$, branching limit)

$\ln v = -4.16 - 2.09 \ln \lambda$ (Measured in D.S. Liebmann and Miller)

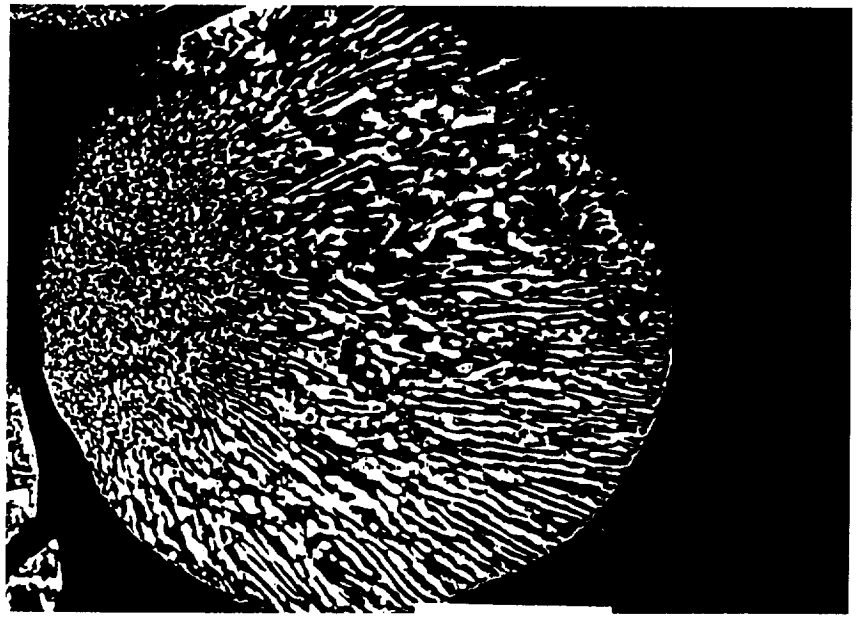
$\ln \lambda = 14.94 - 1.56 \ln \Delta T$ (Measured in droplet ΔT at onset - i.e. T_n)

λ onset $\rightarrow \Delta T \rightarrow v$


(20-30 μm)
(30-44 μm)
(63-88 μm)
HEAT FLOW
(mW)
+5
0
-5
ENDO.
T_N
T_E
300
350
400
450
500
550
TEMPERATURE (°C)

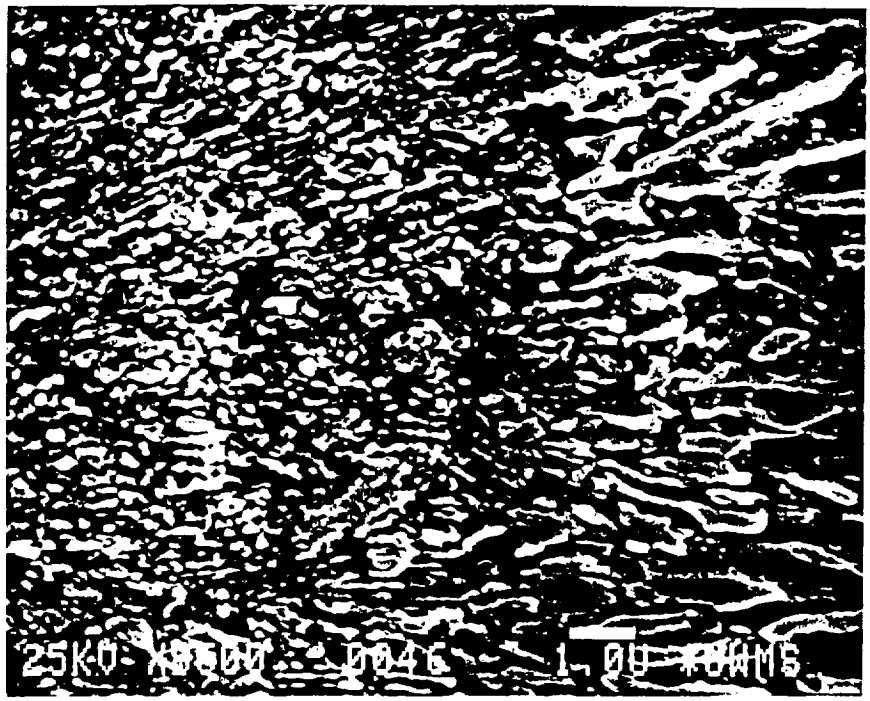


(a)

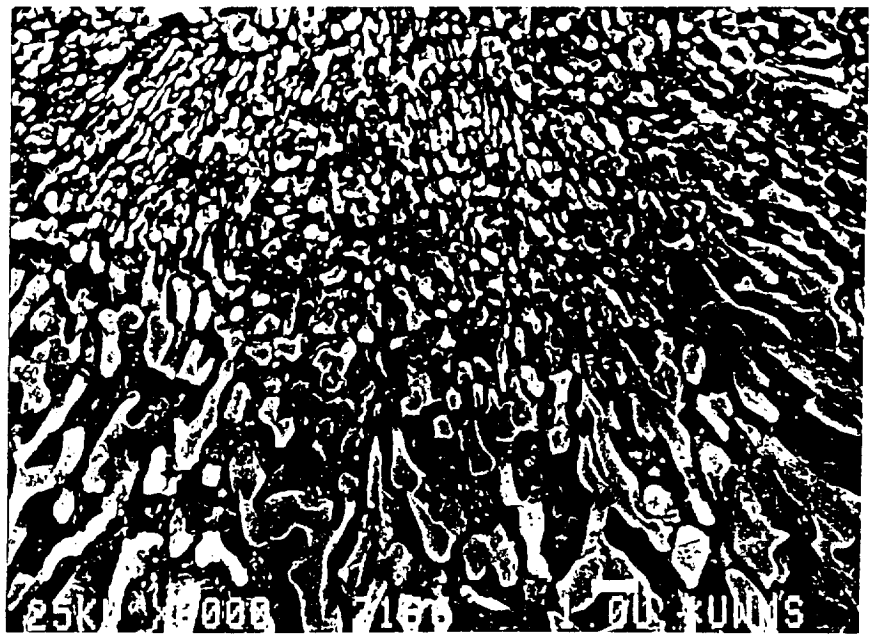


(b)

Figure 51. SEM micrographs showing the solidification structure following DSC Processing. (a) 1300X. (b) 2600X.

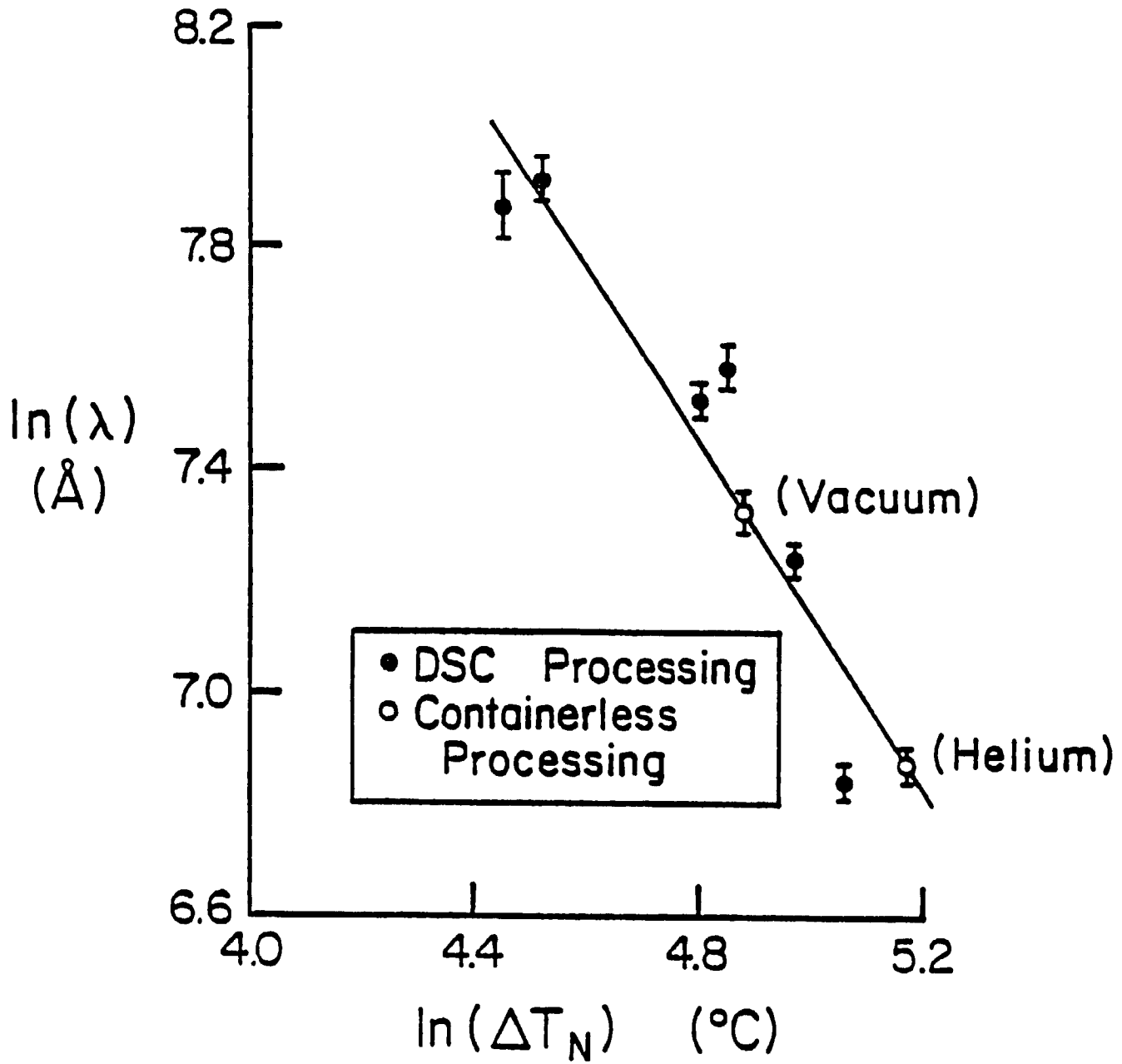


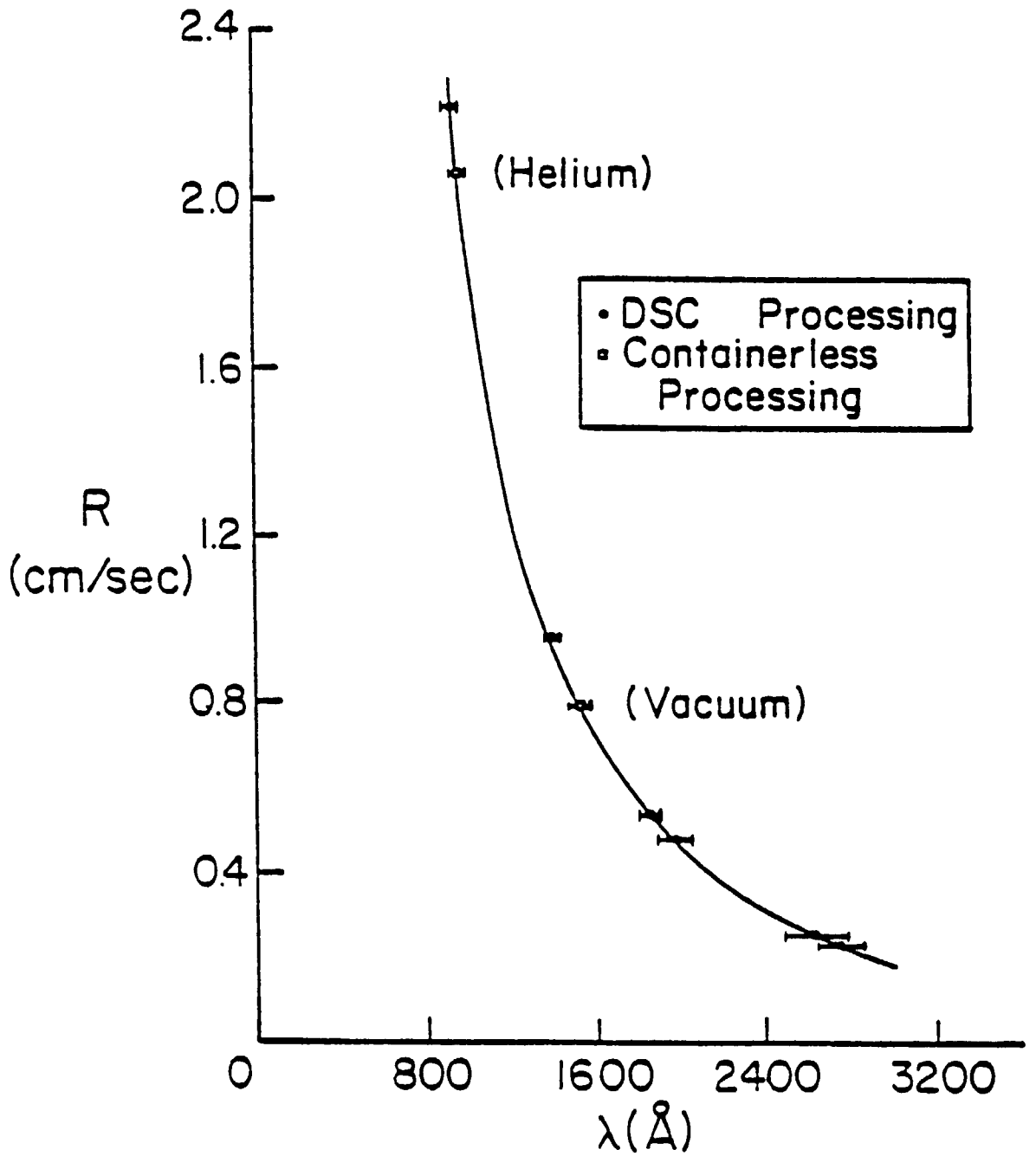
(a)



(b)

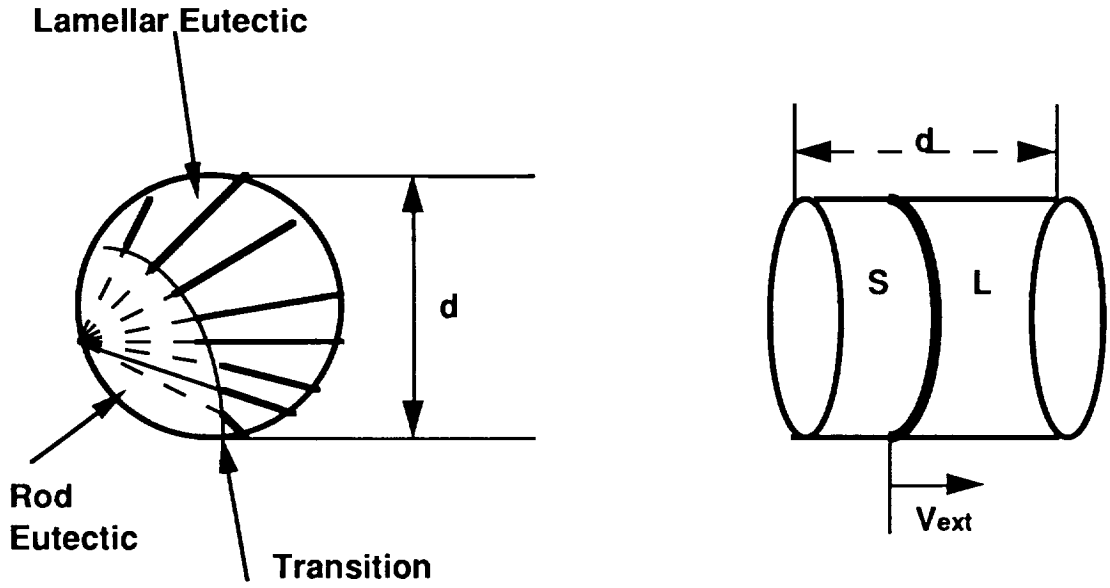
Figure 53. SEM micrographs illustrating the transition from a rod to lamellar morphology. (a) 8600X. (b) 6000X.


8.2
7.8
7.4
7.0
6.6
ln(λ)
(Å)
(Vacuum)
(Helium)
• DSC Processing
o Containerless
Processing
4.0
4.4
4.8
5.2
ln(ΔT_N) (°C)


2.4
2.0
1.6
1.2
0.8
0.4
0
R
(cm/sec)
(Helium)
(Vacuum)
• DSC Processing
▫ Containerless Processing
800
1600
2400
3200
λ(Å)

COMPETITIVE GROWTH KINETICS AND MICROSTRUCTURAL TRANSITION

ROD EUTECTIC----LAMELLAR EUTECTIC



MODEL:

V IS CONTROLLED BY UNDERCOOLING IN ROD EUTECTIC AND BY EXTERNAL COOLING IN LAMELLAR EUTECTIC

AT TRANSITION: $V_{rod} = V_{ext}$

MEASUREMENT:

$\lambda_R = f(\Delta T)$ [Droplet, DTA]

$\lambda_R = f(V_R)$ [D.S.]

$$V_{ext} = \frac{b\, h\, \Delta T_\infty}{\Delta H_f}$$

$\Delta T_\infty = T_{drop} - T_{gas}$

$\Delta H_f = 1252$ joules/cm^3

$$h = \frac{2K_{gas}}{d} + 0.6\left(\sqrt{\frac{u}{d}}\right) c \approx \frac{2k_{gas}}{d}$$

$\frac{vd}{\alpha_2} << 1$ for uniform T

$K_{He} = 2 \cdot 10^{-3}$ j/ s.cm.K

$$B_i = \frac{hd}{K} \approx 1.3 \cdot 10^{-2}$$

$$V_{ext} = 1.9 \cdot 10^{-5} \frac{\Delta T_\infty}{d}$$

$$V_R = 8.8 \cdot 10^{-8} \Delta T^{3.3}$$

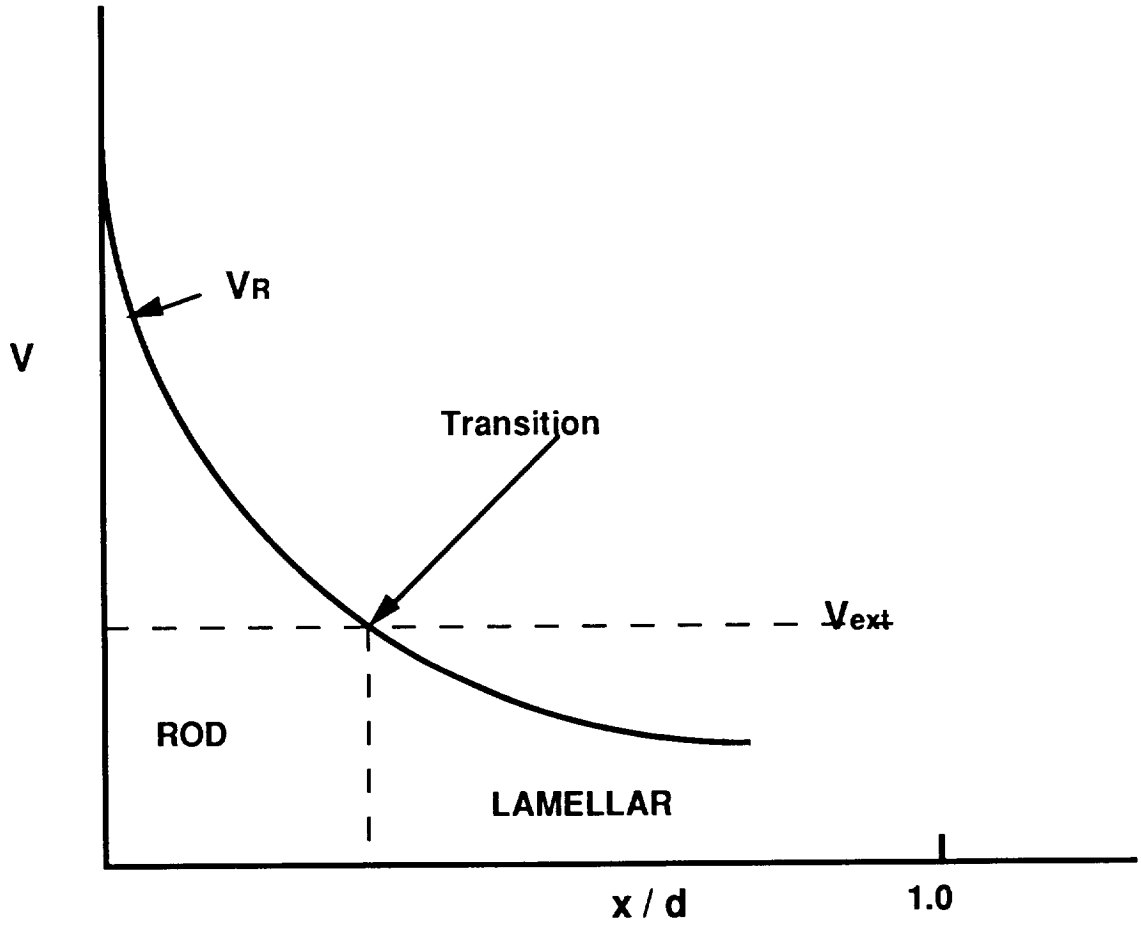
for a 36μ droplet

$\lambda_{onset} = 960A$, $V_{onset} = 2.1$ cm/sec

$\lambda_{transition} = 1100$ Å

$V_{ext} = 1.66$ cm/sec ; $V_R = 1.58$ cm/sec

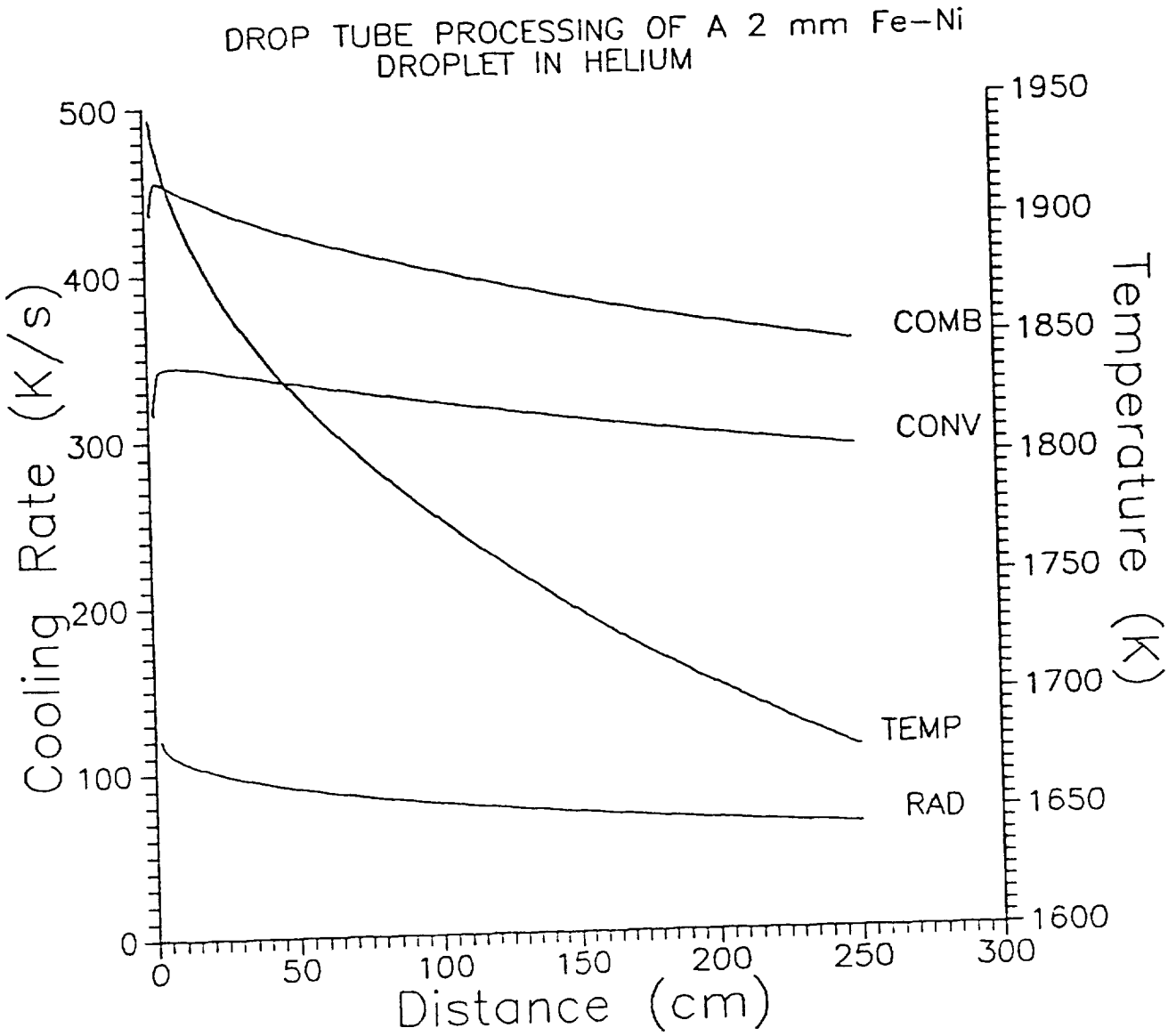
$\therefore V_{ext} \approx V_R$

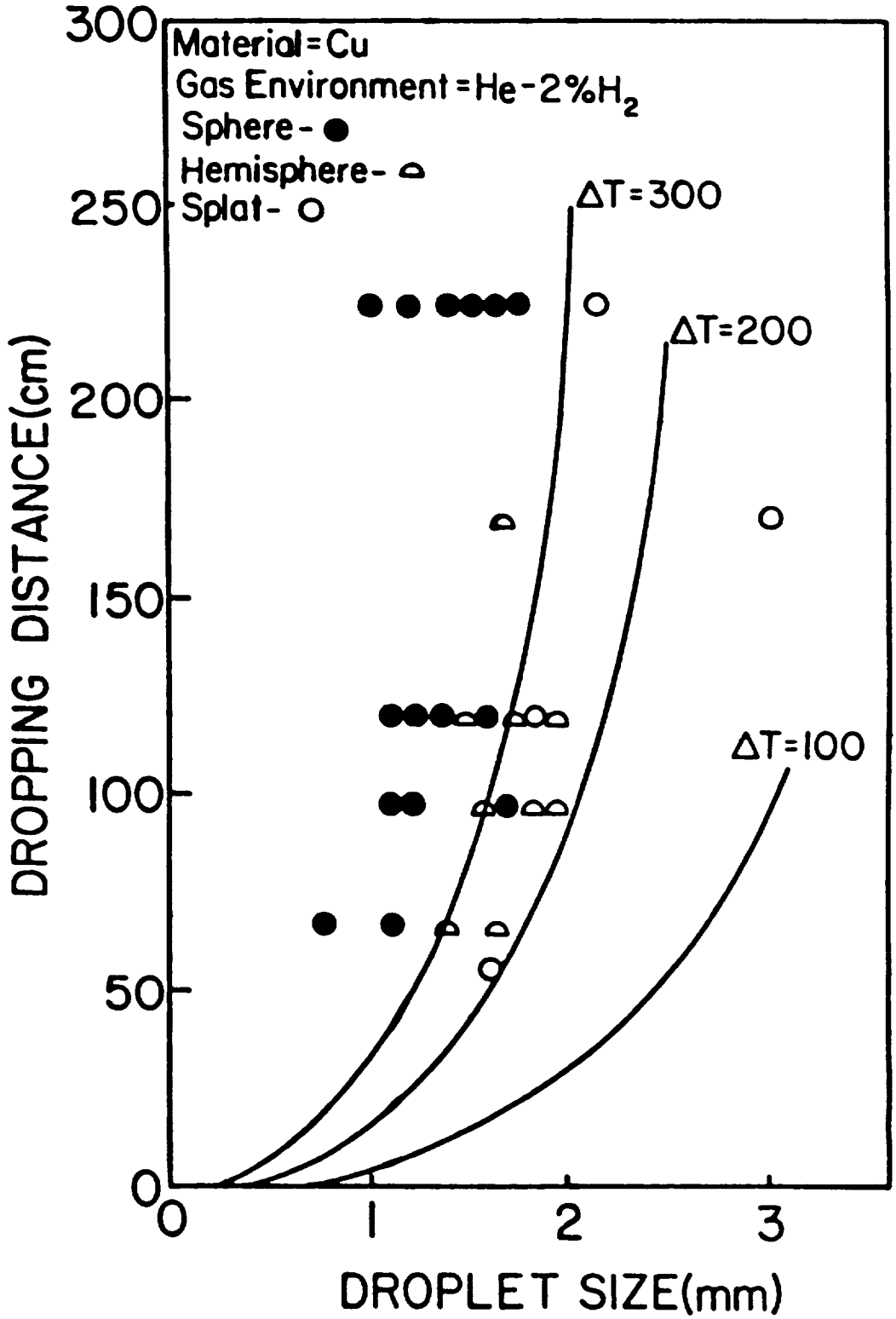


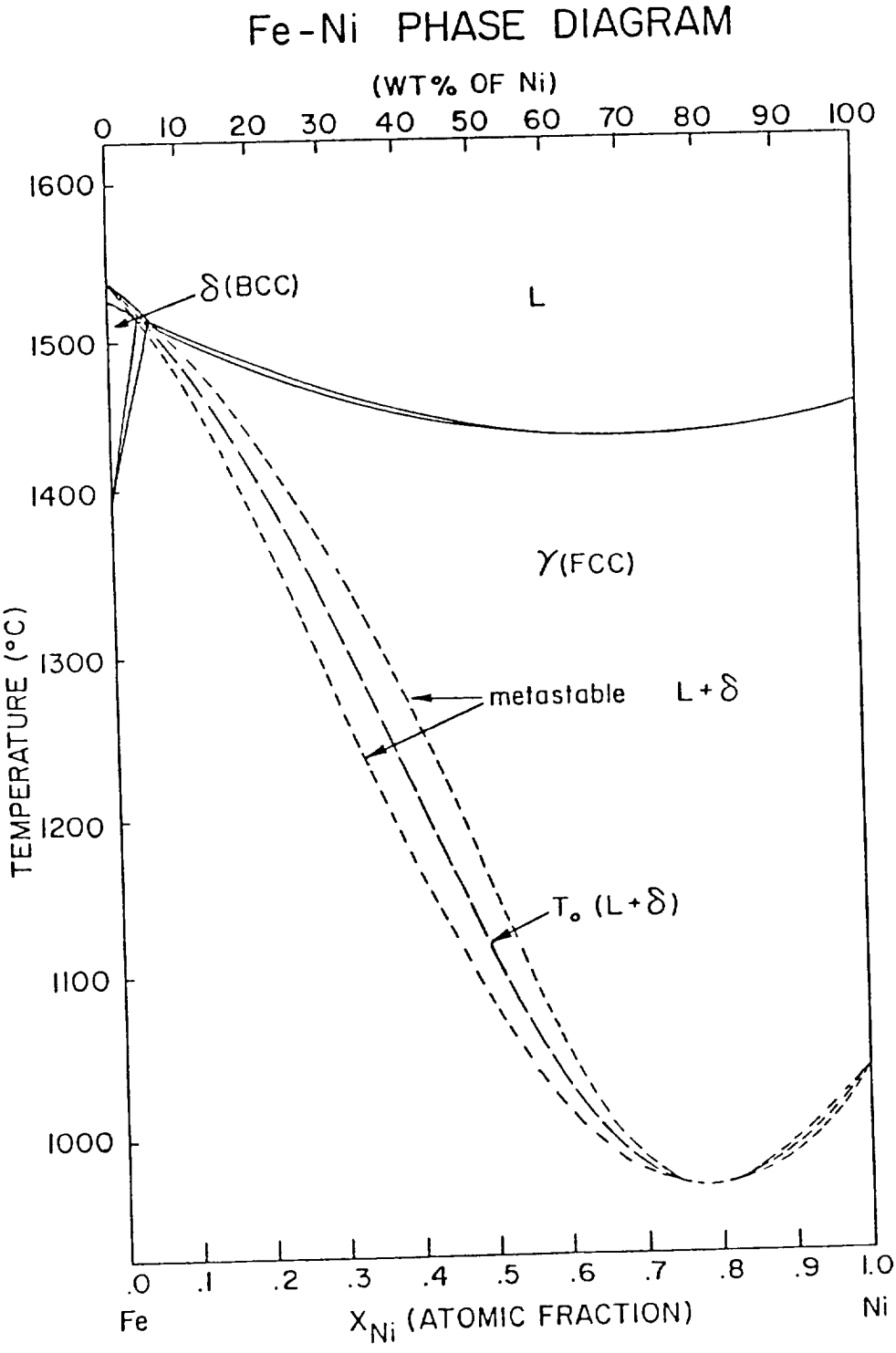
$$\frac{dT}{dt} = -\frac{\varepsilon A \sigma}{m c}\left(T^4 - T_w^4 \right) - \frac{h A}{m c}\left(T - T_a \right)$$

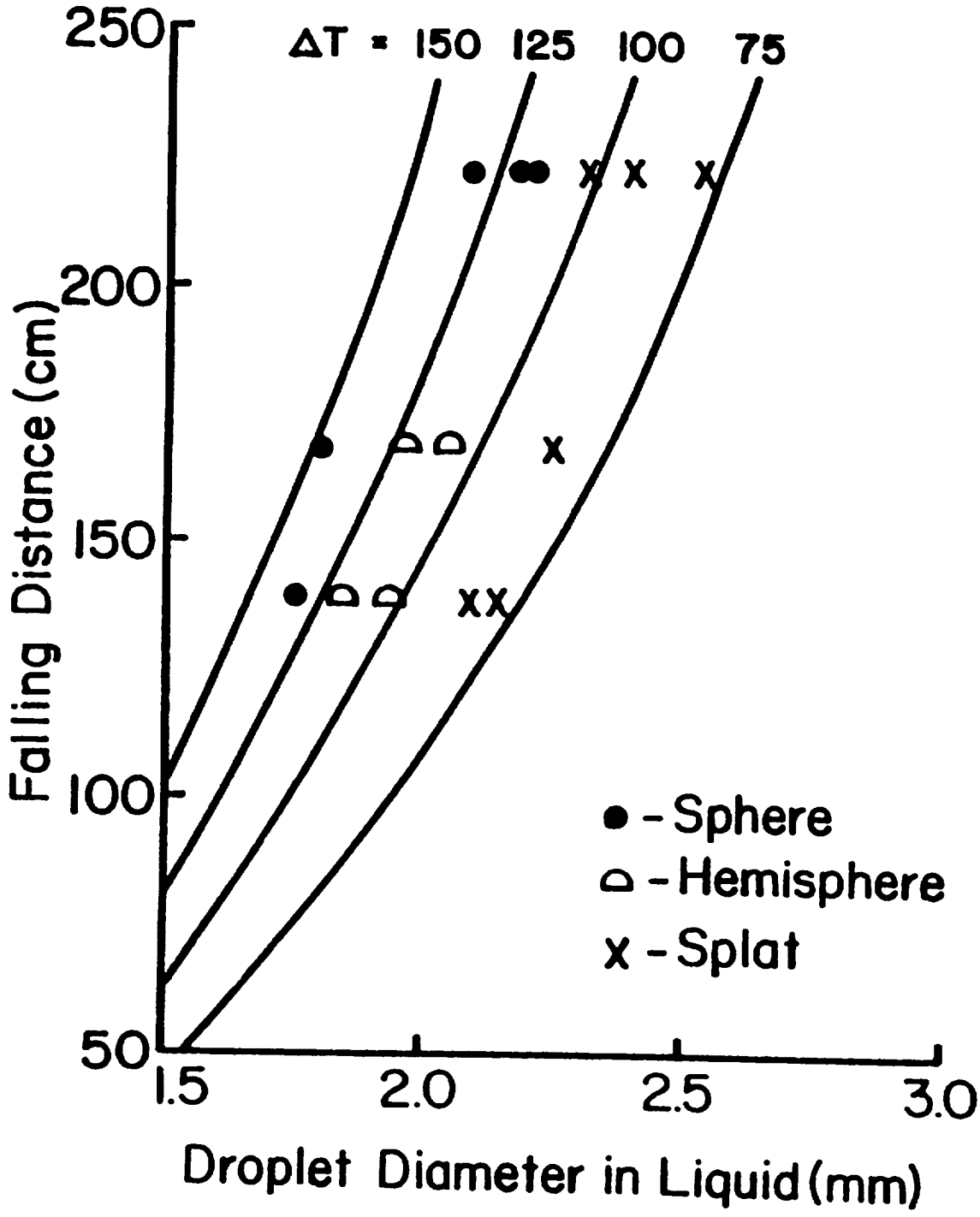
T_a - gas film temperature $(T + T_g)/2$
T_g - gas temperature
T_w - room temperature
ε - emissivity of liquid metal
A - surface area of droplet
σ - Stefan-Boltzmann constant
c - specific heat
m - mass
h - heat transfer coefficient
t - time

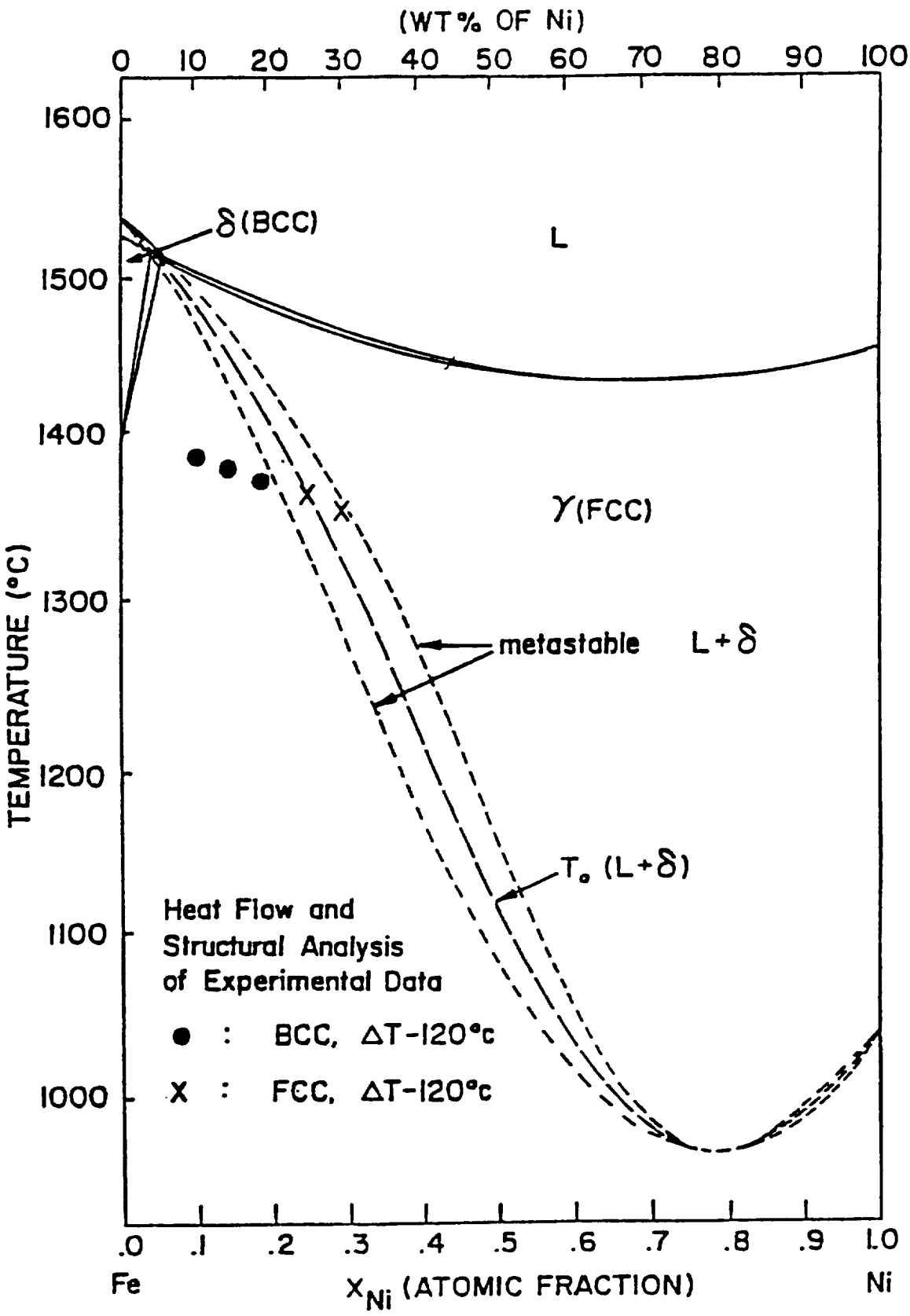
t = f(veloctiy)
v = f(distance, drag forces)


DROP TUBE PROCESSING OF A 2 mm Fe–Ni
DROPLET IN HELIUM
Cooling Rate (K/s)
Temperature (K)
Distance (cm)
COMB
CONV
TEMP
RAD
0
100
200
300
400
500
1600
1650
1700
1750
1800
1850
1900
1950
0
50
100
150
200
250
300


Material=Cu
Gas Environment=He-2%H2
Sphere-
Hemisphere-
Splat-
ΔT=300
ΔT=200
ΔT=100
DROPPING DISTANCE(cm)
DROPLET SIZE(mm)
300
250
200
150
100
50
0
0
1
2
3


Fe-Ni PHASE DIAGRAM
(WT% OF Ni)
0 10 20 30 40 50 60 70 80 90 100
1600
1500
1400
1300
1200
1100
1000
TEMPERATURE (°C)
δ(BCC)
L
γ(FCC)
metastable
L+δ
T₀ (L+δ)
.0 .1 .2 .3 .4 .5 .6 .7 .8 .9 1.0
Fe
X_Ni (ATOMIC FRACTION)
Ni


ΔT = 150 125 100 75
250
200
150
100
50
Falling Distance (cm)
1.5
2.0
2.5
3.0
Droplet Diameter in Liquid (mm)
● - Sphere
○ - Hemisphere
x - Splat


(WT% OF Ni)
0 10 20 30 40 50 60 70 80 90 100
1600
1500
1400
1300
1200
1100
1000
TEMPERATURE (°C)
δ(BCC)
L
γ(FCC)
metastable L+δ
T₀ (L+δ)
Heat Flow and
Structural Analysis
of Experimental Data
● : BCC, ΔT-120°c
X : FCC, ΔT-120°c
.0 .1 .2 .3 .4 .5 .6 .7 .8 .9 1.0
Fe
X_Ni (ATOMIC FRACTION)
Ni

POWDER SOLIDIFICATION KINETICS

* Single Nucleation Mechanism

* Random Arrangements of Nucleation Sites
  (i.e. Poisson Distribution)

$$X = \exp(-AD^n)$$

X : Nucleant-Free Fraction

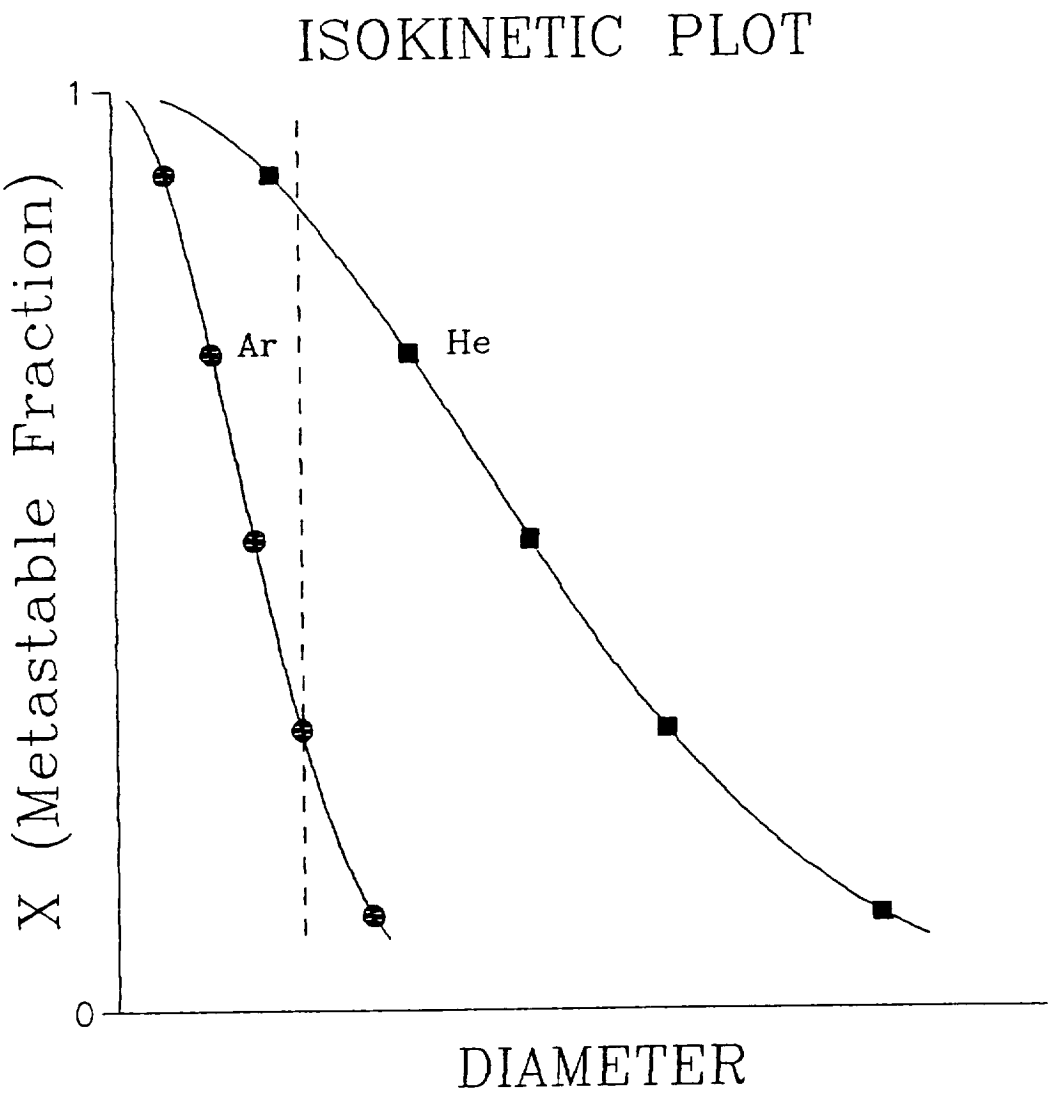
A : Constant

D : Powder Diameter

n: Related to Nucleation Mechanism

n = 2 Surface

n = 3 Volume


ISOKINETIC PLOT
1
0
X (Metastable Fraction)
Ar
He
DIAMETER

ISOKINETIC SHIFT

$$\Delta T^2 = k\ f(\dot{T})$$

Convective cooling...

$$\dot{T} = \frac{6}{\rho\ c_p\ D}\ h\ (T-T_a)$$

Low velocities and small droplet diameters...

$$\dot{T} = \frac{6}{\rho\ c_p\ D}\ \frac{2k_{gas}}{D}\ (T-T_a)$$

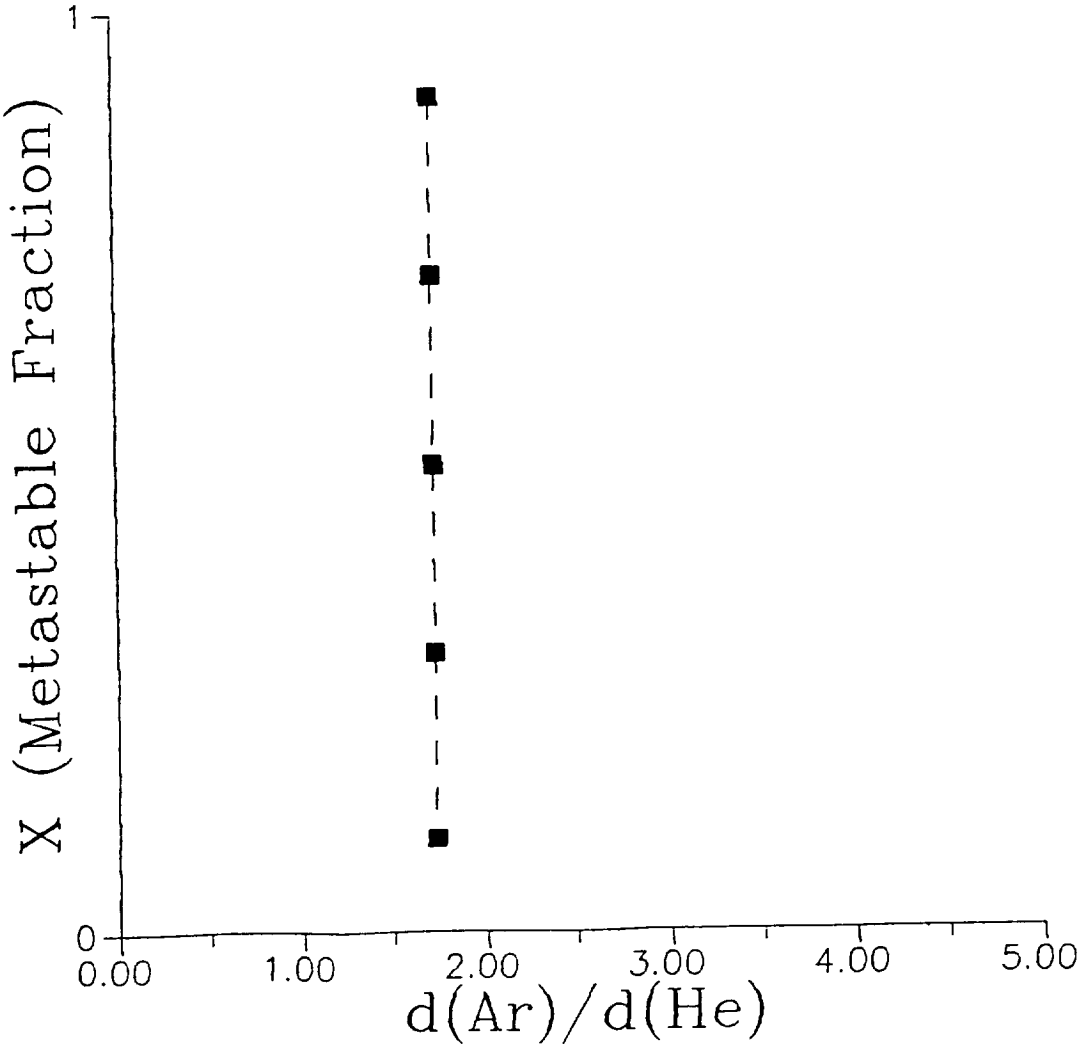
Comparing different gases...

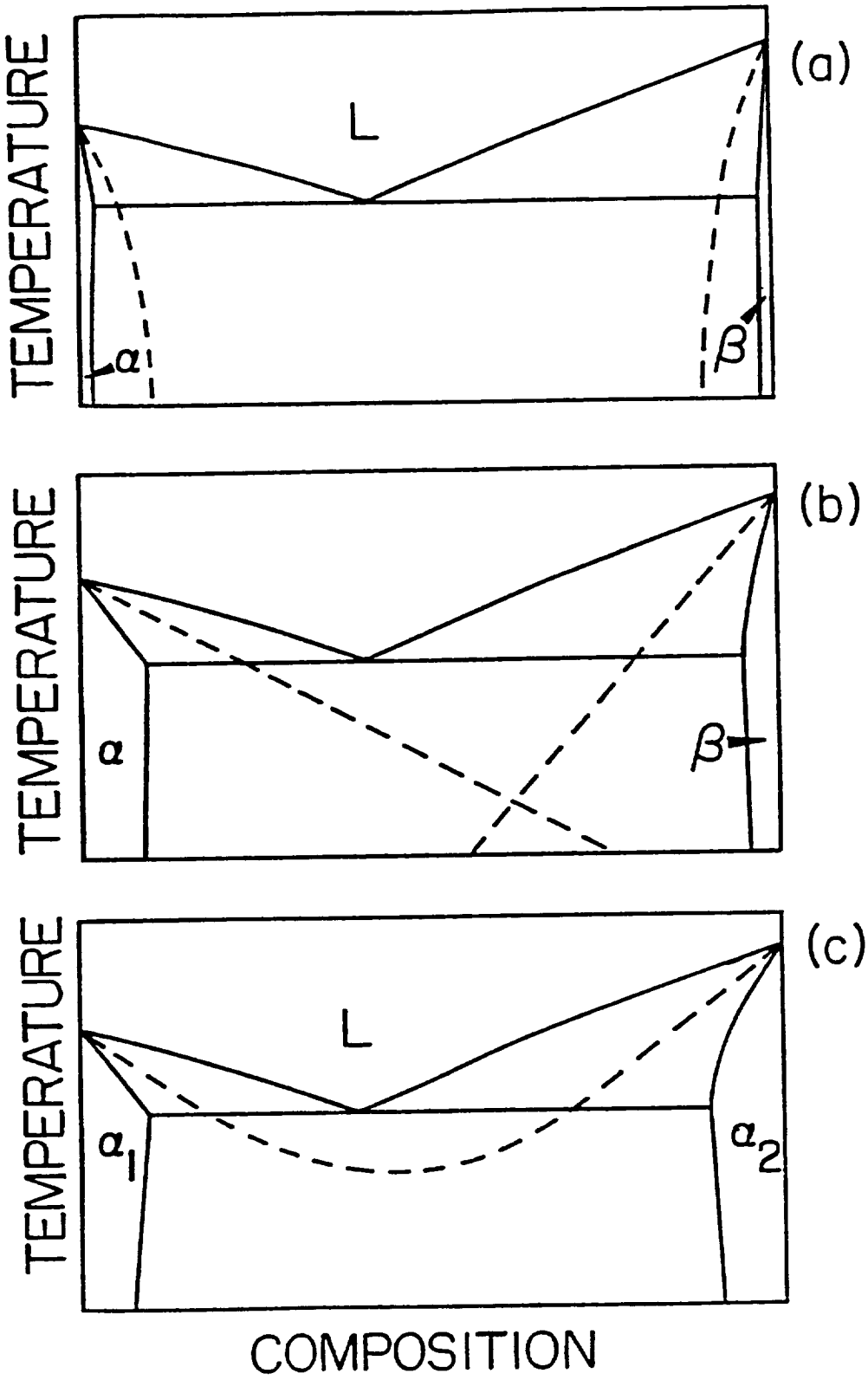
$$\frac{k_1}{k_2} = \frac{D_2{}^2}{D_1{}^2}$$

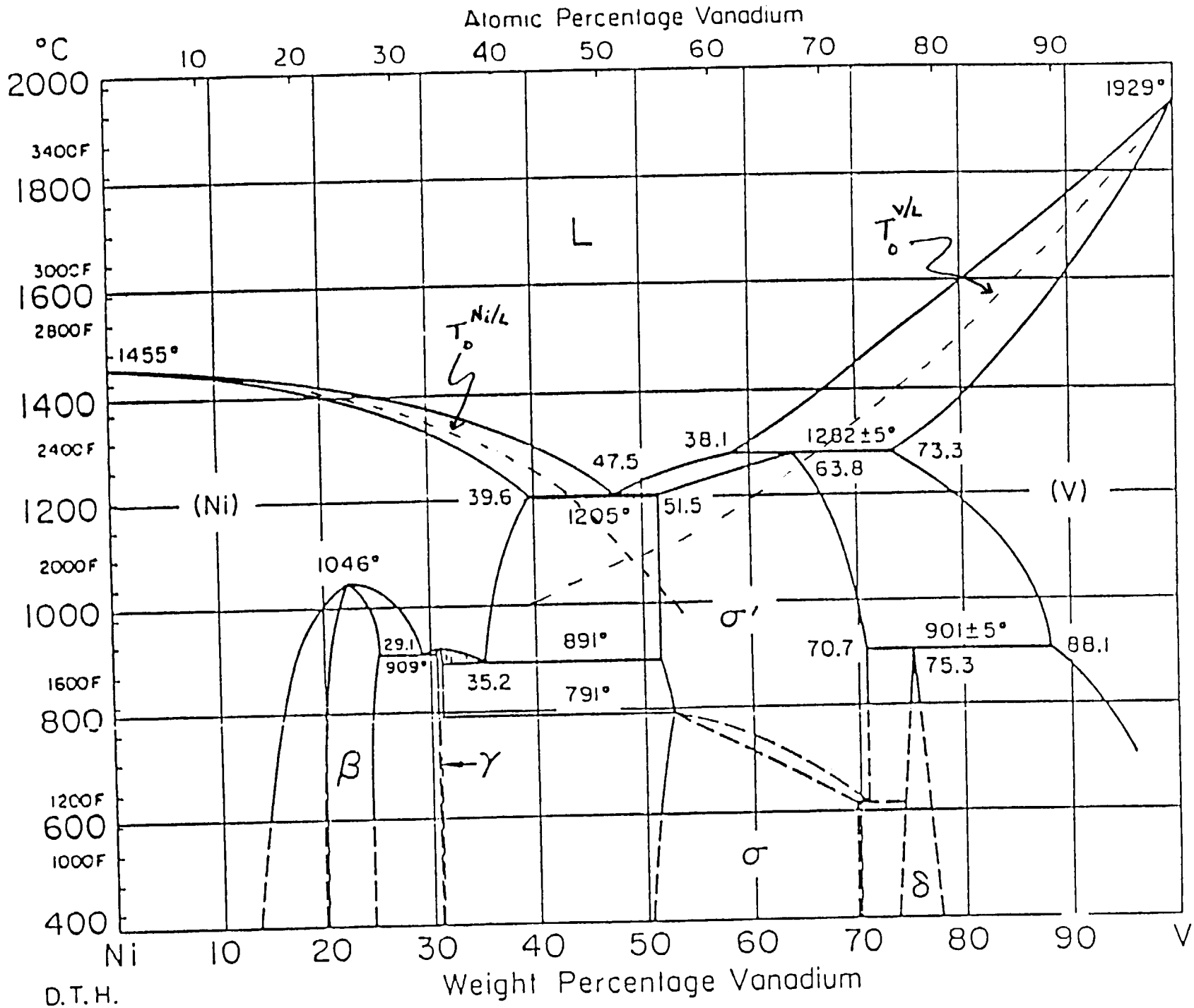
Applying to the Poisson Distribution...

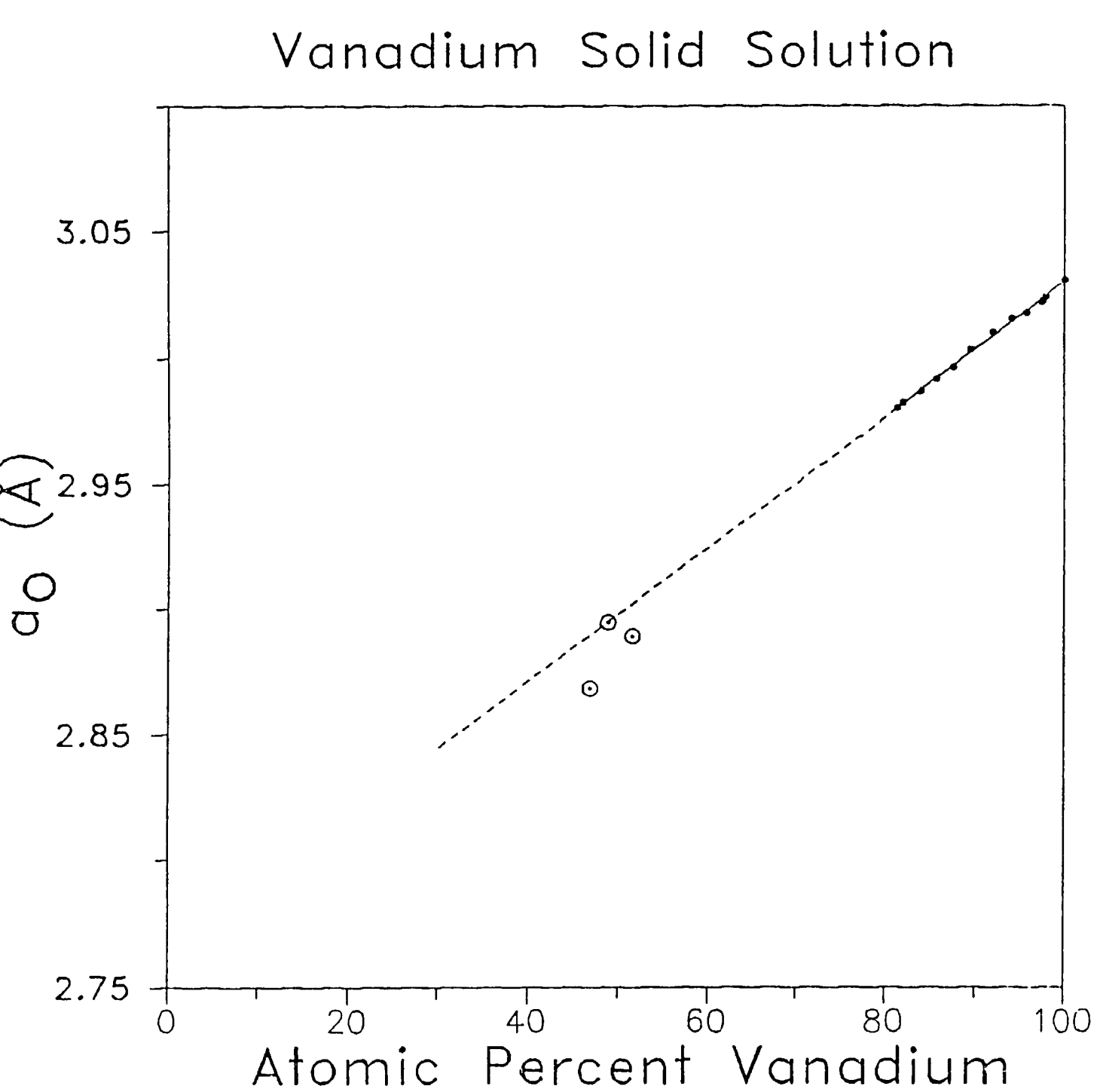
$$X_{He} = \exp\left[-\left(\frac{D_{He}}{D_o}\right)^2\right]$$

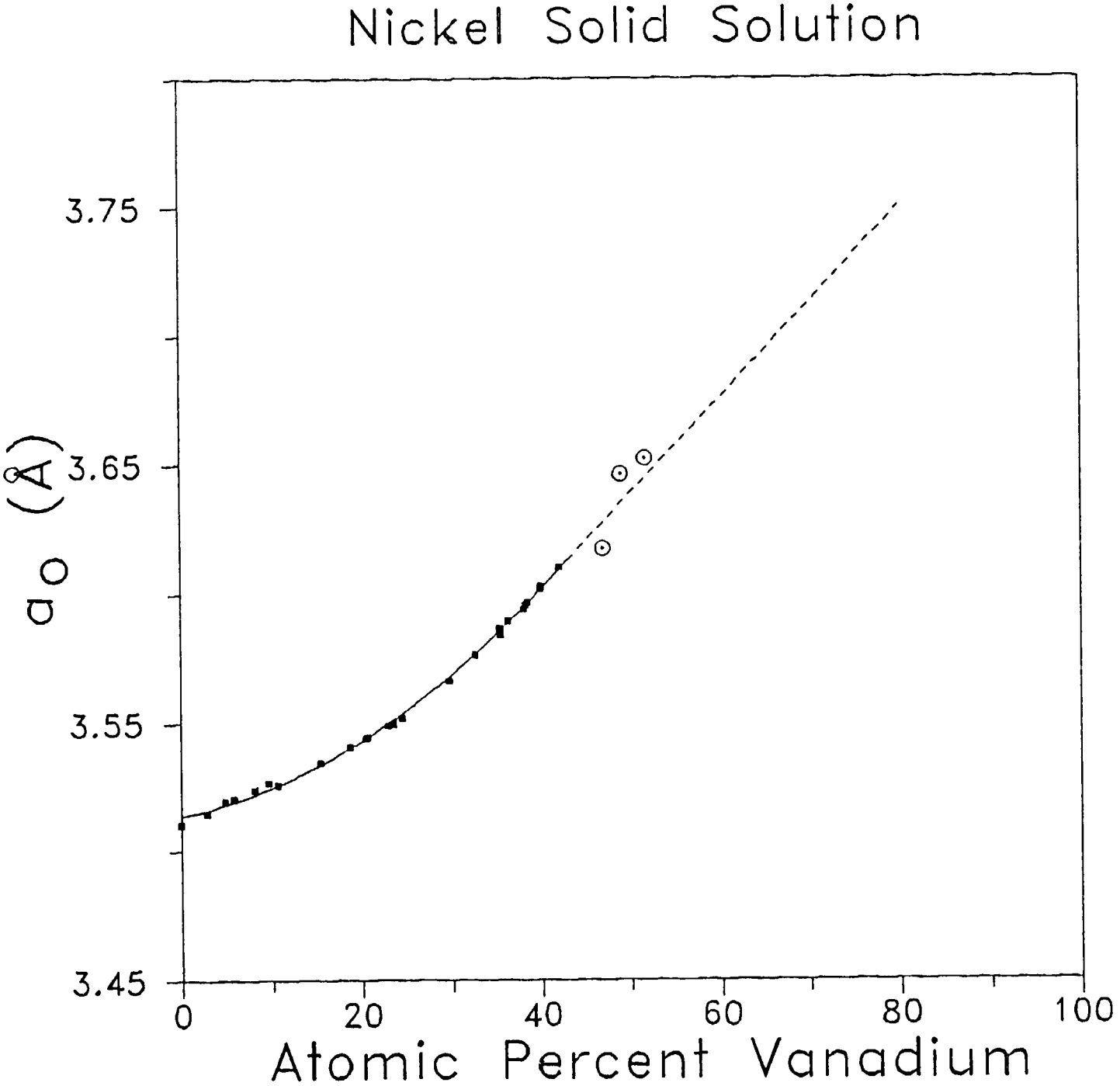
$$X_{Ar} = \exp\left[-\left(\frac{\sqrt{3}D_{He}}{D_o}\right)^2\right]$$


X (Metastable Fraction)
1
0
0.00
1.00
2.00
3.00
4.00
5.00
d(Ar)/d(He)


TEMPERATURE
L
(a)
α
β
TEMPERATURE
(b)
α
β
TEMPERATURE
L
(c)
α1
α2
COMPOSITION


Atomic Percentage Vanadium
°C
2000
3400F
1800
3000F
1600
2800F
1400
2400F
1200
2000F
1000
1600F
800
1200F
600
1000F
400
10 20 30 40 50 60 70 80 90
1929°
L
$T_o^{V/L}$
$T_o^{Ni/L}$
1455°
38.1
1282±5°
73.3
47.5
63.8
(Ni)
39.6
1205°
51.5
(V)
1046°
σ'
29.1
909°
891°
70.7
901±5°
88.1
75.3
35.2
791°
β
γ
σ
δ
Ni 10 20 30 40 50 60 70 80 90 V
Weight Percentage Vanadium
D.T.H.


Vanadium Solid Solution
3.05
2.95
2.85
2.75
a_0 (Å)
0
20
40
60
80
100
Atomic Percent Vanadium


Nickel Solid Solution
3.75
3.65
3.55
3.45
a_o (Å)
0
20
40
60
80
100
Atomic Percent Vanadium

SUMMARY REMARKS

. Undercooled liquids expose a variety of metastable states and Microstructure options

. Containerless processing offers an important background experience to control undercooling, and to process undercooled liquids so that successful microgravity experiments can be undertaken

. Microgravity and Earth-based experiments should be coodinated

SUMMARY

. Progress in Undercooling Control and Analysis

- . Sample size scale-up
- . Surface sensitivity

. Microstructure / Kinetic Transitions

. Metastable Phase Diagrams

- . Interpret non-equilibrium effects
- . Identify microstructural options (Alloy design)
- . Can be accessed experimentally
- . Several variations possible

. Containerless Processing

- . Microstructural probe of undercooling
- . Heat flow analysis
- . Need direct non-contact temperature measurement
- . Measured kinetic response-Processing

N91-21340

CONTAINERLESS SYNTHESIS

OF

INTERESTING GLASSES

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Tucson, AZ 85721

January 17, 1990

CONTAINERLESS GLASS PROCESSING AND STUDIES

Motivations:

*Novel glasses and enhanced glassforming ability

Ultrapure glasses

Gas bubbles and microballoons

Phase separation

Foams

Composite materials (suspensions)

Surface tension

Surface (free) crystallization

High temperature thermophysical properties

OUTLINE

I. Standard Arguments for Containerless Processing (C.P.) of Glasses

II. Flight Results

III. Re-examination of Motivations for C.P.

IV. Two Interesting Candidate Systems

I. Why do containerless processing of glasses?

A. Standard argument

1. Kinetic viewpoint - glass-forming ability, competition of cooling rate vs. crystallization rate
2. Thus, to form new glasses one wishes to suppress crystallization
3. First step in crystallization is nucleation; i.e. preventing nucleation could stop devitrification
4. Nucleation can be greatly aided by liquid being in contact with solid (foreign) surfaces (so called heterogeneous nucleation)
5. The container and impurities from container can be effective heterogeneous nucleation sites

Thus, 6. Containerless processing could prevent crystal nucleation and lead to new family of glasses

B. Required Cooling Rate

1. Define: x = volume fraction of crystals formed

I = crystal nucleation rate

g = crystal growth rate

t = time; T = temperature

R = dT/dT = cooling rate

2. Say for non-isothermal transformation desire $X \leq X_c$ (where $X_c << 1$)

$$X = \frac{4\pi}{3} \int_{t_i}^{t_f} dt\ I(t) \left[\int_{t}^{t_f} g(t')\,dt' \right]^3$$

$$= \frac{4\pi}{3} \int_{T_i}^{T_f} R^{-1}(T)\, I(t)\, dT \left[\int_{T}^{T_f} g(T')\, R^{-1}(T')\, dT' \right]^3$$

3. For $X = X_c$, defines integral equation for R(T)

4. If functional form of R(T) given, then parameters in R(T) can be found

5. For R = constant

$$R_c^4 = \frac{4\pi}{3X_c} A$$

$$A = \frac{4\pi}{3} \int_{T_i}^{T_f} dT \; I(T) \left[\int_{T}^{T_f} g(T') dT \right]^3$$

6. (Aside: cooling rate in microgravity situation not constant and fixed unless special cooling apparatus.)

7. From above we note that as I(T) diminishes, required R_c is reduced

C. Nucleation

1. Steady State Homogeneous

$$I^{\circ}_{Hom} = Z \cdot D^{*} \cdot N^{*}_{e}$$

Z ≡ Zeldovich factor

D* ≡ interfacial transport and attempt term and most important term (for present discussion)

$N^{*}_{e} = N^{\circ}\exp^{(-W^{*}/kT)}$

where W* = thermodynamic barrier to form critical radius

N° ≡ number of sites/volume (available for nucleation)

2. Steady State Heterogeneous

$$I^{\circ}_{Het} = Z' \cdot D^{*} * N'_{e}$$

$$N_e' = n_s \exp(-W^* f(\theta)/kT)$$

n_s = number of sites available for heterogeneous nucleation/volume

$$0 \leq f(\theta) \leq 1, \text{ where } f(\theta) = (2 - \cos\theta + \cos^3\theta)/4$$

θ = contact angle

Thus, the effective lowering of barrier due to $f(\theta)$ makes heterogeneous nucleation the commonly observed crystal nucleation mehanism.

D. Comments:

1. Removal of heterogeneous sites can be very effective in reducing x since I,g overlap important.

2. Above particularly true for marginal glass formers where g expected to be large.

3. Homogeneous nucleation could prevent glass formation, but many compositions "immune" due to meager I,g overlap.

4. Containerless processing may not guarantee homogeneous nucleating conditions.

II. Flight Experiments

A. Delbert Day, P.I.

Mission: D-1 SLM on MEA/A-2

Experiment: 81F01

Sample Characteristics

#	Composition (mol. %)	Diameter (nm)	Rc (earth)
2	$39.3Ga_2O_3$ - 35.7CaO - $25SiO_2$ (hot pressed)	6	11 ± 2 °C/sec.
6	$56Ga_2O_3$ - 44CaO (devitrified melt)	6	550 ± 50 °C/sec.

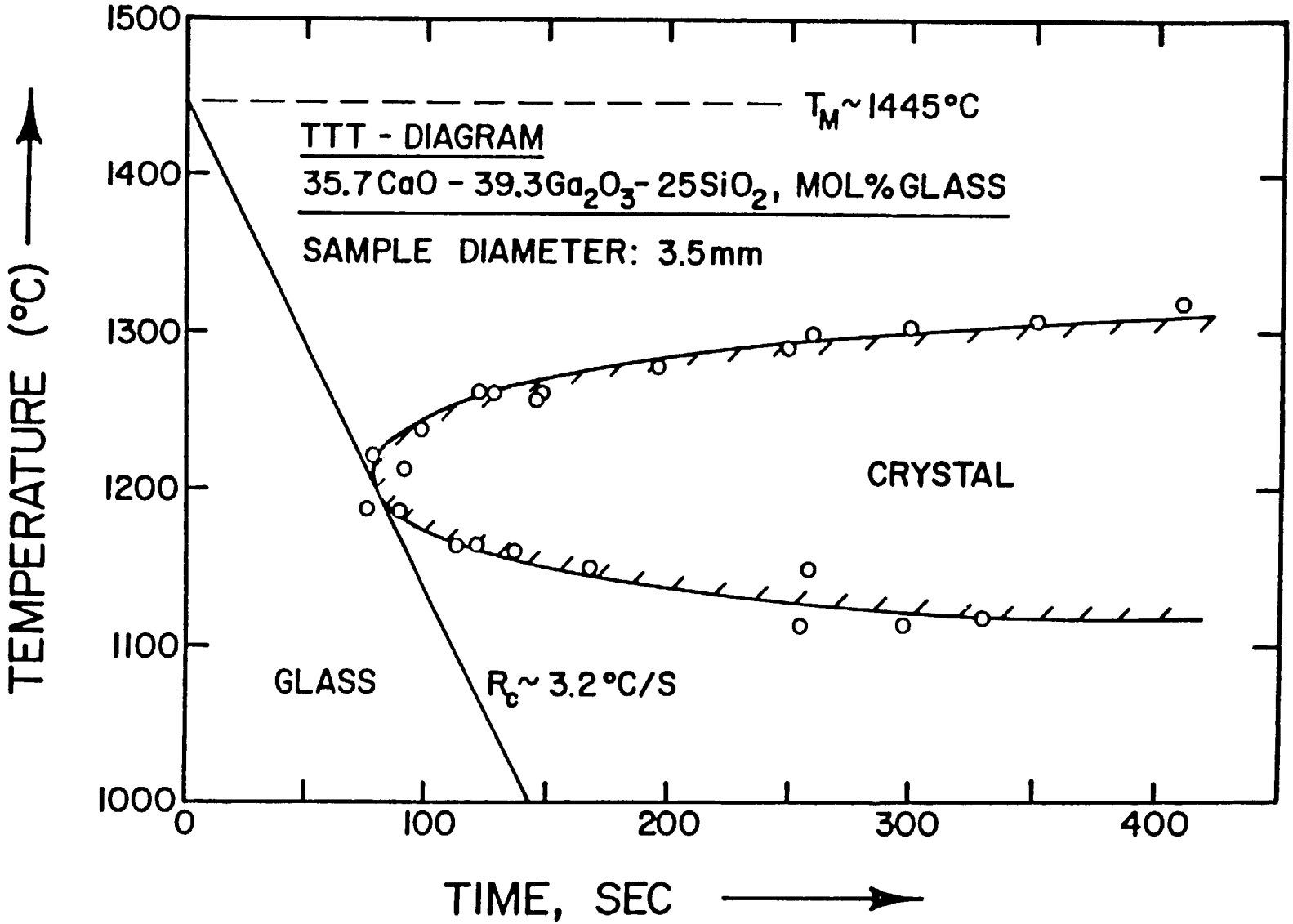

Figure 6. TTT diagram for the 35.7 CaO-39.3 Ga_2O_3-25 SiO_2, mol%, composition for sample diameter ~3.5 mm.

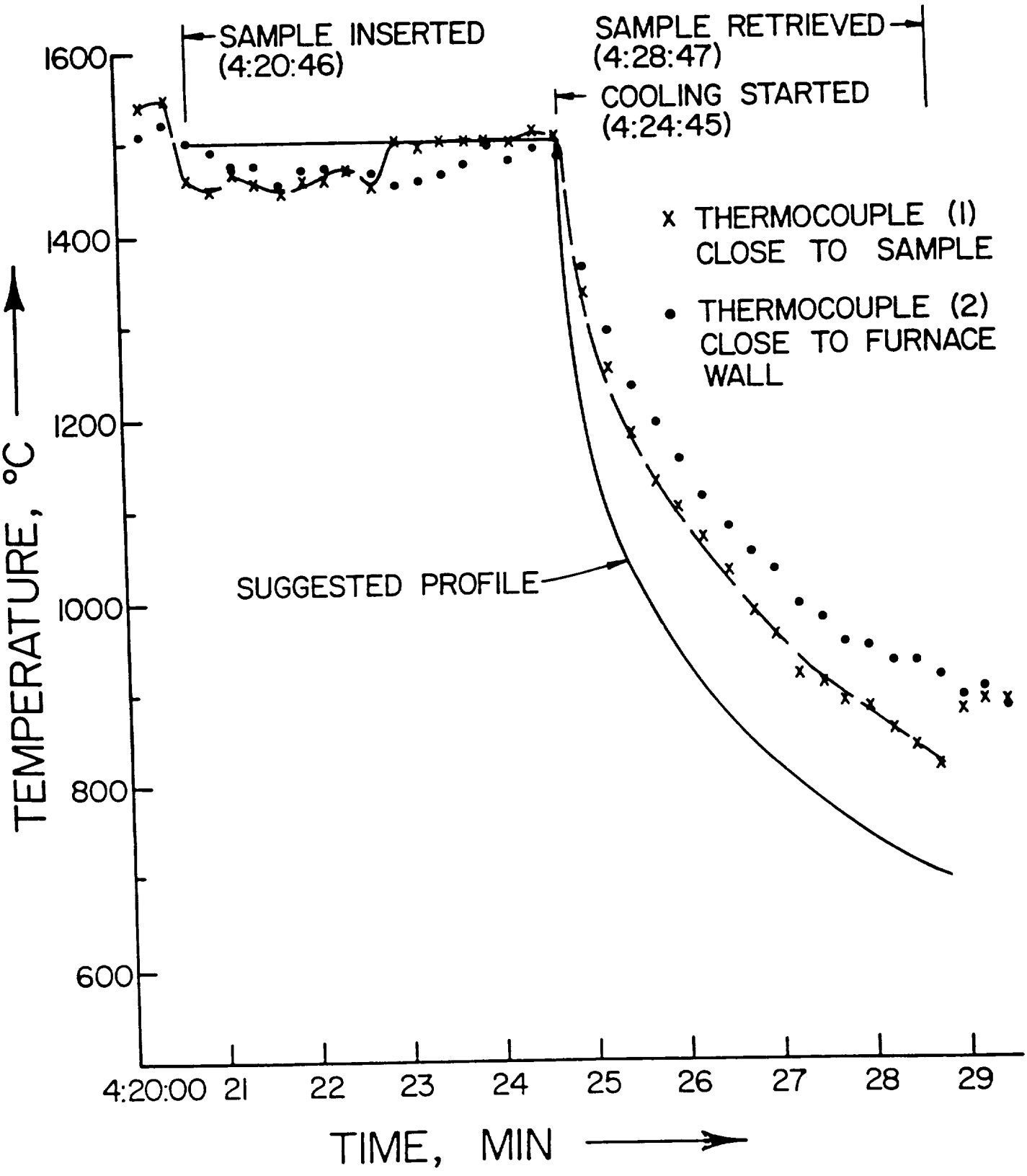

Figure 13. The planned and observed time-temperature profile for 35.7 CaO-39.3 Ga_2O_3-25 SiO_2, mol%, hot pressed sample (±2) when processed in the single axis acoustic levitator, MEA/A-2.

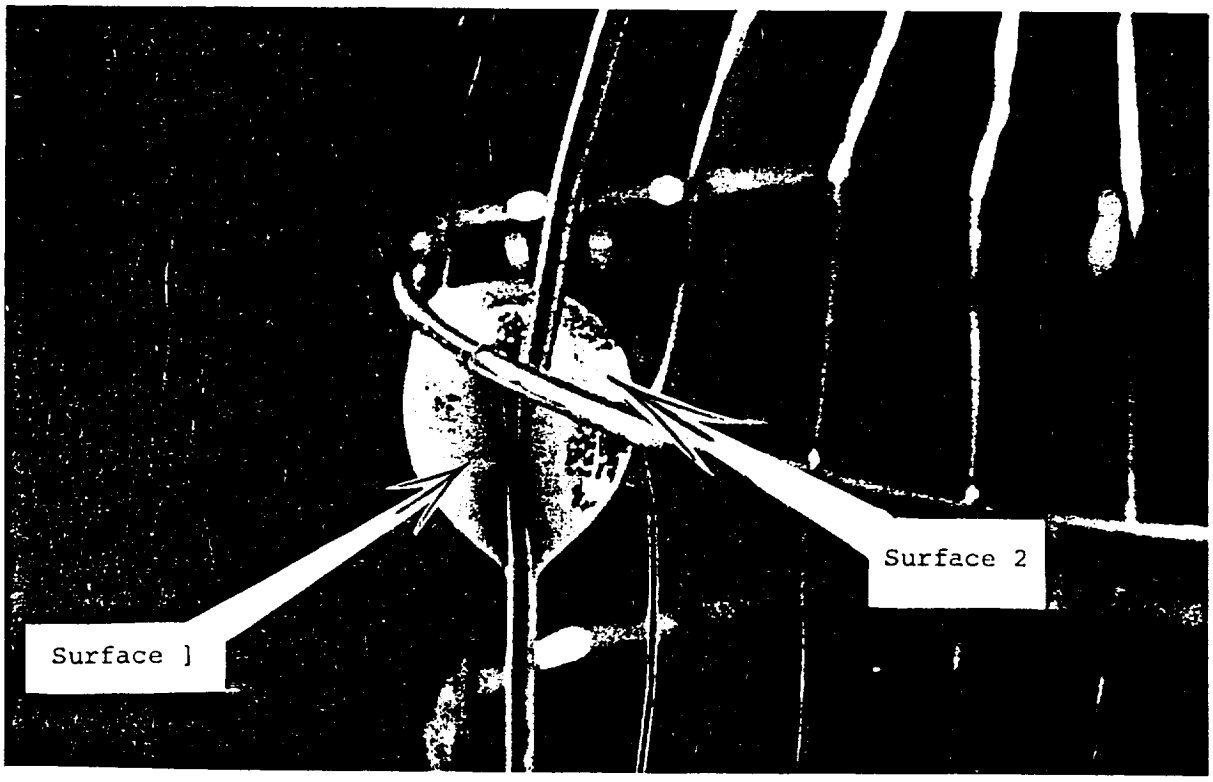

Figure 11. Flight sample 2 (35.7 CaO-39.3 Ga_2O_3-25 SiO_2, mol%, hot pressed) stuck to platinum wire cage while levitated and melted in space (MEA/A-2). The sample crystallized where it contacted the cage wires.

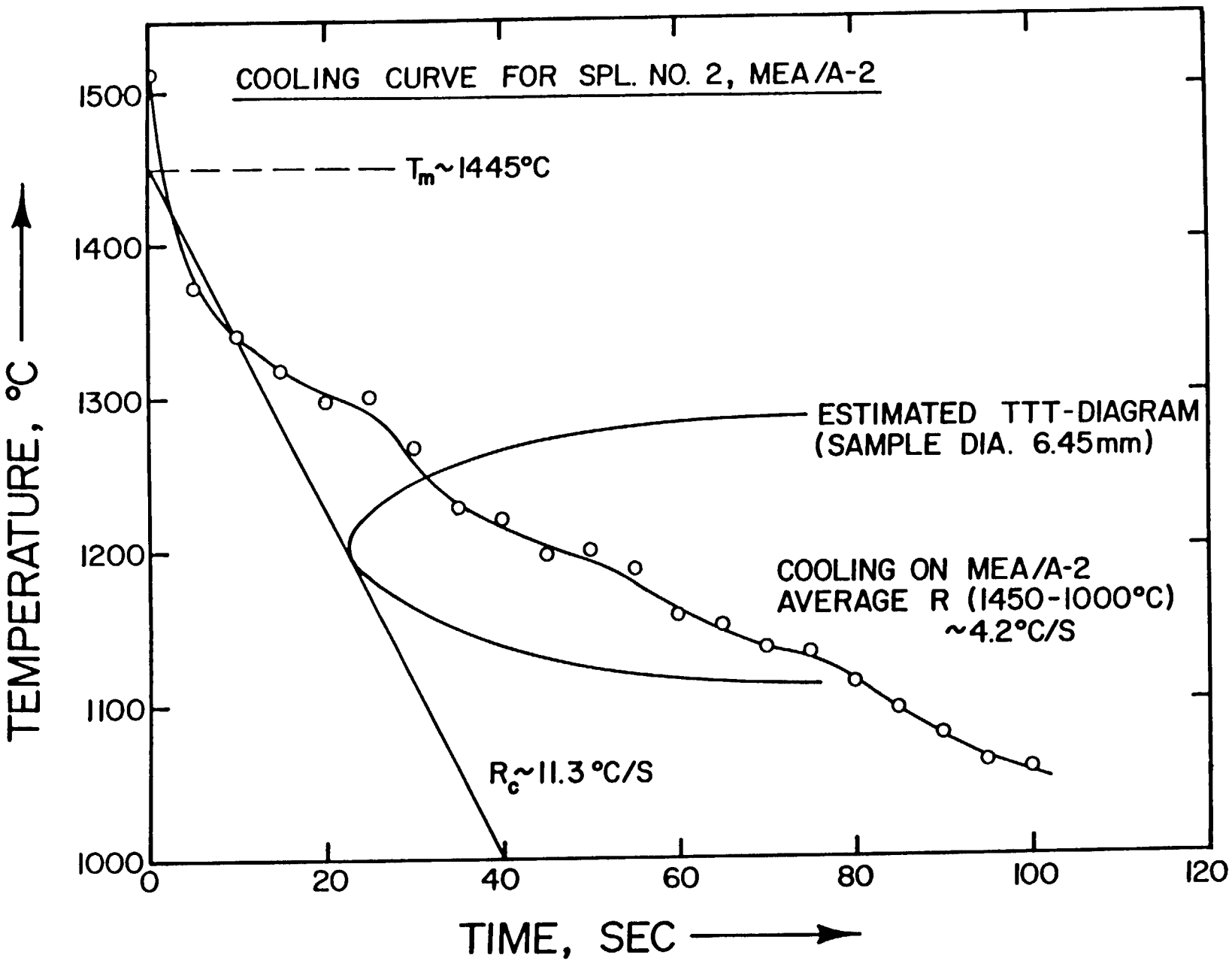

Figure 25. The estimated TTT diagram for the 35.7 CaO-39.3 Ga_2O_3-25 SiO_2, mol%, composition for sample diameter ~6.5 mm and the cooling rate measured for sample 2 in MEA/A-2.

TABLE XII. Results for the hot pressed calcia-gallia-silica sample (#2) processed on MEA/A-2 experiment.

1. The sample escaped the acoustic energy well and stuck to the cage wires while levitated in the SAAL in space. Escape was clearly associated with the opening of the SAAL cooling shroud gate.

2. 70 to 80% of the sample was glassy. Partial crystallization (≈20%) was observed where the melt touched the platinum wires of the cage. Examination by SEM and EDAX showed that the crystalline phases were Ga_2O_3 and $Ca_2Ga_2SiO_7$.

3. Comparison of the critical cooling rate for glass formation of this glass on earth with the cooling rate used in the flight experiment, showed a 2 to 3 times enhancement of glass formation for this composition melted in space.

III. The Other Side of The Coin

A. Since glass-forming is a contest between cooling and crystallization, why not rapid cooling (on earth)?

B. It has been employed:

Technique	Typical Compositions
Liquid Quenching	TiO_2 + RO, Al_2O_3 + Gd_2O_3
Splat Cooling	Metal Alloy Glasses; L_2O_3-Al_2O_3
	L = La, Nd, Gd, Er, . . .
Cold Rollers	R_2O-TaO_3; R_2O-NbO_3
	R = Li, Na, K
Spinning Wheel	Metal Alloy Glasses
Laser Film Melting	Ge, Si, Mixed Chalcogenides

C. Other Novel-"Non-Melting" Techniques

1. Sol-gel

2. Gas Phase Deposition Methods

   (a) Evaporation

   (b) Sputtering

   (c) Reactive Sputtering

   (d) CVD

   etc.

3. Solid State Methods

   (a) High Pressure

   (b) Shock Tube

   (c) Solid State RXS

Ask again: Why do containerless processing of glasses?

Let us primarily focus on rapid cooling techniques--

A. Limited to ribbon, fragments, etc. - no bulk material

B. No contact could be of greater importance than splat cooling

e.g. Li_2O-B_2O_3 glass

C. Glass properties can depend on sensitively upon history; i.e. rapidly cooled glass will behave differently from ordinary glass. Also, more generally properties can depend on how glass is made.

e.g. Weeks et. al. - electrical conductivity of GeO_2 can depend upon

melting T

Galeener et. al. - Raman scattering function of Fictive Temperature

Hench et. al. - Sol-gel SiO_2; different ρ, uv-cutoff

D. Difference in Properties Imply Structural Differences

i.e. One is preparing different materials

IV. Two Examples of Interesting Glass Systems

A. $CaO\text{-}Al_2O_3$ System

1. Why interesting?

Technological: (a) very good IR transmission for oxide glass

cutoff ~ 6 μ

(b) High Young's Modulus - use for reinforcement in structural composites

Scientific: (a) Unusual glass-forming system

(b) Questions regarding structure

2. Glass-Forming Ability

(a) By normal coating: Narrow region around 65 mole % CaO

(b) By melt quenching: CaO (59-70%)

(c) By Splat Cooling (Slivers): CaO (19-81%) (super-quench)

3. Structure/Properties

(a) Structural studies mainly by Raman

(b) Thermal expansion and index measurements of "stable" glasses

(c) Studies have been performed on ternaries (SiO_2 or CaF_2 additions)

4. Future Work

Ground based:

(a) Alternate preparation methods

(b) Study of crystallization behavior

(c) More detailed structural studies (NMR)

Flight opportunities:

(a) Glass-forming ability

(b) Bulk samples for structural studies and mechanical property measurements

(c) Optical performance

B. Li_2O-B_2O_3 System

1. Why interesting?

(a) Base composition for wide variety of FIC glasses

(b) Borate structures more varied and complex than silicates due to 3 or 4 fold coordination of B

(c) LB_2 first simple borate which seems to exhibit homogeneous crystal nucleation

2. Glass-forming Ability

$$XLi_2O \cdot (1\text{-}x)B_2O_3$$

(a) Bulk samples ($0 \leq x \leq .42$) (but special care for larger x)

(b) Splat cooling (virtually no advantage)

(c) Quench crucible + dry N_2 ($0 \leq x \leq .6$)

(d) Roller method ($0 \leq x \leq .7$)

3. Structure/Properties

   (a) Activation energies for DC conductivity appear to decrease monotonically with increasing Li_2O content

   (b) High Li_2O content glasses appear to devitrify via surface mechanism - avoidance of contact with substrate important

   (c) Detailed IR, Raman and NMR studies have been performed as function of composition

4. Future Work

   Ground based:
   (a) Crystallization studies as a function of composition
   (b) Effects of quench rate on structure and properties (electrical)
   (c) Effect of nucleation upon conductivity

   Flight opportunities:
   (a) Glass-forming ability
   (b) Crystallization behavior
   (c) Structure/properties of slowly cooled glasses

V. Summary

1. We have focused on one aspect of containerless glass experimentation: glass-forming ability

2. We have argued that although containerless processing will abet glass formation, other ground-based methods can do the "job" better

3. However, these methods have limitations re: sample dimensions and concomitant ability to make property measurements (e.g. fracture)

4. Most significantly, perhaps, is the observation that glass properties are a function of preparation procedure

5. Thus, in conclusion, it seems as though there still is an argument for use of containerless processing for glass-forming

ACKNOWLEDGEMENTS

The author wishes to express his gratitude to the Jet Propulsion Laboratory and the Division of Microgravity Sciences and Applications of NASA for the financial support of our investigations concerning containerless processing of glasses.

N91-21341

CONTAINERLESS EXPERIMENTS IN FLUID PHYSICS IN MICROGRAVITY

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CALIFORNIA INSTITUTE OF TECHNOLOGY

INTRODUCTION

The basic favorable conditions for low gravity studies of fluid physics are the removal of the overwhelming influence of sedimentation and of natural convective effects. If these conditions are combined with the elimination of the unwanted interaction of free-liquid surfaces with solids, such as those found in the sample containers, then experimental studies of unfettered capillarity-dominated phenomena become possible. The removal of the complicated and material-specific boundary conditions made possible by the containerless environment also renders the analysis of these phenomena more tractable and, therefore, allows the verification of theories and models that have long been the only means to advance our understanding of these areas of fluid physics.

This more beneficial environment will, however, not guarantee the successful performance of rigorous experiments if the appropriate instrumentation and physical parameter measurement and control are not available to the microgravity experimenter. Because the magnitudes of the forces and perturbations are small, tighter control of environmental influences such as the residual acceleration background, the thermal distribution field, the material species concentration distribution, and other extraneous effects introduced by the noncontact sample positioning fields must be exercised. On the other hand, one of the major advantages of the microgravity environment is the inherent ability to reduce the effects of the positioning fields to a very low level, and thus to significantly decrease their perturbing effects. Novel non-invasive sample and environmental properties measurement techniques must also be developed and implemented specifically for the on-orbit environment, and they should not add further perturbation to the sample.

The areas of interest in microgravity fluid physics cover multiple disciplines and are relevant to a variety of environmental conditions, from the more benign, ambient manned surroundings to conditions of ultra-high temperature and high pressure or vacuum. Basic study of the behavior of freely suspended liquids in gas or vacuum environments that are free from the Earth's gravitational field is a new area of investigation, and the methodology involved has not yet been completely mastered and understood. It, therefore, would be logical to develop the experiment concepts and instrumentation to carry out studies in more controlled and well-characterized conditions prior to attempts to access more restricted and adverse environments at ultra-high temperature. The ambient environment studies will serve to verify models that could then be applied in any environment. Of course different instrumentation and measurement techniques will be required to accommodate different environmental conditions, but the basic physical phenomena dominated by thermocapillary interactions will be described by the same models.

Some of the possible areas of research in microgravity containerless fluid physics studies will be suggested below. The current work carried out by researchers both in 1 G and in low gravity will then be described. Applications of the results of these low gravity studies to the basic understanding of the dynamics of free-liquid surfaces, of the transport processes involved in phase transitions, and of the properties of metastable liquids will also be discussed.

POSSIBLE AREAS OF INVESTIGATION

The fundamental physical system available for investigation is a free liquid in spherical or near-spherical shape under the predominant influence of capillary forces. The thermal environment is ideally uniform but can be varied in a controlled manner to create surface tension gradients or to uniformally elevate the sample temperature. Noncontact positioning techniques using acoustic, electrostatic, electromagnetic, or aerodynamic forces allow the controlled manipulation of the specimen: static deformation, solid body and differential rotation, driven and free shape oscillations can all be induced as desired. The remote positioning of the specimen also allows it to be largely decoupled from residual acceleration transients specific to the microgravity environment. A variety of processing environments that are specific to the desired investigation can be accommodated with controlled gas additions or partial evacuation or variation of humidity levels. The totally free-liquid surface can thus respond to any external stimulus in a manner characteristic of its surface properties: the natural response of a fluid sphere is then accessible to experimental examination, and the effects of controlled addition of surface adulterants can be specifically measured. In addition, partial contact with specific, well-characterized solid surfaces can be achieved to provide specific measurement of liquid-solid interaction in the absence of gravity.

Containerless experimentation is an additional opportunity for scientific study and offers a new approach to heterogeneous nucleation investigation by allowing the virtual removal of external contact sources that could serve as a basis for initiating an unwanted phase transition. This additional delay of the nucleation of a new phase will allow access to the metastable regions of the liquid state and the subsequent experimental measurements of their physical properties.

A. The Dynamics of Oscillating and Rotating Free Drops

Perfect sphericity of a liquid sample can only be achieved through the undisturbed influence of surface tension; this condition can be very closely approximated if the Earth's gravitational field effects are drastically reduced and if the residual influence of sample positioning forces is negligible. In practice, noncontacting stress fields are needed to probe the response of the drop-to-perturbation and to manipulate it. The vibrational dynamics of free drops can then be studied in both the linear small amplitude region as well as in the nonlinear regime. The transition from the ordered to the chaotic behavior of this simple mechanical system is thus accessible to controlled experimentation. The static and dynamics of uniformally and differentially rotating drops can be investigated in detail.

In addition to simple drops, compound liquid-liquid and gas-liquid combinations are of fundamental interest both in terms of basic physical reasons as well as for the various technological applications based on such systems. The removal of gravitational body forces allows experimental access to ideal geometries with perfect symmetry and concentricity amenable to theoretical analysis.

The behavior of both Newtonian and non-Newtonian liquids can be investigated using the same experimental capabilities at room temperature or at slightly elevated temperature. Physical parameters such as surface tension and density can easily be determined if the appropriate controls are provided.

Current ground-based and flight investigations are being carried out both theoretically and experimentally. Some of the areas covered are listed below.

* R. Brown at MIT has been analytically and numerically analyzing the static and dynamic shapes of free drops and has predicted the stability limits of the equilibrium shapes of rotating drops in gyrostatic equilibrium (1). In addition, the nonlinear large amplitude shape oscillation regime

has also been extensively investigated for both electrically charged and uncharged drops (2). Nonlinear characteristics that are indicative of chaotic behavior in the shape oscillations of free drops have been uncovered (3).

* Experimental investigations have been carried out at the Jet Propulsion Laboratory, Yale, and, subsequently, at Vanderbilt University (4–6). Ground-based experiments in immiscible liquid media using ultrasonic radiation pressure have investigated the linear and nonlinear regimes of free drop shape oscillations. The rotational behavior of simple and compound levitated drops are under current scrutiny in preparation to a space flight experiment. T. Wang performed microgravity experiments during the flight of Spacelab 3 in 1985. In addition to demonstrating the viability of acoustic containerless positioning techniques in space, he has obtained intriguing results on the shapes of rotating drops that have raised further questions about the influence of dynamic phenomena on these shapes (7). A refinement and extension of these experiments will be carried out during the USML-1 Spacelab flight in 1992.

B. Mass and Heat Transport Phenomena

The direct three-dimensional interfacing of a liquid surface to a gaseous or vacuum environment also allows the simplification of analytical studies of heat and mass transfer problems in the spherical or near-spherical geometry. Control of the liquid surface properties, the composition, pressure, and temperature of the environment coupled with the drastic reduction of uncontrolled natural convection will allow experimental access to a much wider parameter space. Small transfer coefficients can be measured, and the effect of forced convection can be experimentally decoupled.

Ground-based experiments using acoustically levitated droplets have been carried out at the Naval Research Laboratory, Battelle (Frankfurt) laboratory, and at the Jet Propulsion Laboratory to measure the liquid evaporation rate (8,9). The interference of the high intensity acoustic field and of the resulting forced convective flows cannot be eliminated in experiments carried out in 1 G. These investigations have concentrated, therefore, on the overall mass transfer rate or on the technological feasibility of accurately controlling the levitated drop volume. A team of researchers from the NASA Lewis Research Center and the University of Michigan are currently investigating the feasibility of a controlled drop evaporation experiment paying particular attention to the induced convective flow inside the freely suspended drop in microgravity (10).

C. Surface Rheology and Thermocapillary Phenomena

Containerless experimentation should allow the control of the liquid free surface composition either by postponing the contamination onset or by deliberately altering its properties by the addition of various surfactant. Time-dependent or steady-state mechanical and transport properties can then be experimentally investigated through various noncontact techniques. The verification of simpler theoretical models dealing with the properties of surface elasticity and viscosity will become possible with microgravity experiments.

Non-uniform thermal distributions on the drop surface can be generated, and the resulting thermocapillary phenomena can be observed and measured inside transparent droplets. Marangoni-flow phenomena can then be investigated in the absence of other body forces or external convective flows in microgravity.

* Current and past ground-based studies of the surfactant effects on the surface tension and viscosity of droplets levitated in an immiscible liquid medium have been carried out at Yale

University. An anomalous time dependence of both these parameters has been discovered. Preparations for a flight experiment in 1992 are being made (11).

* S. Subramanian at Clarkson University has carried out both theoretical and experimental studies of the thermocapillary flow generated due to a surface tension gradient on a free drop surface. Applications to the control of bubble motion within a melt in a containerless situation and in low gravity have been a driving motivation (12).

D. Measurement of Thermophysical Properties

Nonperturbing, remote measurement techniques based on containerless positioning techniques as well as other optical or spectroscopic methods should be made available in order to fully exploit the possibilities raised by the microgravity containerless experimentation program. Thermophysical properties currently being emphasized are density, surface tension, viscosity, specific heat, thermal diffusivity, and absolute temperature. Room temperature and elevated temperature measurements are required depending upon the materials or processes of interest.

* Investigators at the Jet Propulsion Laboratory have developed and validated techniques to measure the surface tension, density, viscosity, and index of refraction of levitated liquid specimens (13). Both room temperature and higher temperature experiments in microgravity are planned for the USML-1 flight to determine the accuracy of these methods in space.

* Other researchers at MIT and in Europe are planning to measure surface tension and viscosity of high-temperature metallic melts (between 1,000 and 1,700°C) using the same techniques (14). Flight experiments are planned for the IML-2 mission scheduled for 1993.

E. Undercooling, Nucleation, and Solidification Studies

The postponement of liquid to solid-phase transformation can be facilitated by containerless processing liquid samples. This postponement can easily be attributed to the removal of obvious heterogeneous nucleation of container surfaces. Although other factors such as suspended impurities and "dynamic" effects are still obstacles to the homogeneous nucleation limit, containerless experimentation widens the parameter space open to experimental investigation of the metastable (undercooled and superheated) liquid state. Controlled experiments in microgravity or even in 1 G will also allow detailed examination of the various factors responsible for the premature nucleation of the solid phase from the undercooled melt.

* Ground-based levitation undercooling of ordinary liquids, low melting metals and alloys have been obtained using ultrasonic and electromagnetic levitation (15,16), and drop tube studies have allowed quite significant undercooling of millimeter-size refractory metal alloys (17). Both 1 G and flight experiments are either being carried out or being planned for a future Spacelab flight of a containerless processing instrument.

* Containerless solidification from melts and from solution has been under scrutiny in ground-based laboratories for some time. High-temperature solidification velocities in metal alloys have been measured from electromagnetically levitated melts (18). Preliminary measurements of surface dendritic growth velocity at near-ambient temperature has been obtained for slightly undercooled Succinonitrile, and measurements at higher undercooling are being carried out at the present time (19). Flight experiments are also being planned for the IML-2 Spacelab mission by European investigators.

CONCLUSION

The physical phenomena associated with the behavior of liquid samples freely suspended in low gravity must be thoroughly understood prior to undertaking detailed scientific studies of the materials under scrutiny. The characteristics of molten specimens under the action of containerless positioning stresses must be identified and separated from the specific phenomena relating to the absence of an overwhelming gravitational field. The strategy designed to optimize the scientific return of reliable experimental data from infrequent microgravity investigations should include the gradual and logical phasing of more sophisticated studies building on the accumulated results from previous flight experiments. Lower temperature fluid physics experiments using model materials can provide a great deal of information that can be useful in analyzing the behavior of high temperature melts. The phasing of the experimental capabilities should, therefore, also include a gradual build-up of more intricate and specialized diagnostic instrumentation and environmental control and monitoring capabilities. Basic physical investigations should also be distinguished from specific materials technology issues. The latter investigations require very specific high temperature (and high vacuum) devices that must be thoroughly mastered on the ground prior to implementing them in space.

ACKNOWLEDGEMENTS

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Figure 1

Comparison between theoretical predictions on the nonlinear characteristics of the fundamental mode of uncharged simple drop shape oscillations. Numerical results predict an increasing percentage of the oscillation period spent in the prolate shape as the oscillation amplitude increases. This was confirmed by experimental results. Theoretical results predicting of a soft nonlinearity for large amplitude drop shape oscillations are also in agreement with experimental data.
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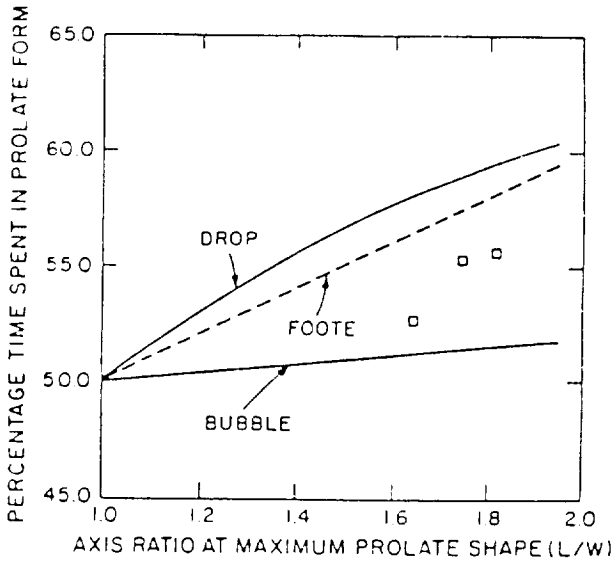

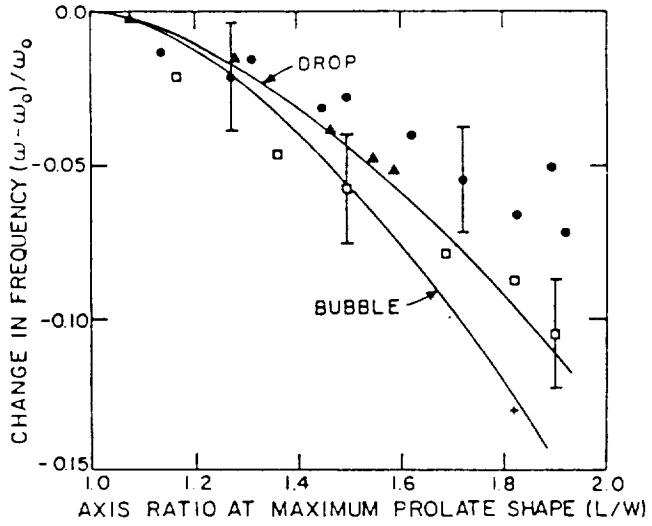

Figure 2
Results from space experiment carried out during the Spacelab 3 flight. The observed equilibrium shape of an acoustically rotated freely suspended drop of an aqueous solution of glycerin deviates from the predicted behavior at the point of transition between the axisymmetric and two-lobed shape (bifurcation). Dynamic, surfactant, and differential flows could be at the origin of this intriguing result.
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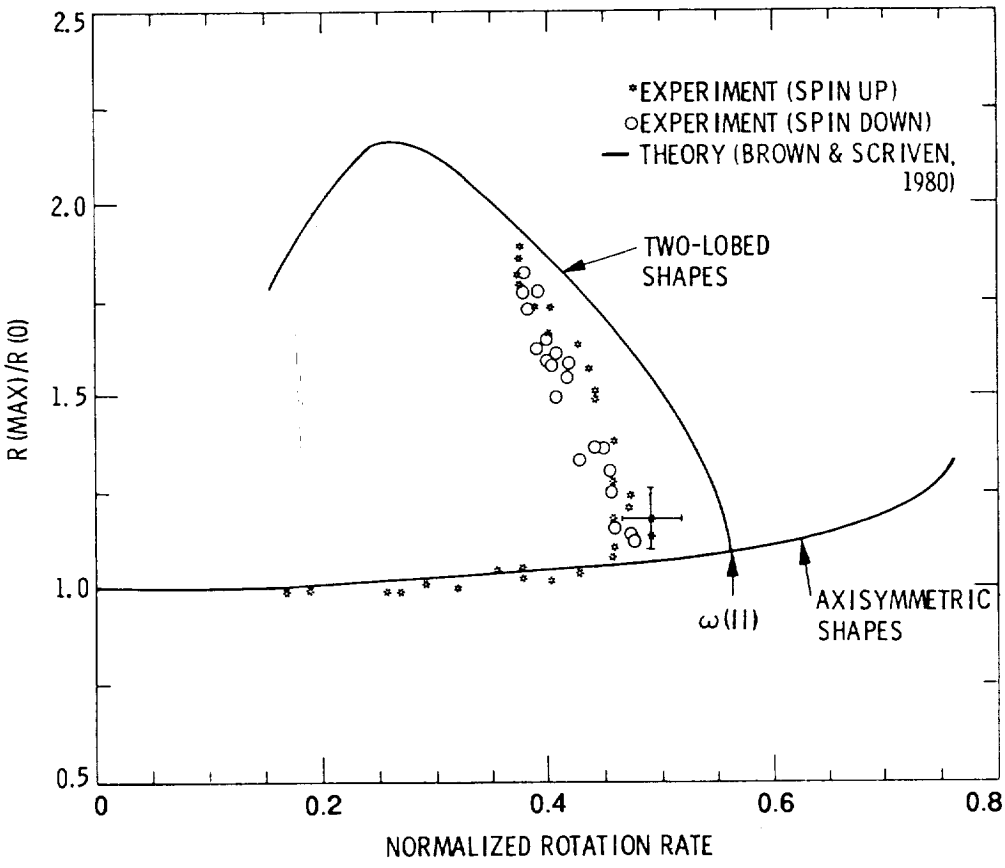

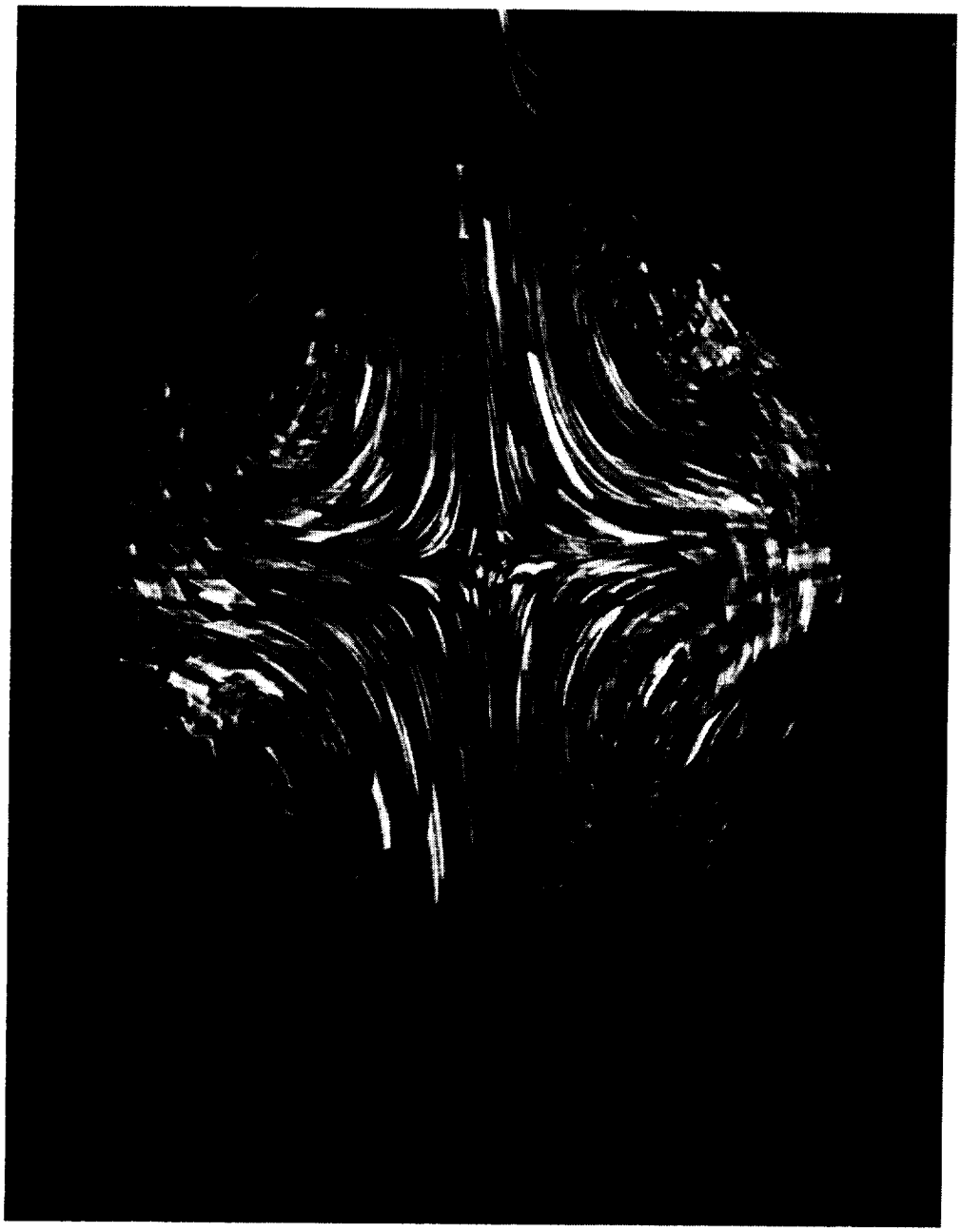

Figure 3
Tracer particles-based visualization of the internal flow of a freely suspended drop undergoing large amplitude drop shape oscillations. Steady convective flows have been observed in addition to the oscillatory motion.
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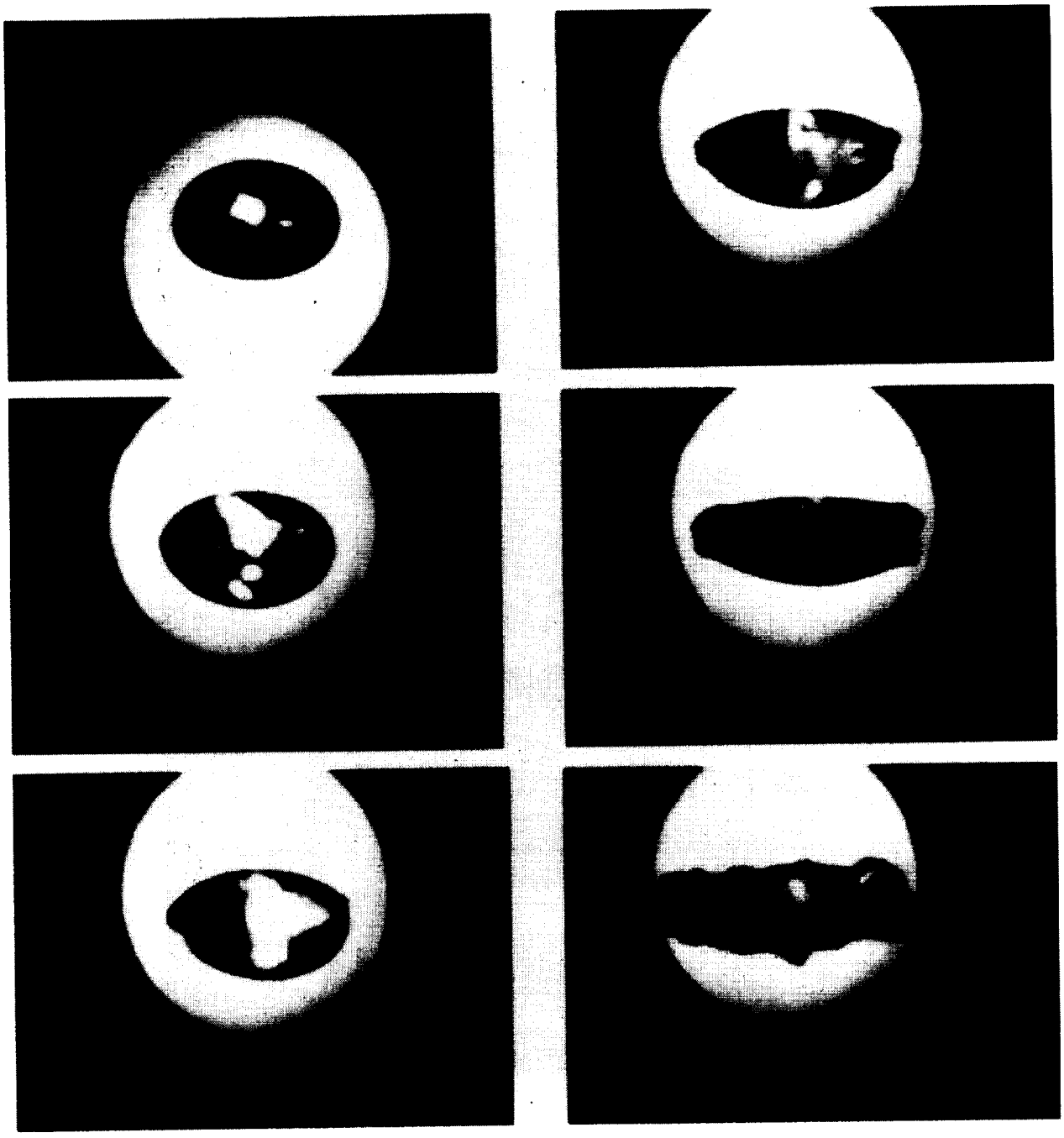

Figure 4
Containerless crystallization of levitated initially undercooled O-Terphenyl sample in 1 G.
Data from E.H. Trinh, Jet Propulsion Laboratory

Figure 5
Hypothesis regarding evaporation mechanism for a freely suspended liquid drop under diffusion-controlled regime and under convective flow.
From A.T. Chai and J.C. Duh, ESA publication SP 295, 492 (1990)

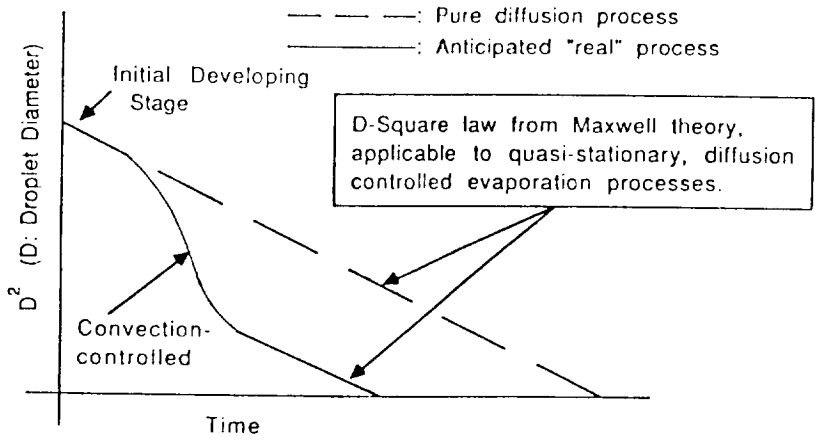

Figure 6
Theoretically predicted Marangoni flow patterns in a freely suspended spherical drop under a non-axisymmetric themal gradient.
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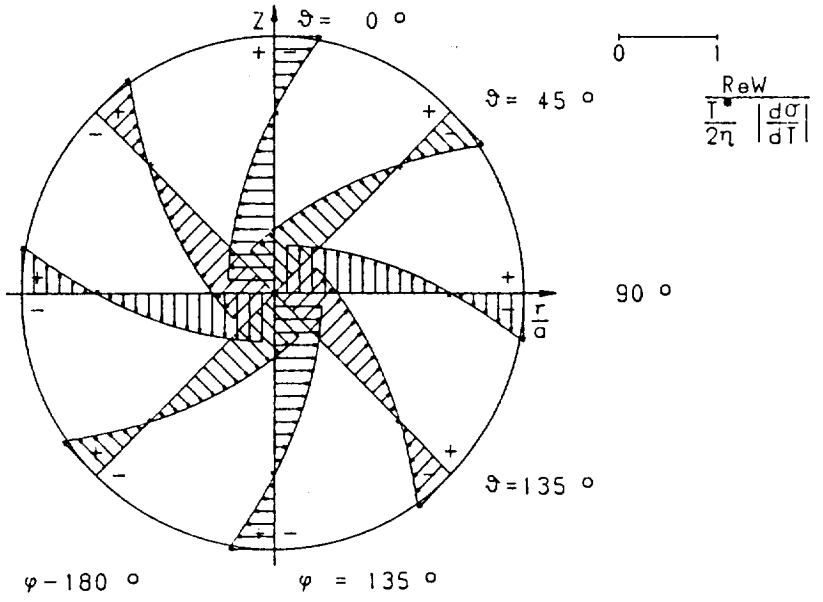

Fig. 2(g)

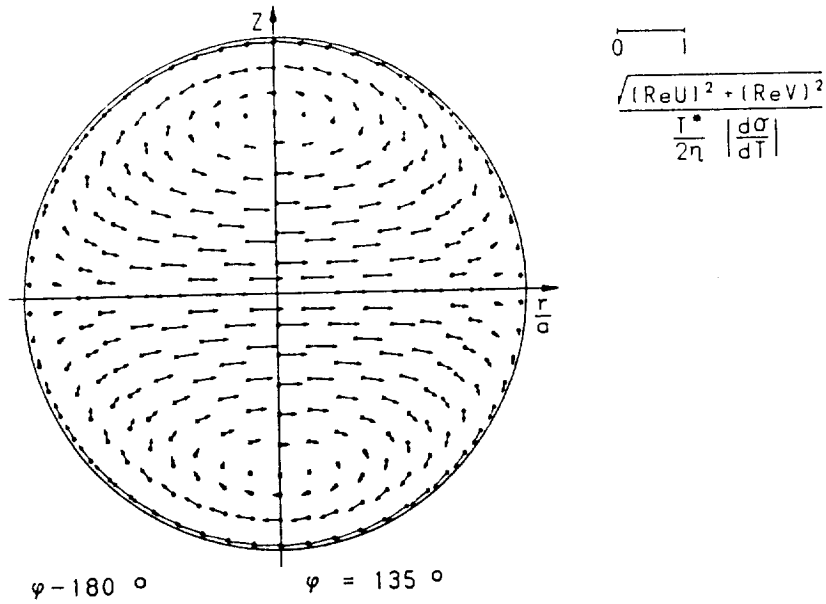

Figure 7
Equilibrium shapes of acoustically rotated charged drops under levitation in an electrostatic field.
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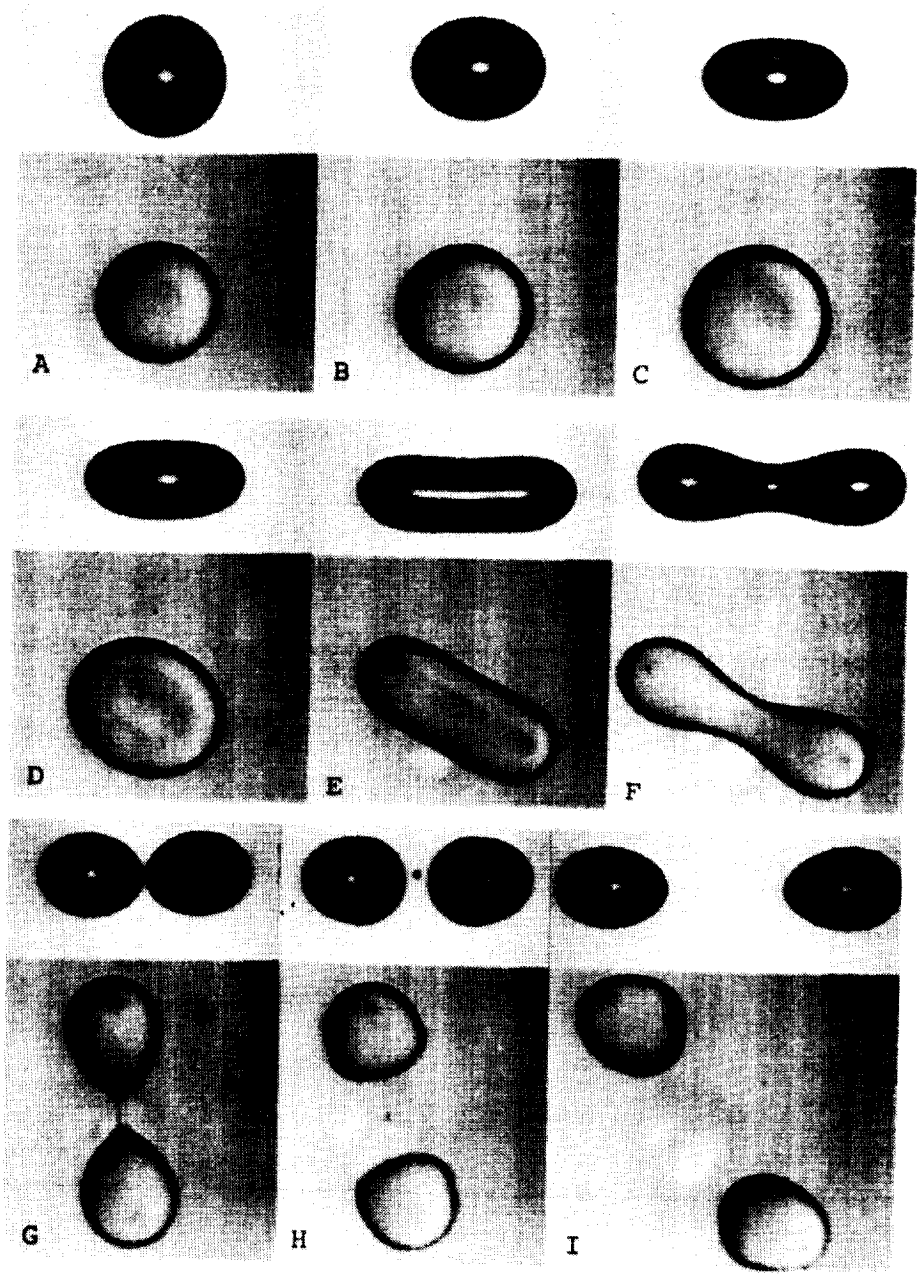

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Figure 8
Time history of the frequency and damping constant for a Hexane drop of approximately 1 mm in diameter in water at 22 C. The solid curves are theoretical predictions.
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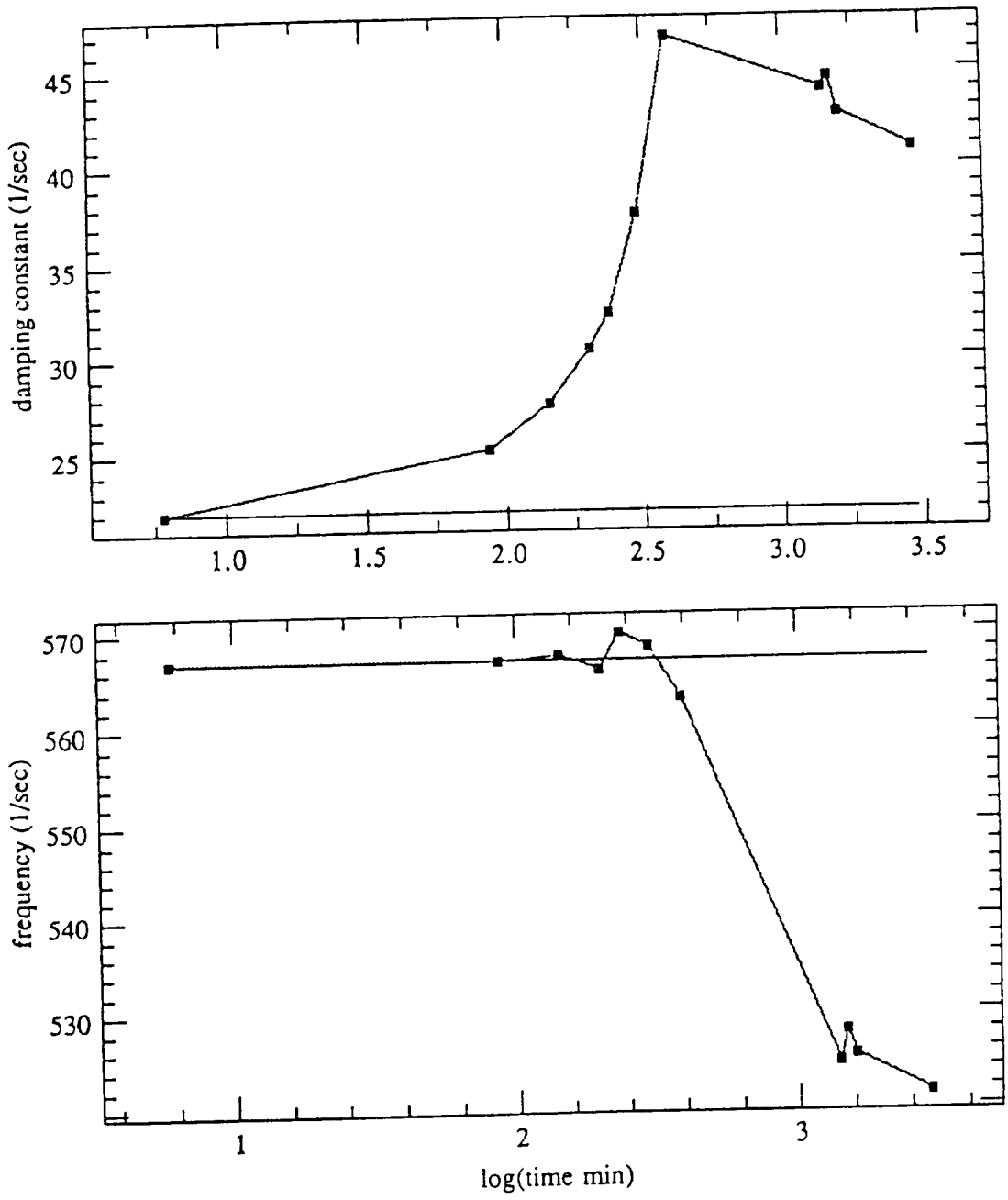

SPLINTER SESSION 1

Technical Papers

N91-21342

Real-Time Measurement of Materials Properties at High Temperatures by Laser Produced Plasmas

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Determination of elemental composition and thermophysical properties of materials at high temperatures, as visualized in the context of containerless materials processing in a microgravity environment, presents a variety of unusual requirements owing to the thermal hazards and interferences from electromagnetic control fields. In addition, such information is intended for process control applications and thus the measurements must be real time in nature. We will describe a new technique which we have developed for real time, in situ determination of the elemental composition of molten metallic alloys such as specialty steel. The technique is based on time-resolved spectroscopy of a laser produced plasma (LPP) plume resulting from the interaction of a giant laser pulse with a material target. The sensitivity and precision have been demonstrated to be comparable to, or better than, the conventional methods of analysis which are applicable only to post-mortem specimens sampled from a molten metal pool. The LPP technique can be applied widely to other materials composition analysis applications.

The LPP technique is extremely information rich and therefore provides opportunities for extracting other physical properties in addition to the materials composition. The case in point is that it is possible to determine thermophysical properties of the target materials at high temperatures by monitoring generation and transport of acoustic pulses as well as a number of other fluid-dynamic processes triggered by the LPP event. By manipulation of the scaling properties of the laser-matter interaction, many different kinds of flow events, ranging from shock waves to surface waves to flow induced instabilities, can be generated in a controllable manner. Time-resolved detection of these events can lead to such thermophysical quantities as volume and shear viscosities, thermal conductivity, specific heat, mass density and others.

A Summary of the Paper Presented at the Workshop on
Containerless Experimentation in Microgravity
Jet Propulsion Laboratory, Pasadena
17 - 19 January 1990

Real-Time Measurement of Materials Properties at High Temperatures by Laser Produced Plasmas

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9 March 1990

Determination of elemental composition and thermophysical properties of meterials at high temperatures, as visualized in the context of containerless experimentation in a microgravity environment, presents a variety of unusual requirements owing to the thermal hazards and interferences from electromagnetic control fields. In addition, such information is transient in the sense that the target specimen is undergoing a change of state either due to material processing or it is stimulated for certain responses, and thus the measurements must be real-time in nature. We describe a new technique developed for real-time, in-situ determination of the elemental composition of molten metallic alloys such as specialty steel. The technique is based on time-resolved spectroscopy of a laser produced plasma(LPP) plume resulting from the interaction of a giant laser pulse with a material target. The sensitivity and precision have been demonstrated to be comparable to, or better than, the conventional methods of analysis which are applicable only to post-mortem specimens sampled from a molten metal pool. The LPP technique can be applied widely to other materials composition analysis applications.[1-5]

The basic concept is to determine elemental composition by time- and space-resolved spectroscopic analysis of a plasma plume, as produced by a high power laser pulse incident on molten metal, under real-time in-situ conditions. Analysis of molten metal composition by laser produced plasmas(LPP), as conceptualized above, represents a creation of completely new technology from the very fundamental level physics of laser-matter interactions. As such, the research program has had to deal with the extraordinary challenge of making an efficient connection between the newly gained knowledge of the dynamical properties of laser produced plasmas and the task of design and construction of applications hardware for a physically brutal environment of metals production furnaces.

The question then boils down to how to produce the LPP plume

off the molten metal surface such that elemental abundances can be determined in a highly reproducible manner, uninfluenced by the variabilities in the thermodynamic and environmental variables. It is understood that each full anlysis is carried out with a single laser pulse. Each LPP plume must consist of elemental consitituents in exact proportion to the elemental composition in the condensed phase of the target material. This has required extensive experimental and theoretical studies on laser-matter interaction and the lessons from them have indeed been reduced to a rule of thumb.

The LPP analysis concept has been implemented in the form of a prototype sensor-probe.

In order to make a connection between the new LPP methodology and the long-standing analysis establishment, we have first directed attention to analysis of solid alloy specimens. The elemental composition data from the LPP method have been successfully compared with the results for the same specimens analyzed by the conventional analytical techniques such as spark discharge emission spectroscopy and X-ray fluorescence.

Extensive studies have been made of the various issues of LPP analysis of molten metals using a number of different sources. Combining the experimental investigation with our own numerical code studies, we have developed a general picture of the differences between the response to a laser excitation when an alloy specimen is in a liquid form and that when in a solid form. We have also confirmed that all the requisite spectroscopic properties for elemental analysis exist of the LPP plume off the molten metal surface. The prototype LPP sensor-probe has been successfully demonstrated as a survivable real-time analysis tool under full exposure to a steelmaking furnace.

It is worth stating that the LPP method opens up new opportunities for materials analysis because of its very information-rich nature. It is possible to make a strong connection between the microscopic morphology of an alloy, as can be interrogated by the LPP mehtod, and its macroscopic performance properties, such as resistance against wear, corrosion or fracture. It is also possible to explore the interface properties of composites by the LPP method because, on one hand, an LPP event can lead to a thermal pulse, acoustic pulses or a shock wave, which can be used for diagnostics, and, on the other, the interfaces can influence the growth of the LPP plume.

From the standpoint of the containerless experimentation under microgravity, the LPP methodology can play many useful roles. The case in point is that it is possible to determine thermophysical properties of the target materials at high temperatures by monitoring generation and transport of acoustic pulses as well as a number of other fluid dynamic processes

triggered by the LPP event. By manipulation of the scaling properties of the laser-matter interaction, many different kinds of flow events, ranging from shock waves to surface waves to flow induced instabilities, can be generated in a controllable manner. These phenomena are in themselves of considerable interest in the microgravity context. In addition, time-resolved detection of these events can lead to such thermophysical quantities as

viscosity
thermal conductivity
specific heat
mass density
surface tension
latent heat of melting
critical properties
elastic properties and others.

Of particular interest is the fact that the LPP method can create a supercritical state of the target, which can be studied at the same time.

The LPP method can play a crucial role in unravelling the near-the-surface density profile and chemical reactions at high temperatures because of its intinsic ability to interogate sub-nanosecond and micron-size features.

On the other end of the possibilities, the LPP method can provide an extremely effective means for exacting manipulation of the target specimen for rotation and translation, without requiring any materials injection.

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N91-21343

THERMODYNAMIC PROPERTIES AND CRYSTALLIZATION KINETICS AT HIGH LIQUID UNDERCOOLING

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Abstract

The heat capacities of liquid and crystalline Au-Pb-Sb alloys in the glass-forming composition range were measured with droplet emulsion and bulk samples. Based on the measured C_p data, the entropy, enthalpy, and Gibbs free energy functions of the eutectic, solid mixture and undercooled liquid were determined as a function of undercooling and compared with theoretical predictions. The results indicate an isentropic temperature at 313 ± 5 K, which agrees well with experimental data for the glass transition. A kinetics analysis of the nucleation undercooling response suggests that the proper choice for the Gibbs free energy change during crystallization is most important in analyzing the nucleation kinetics. By classical nucleation theory, the prefactors obtained, based on a variety of theoretical predictions for the driving force, can differ by six orders of magnitude. If the nucleation rates are extrapolated to high undercooling, the extrapolations based on measured heat capacity data show agreement, whereas the predicted nucleation rates are inconsistent with results from drop tower experiments. The implications for μg experiments are discussed.

Introduction

In order to predict the crystalline or amorphous transformation products during solidification from an undercooled liquid, knowledge of the thermodynamic properties of the metastable liquid is necessary. In particular, the entropy and enthalpy differences between the crystalline and undercooled liquid phases are of interest and can be evaluated from available heat capacity data. Experimentally, deep undercooling levels of a liquid can be attained by slow cooling of a droplet emulsion [1]. Droplet samples were prepared by shearing a mixture of carrier fluid and liquid alloy (99.99% purity) under an inert atmosphere into an emulsion ($\sim 10^7$ droplets) with an average diameter of 10 μm, thus effectively isolating potential nucleation sites. Applying a relatively noncatalytic coating, crystallization can be prevented in a temperature range up to 0.3 to 0.4 T_m on a time scale long enough to allow measurements of thermophysical properties of the undercooled liquid. The emulsification approach allows glass formation in droplet samples during slow cooling (10–20 K/min) in a few alloy systems [1]. This method has been applied to Au-Pb-Sb alloys [2] with compositions close to the ternary eutectic and within the experimentally determined glass formation range [3].

The undercooling, heat capacity C_p, and reaction kinetics were measured with a well-calibrated differential scanning calorimeter, Perkin-Elmer DSC7, under computer control. For C_p measurements, three different scans are compared: (1) an empty pan of equivalent mass, (2) sapphire as standard for calibration, and (3) the sample itself. Bulk samples are used for C_p measurements of the stable liquid and crystalline phases. The C_p measurements of the undercooled liquid are based on a differential measurement of C_p by comparing one sample containing carrier fluid, liquid, and a small amount of X of solid with a second sample containing carrier fluid and the liquid completely crystallized [2]. From the knowledge of X (~10%), the specific heat of the crystalline phase C_p^x and the measured heat capacity difference ΔC_p, C_p of the undercooled liquid is obtained (within ± 5%) as $C_p^L = [\Delta C_p + (1 - X)C_p^x]/(1 - X)$.

For evaluation of the nucleation kinetics during crystallization from the highly undercooled liquid, the cooling rate has been varied between 0.16 and 5 K/s and is related to the measured nucleation temperatures taken at the onset of the nucleation exotherm. With the nucleation temperatures corrected for instrumental temperature lag, the reaction kinetics are analyzed in terms of classical nucleation theory. During continuous cooling at low rates, the onset of solidification occurring within the time t in a droplet of catalytic surface area a, the following relation holds [4]:

$$\ln t = - \ln A + B/\Delta G_v^2 T \qquad (1)$$

with the prefactor term $A = a\Omega_a/[K\eta(T)]$ and the interfacial energy related term $B = b\sigma_{xL}^3 f(\theta)/k_B$. Here, Ω_a is the prefactor of the nucleation frequency; K is a constant on the order of 100; the liquid shear viscosity η is given by $\eta(T) = 10^{-3.3}\exp[3.34\ T_E/(T - T_{go})]$ with T_{go} being the ideal glass transition temperature [5]; $b = 16\pi/3$ is a geometrical factor; $\sigma_{xL}f(\theta)$ is the crystal/liquid interfacial energy; and ΔG_v is the free energy difference per unit volume between the undercooled liquid and crystalline phase. The dependence of the cooling rate $\dot{T}$ on the undercooling level ΔT (temperature difference between the eutectic temperature T_E and nucleation temperature T_N) is measured by DSC with the time t corresponding to $\Delta T/\dot{T}$. This allows us to determine the operating nucleation kinetics in terms of A and B from the experimental data.

Results and Discussion

Using droplet samples, a maximum undercooling level of ~ 0.3 T_L (175 K) below the liquidus temperature T_L was achieved at a cooling rate of 0.33 K/s, resulting in a single, well-defined crystallization exotherm [2]. The composition closest to the ternary eutectic composition and still in the glass-forming range [3] was found as $Au_{53.2}Pb_{27.5}Sb_{19.3}$ with the eutectic temperature T_E at 523 K and T_L at 573 K. The heat of fusion ΔH_f of this alloy is ΔH_f = 8.25 kJmole^{-1} with a corresponding entropy of fusion ΔS_f = 15.78 JK^{-1}mole^{-1}.

The measured heat capacities of bulk and droplet samples are fitted numerically by the following equations: $C_p^L = -47.4 + 9.4\cdot10^{-2}\,T + 1.11\cdot10^{7}\,T^{-2}$ JK^{-1}mole^{-1} and $C_p^x = 29.1 + 5.9\cdot10^{-3}\,T$ JK^{-1}mole^{-1}, and are shown in Fig. 1. The heat capacity of the undercooled liquid exhibits a continuous rise of C_p with decreasing temperature. This effect is much more pronounced for glass-forming alloys like Au-Pb-Sb than for pure metals [6] and has important consequences for the thermodynamic properties of the undercooled liquid and evaluation of the crystallization kinetics.

From the C_p data, the thermodynamic properties of the undercooled liquid L and eutectic solid X can be derived below the eutectic temperature T_E. For the current purpose, the differences in entropy ΔS (integration over $\Delta C_p^L/T\ dT$), enthalpy ΔH (integration over $\Delta C_p^L\ dT$), and free energy ΔG (= $\Delta H - T\Delta S$) between undercooled liquid and crystalline solid are determined. The entropy difference is considered as one of the main parameters describing the ability of an alloy to form a glass, and is shown in Fig. 2. The entropy of the undercooled liquid decreases faster than the entropy of the stable crystalline phase when the temperature is reduced. If the entropy values are extrapolated beyond the experimentally determined range (indicated by the dashed line in Fig. 2) an isentropic temperature $T_{\Delta S = 0}$ is found at 313 ± 5 K (within the error range of the C_p measurement). Below this temperature the entropy of the undercooled liquid would become smaller than that of the crystal. This situation can be avoided

by massive freezing of the liquid in a glass (Kauzmann paradox [7]). Thus, the isentropic temperature $T_{\Delta S = 0}$ indicates the ultimate undercooling limit of a liquid in order to prevent an "entropy crisis," and corresponds to the ideal glass transition temperature T_{go}. The estimated temperature T_{go} agrees well with the experimentally observed glass transition temperature by heating an Au-Pb-Sb glass through T_g [2]. It corresponds to 0.6 T_E (the reduced-glass transition temperature) in comparison to 0.25 T_m for pure metals [8], thus characterizing the easy glass-forming ability of Au-Pb-Sb alloys.

The enthalpy difference ΔH is also included in Fig. 2. It is found that solidification under adiabatic conditions [9] becomes possible below T_h = 365 K. This temperature marks the start of the hypercooled regime, where the enthalpy of the undercooled liquid equals the enthalpy of the eutectic solid at the eutectic temperature and is only about 35 K below the average nucleation temperature in the droplet population at a cooling rate of 0.33 K/s.

In addition to the determination of the thermodynamic properties in the metastable regime, the droplet samples have also been used for determination of the crystallization kinetics. For these samples, the nucleation temperature decreased from 425.2 K to 419.9 K if the cooling rate was varied from 0.16 K/s to 5 K/s. The kinetics measurements are analyzed by plotting the measured response time t (= $\Delta T/\dot{T}$) versus $(\Delta G_v^2 T)^{-1}$ based on the experimentally determined heat capacity data. From classical nucleation theory, Eq. (1), A and B are then given as $A = 1.8 \cdot 10^{16}$ sec^{-1} and $B = 1.02 \cdot 10^8$ KJ^{-2}cm^{-6}.

An important parameter in the analysis of the kinetics of crystallization at high undercooling is the Gibbs free energy difference between metastable liquid and crystalline solid ΔG_V and its dependence on temperature. Because C_p is unknown in most cases, different models have been proposed for ΔG_V using such easily accessible data as the heat of fusion ΔH_f, the heat capacity difference ΔC_p^f at the eutectic temperature T_E, and the undercooling level $\Delta T = T_E - T$ below the eutectic temperature. Several approaches for crystallization of a single solid phase are given below, such as

$$\Delta G_V = \Delta H_f \Delta T/T_E \qquad (2a) \quad [10]$$

$$\Delta G_V = \Delta H_f \Delta T/T_E - [\Delta C_p^f (\Delta T)^2 (1 - \Delta T/6T)]/2T \qquad (2b) \quad [11]$$

$$\Delta G_V = \Delta H_f \Delta T \cdot 2T/[T_E(T_E + T)] \qquad (2c) \quad [12]$$

$$\Delta G_V = \Delta H_f \Delta T \cdot T/(T_E)^2 \qquad (2d) \quad [13]$$

Figure 3 represents different approximations proposed for ΔG_V including the experimentally derived values. It is seen that at low undercooling, all formulae predict basically the same ΔG_V in excellent agreement with the experimental curve. At modest undercooling, the best correlation between experimental data and the proposed models over the measured temperature range is obtained with Eq. (2b) [11] and Eq. (2c) [12]. As has been found earlier, Turnbull's approximation, Eq. (2a), gives an upper limit for ΔG_V and is close to values obtained for pure metals [6], whereas Hoffman's expression underestimates ΔG_V in the case of glass-forming metallic alloys. In addition, the Kauzmann temperatures $T_{\Delta S = 0}$, where $-\partial \Delta G_V/\partial T$ equals 0, are indicated in Fig. 3 and summarized in Table I. They are all lower than the Kauzmann temperature extrapolated from the measured C_p data and differ considerably for the different models ranging from $-\infty$ Eq. (2a) [10] to 261 K Eq. (2d) [13].

One consequence of the correct evaluation of ΔG_V relates to the proper interpretation of crystallization kinetics [4]. The measured prefactor term A is in good agreement with the classical nucleation theory for heterogeneous nucleation ($\Omega_a = \sim 10^{23}$ cm^{-3}sec^{-1} [5]) if only a small portion (10^{-3}) of the surface area presents an active catalytic site for crystallization. Using the same type of analysis as outlined above for the experimentally derived nucleation data and

basing ΔG_V on Eqs. (2a) to (2d) would lead to a large difference in the prefactor term A in the nucleation rate expression as given in Table I. Whereas ΔG_V differs only about 20% at 420 K for the different models, the derived constants A can vary by six orders of magnitude ranging from $A = 2.4 \cdot 10^{13}$ sec^{-1}, Eq. (2a), to $1.1 \cdot 10^{19}$ sec^{-1}, Eq. (2d), in comparison to $1.8 \cdot 10^{16}$ sec^{-1} for the experimentally derived value. Furthermore, the slope B shows only a small dependence on the model with a variation of less than 10%.

Based upon the factors A and B given in Table I, it is possible to develop the transformation diagrams that are described by Eq. (1). Especially for glass-forming alloys, the temperature dependence of the prefactor term A, i.e., the viscosity change of the undercooled liquid as a function of undercooling, should be taken into account. This can be done by appropriately correcting the factor A, which was determined in a narrow range of the nucleation temperatures T_N as $A(T) = A(T_N)\eta(T_N)/\eta(T)$. Then the steady-state transformation diagrams are calculated using the modified prefactors A(T), and the corresponding extrapolated Kauzmann temperatures $T_{\Delta S = 0}$ taken as critical temperature for the viscosity T_{go}.

Table I: Analysis of nucleation kinetics according to classical nucleation theory Eq. (1) and different models for the Gibbs free energy difference ΔG_V. The Kauzmann temperature $T_{\Delta S = 0}$ is given for the different ΔG_V models.

ΔG_V (J/cm^3)	$T_{\Delta S = 0}$ (K)	A (sec^{-1})	B (K J^{-2} cm^{-6})
experimental	313	$1.8 \cdot 10^{16}$	$1.02 \cdot 10^{8}$
(2a) Ref. 10	$-\infty$	$2.4 \cdot 10^{13}$	$1.03 \cdot 10^{8}$
(2b) Ref. 11	209	$2.1 \cdot 10^{15}$	$0.99 \cdot 10^{8}$
(2c) Ref. 12	216	$1.2 \cdot 10^{16}$	$0.97 \cdot 10^{8}$
(2d) Ref. 13	261	$1.1 \cdot 10^{19}$	$0.93 \cdot 10^{8}$

Whereas a linear approximation for ΔG_V is not applicable for glass-forming alloys ($T_{go} = -\infty$), all other approximations [11–13] depict the "nose" of the transformation curve at considerably shorter times (between $3 \cdot 10^{-4}$/s [13] and $5 \cdot 10^{-6}$/s [11]). According to this analysis, much higher cooling rates would be required for curves (b), (c), and (d), in order to avoid crystallization than the experimentally determined critical cooling rate of ~ 10^3 K/s using drop tube processing [2]. The only curve that is consistent with the experimental observation that crystallization is avoided at cooling rates of ~10^3 K/s is represented by the curve (exp) that is based on the measured C_p data. Consequently, the ΔG_V models as given by Eqs. (2a) to (2d) are inappropriate for analysis of the crystallization kinetics of glass-forming Au-Pb-Sb alloys at high undercooling.

Moreover, the effects of non-steady-state nucleation have been considered [14]. Such transient effects are becoming important at temperatures below 400 K for this alloy system with the "noses" in Fig. 3 only slightly shifted to shorter times. These effects are negligible above 400 K and, therefore, do not affect the critical cooling rate required for glass formation in Au-Pb-Sb. Nevertheless, due to the Stokes–Einstein relation commonly used for non-steady-state nucleation analysis, the viscosity and transient nucleation effects are strongly coupled. Because diffusivity data are unknown in the highly undercooled state, it is very difficult to separate the two effects at high undercooling. Because of the lack of an appropriate theory for the glass transition itself and the lack of knowledge of many important dynamical properties of highly undercooled liquids including the viscosity η, diffusion coefficients D, and transient times τ, the correct description of the crystallization kinetics or glass formation in the highly undercooled regime cannot be simply reduced to existing models for the free energy difference ΔG_V. It is shown here that the best extrapolation possible is when experimental data for the

nucleation rates are available based on crystallization kinetics and thermodynamic data for the free energy functions measured at low and modest undercooling.

Implications for Microgravity

The described emulsification method is applicable for metals and alloys with low melting points and gives insight into the fundamentals of crystallization and glass formation. Due to the lack of appropriate carrier fluids, this method is not generally applicable to metals or alloys with high melting points that are of more substantial technological interest. Therefore, we propose to use containerless processing methods, such as levitation melting under UHV conditions, preferably in a μg environment, to study the thermophysical properties of the undercooled liquid state of alloys with high T_m over sufficiently long time scales. It turns out in the above analysis that the most important parameter for a correct description of crystallization kinetics and glass formation is the heat capacity of the undercooled liquid which allows estimates of ΔG_V and T_{go} (the ideal glass transition temperature and critical temperature for the viscosity in the Vogel–Fulcher equation). A method has been proposed to measure C_p of the undercooled liquid state under μg by an a.c. pulse method described in detail elsewhere [15]. Together with the knowledge of additional thermophysical properties including the viscosity and diffusion coefficients, the above analysis can then be applied to a broader class of materials. Glass-forming alloys exhibit a more pronounced temperature dependence of C_p and η than pure metals and, therefore, appear to present the best candidates for thermophysical property measurements of the undercooled liquid state under μg conditions.

ACKNOWLEDGMENTS

The author would like to thank Prof. J. H. Perepezko for the initiation of this work and continuous encouragement, Prof. W. L. Johnson, Dr. M. C. Lee, Dr. K. Ohsaka and Dr. E. Trinh for fruitful discussions, and NASA for financial support (NAS 496954MG3203550).

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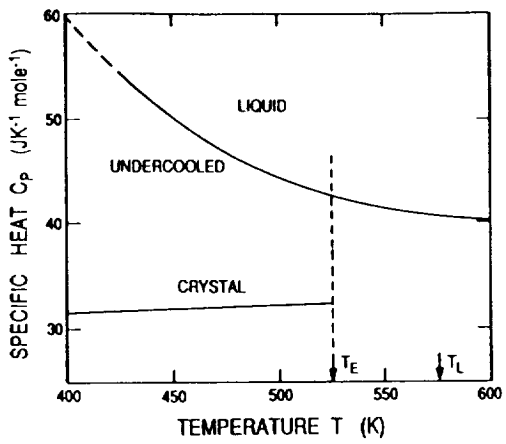

Fig. 1: Heat capacity data measured for liquid and crystalline $Au_{53.2}Pb_{27.5}Sb_{19.3}$ alloys.

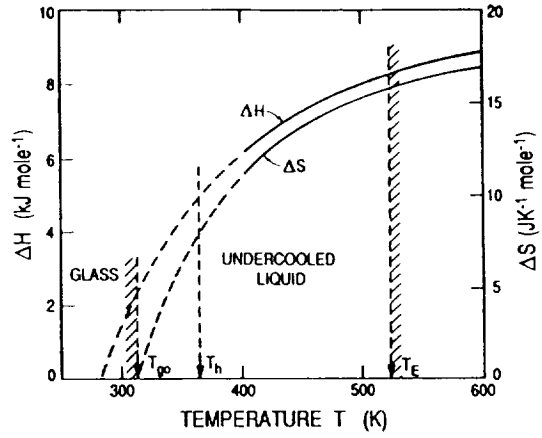

Fig. 2: The entropy and enthalpy differences ΔS and ΔH between undercooled liquid and crystalline eutectic solid based on measured (solid line) and extrapolated (dashed line) C_p data. The isentropic temperature T_{go} is obtained as 313 ± 5 K, close to the experimental glass transition temperature. The hypercooling regime starts below the isenthalpic temperature T_h = 365 K.

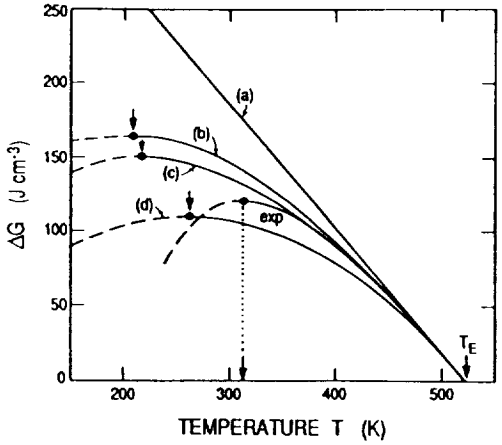

Fig. 3: Free energy difference between ΔG_V of the undercooled liquid and eutectic solid per unit volume based on different approximations including the experimental data. Curves (a) to (d) refer to Eqs. (3a) to (3d) respectively. The ideal glass transition temperature is indicated by the zero slope for ΔG_V.

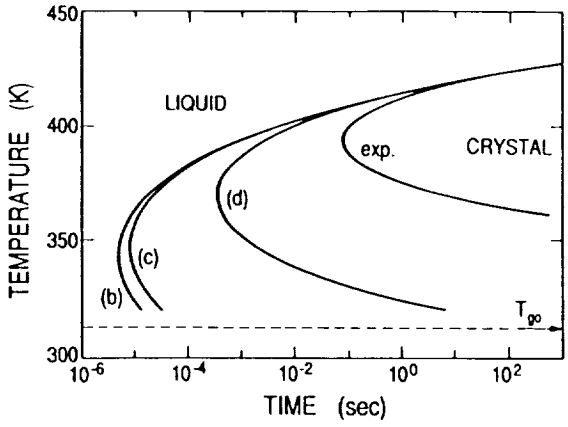

Fig. 4: Calculated steady-state heterogeneous nucleation kinetics of glass-forming Au-Pb-Sb alloys based on Eq. (1) for experimental C_p data (exp), and approximations for ΔG_V and $T_{\Delta S=0}$ according to Eqs. (3b) to (3d). Consistency with drop tower experiments (glass formation at ~10^3 K/s) is only found for the curve (exp).

N91-21344

Workshop on Containerless Experimentation in Microgravity

Thermophysical Property Measurements in Electromagnetic Levitators

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Rice University & Houston Area Research Center

Proper measurements of thermophysical properties of hot levitated liquid drops require the following:

1. Accurate Temperature Measurement
   a. Brightness measurement
   b. Emissivity measurement

2. Precise Drop Shape Measurements with Submillisecond Time Resolution
   a. Density determination
   b. Rotational and vibrational shape information

3. Precise Control of Drop Shape
   a. High symmetry variable gap levitators

4. Accurate Energy Transfer Measurements
   a. Direct measurements of energy transfer rates for defined gas flows over samples with Quantitative measurements of energy transfer rates for defined gas flows over samples with known shapes

5. Precise Measurements of Repetitive Sample Motions
   a. Rapid repetitive shape measurements
   b. Frequency measurements with reflected laser light
   c. Measurements in the levitator and as a freely falling drop

Recent advances in coil design and control of sample rotation in an electromagnetic levitator will be discussed with respect to the above requirements

The Primary Electromagnetic Levitation Problem has been Uncontrolled Sample Motion

Uncontrolled sample motion is thought to result from a number of causes either singly or in combination as follow.

Flux Inhomogeneity

Inhomogeneity of the coil magnetic flux field results from lack of physical symmetry of the coil. Inhomogenous fields can result in large sample oscillations during melting and nonsymmetric drop shapes.

Nonuniform Heating

Non-uniform sample heating can result in a net circular mass flow within the sample. Non-uniform heating will result from nonuniform flux fields and non-spherical sample shapes such as that caused by the earth's gravitational field.

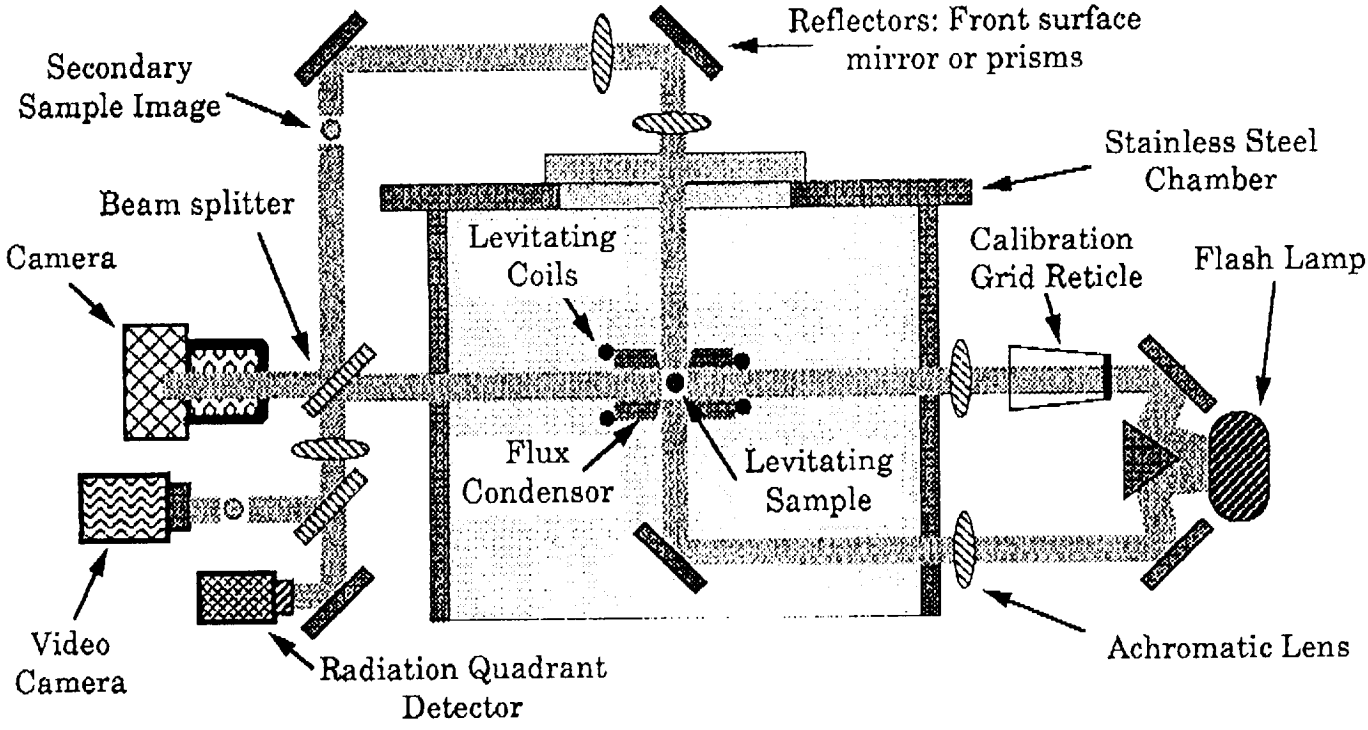
Coil Vibration

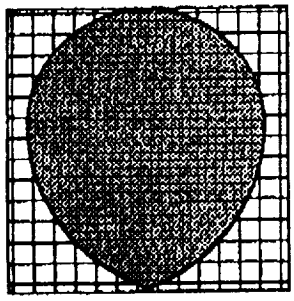
The importance of physical vibration of the coil is unknown but can be quite important since levitation coils are usually suspended on long lever arms which can cause the coil to be sensitive to vibrations present in the chamber.

RF Source Modulation

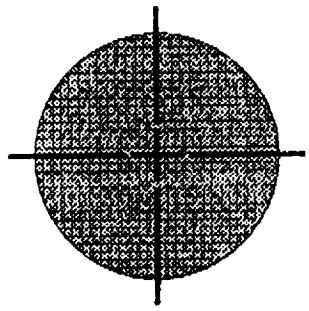
Low frequency modulation of the RF power supply, sixty cycle and overtone modulation of the high power RF supplies is often present due to incomplete rectification of the line sixty cycle power. Since levitation forces are directly dependent on the RF power its modulation will directly couple energy into the oscillations of the sample.

Liquid Drop Density Measurements In An Electromagnetic Levitator

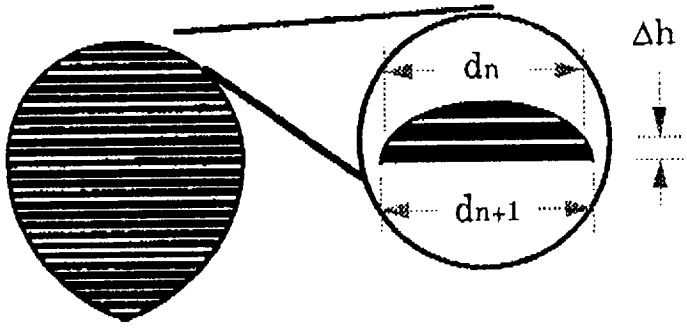




Profile of levitating sample as seen in Camera with Calibration Grid



Top View of levitating sample as seen in Camera & RQD

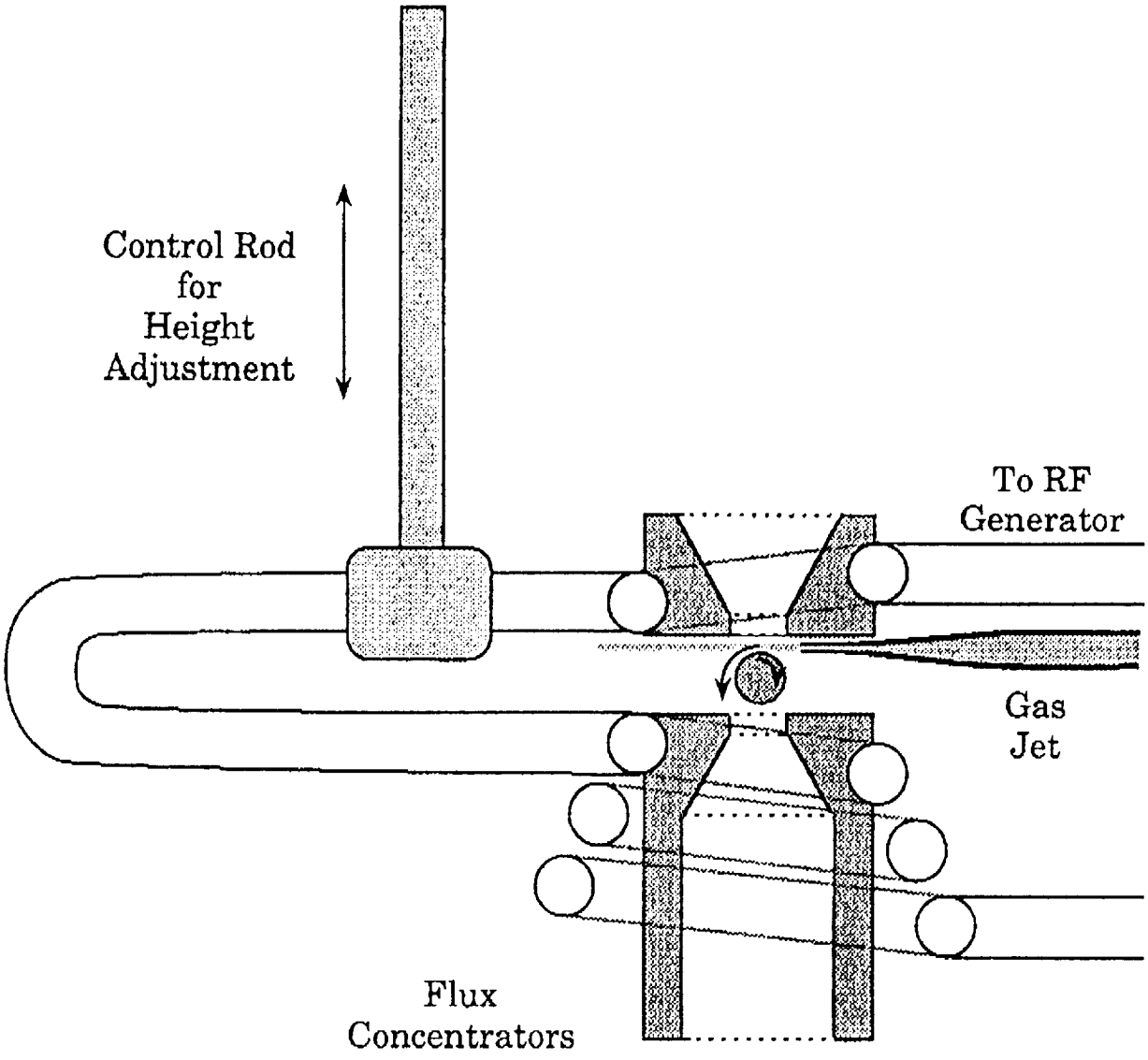


Scanned sample image equipartitioned for volume calcalation

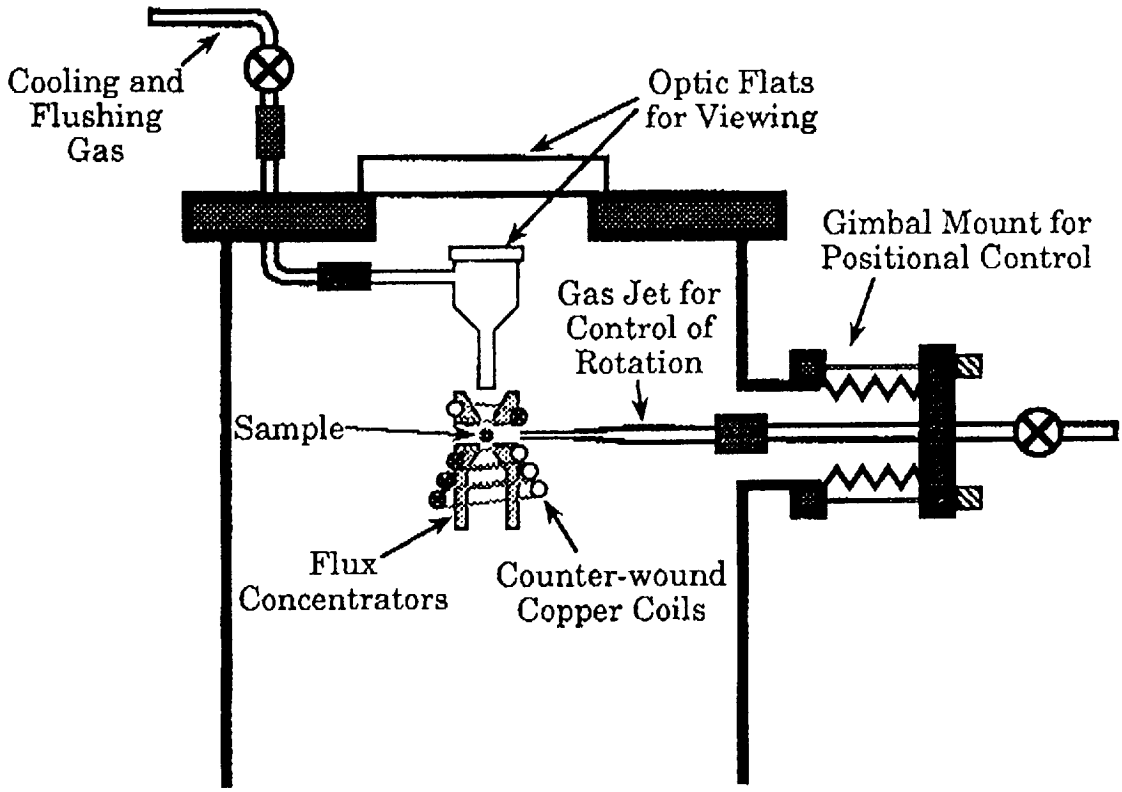
$$\text{Total Volume} = \sum \text{all slices}$$

$$\text{Volume of each slice} \approx \pi\left((d_n + d_{n+1})/4\right)^2 \ (\Delta h)$$

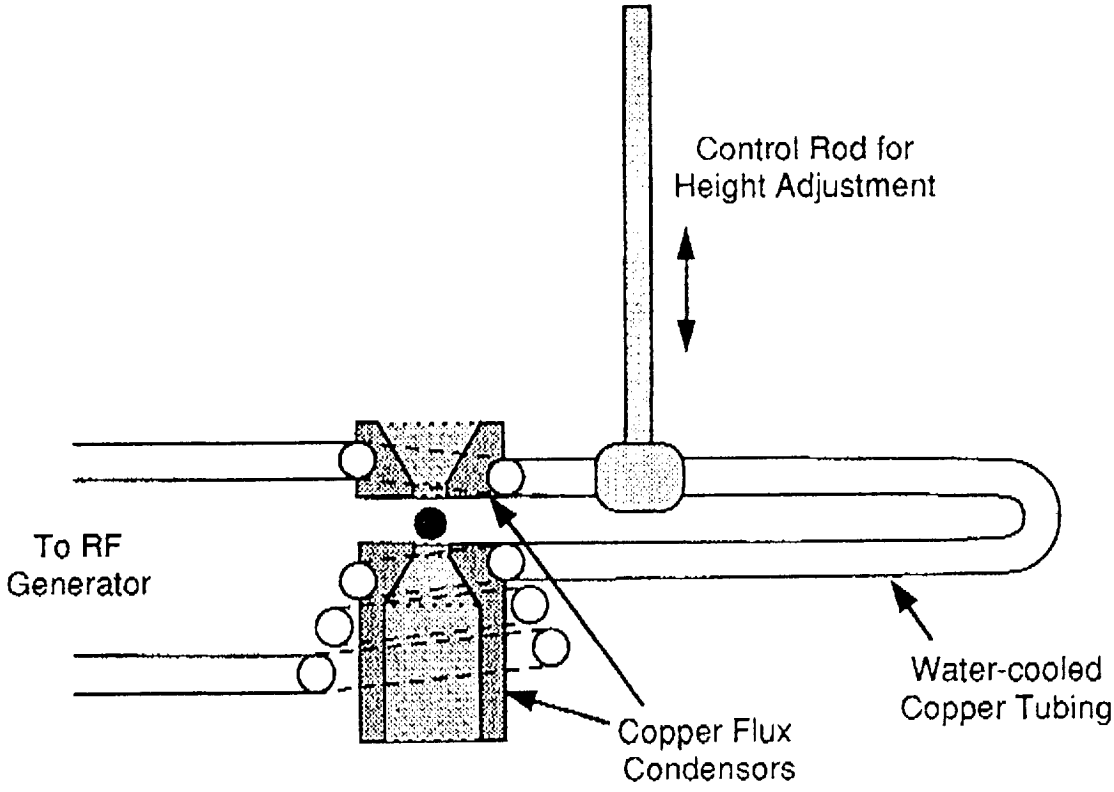
Modifications for Drop Shape and Spin Control (Expanded View)



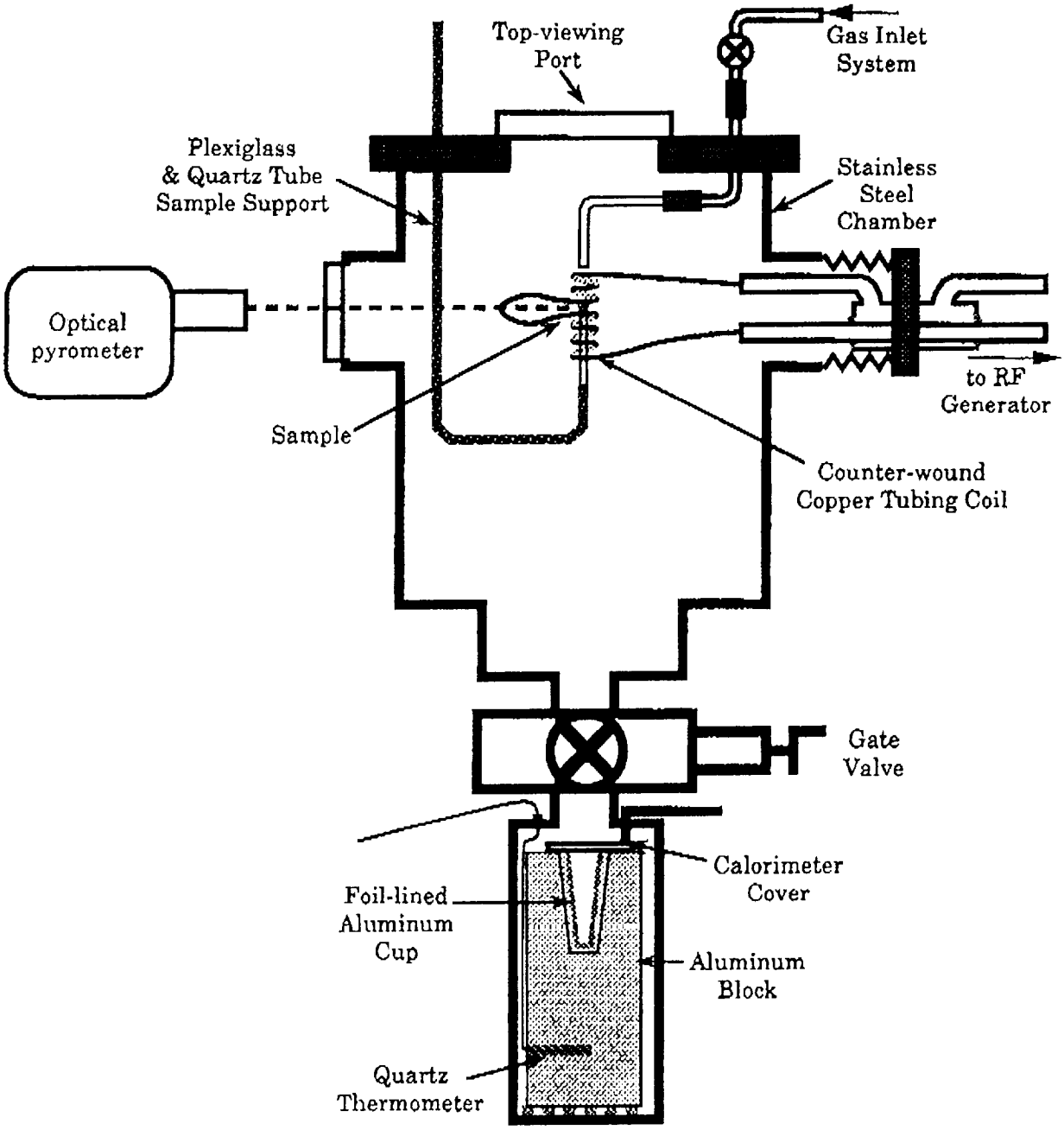
Modifications for Drop Shape and Spin Control



Flux Concentration Levitator with Variable Gap



Calorimetry Set-up



Suggestion For Improved Coil Symmetry, Coupling to the Sample & Sample Control

Use of Flux Contentration

Magnetic flux homogeneity can be achieved with a technique of flux concentration. This involves the use of split copper plates or tubes with center holes. Each plate is attached to and cooled by a turn of the solenoid coil nearest the sample. The RF current flows primarily around the inner surface of the center hole. This serves to concentrate the flux and also to give a uniform radial distribution. When plates are attached to both the upper and lower coil, the flux radial uniformity is expected to be much improved over those present in existing coils.

Variable Gap Coils

Efficient sample heating will ultimately permit smaller RF generators to be used. A factor which critically affects sample heating; is the distance between the sample surface and the coil. A variable gap between the upper and lower coils of a levitation coil allows for optimization of sample-coil distances.

Sample Rotational Control

Rotational control of the sample can be achieved with gas jets. A moveable point source of high velocity gas which is located to either side of the sample induces a counter-rotation of bath gas around the sample. This allows one to either decrease or increase sample rotation rate.

N91-21345

NCTM of Liquids at High Temperatures using Polarization Techniques

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ABSTRACT

Temperature measurement and control is extremely important in any materials processing application. However, conventional techniques for non-contact temperature measurement (mainly optical pyrometry) are very uncertain because of unknown or varying surface emittance. Optical properties like other properties change during processing. A dynamic, in-situ measurement of optical properties including the emittance is required. Intersonics is developing new technologies using polarized laser light scattering to determine surface emittance of freely radiating bodies concurrent with conventional optical pyrometry. These are sufficient to determine the true surface temperature of the target.

Intersonics is currently developing a system called DAPP, the Division of Amplitude Polarimetric Pyrometer, that uses polarization information to measure the true thermodynamic temperature of freely radiating objects. This instrument has potential use in materials processing applications in ground and space based equipment. Results of thermophysical and thermodynamic measurements using laser reflection as a temperature measuring tool will be presented. The impact of these techniques on thermophysical property measurements at high temperatures will be discussed.

RESULTS: The results presented at the workshop are summarized in the next four pages. The first figure lists the various materials that have been studied in the solid, liquid and undercooled states. The second slide presents illustrates the accuracy of the technique by comparison of data with that obtained by DeVoss on tungsten. It can be seen that the accuracy is about 1% in the value of the spectral emissivity. The third slide illustrates the temperature dependence of the spectral emissivity for liquid platinum and the last slide shows recent preliminary results on solid and liquid niobium.

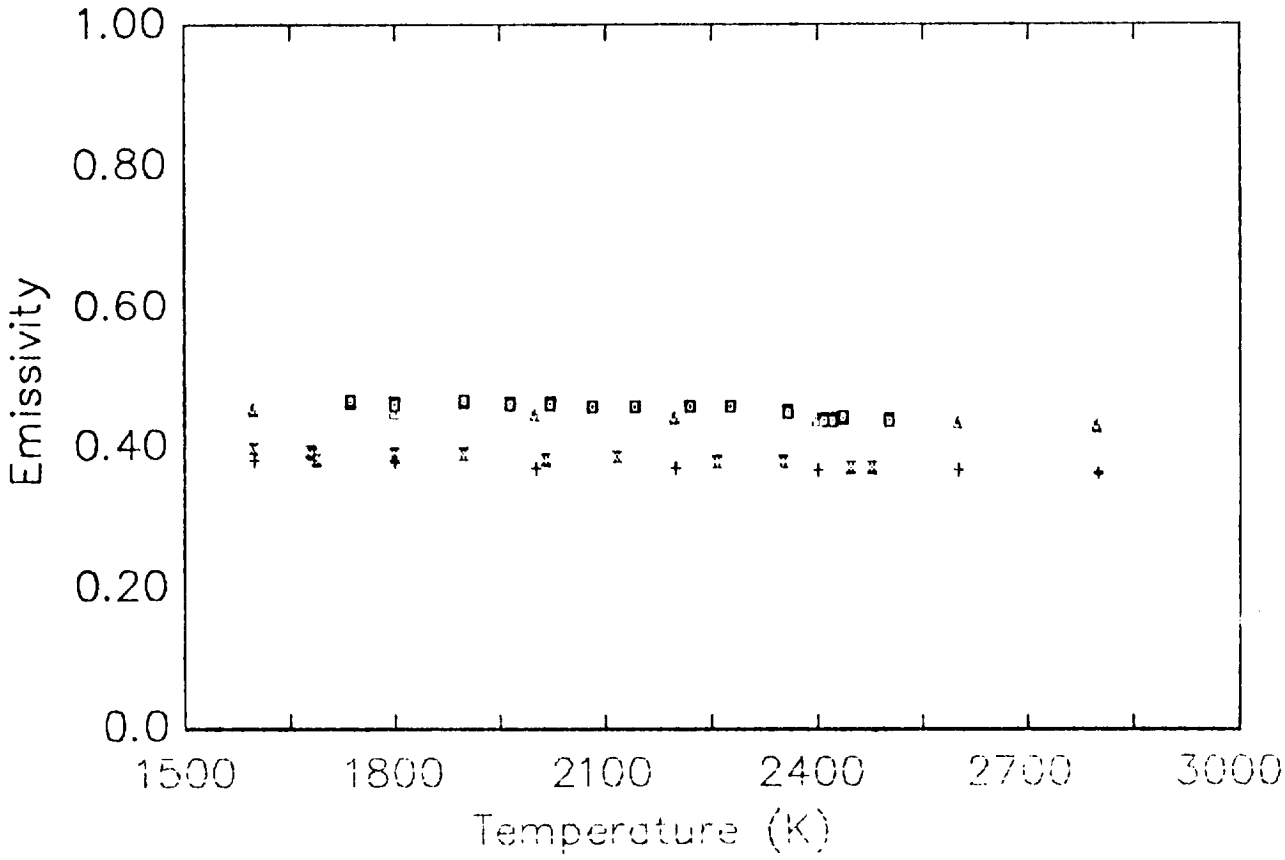
CONCLUSIONS: Extremely accurate (1%) spectral emissivities and optical properties of various materials at high temperature can be obtained using polarization techniques. The technology has been successfully applied to solids, liquids, and undercooled liquids for emissivity and true temperature measurement. The technique has been developed mainly for containerless processing for solids and liquids and Intersonics is currently developing the DAPP, the Division of Amplitude Polarimetric Pyrometer for the NASA Microgravity Sciences Program.

Materials whose optical properties have been studied using Polarization Techniques include:

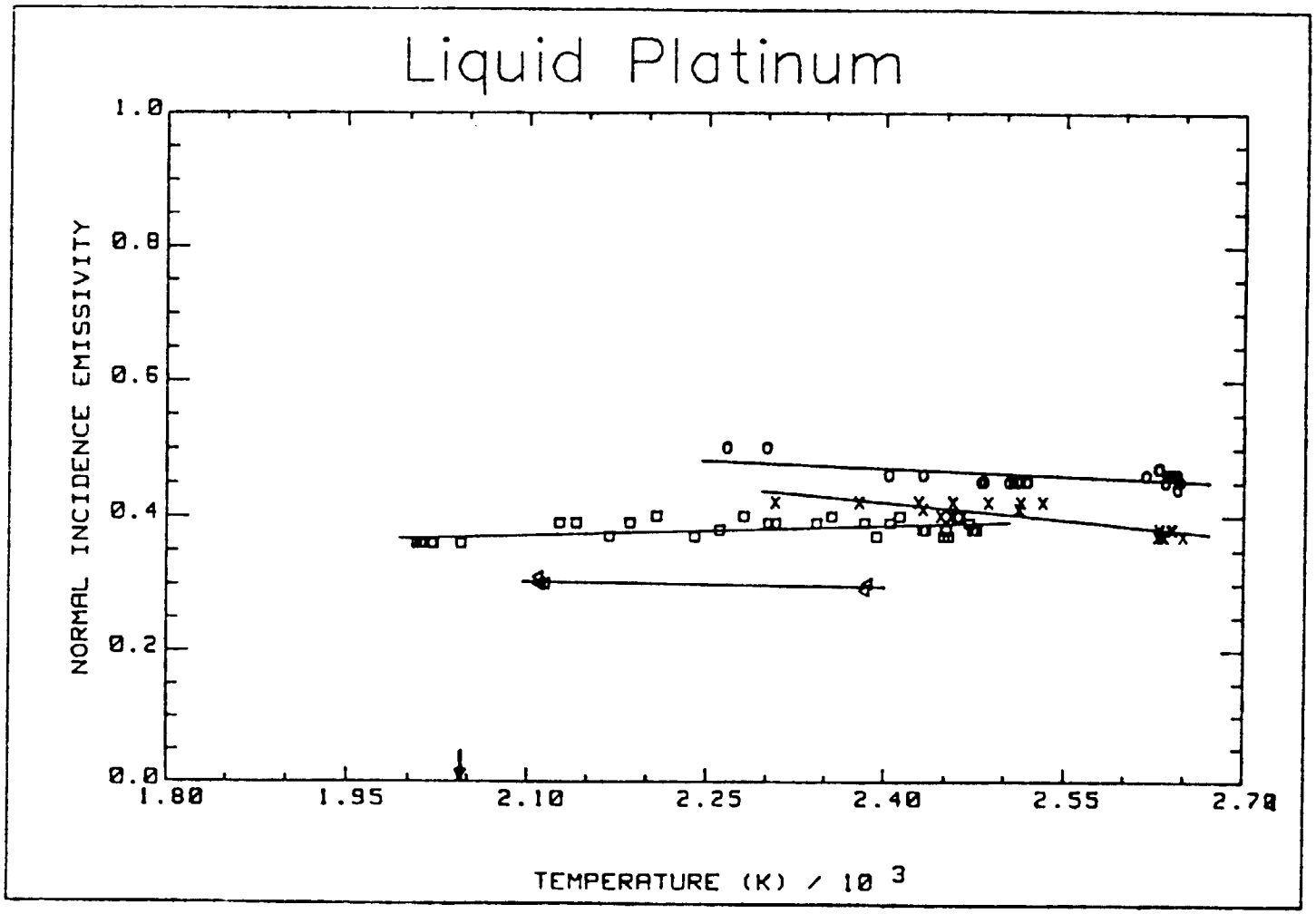
SOLIDS: Si, Ti, Ir, Nb, Mo, Ta, V, Pd, Pt, W, WC, TaC, C(graphite)

LIQUIDS: Si, Cu, Ag, Au, Ni, Pd, Pt, Zr, Al, Ti, Nb

UNDERCOOLED: Si, Pt, Nb, Zr, Pd



The spectral emissivity of tungsten at λ = 1064 (▫) λ = 633 nm (▫) as a function of temperature. The data measured by De Vos at 1064 (+) and 633 nm (×) are shown for comparison.



Normal incidence spectral emissivity of liquid platinum as a function of temperature at 1064 (Δ), 633.8 (□), 514.5 (X), and 488 nm (O). Solid line represents the least squares fit to the data. Melting point indicated by arrow.

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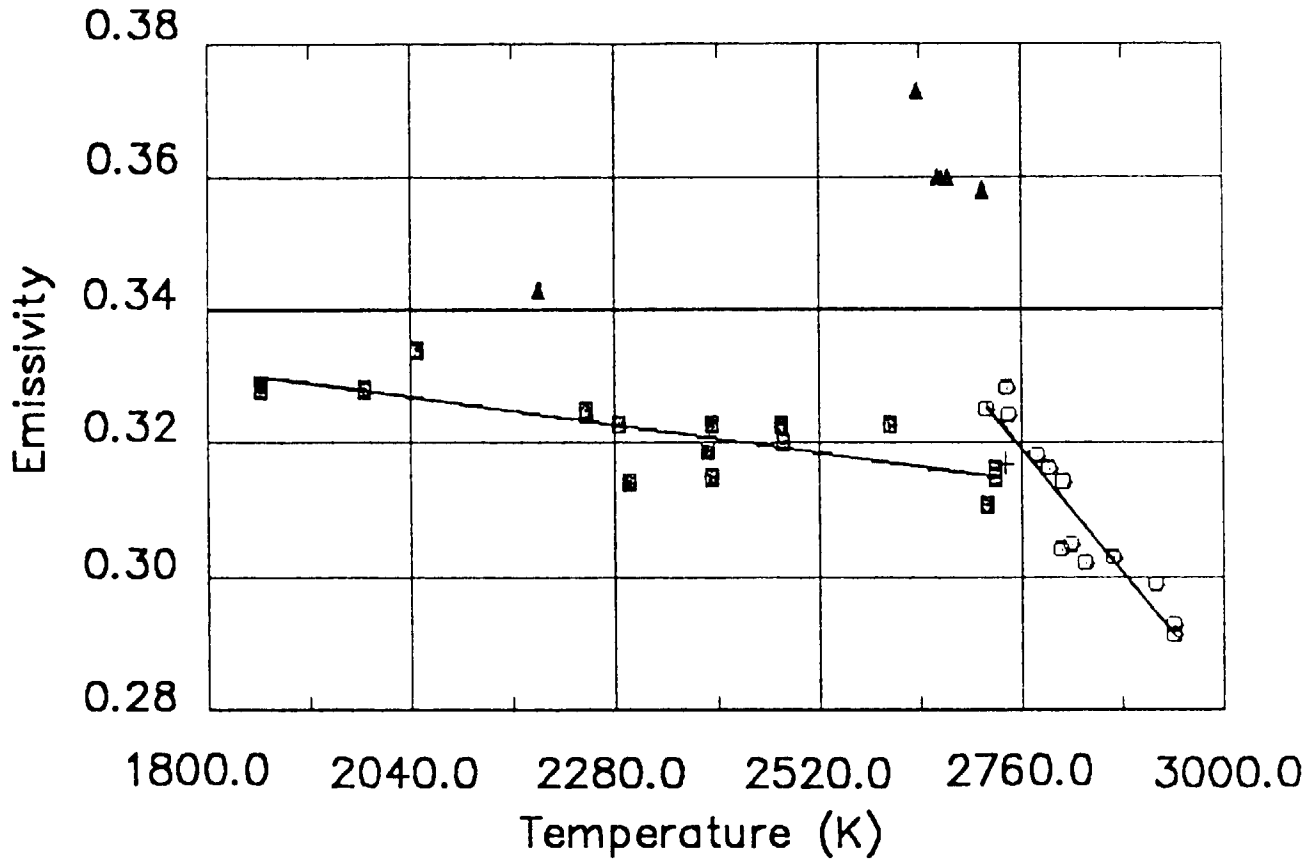
The normal spectral emissivity of Niobium as a function of temperature at 0.6328 μm.



○ — Liquid Niobium ■ — Solid Niobium ▲ — Solid Niobium before clean up
\+ — Values at Tm and 0.65 μm (D. W. Bonnell)

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N91-21346

SURFACE TENSION AND VISCOSITY FROM DAMPED FREE OSCILLATIONS OF VISCOUS DROPLETS

by

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Damped oscillations of a viscous droplet in vacuum or in an inert gas of negligible density are considered. The dependence of the complex decay factor on the properties of the liquid is investigated for the first time, and numerical results are compared with earlier studies for special cases. A new method is developed to determine both surface tension and viscosity from a single experiment in which the damping rate and frequency of oscillations are measured. The procedure to determine surface tension and viscosity from oscillating levitated liquids is outlined, and results presented for various modes of shape oscillations.

Key Words: Levitation, oscillating droplets, surface tension, viscosity

N91-21347

A DYNAMIC TECHNIQUE FOR MEASURING SURFACE TENSION AT HIGH TEMPERATURES IN MICROGRAVITY ENVIRONMENT

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Gaithersburg, MD 20899

Abstract

The feasibility of a dynamic technique for measuring surface tension of liquid metals at high temperatures in a microgravity environment has been demonstrated. The basic method involves heating a tubular specimen resistively from ambient temperature through its melting point in about 1 s by passing an electrical current pulse through it, while simultaneously recording the pertinent experimental quantities. Static equilibrium for the molten specimen is achieved in a microgravity environment by splitting the current after it passes through the specimen tube and returning a fraction along the tube axis, and the remaining fraction outside the specimen. Adjustments to the current split enable a balance between the magnetic and surface tension forces acting on the specimen. Values for surface tension are determined from measurements of the equilibrium dimensions of the molten specimen tube, and the magnitudes of the currents. Rapid melting experiments, performed during microgravity simulations with NASA's KC-135 aircraft, yield a value for the surface tension of copper at its melting point which is in agreement with literature data. Measurements of surface tension of a refractory metal (tantalum) are underway.

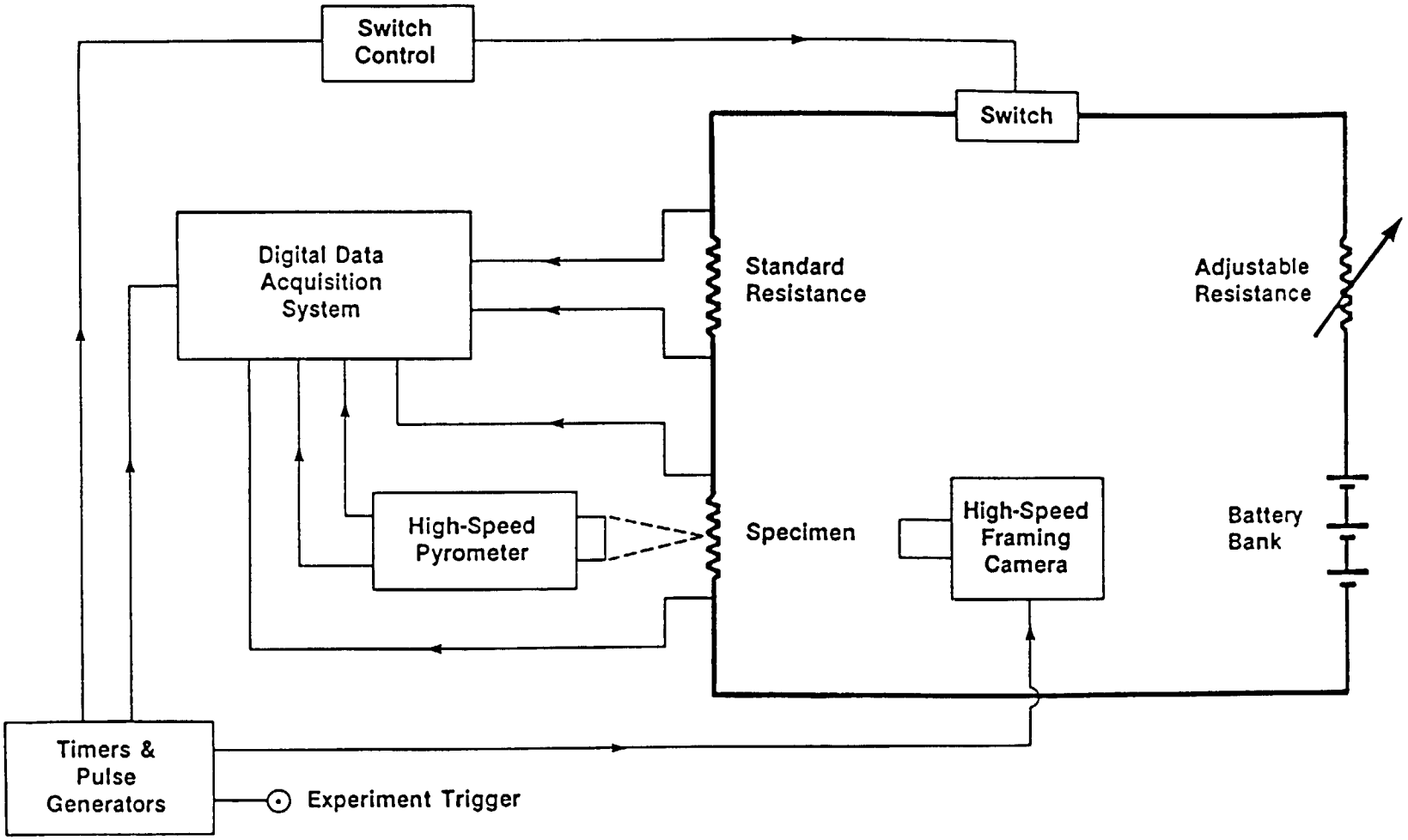

Fig. 1. A functional diagram of a compact pulse-heating system designed for rapid-melting experiments during microgravity simulations with the KC-135 aircraft.

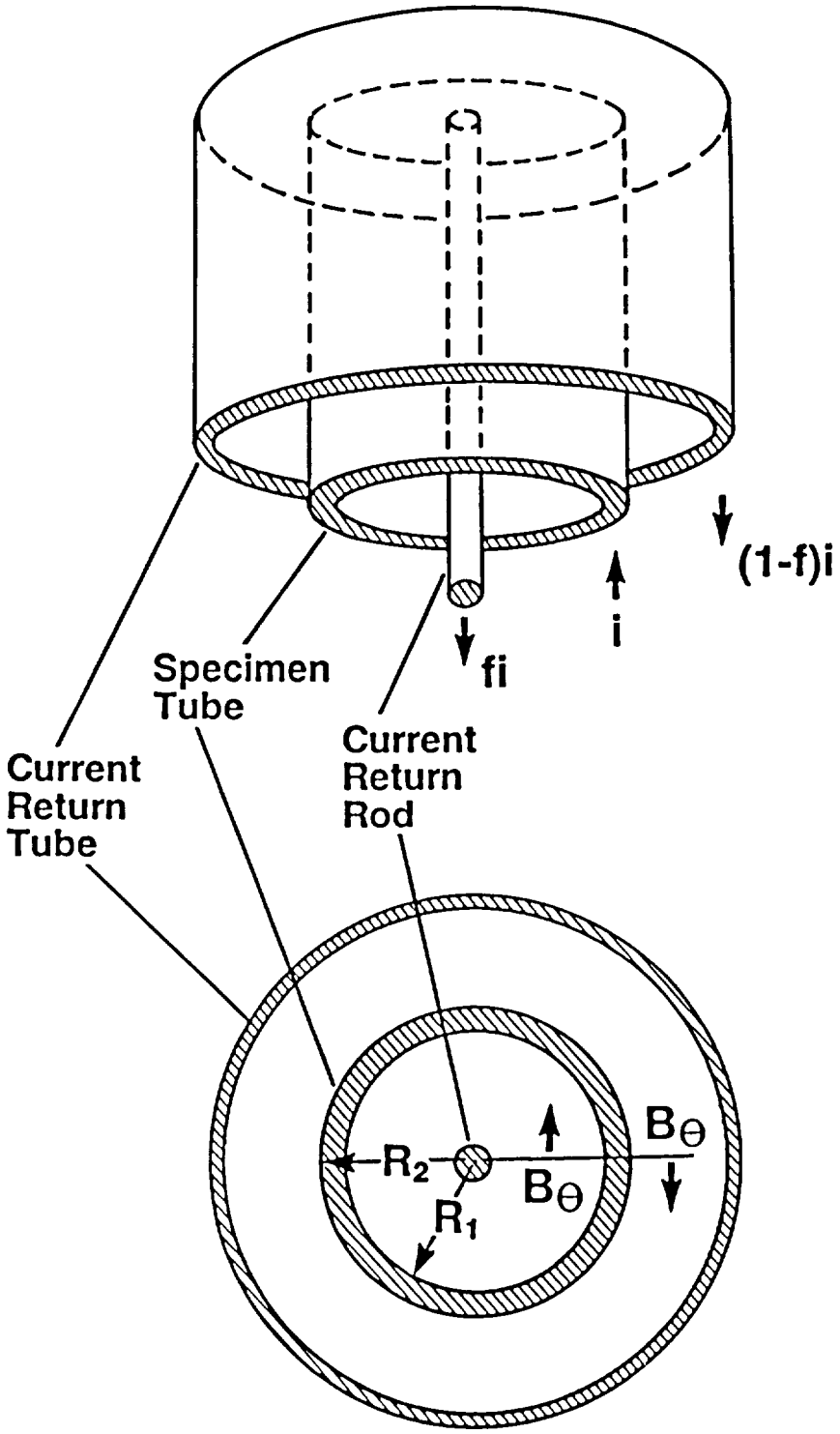

Fig. 2. A schematic diagram of the triaxial configuration in which a tubular specimen in mounted concentrically with respect to the current return paths. During melting, the (inward) pressure due to surface tension may be counter-balanced by selecting a suitable return current split f to provide a net (outward) magnetic pressure.

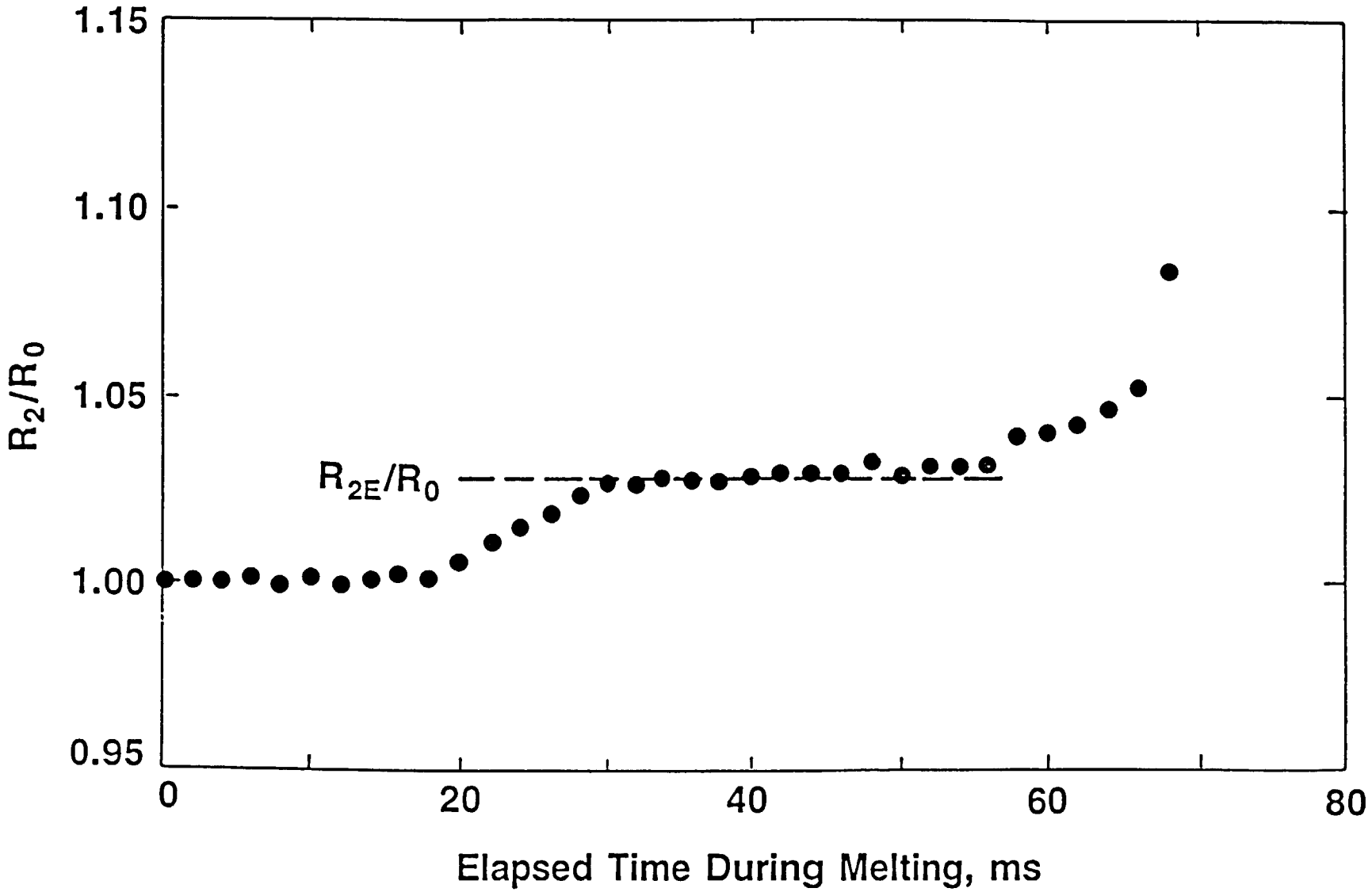

Fig. 3. Variation of the melt zone radius R_2 of a thin-walled copper tube during rapid melting under microgravity conditions; R_o is the tube radius just before melting and R_{2E} is the tube radius at static equilibrium during melting.

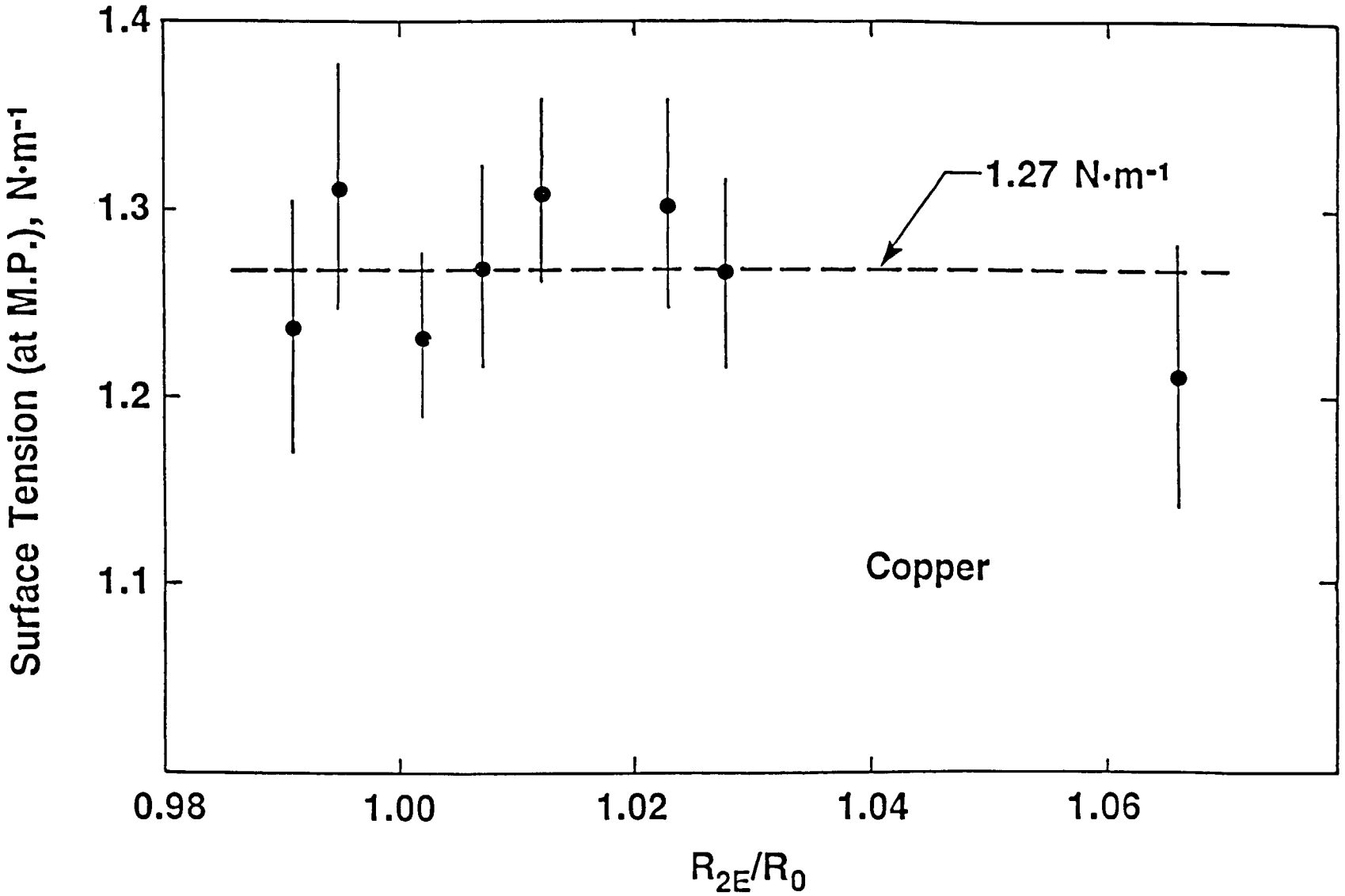

Fig. 4. Results from eight microgravity experiments for surface tension of copper (at its melting point) as determined from measurements of the equilibrium dimensions of the molten tube and the magnitudes of the currents.

N91-21348

Experiments for Electromagnetic Levitation in Microgravity

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and

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Containerless Processing is a promising research tool for investigating the properties of undercooled melts and their solidification. For conducting samples RF-electromagnetic levitation offers the possibility to obtain large undercoolings by avoiding heterogeneous nucleation at container walls.

On earth, however, strong magnetic fields are needed to compensate the gravitational force which imposes a lower limit on the available temperatures and on the accessible undercooling range. Under microgravity conditions the magnetic positioning fields can be minimized and hence, undercooling becomes feasible under ultra high vacuum conditions and lower temperatures become accessible.

In contrast to other undercooling and solidification techniques, electromagnetic levitation allows for diagnostic measurements during the early steps of nucleation and phase selection. Experiments cover a wide field of research topics: nucleation, directional solidification at a high velocities, generation of metastable phases, evolution of microstructures, properties of undercooled liquids. Examples from these classes including experiments selected for the IML-2 mission will be discussed with emphasis of technical requirements. An overview will be given on the German TEMPUS (Electromagnetic levitation facility) program.

EXPERIMENTS FOR ELECTROMAGNETIC LEVITATION IN MICROGRAVITY

R. Willnecker [1], I. Egry [2]

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2) Institute for Space Simulation, DLR, 5000 Cologne 90, FRG

Workshop on Containerless Experimentation in Microgravity
Pasadena, January 17 - 19, 1990

Experiments for Electromagnetic Levitation in Microgravity

R. Willnecker, I. Egry (DLR)

INTRODUCTION

- o General Aspects of Electromagnetic Levitation Techniques
  - Advantages of Experiments under Microgravity
- o Experiment Classes and Scientific Objectives
- o Scientific Hardware Requirements
- o TEMPUS Development Program

ADVANTAGES OF ELECTROMAGNETIC LEVITATION

- Contactless Positioning
- Contactless Heating
- *in situ* – Diagnostics
- Bulk Samples
- High Temperatures (up to 2500°C)
- Pure Environment

Limitation

- Conducting Sample

ADVANTAGES OF ELECTROMAGNETIC LEVITATION UNDER MICROGRAVITY

- Less R.F. power necessary for positioning
- Separation of positioning and heating
- Investigation of low melting metals
- UHV environment
- No shape deformation
- Reduced magnetic damping
- Stirring effects will be considerably weaker

Experiment Classes

1. Undercooling and Nucleation

2. Non - Equilibrium Solidification

3. Metastable Phases

4. Thermophysical Properties of Undercooled Melts

Scientific Objectives

1. Undercooling and Nucleation

- max. undercoolings
- "hypercooling"
- nucleation frequencies
- heterogenous and homogeneus nucleation
- nucleation control
- effects of microgravity environment on undercooling experiments

2. Non - Equilibrium Solidification

- measurements of solidification velocities
- directional dendritic and eutectic solidification
- solute trapping and segregation free solidification
- microstructure evolution
- coarsening and ripening effects

Scientific Objectives

3. Metastable Phases

- generation of metastable phases
- metastable phase diagrams
- phase selection processes
- formation of quasicrystaliine phases
- metallic glass research
- thermodynamics of metastable phases

4. Thermophysical Properties

- surface tension
- viscosity
- specific heat

Experiment Hardware Requirements

Generic Requirements

Pure Environment	UHV $\leq 10^{-8}$ mbar, pure inert gases
Two Frequency Generators	two coil system tunable input power 0-100%
Stable Sample Positioning	stable against 10^{-2} g_o damped rotations and oscillations
Evaporation Shielding	

Specific Requirements

	Nucleation	Growth	Metastable Phases	Properties
Pyrometry	$\leq$ 1 µs; < 2570 C	$\leq$ 1 µs; = 90	$\leq$ 1 s; $\geq$ 400 C	#
Power Modulation				10 W/s
Nucleation Trigger		#	#	
Video System		$\leq$ 500 Hz		$\leq$ 200 Hz; top and side view
Quenching Device		#	#	

TEMPUS DEVELOPMENT PROGRAM

STEP O:	- Pre-Developments	Since 1983	DLR Inst. for Space Simulation
STEP 1:	- Laboratory-type Model	3/86 - 11/87	Contractor: Dornier
	o KC-135 Tests	11/1987, 5/1988	
	o coil development	-1989	
	o ground support program (TEXUS)	1988	
	o temperature diagnostics (contamination study)	1989/1990	
	o user support program (IML-2)	1990 - 1993	
STEP 2:	- TEXUS-Model	5/1987 - 6/1989	
	Facility Test / Scientific Experiment		
	First Mission	4/1989	
	- Spacelab Model Phase B	6/1988 - 3/1990	
STEP 3:	- Spacelab Model Phase C/D	4/1990 - 1/1992	
	o First Mission : IML-2	Jan. 1993	

Experiments for TEMPUS on IML-2

PI	Affiliation	Title
Bayuzick	Vanderbilt Univ.	"Effects on Nucleation by Containerless Processing in Low Gravity"
Flemings	MIT	"Alloy Undercooling Experiments"
Szekely	MIT	"Measurements of the Viscosity of the Undercooled Melts Under the Conditions of Microgravity and Supporting MHD Calculations"
Johnson	California Inst. of Technology	"Metallic Glass Research in Space"
Egry	Inst. for Space Simulation, DLR	"Viscosity and Surface Tension of Undercooled Melts"
Herlach	Inst. for Space Simulation, DLR	"Non-Equilibrium Solidification of Largely Undercooled Melts"
Urban	Inst. for Solid State Research, KFA Jülich	"Structure and Solidification of Largely Undercooled Melts of Quasicrystal-Forming Alloys
N.N.		

N91-21349

Electromagnetic Processing Onboard Spacelab

H. Lenski and R. Wilnecker
Dornier GmbH and DLR

The electromagnetic containerless processing facility TEMPUS has recently been assigned for a flight on the IML-2 mission.

In comparison to the TEMPUS facility already flown on a sounding rocket, several improvements had to be implemented. These are in particular related to:

- Safety
- resource management
- the possibility to process different samples with different requirements in one mission.

The basic design of this facility as well as the expected processing capabilities will be presented.

Two operational aspects turned out to strongly influence the facility design:

a) control of sample motion

First experimental results indicate that crew or ground interaction will be necessary to minimize residual sample motions during processing

b) exchange of RF-coils

During processing in vacuum, evaporated sample materials will condense at the cold surface and may force a coil exchange, when a critical thickness is exceeded.

TEMPUS-A FACILITY FOR CONTAINERLESS ELECTROMAGNETIC PROCESSING ONBOARD SPACELAB

H. Lenski
Dornier GmbH

R. Willnecker
DLR

The TEMPUS facility has been designed for containerless processing of metallic samples in weightlessness.

The main design driving requirements are:
- Melting and undercooling of very different sample types with the same RF system (generators, coils), for example:
    - o 6mm niobium spheres (T max > 2550°C)
    - o 10mm aluminium spheres (T min < 600 °C)
- sufficient visual accessibility to observe sample oscillations
- high symmetry of RF fields to minimize residual sample motions

The facility development started with a set of basic requirements. In due course of the development the performance range has been extended as far as possible. In particular improvements of the RF-system turned out to be necessary.

The design for the S/L-version of TEMPUS is characterized by:
- use of two independent coaxial coils (dipole, quadruple) running at two frequencies (420kHz and 140kHz)
- free oscillating circuits coupled to a low-voltage (0.20V) RF generator via a transformer

Dornier
Deutsche Aerospace

T E M P U S

A Facility for Containerless Electromagnetic Processing onboard Spacelab

January 1990

The facility has been developed under contract of DLR,
acting on behalf of the German Ministry of Research and Technology.

The design is based on experimental and theoretical work
performed by the Institute of Space Simulation (DLR).

TEMPUS Facility Characteristics:

- sample positioning and heating by high frequency electromagnetic fields
- processing in an ultraclean environment (UHV or noble gas)

Development Strategy:

Start with a set of basic requirements and try to extend the performance range as far as possible.

Flight experience:

- parabolic flights on KC-135
- sounding rocket flight (TEXUS 22)

Design Driving Requirements:

Experimental requirements:

- temperature range: melting of a 6mm Nb sphere
  undercooling of a 10mm Al sphere
- sufficient visual accessibility to observe sample oscillations
- minimized residual motion (high symmetry of RF fields)

General:

- use of the same RF system (coil, generator) for all experiment types
- limited power (two 60 A lines for S/L versions)

The development of the RF system was crucial for the success of the TEMPUS concept.

Necessary Improvements for TEMPUS on Spacelab

- Processing capability extended from 1 sample /1 cycle to 22 samples / 100 cycles
  - o sample storage magazine
  - o enlarged evaporation shielding capability
  - o more process gas
  - o turbomolecular pump
  - o data reduction for 1 MHz data/ extended intermediate memory
- More complex process control (different experiment types)
- additional diagnostics (radial IR sensor, fast video)
- improved efficiency of RF system
- increased reliability of high power electronics (derating)
- additional safety devices

TEMPUS Performance Data (Basic Design for IML-2)

sample temperature:	300°C-2500C°
sample diameter:	max.: 10mm (coils optimized for 10mm)
No. of samples (stored in vacuum):	22 (note: samples will be processed in wire cages)
vacuum quality:	ultimate pressure < 1.10^{-9} mbar
gas atmosphere	He/Ar, impurities < 1ppm
RF system:	superposition of quadrupole and dipole field
combined RF power:	1980 W (quadrupole: dipole ratio about 1:1)
heating efficiency for metals:	quadrupole field 0,3% to 1,7% dipole field 5% to 37 %
power adjustment range:	0 to 100%
frequency monitoring accuracy:	0,05%
DC magnetic field damping:	inhomogeneous field adjustable, 1 to 50 mT

Sample Temperature Measurement:

pyrometer type:	Two or three colour
temperature range:	300°C to 2400°C (ε = 0,05 to 1) for at least two colours, 2600°C for one colour
accuracy:	± 5°C
resolution:	better than 0,1°C (100Hz)
max. frequency:	1 MHZ (250.000 readings internally stored)
evaporation shielding:	CaF_2 windows or double mirrors

Additional Diagnostics

video observation:	2 CCD cameras (b/w), side and top view
frequency:	up to 500 half pictures per second
	(side view only, reduced field of view)

radial IR sensor

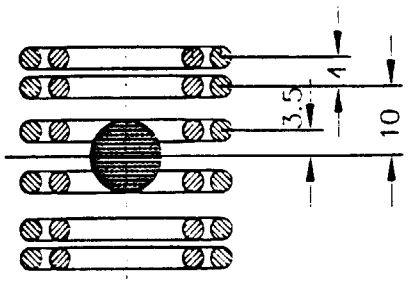
temperature range:	same as pyrometer
measuring frequency:	1MHz
internal storage:	250.000 readings

Optimization of the RF system

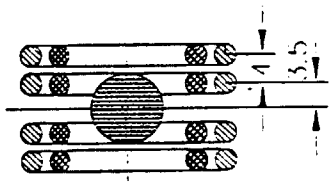
The full range of performance can only be achieved by superposing a quadrupole and a dipole field, both individually controllable.

Technical alternatives:

A) 2 independent coils,
two frequencies



B) 2 coils, identical frequency
adjustable phase shift.



Alternative B) has been calculated to be more efficient .
However, no technical solution has been found up to now.

Optimization of the RF Generators

Note: To achieve sufficient efficiency free resonant circuits have to be used

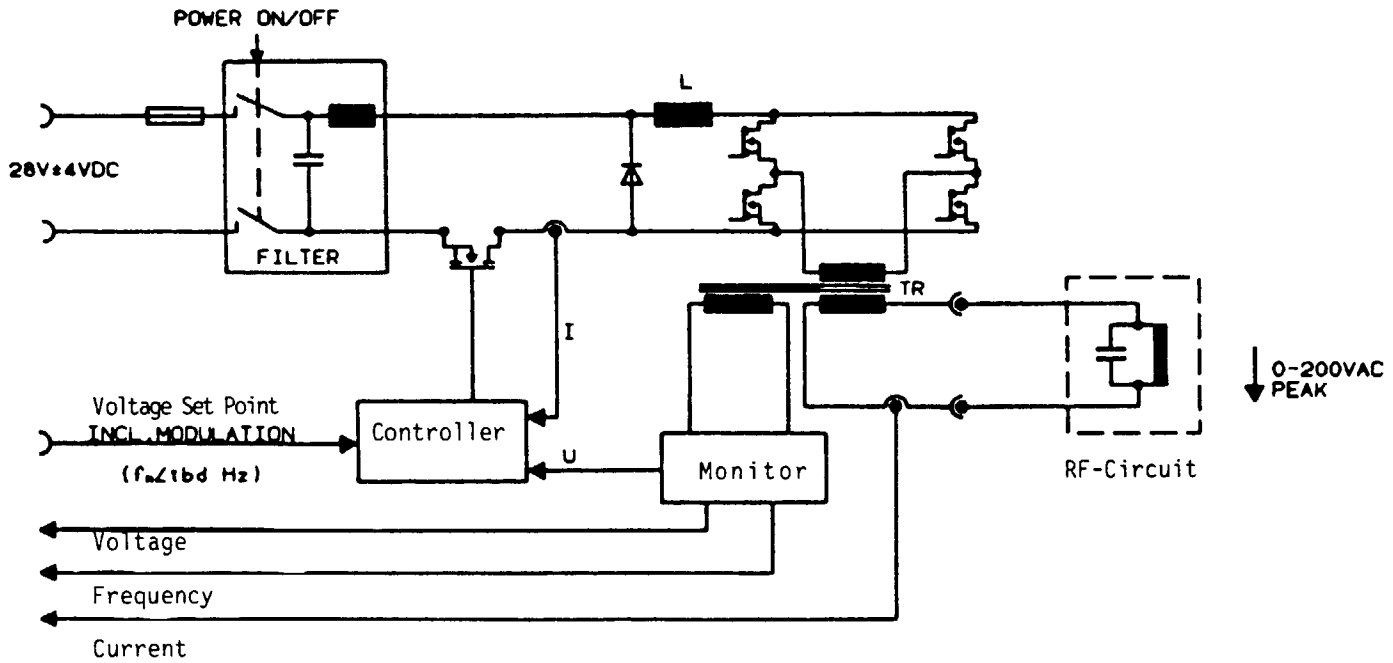
Concept for TEMPUS-TEXUS:

- RF power generated at high voltages and directly fed into the RF circuit
- input voltage 25 to 100V (by step-down converter from battery package) results in circuits voltage (peak to peak) of about 50 to 200 V

Improved concept for TEMPUS-Spacelab:

- RF generator directly powered by 28VDC bus (unregulated)
- RF output voltage 0 to 20 V, coupled to RF circuit via transformer

Advantages:
- increased efficiency (better than 80%)
- lower switching voltages, resulting in low stresses to electrical parts
- RF generator clearly separated from resonant circuit



RF CONVERTER BLOCK DIAGRAM

TEMPUS Control Concept

Facility control by dedicated processor (80386)

- data acquisition and transfer to ground (1Hz)
- interaction with flight and ground crew
- process control
- subsystem supervision including safety features
- selection of relevant 1 MHz data and transfer to ground

Fully automatic processing is possible.

Change of process parameters by flight or ground crew via serial RAU channel.

Additional manual control of some parameters to optimize dynamic positioning stability.

Control of Sample Oscillation and Rotation

sample motion is induced by

- non-symmetric fields
- rapid changes of dipole field
- low-frequency accelerations
- release from sample holder (initial energy)

passive damping foreseen by DC magnetic field

- not very efficient for low frequency oscillations

experience from TEXUS flight (FeNi sample)

- slight oscillation (rotation ?) starts immediately after sample release (± 0,25 mm)
- amplitude increased stepwise due to switching of dipole field (up to ± 0,5mm)
- no significant damping or acceleration due to other effects could be observed

Control of Sample Oscillation and Rotation (contd.)

Operational Improvements for TEMPUS on Spacelab:

- further optimization of coil system is difficult (if necessary at all)
- optimum ratio of quadrupole / dipole field has to be determined
- initial sample motion has to be avoided

As a consequence the following parameters shall be manually controllable by the flight crew:

- sample holder / cage linear motion
- quadrupole field power
- dipole field power

In Orbit Coil Exchange

Not foreseen for IML-2 but will become necessary for long duration missions because

- the coil cannot be shielded against evaporation of sample material in vacuum, thick layers will increase the coil resistance and/or flake off

- the present coil design is a compromise, optimized dedicated versions may be necessary for some experiments
  (smaller/larger samples, better observation, additional stimuli/diagnostics)

In Orbit Coil Exchange (contd.)

The present design allows exchange of the complete RF circuit including

- coils
- feed-through flange
- capacitors and transformer incl. housing

Interfaces:

- mechanical:	CF vacuum flange
- thermal	water cooling line
	(quick disconnects at capacitor housing)
- electrical	two 60 A power plugs

Open problem:

Safe containment of (toxic) metal dust/flakes during exchange

N91-21350

TEMPUS - First Results

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Institute for Space Simulation, DLR, 5000 Köln 90, FRG
*Dept. PT-PM, DLR, 5000 Köln 90, FRG

The electromagnetic levitation facility TEMPUS developed by Dornier under the contract of BMFT is designed to operate under microgravity conditions. Compared to terrestrial levitation, μg offers the possibility to melt and undercool in ultra high vacuum thereby providing an ultra clean environment.

The technical concept of the TEMPUS facility has been tested on two KC 135 flights and in the Texus 22 mission. Preparative investigations concerning the coil system and the heating and positioning efficiencies have been carried out in the TEMPUS laboratory version. Furthermore, temperature - time profiles have been determined under various boundary conditions.

As a consequence of processing liquid metals under UHV, correct temperature measurement arises as the most critical problem. Experiences with experiments in the TEMPUS laboratory module show that due to the evaporation losses of the sample, the transmission of the CaF_2 shielding windows changes drastically during the processing time. We have started to investigate the effect of contamination on pyrometry and are developing alternative evaporation shielding methods.

During the second KC 135 flight it was possible to heat up and melt an FeNi sample under He atmosphere. Oscillations of the molten sample, which were excited by switching out the magnetic heating field, could be detected and afterwards analyzed. From the frequency of these oscillations the surface tension of the sample material could be derived. The measurement of the surface tension and viscosity of an undercooled metal is proposed for TEMPUS on IML-2.

TEMPUS - FIRST RESULTS

P. Neuhaus, G. Lohöfer, I. Egry, R. Willnecker*

Institute for Space Simulation
*Dept. PT-PM
German Aerospace Research Establishment
5000 Köln 90, FRG

- PYROMETRY, EVAPORATION PROBLEM
- KC 135 RESULTS, SURFACE TENSION

Technical Workshop on
Containerless Experimentation in Microgravity
Jet Propulsion Laboratory, Pasadena
January 17 - 19, 1990

DLR

THE TEMPUS PROJECT

TEMPUS is an electromagnetic levitation facility,
designed to operate in microgravity

- two coil, two frequency concept:
  ⇒decoupling of heating and positioning
- two colour, fast (1MHz) pyrometer
- two axis visual observation by video
- processing under UHV possible

- laboratory model successfully tested in parabolic flights
- Texus version tested on a sounding rocket
- scheduled for IML-2 Spacelab mission
- under consideration for space station

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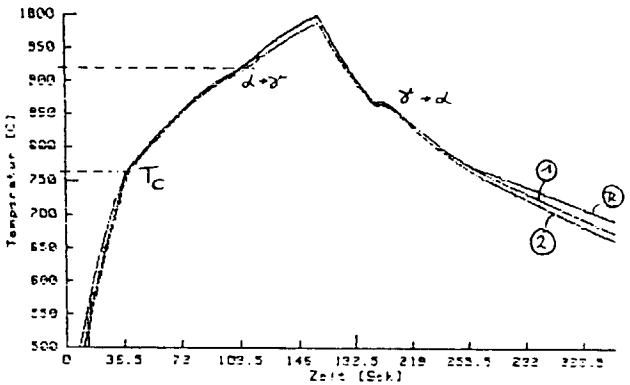
PYROMETRY

Infra-red pyrometer

- two one-colour signals, one ratio signal
- temperature range 300 °C to 2400 °C
- wavelength: 1 to 2 μm and 3 to 4 μm



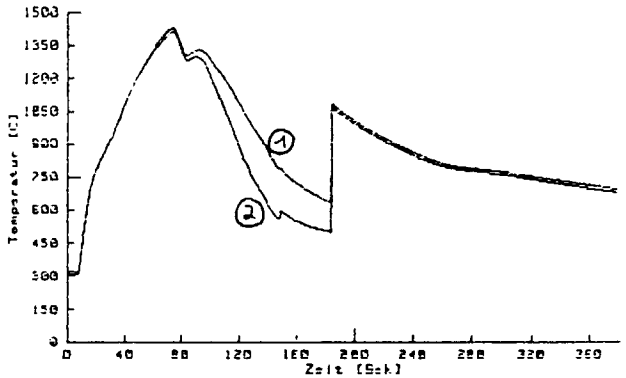
temperature - time diagram of Fe

- temperature calibration at magnetic and structural phase transitions
- determination of the emissivity coefficients

EVAPORATION PROBLEM

- evaporation losses of sample material
- contamination of the pyrometer window

⇒decrease of the temperature signal, dependent on the wavelength



temperature - time diagram of Fe

consequence: no temperature calibration at the melting point possible
no calibration and determination of the emissivity in the liquid sample

POSSIBLE SOLUTIONS

1. Shielding Windows
- the window in front of the pyrometer will be replaced when contamination becomes untolerable

2.Shutter or Rotor
- reduction of contamination at the expense of no continuous temperature measurement

3. Mirror Optics
- a double mirror system is placed in front of the pyrometer avoiding direct view on the sample

4. Diffraction Gratings
- putting a diffraction grating into the optical path and detecting only the diffracted light, contamination in front of the pyrometer can be avoided

PARABOLIC FLIGHT EXPERIMENT

MAIN OBJECTIVE
- verification of technical concept could be achieved (positioning cannot be tested under 1g)

SIDE RESULTS (not planned)
- levitated FeNi sample could be melted
- oscillations of the liquid sample could be excited
- surface oscillations detectable

FREQUENCY and DAMPING of the surface oscillations are related to SURFACE TENSION and VISCOSITY.

DIFFICULTIES UNDER 1G
- shape deformation
- stirring
- magnetic damping

EXPERIMENT ANALYSIS

Oscillations of a viscous sphere:

$R_n \sim \cos(\omega_n t) \exp(-\gamma_n t) P_n(\cos\theta)$

where

$\omega_n^2 = (n-1)n(n+2)\sigma/(\rho R_0^3)$

$\gamma_n = (n-1)(2n+1)\eta/(\rho R_0^2)$

P_n = Legendre polynomial

R_0 = unperturbed radius of sphere

η = viscosity

σ = surface tension

ρ = density

n = 2 fundamental mode

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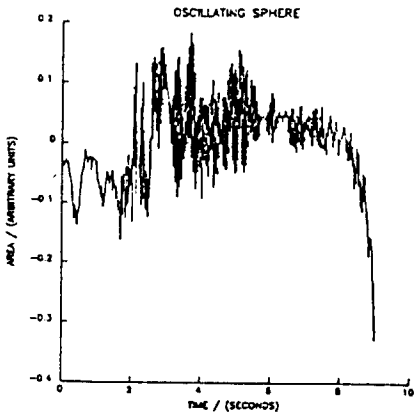
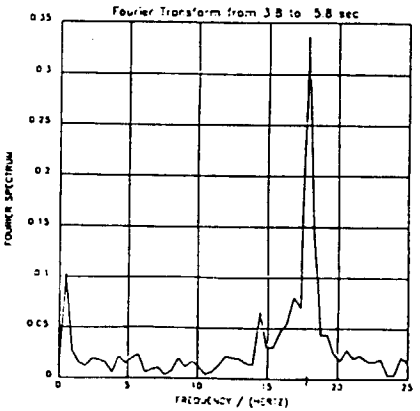
RESULT





$\gamma = 17.8$ Hz

$m = 4.2$ g

$G = 1.6$ N/m

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CONCLUSIONS

- EVAPORATION PROBLEM CAN BE HANDLED

- SURFACE OSCILLATIONS CAN BE EXCITED AND DETECTED

SURFACE TENSION AND (HOPEFULLY) VISCOSITY CAN BE DETERMINED

DLR

N91-21351

Reluctant Glass Formers and Their Applications In Optical Lens Design

Dr. E. C. Ethridge
Space Science Laboratory
NASA - Marshall Space Flight Center

and

Prof. R. B. Johnson and Dr. C. Feng
Center for Applied Optics
University of Alabama in Huntsville

Over ten years ago it was shown that glasses with high index of refraction and some with low dispersion could be produced from a number of pure refractory oxides including the lanthanides by containerless processing. By containerless processing it is possible to minimize surface heterogeneous nucleation and produce larger glass samples of the materials than by other methods. The use of proposed high temperature containerless processing facilities in space will permit the fabrication of benchmark samples of new unique glass compositions for optical property determination as well as for glass formation.

It has been shown that glasses with high refractive indices and large Abbe numbers can be formulated using this technology. These glasses lie in the classical "forbidden" region of the glass map. Preliminary study of the impact of having such unusual glasses available for the lens designer has been made. Results indicate that significant improvements can be realized over the use of only conventional glasses. A cursory survey of a number of nationally recognized lens designers indicated a general agreement that such glasses would be highly desired and could be expected to lead to completely new designs as well as simplifying existing designs.

Reluctant Glass Formers and Their Applications in Optical Lens Design

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The art of optical lens system design is very old. Over a hundred years ago flint glasses (lead-alkali-silicates) were discovered to correct the color aberration of the crown glasses (soda-lime-silicates) making the first achromatic color corrected multi-element lens systems possible. Over the following years demands for better quality lenses led to a multitude of glass compositions with unique combinations of index of refraction and dispersion (inverse of the Abbe number) that have made possible the increasing complicated optical systems.

The performance of optical lenses is highly dependent upon the specific selection of glasses incorporated into the design and the properties of these glasses. Over the years, lens designers have observed that the image quality of most lens configurations significantly benefit from the use of glasses with high refractive index. The difficulty experienced by designers is that the dispersion (variation of refractive index with wavelength) of such glasses rapidly increases as the refractive index increases. Although color correction of lenses is accomplished by using combination glasses having both low and high dispersive characteristics, the difference in refractive index at the reference wavelength often cannot be very great without adversely impacting the potential state of correction of the optical aberrations. In order to exploit the anticipated advantages of using high refractive index glasses, it appears to be generally necessary for high refractive index, low dispersion glasses to be available. The technically refraction plotted vs. Abbe number. New compositions with properties up and to the left with high index of refraction and low dispersion are highly desirable. Moreover, for good correction of secondary chromatic aberration, it is often necessary to have glasses available with anomalous dispersion (non-normal partial dispersion).

Over a decade ago Ralph Happe of Rockwell International advanced the science of optical glasses by demonstrating that by containerless processing, small samples of refractory reluctant glass formers could be produced from a large number of nontraditional glass formers. These glasses with high index of refraction and low dispersion could be produced from a number of pure refractory oxides (including the lanthandes) and from eutectics formed from two or more of the oxides. This was done by laser spin melting appropriate compositions of the oxides which solidified in the short free fall time as amorphous small spheres of glass. The small size of the samples limits the determination of the index of refraction of the material to sufficient accuracy to determine reliable dispersion values but a few compositions, however, were found to have both high index of refraction and low dispersion residing the currently forbidden but desirable region of the optical property diagram. Based on the expected values for index of refraction and dispersion it was proposed that new glasses from regions 1,2 and 3 could be mixed to produce virtually any intermediate property between the three new property regions. Containerless processing makes it possible to minimize surface heterogeneous nucleation and produce larger glass samples of the materials than by other methods. The use of proposed high temperature containerless processing facilities in space will permit the fabrication of benchmark samples of new unique glass compositions for optical property determination as well as for glass formation studies.

First results from a preliminary study of the impact of having such glasses available has been

made through the study of several simple and common lens configurations. The result of the computer modeling of these lenses showed impressive enhancement of the optical performance. For example, a Tessar lens was first optimized using four conventional glasses and then redesigned by substituting two "new" glasses in the design. Transverse ray aberrations on-axis and at three off-axis field angles for both the conventional design and the "new" design. The improvement in image quality is evident. Further study of the use of "new" glasses in lens design is being directed towards the determination of specifications of specific optical properties for these glasses that can serve as engineering goals for the materials scientist. A cursory survey of a number of nationally recognized lens designers indicated a general agreement that such glasses would be highly desirable and could be expected to lead to completely new designs as well as simplify existing designs.

Production of these new reluctant refractory glasses could be made possible utilizing containerless processing to prevent the contamination of th high temperature melt but more importantly by the suppression of heterogeneous surface nucleation of crystallization during cooling. If heterogeneous nucleation could be minimized, homogeneous nucleation would not occur till much lower temperatures where the systems would be much less susceptible to crystallization due to the increased viscosity of the melt. Critical cooling rates to form glass could be much smaller than with samples cooled in a mold in contact with a container wall. Glasses that are virtually impossible to form in the confined conditions (in lg of Earth) could be possible to form under containerless conditions (low g of space).

A high temperature containerless processing furnace is necessary in order to investigate these new types of optical glasses. One of the authors (EE) has investigated terrestrial containerless processing methods for the past 13 years. Each method has limitations that restrict the size or type of samples that can be processed. None of the methods has proven useful for containerless processing samples large enough to make the necessary optical property measurements to sufficient accuracy.

Due to the upper temperature limits of existing low gravity containerless processing facilities (1475°C in Single Axis Acoustic Levitator SAAL). The Gallia Calcia system was selected as a low melting (1323°C) model of these refractory reluctant glass formers. It has optical properties within the known region of the diagram but is a "low temperature model" for the new glass systems. It is a reluctant glass former that is without the traditional glass formers. Gallia-Calcia may have unique partial dispersion characteristics. From the limited optical property measurements of this material there is an indication that there may be analmous dispersion in this new glass system which implies more fundamental differences in the partial dispersion of this glass compared with the closest commercial glass. Virtually all glasses from the traditional glass formers are concave up. With Gallia-Calcia the curve is concave down. The new families of noncross bred glasses offer possibilities for unique partial dispersion characteristics which could correct the secondary spectrum.

Of the several hundred compositions of potential refractory reluctant glass formers that have been investigated to date very few compositions can be processed in the existing SAAL. Using one potential system as an example, the niobium calcium titanate ternary has a reasonably large glass formation region through the diagram as determined by laser spin melted samples. Only two small regions are molten below 1500°C.

The high temperature requirement for containerless processing is further increased due to the fact that the melts of these materials must be superheated in order to achieve optimal undercooling conditions. It is expected that the melt should be heated at least 200°C above the thermodynamic melting point to minimize nucleation upon cooling. Obviously the superheat required in order to optimize undercooling would have to be determined for each system.

High temperature containerless processing of non-electrical-conducting melts is required for further development of unique optical glasses. Samples on the order of 5 mm to 1 cm in size are required for optical property measurements. Cooling rates need to be very fast in order to avoid nucleation and crystallization. Few (if any) meaningful experiments can be done with a furnace capability below 1500°C. Temperature capabilities to 2000°C is a minimum requirement for such a furnace with 2200°C being a goal. If the production of these glasses can be proven possible, a revolution in optical glasses could result in completely new optical lens systems.

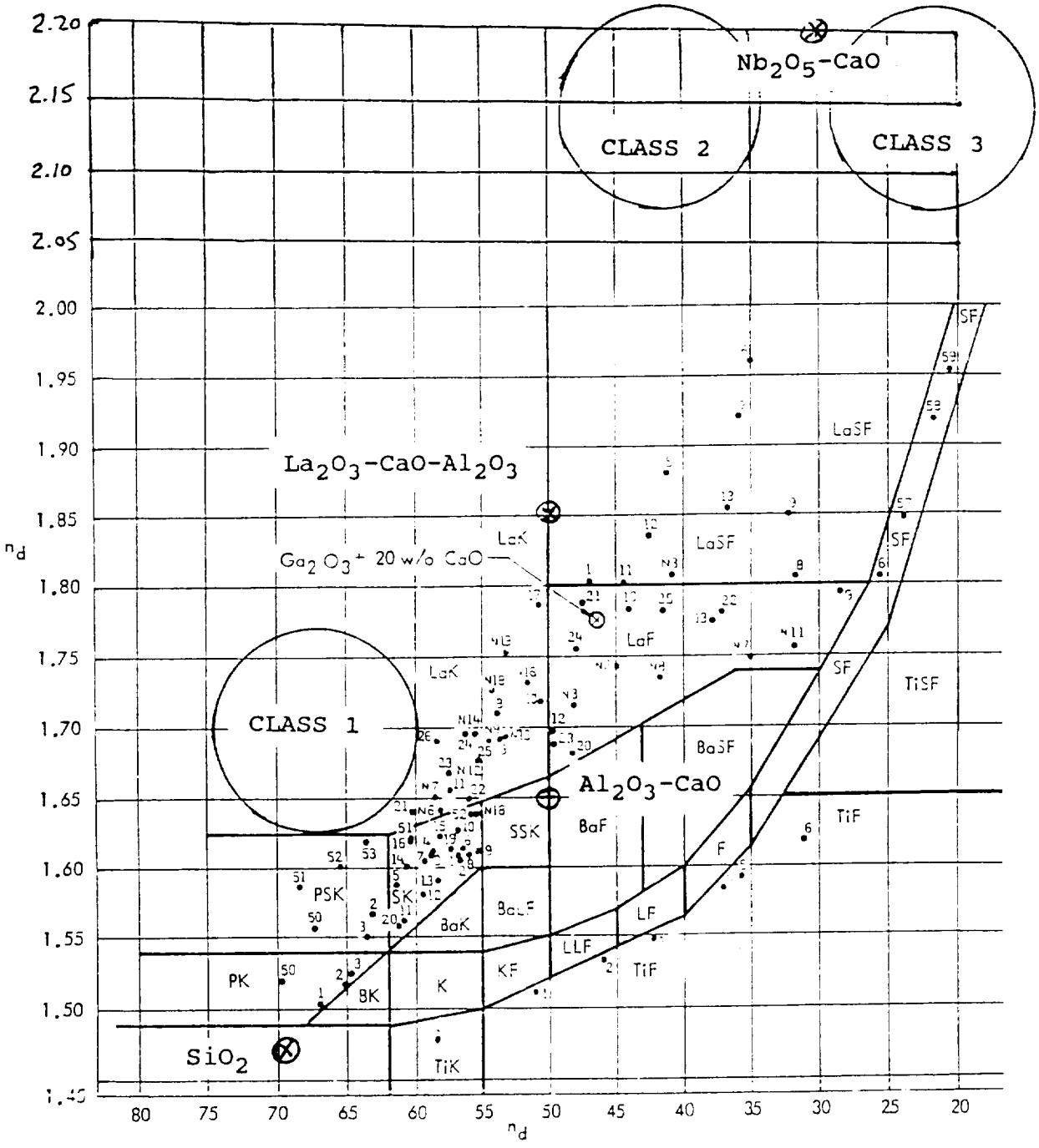
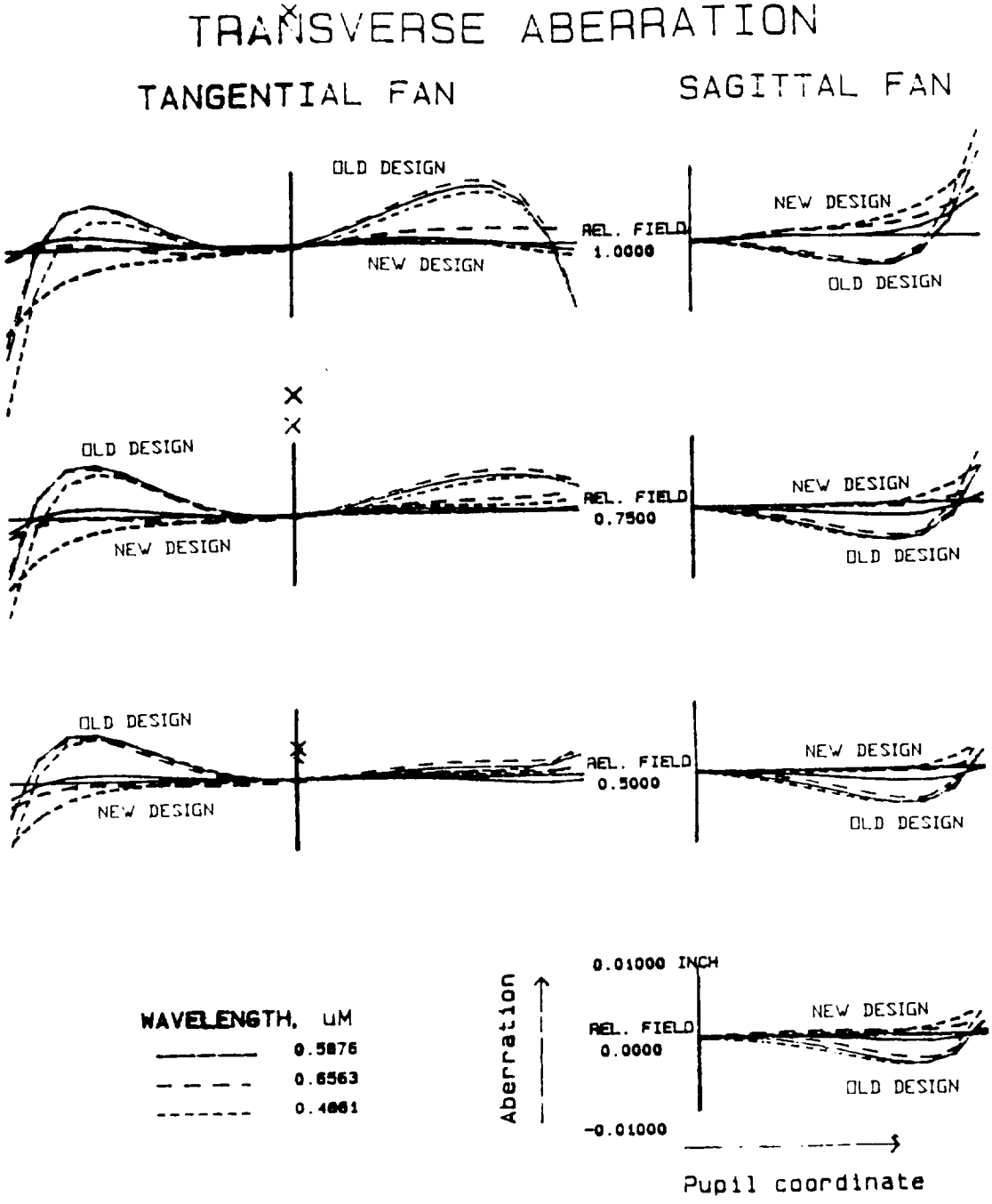


Figure 5. Diagram of Optical Glasses

TRANSVERSE ABERRATION



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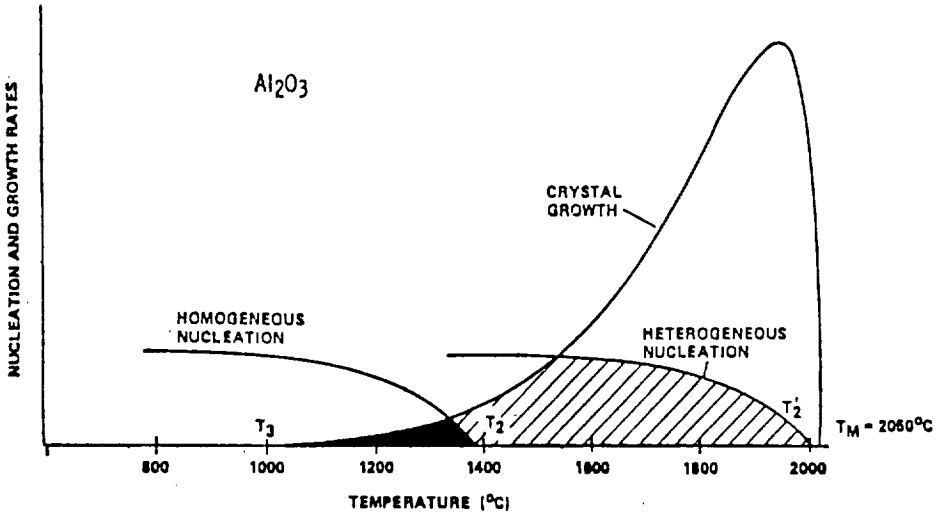
Al_2O_3
NUCLEATION AND GROWTH RATES
CRYSTAL GROWTH
HOMOGENEOUS NUCLEATION
HETEROGENEOUS NUCLEATION
T_3
T_2
T_2'
T_M = 2050°C
800
1000
1200
1400
1600
1800
2000
TEMPERATURE (°C)

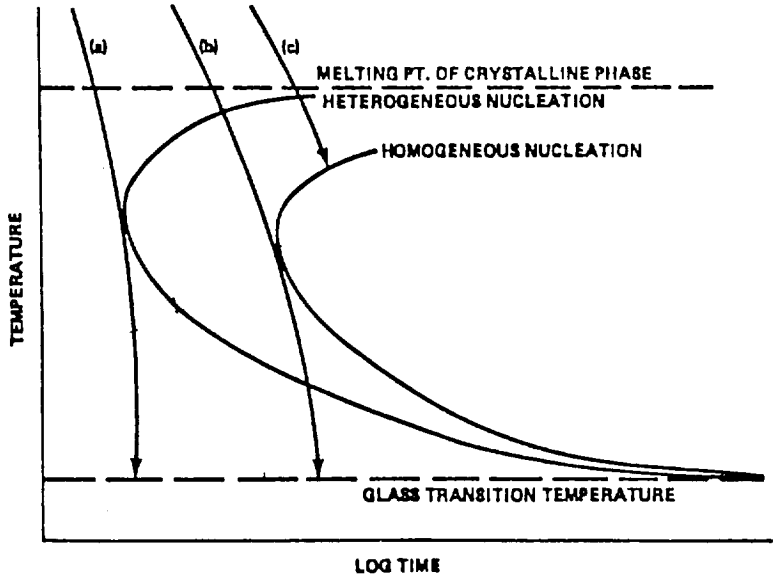
(a)
(b)
(c)
MELTING PT. OF CRYSTALLINE PHASE
HETEROGENEOUS NUCLEATION
HOMOGENEOUS NUCLEATION
GLASS TRANSITION TEMPERATURE
TEMPERATURE
LOG TIME

TABLE I

GROUND-BASED PROCESSING EXPERIMENTS

METHOD	TEMPERATURE LIMIT	COOLING RATE °C/Sec	ADVANTAGES	DISADVANTAGES	EXPERIMENTS
Short Drops Laser Spin Melting	2000°C	1000	Containerless Solidification	No temperature data Very fast quenching Very Small Samples	Refractory rare Earth oxides with alkaline Earth oxides
Laser Drop Melting	2000°C	100 - 500	Large Samples Most oxides	Uncertain quench rate Temperature unknown Large thermal gradients	Ga2O3-CaO, Al2O3-SiO2
Long Drop Tube	1800°C	250 - 1000	Containerless Solidification	No temperature data Refractories Difficult	None
Air Jet Levitation/	2000°C	0.1 - 250	Containerless Solidification	Unstable Levitation High Surface Tension	Molten Al2O3, Al2O3-SiO2 Glass, Glass Microspheres
Acoustic Levitation/ CO2 Laser Melting	1000°C	0.1 - 250	Containerless Solidification	Very Unstable Not reproducable	Recrystallized Amorphous Al2O3-SiO2 at 950°C
Thermocouple/ Focused Heating	1750°C	0.1 - 1000	Good Temperature Data	Not containerless Heterogeneous Nucl. Temperature Limits	Ga2O3-CaO, Ga2O3-CaO-SiO2, Al2O3-CaO

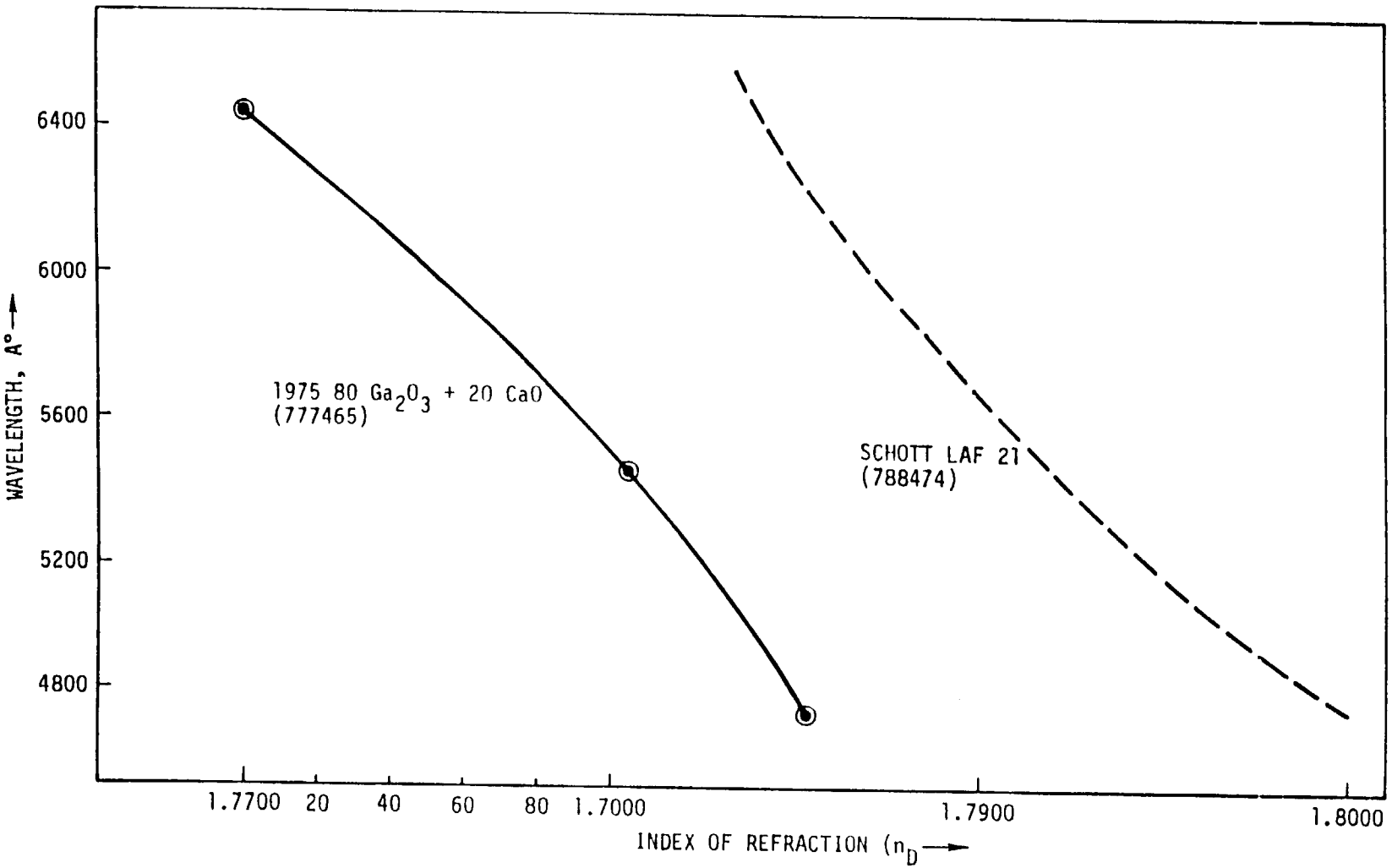

Figure 6. Wavelength Versus Index of Refraction for Ga_2O_3 - CaO Glass and Schott LaF 21 Glass

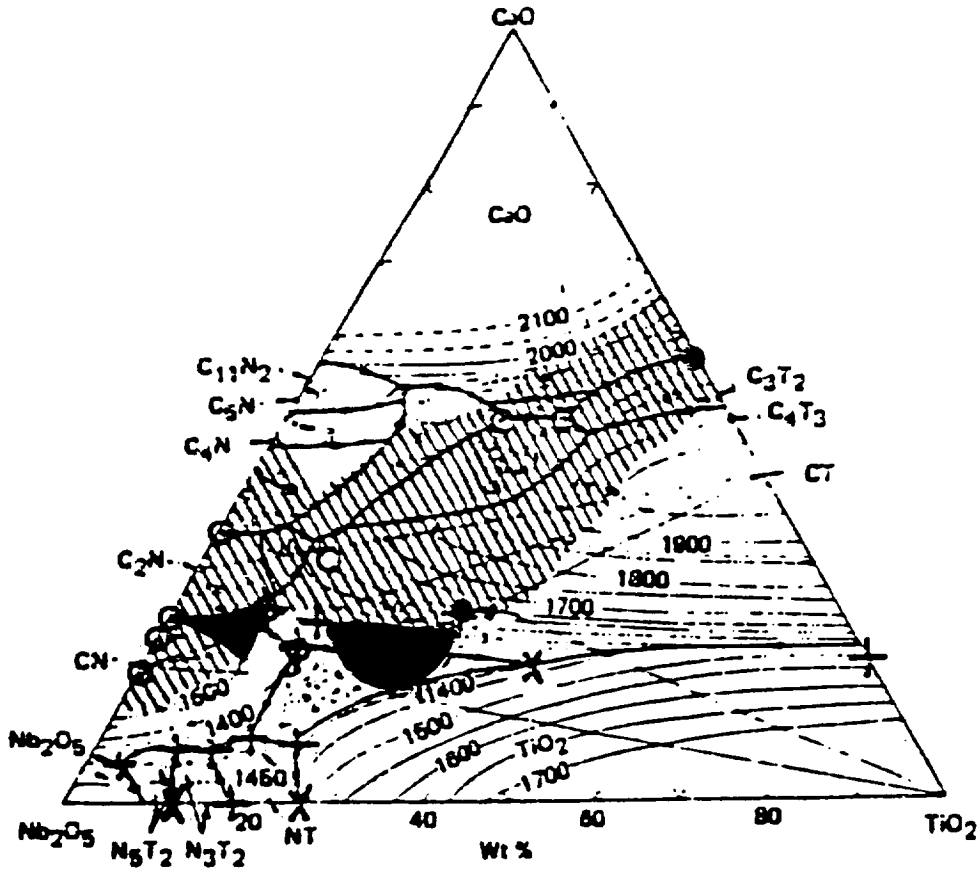

Fig. 5 Glass Formation Region in the Nb_2O_5-TiO_2-CaO System (Phase Diagram in Ref.10) \\\\

Glass Formation Region in the Above System with T_m < 1500°C

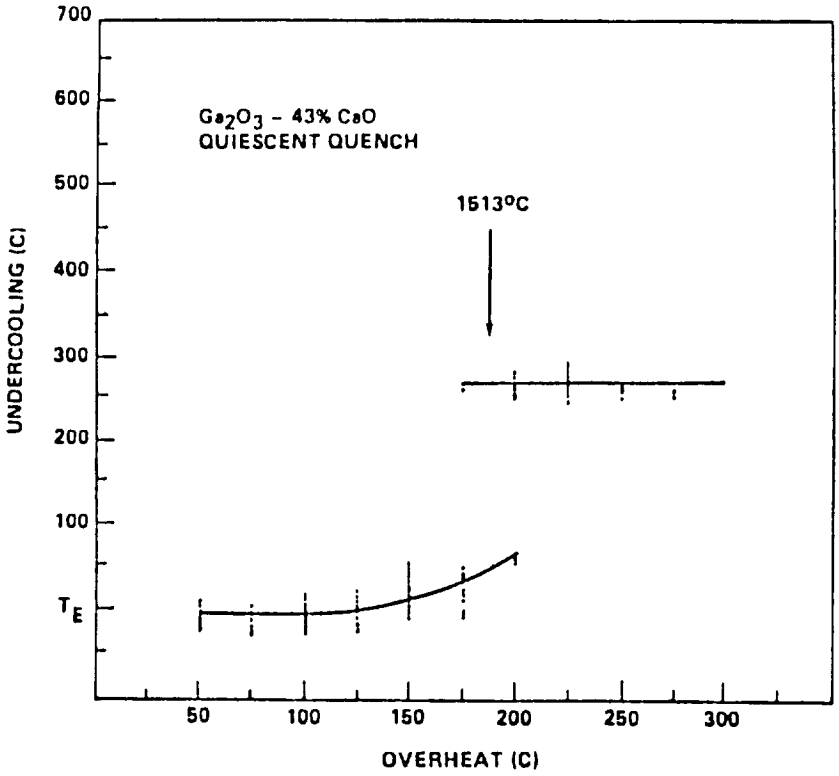

Fig. 4. Plot of undercooling vs overheat for quiescently quenched samples.

N91-21352

Containerless Protein Crystal Growth Technology: Electrostatic Multidrop Positioner

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The rationale behind the containerless protein crystal growth method is to provide a simple and clean environment for protein crystal growth in space. The method is simple in that freely floating liquid drops form spherical shapes as a result of their own surface tension and, therefore, expose themselves to isotropic thermal as well as vapor diffusion fields. These simple shapes enable accurate drop-sizing that can be continuously monitored and controlled in a programmed way to achieve an optimal protein-saturation level for crystallization.

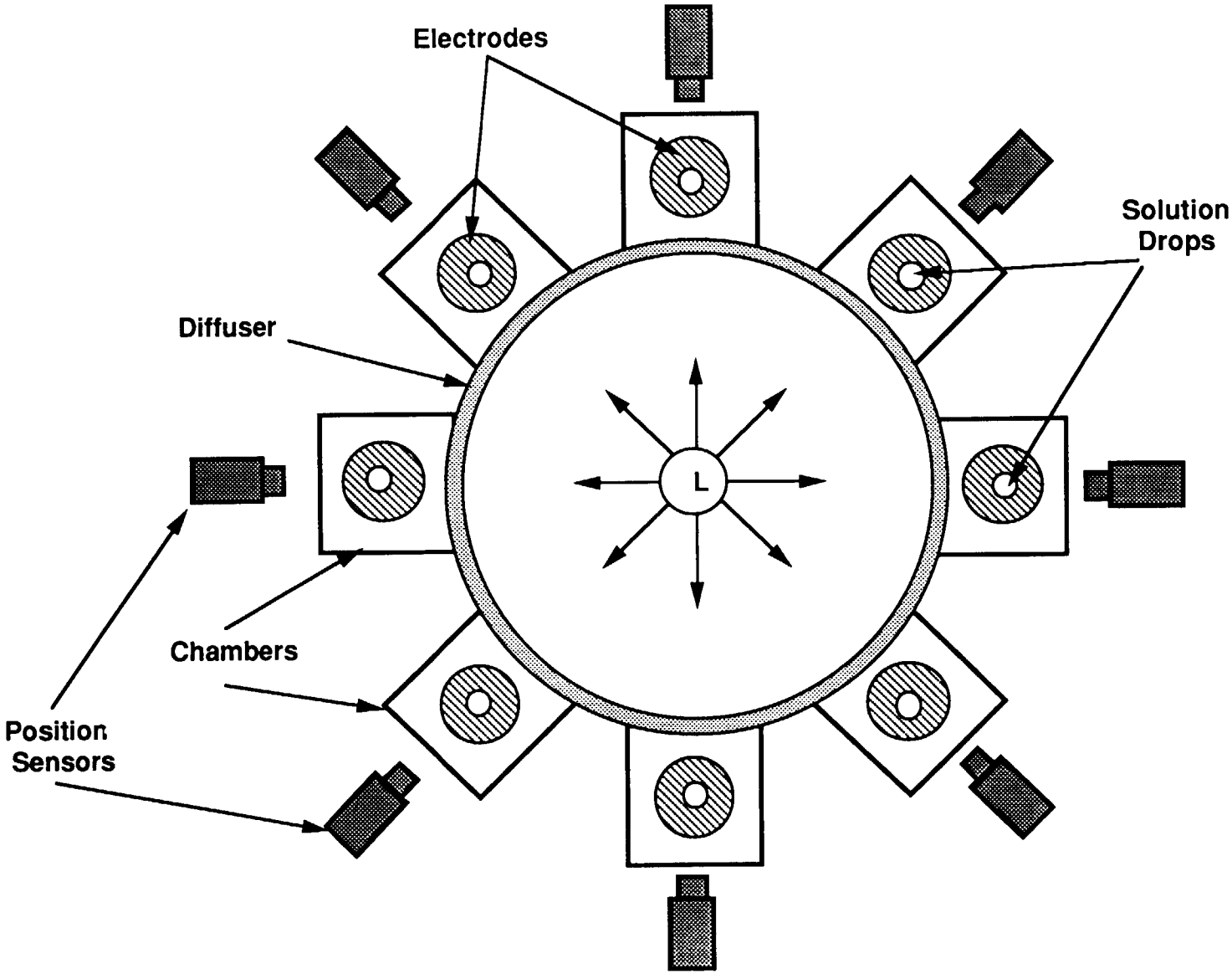
The containerless method is clean in two different ways. First, the sample is not in physical contact with container walls that might induce uncontrollable nucleation. Second, through simple programming of control forces, the sample drops can be isolated from much of the oscillatory and impulsive forces that are known to reside in space laboratories. With the perturbing forces filtered out, the drops will experience a true micro-g environment. Furthermore, with the drops freed from undesirable container walls and disturbing forces, parameters recorded in the course of the experiments will have good correlations with the final results; this capability will even allow investigators to dictate the course of the experiments interactively toward the growth of large, high-quality protein crystals. Because there will be virtually no limitation in drop size or in producing compound drops that will initially have a well-defined interface between two different liquids, the containerless method will be able to accommodate most conventional methods—such as the vapor diffusion method, the temperature control method, or even the liquid-liquid interfacial diffusion method—all in the same drop positioning system.

In this presentation, the electrostatic multidrop positioner will be described for its present capabilities, limitations, and future prospects as an advanced facility for protein growth in space. This presentation will include 1) the general principle of electrostatic positioners, 2) the architecture of multidrop positioners, 3) the drop-launching and collecting method, 4) the drop-sizing method, 5) a method for controlling protein-saturation levels, 6) the optical detection of the onset of nucleation, 7) the vibration isolation of levitated drops, and 8) the drop-manipulation capabilities. A demonstration of a four-drop levitator is presented during the workshop.

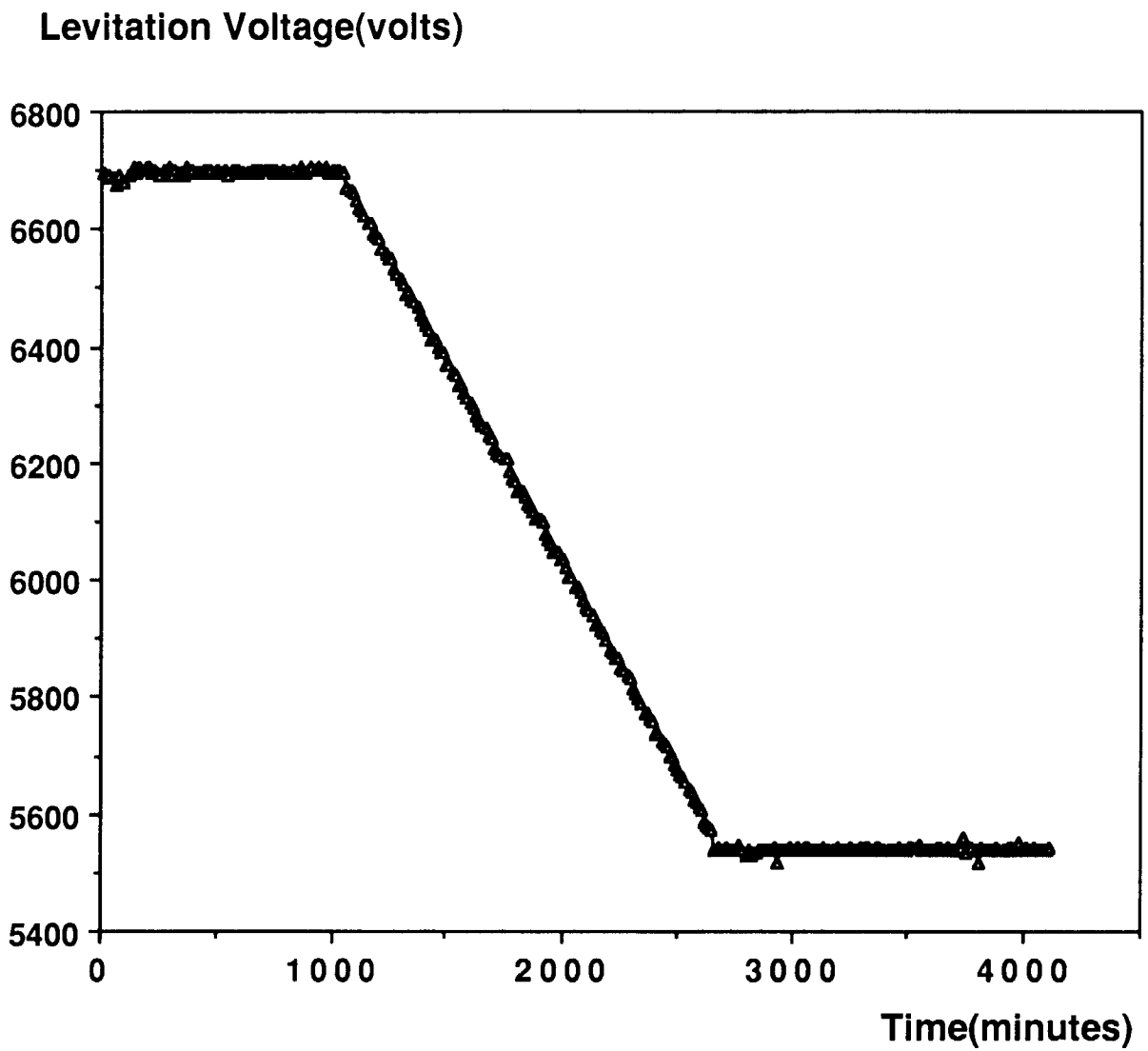
Containerless Protein Crystal Growth Method

- Removes container induced nucleations
- Vibration isolation
- Accurate drop sizing from spherical drops
- Isotropic temperature and vapor diffusion field
- Wide range in drop size (10 ~ 1000 micro-liter)
- Multi-drop positioning capability (~ 12 drops or more)
- Protein crystal growth in the actively controlled environments
  - Controlled vapor diffusion method
  - Temperature controlled method
  - Liquid-liquid interfacial diffusion method

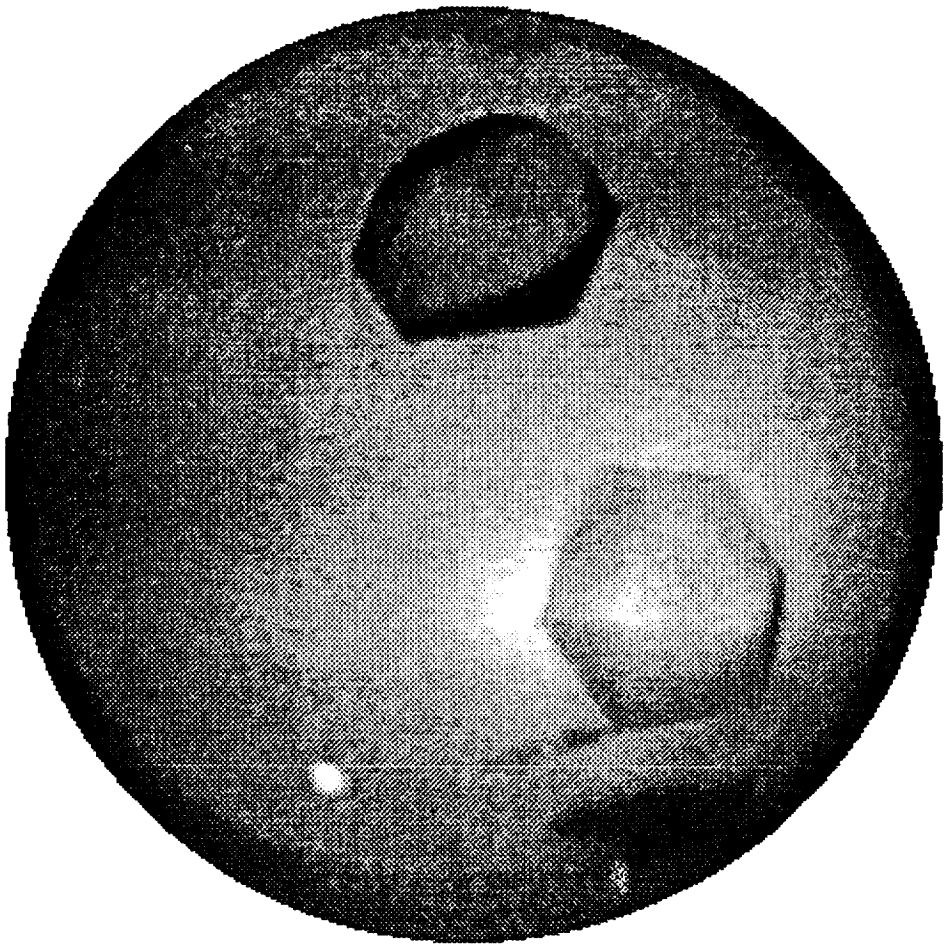
Electrostatic Multi-Drop Positioner for Protein Crystal Growth Experiments



Controlled Protein Concentration in a Levitated Drop



Lysozyme Crystals Growing in a Drop



N91-21353

A Systematic Investigation of the Preparation and Properties of Composite Carbon Molecular Sieves Containing Inorganic Oxides

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Newark, Delaware 19711

The objective of this research is to define the methodology for the preparation and characterization of new carbon-based molecular sieves with composite structures. Carbon molecular sieves have found increasing application in the field of separation and purification of gases. These materials are relatively easy to prepare and their surfaces can be modified to some extent. It is expected that by combining inorganic oxides with the carbonaceous structure one can begin to design composite materials with a wider range of possible chemical and physical properties. In this way the IOM-CMS materials may confer distinct advantages over pure carbon molecular sieves, not just for separation, but also for catalysis. Our most recent results in the design and characterization of these IOM-CMS materials will be reviewed and summarized. Directions for further research will also be explored.

N91-21354

Supercritical Microgravity Droplet Vaporization

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University of Wisconsin-Madison

Supercritical droplet vaporization is an important issue in many combustion systems, such as liquid fueled rockets and compression-ignition (diesel) engines. In order to study the details of droplet behavior at these conditions an experiment has been designed to provide a gas phase environment which is above the critical pressure and above the critical temperature of a single liquid droplet. In general the droplet will begin as a cold droplet in the hot, high pressure environment. In order to eliminate disruptions to the droplet by convective motion in the gas, forced and natural convection gas motion are required to be small. Implementation of this requirement for forced convection is straightforward, while reduction of natural convection is achieved by reduction in the g-level for the experiment. The resulting experiment consists of a rig which can be stably position a droplet without restraint in a high-pressure, high temperature gas field in microgravity.

The microgravity field is currently achieved by dropping the device in the NASA Lewis 2.2 second drop tower. The performance of the experimental device and results to date will be presented.

N91-21355

Using a Microgravity Environment to Probe Wave Turbulence

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and

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Pasadena, CA 91109

The experimental key to observing stochasticity or turbulence in a distribution of interacting propagating waves is (a) the achievement of high amplitude and (b) the use of a medium with a large coefficient of nonlinearity. Our research indicates that capillary waves are the best means of observing this phenomenon, however gravitational modifications of the capillary wave dispersion law greatly reduce (b). Thus we intend to search for wave turbulence in a large drop of fluid that is positioned in a microgravity environment. Capillary waves that run around the surface of the drop will be excited and their power spectrum and higher order correlations will be analyzed for wave turbulence. Our theoretical calculations indicate that modulations of the power spectrum should propagate as second sound waves. These issues have consequences for signal processing and plasma confinement.

Turbulence

- reversible nonlinear processes beat out linear transport.
- Density of states >> nonlinear rollover time

Vortex: $\vec{\nabla} \times \vec{v} \neq 0, \vec{\nabla} \cdot \vec{v} = 0$

- Stirred liquids; Kolmogorov

Wave: Dispersion law

$\vec{v} = \vec{v}' \exp(i\mathbf{k}\cdot\mathbf{r} - i\omega t)$

- Sound waves
- Surface g waves
- Alfven waves
- Capillary waves
- SAW = Love/Rayleigh waves
- Flexing waves (e.g. gongs)

Wave Turbulence: A. Larraza, P.H. Roberts. Possible experiments being considered by S. Garrett, Gary Williams, M. Barmatz

Note: No controlled lab experiments on either fully developed, wave or vortex turbulence.

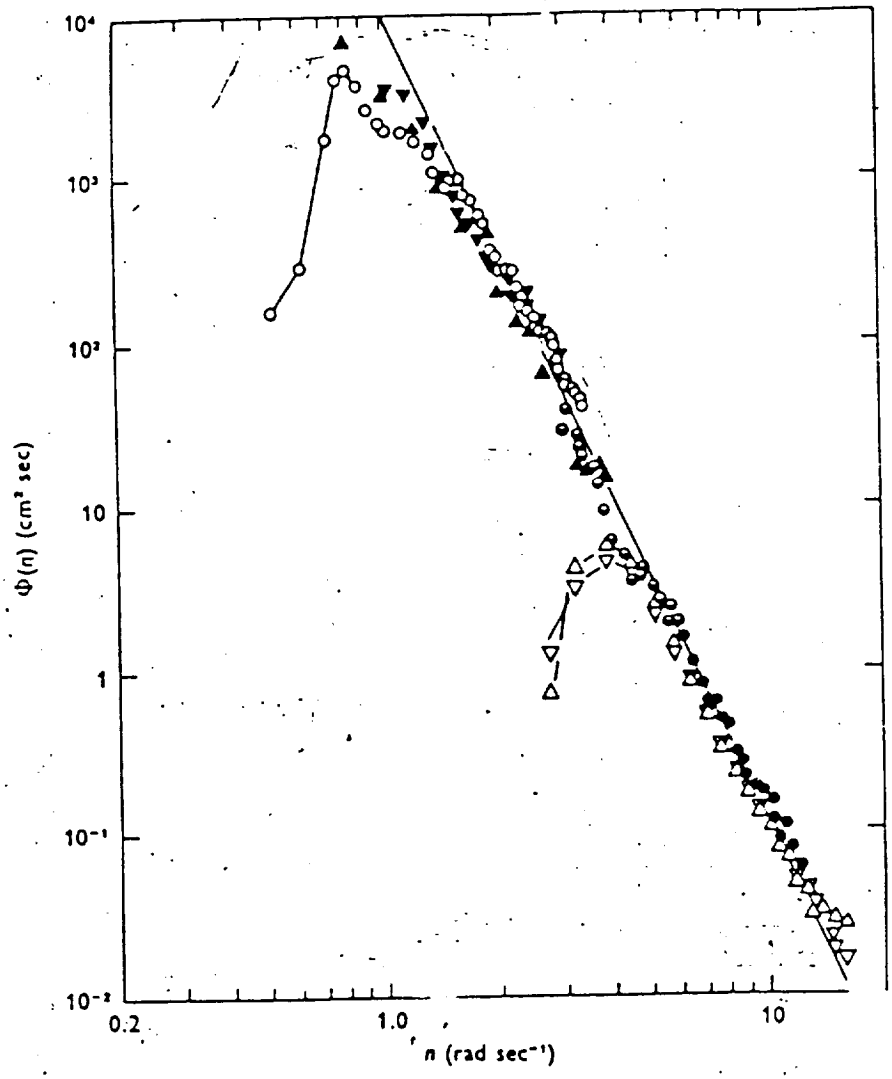


Fig. 4.8. The equilibrium range of the frequency spectrum of wind-generated waves. The logarithmic vertical scale covers six decades. The shape of the spectral peak is included in only three cases; otherwise only the saturated part of each spectrum is shown. Measurements by:

○	Stereo-Wave Observation Project (Pierson, 1962)	Floating wave spar	1 spectrum
▲	Longuet-Higgins *et al.* (1963)	Accelerometer	1 spectrum
▼	DeLeonibus (1963)	Inverted fathometer	Mean of 6 spectra
△	Kinsman (1960) November series	Capacitance probe	Mean of 16
▽	Kinsman (1960), July series	Capacitance probe	Mean of 16
●	Burling (1959)	Capacitance probe	Mean of 11
⊖	Walden (1963)	Probe and cinematograph	1 spectrum

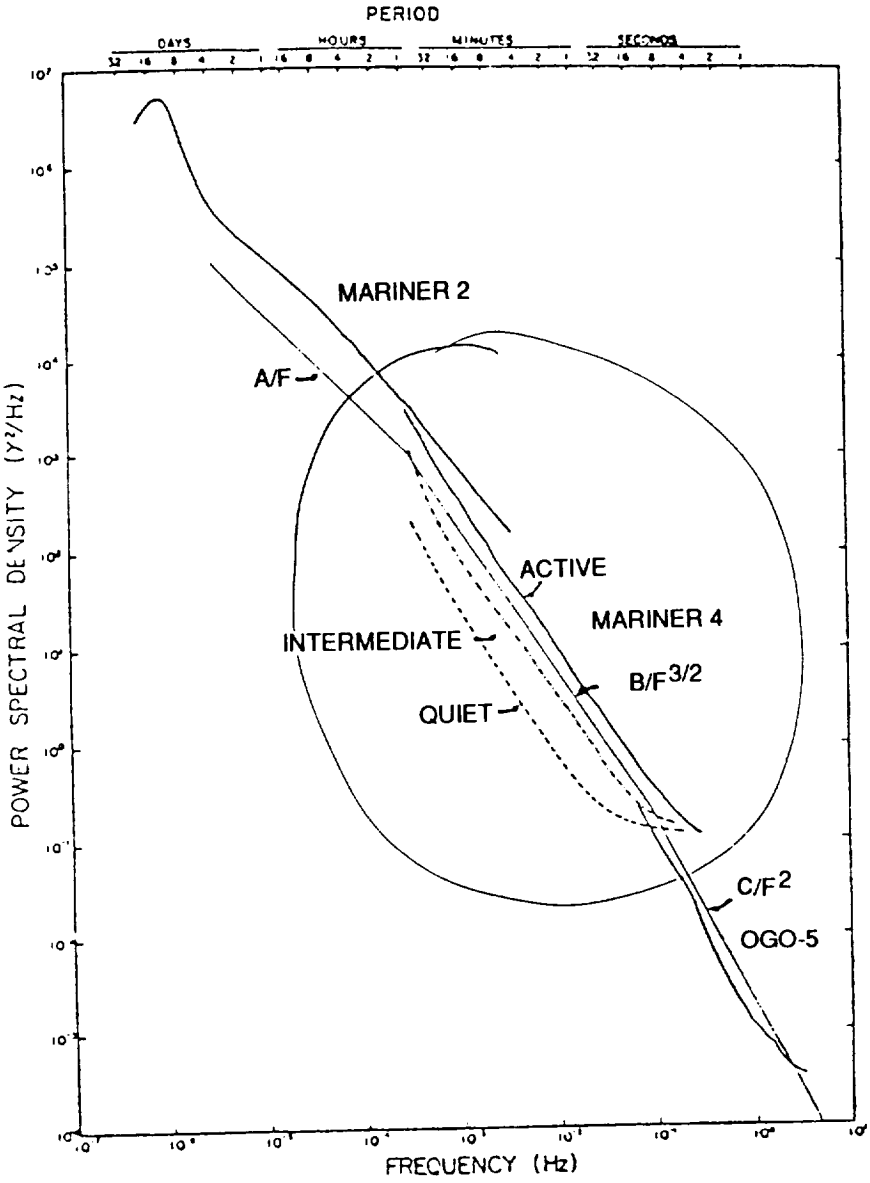


Figure 8. A composite spectrum of the radial component of the interplanetary magnetic field as observed on Mariner 2 [Coleman, 1968], on Mariner 4 [Siscoe et al., 1968], and on OSO - 5. Three spectra illustrating the range of variability of the interplanetary spectrum are shown for Mariner 4. Since the Mariner 2 data are consistently higher than the Mariner 4 data in the overlapping range of frequencies, it is assumed that the Mariner 2 data were obtained during an usually disturbed period of time and the typical spectrum has lower power. Three straight line segments have been drawn with slopes of - 1, - 1.5, - 2 to roughly represent the expected average spectrum near 1 AU.

Capillary Waves

$$\left[\frac{\partial e(\omega)}{\partial y}\right]_{+} \approx \frac{G^2\omega^2}{\sigma}[e\omega]^2 + \nu k^2 e(\omega)$$

= rate at which erg/cm^2 leave ω due to nonlinearity and damping

- σ = surface tension
- ν = kinematic viscosity

When nonlinearities dominate

$$e(\omega) \cong \left[\frac{q}{G^2}\right]^{1/2} \frac{1}{\omega^{3/2}}$$

Dispersion Law

$$\omega^2 = gk + \frac{\sigma}{\rho} k^3$$

Quality Factor

$$Q_\omega = \frac{1}{2\nu}\left[\frac{\sigma\lambda}{2\pi\rho}\right]^{1/2}$$

Mach # = ζ/λ ; ζ = displacement amplitude

Turbulence $\Leftrightarrow$

$$M_\omega^2 >> \frac{1}{Q_\omega G^2}$$

If ν irrelevant then classical system far off equilibrium has 2nd sound

Why low g?

- Spherical drop
- Large drop

  1.mm vs 4. cm.

- Large wavelengths $\Leftrightarrow$

  low damping

Key requirement $$gk < \frac{\sigma}{\rho} k^3$$

σ = surface tension

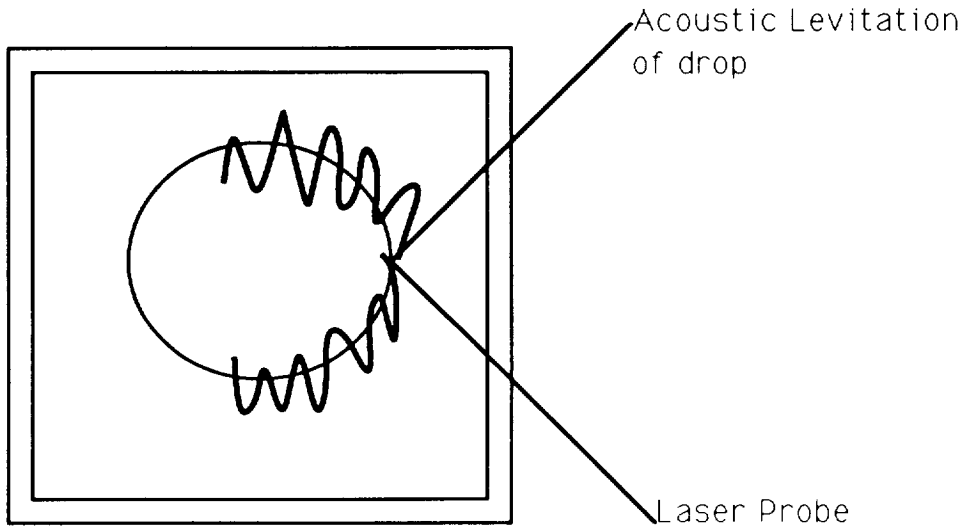
ρ = density

$2\pi / k$ = wavelength

WHY USE CAPILLARY WAVES TO STUDY TURBULENCE?

USE A DROP:

CLOSED SPHERICAL RESONATOR
LARGE MACH NUMBERS ARE POSSBILE COEFF. OF NONLINIARITY IS HUGE



Why do experiments on wave turbulence?

1	Universal power spectra
2	Higher order correlations
3	Some reasonable theory exists
1+ 2 +3	Signal Processing
4	Transition from weak to strong nonlinear effects
5	Second Sound-elasticity of turbulence - controlled fusion

He^4 $\chi \equiv \frac{K}{e} \sim 10^{-4} \frac{cm^2}{sec}$ Normal

$\chi \equiv \frac{K}{e} \frac{d^2c^2}{\nu} \sim 10^{12} \frac{cm^2}{sec}$ 2nd Sound

ν	=	kinematic viscosity
c	=	geometry
x	=	thermal diffusivity

N91-21356

Drop Evaporation in a Single-axis Acoustic Levitator

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A. P. Croonquist
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Pasadena, California

A 20 kHz single-axis acoustic positioner is used to levitate aqueous-solution drops (volumes $\lesssim$ 100 micro-liters). Drop evaporation rates are measured under ambient, isothermal conditions for different relative humidities.

Acoustic convection around the levitated sample enhances the mass loss over that due to natural convection and diffusion. A theoretical treatment of the mass flow is developed in analogy to previous studies of the heat transfer from a sphere in an acoustic field.

Predictions of the enhanced mass loss, in the form of Nusselt (Sherwood) numbers, are compared with observed rates of drop shrinking.

The work is part of an ESA study on crystal growth from levitated solution drops.

DROP EVAPORATION IN A SINGLE-AXIS ACOUSTIC LEVITATOR

APPLICATION BACKGROUND: (LABORATORY AND MICRO-G CONDITIONS)

- ESA STUDY ON CRYSTAL GROWTH FROM LEVITATED-SOLUTION DROPS
- LARGER AND BETTER SINGLE CRYSTALS IN CONTAINERLESS PROCESSING

SAMPLE AND ENVIRONMENTAL CONDITIONS:

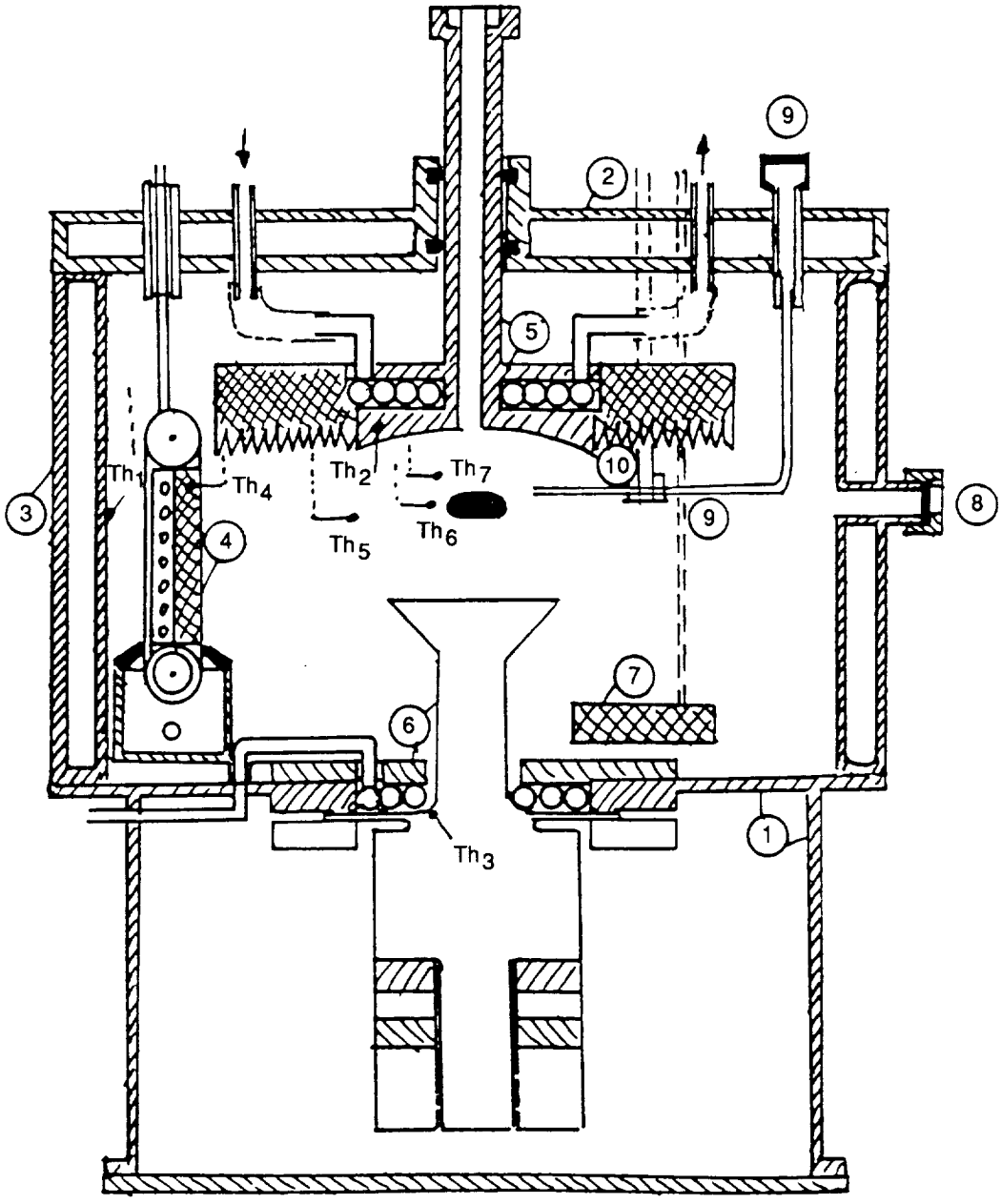
- WATER SOLUTIONS OF INORGANIC AND ORGANIC MATERIALS (PROTEINS), ALSO OTHER SOLVENTS
- DROP SIZE: 10 ul < V < 100ul (2.5mm < d < 6mm)
- ENVIRONMENT: AIR AT AMBIENT PRESSURE (1 atm)
  TEMPERATURE: (0 C) 4 C < T < 40 C (70 C)
  RELATIVE HUMIDITY: 0 < h_r < 100%
- SOUND PRESSURE LEVEL (FOR 1-G): 160 < SPL < 165 dB

HARDWARE FOR EXPERIMENTS:

- SINGLE-AXIS ACOUSTIC STANDING WAVE LEVITATOR (21 kHz)
- ISOTHERMAL PROCESSING CHAMBER (T = +/- 0.1 K)
- HUMIDIFIER AND HUMIDITY SENSOR
- CCD CAMERA FOR DROP OBSERVATION AND MONITORING
- SOPHISTICATED OPTICS FOR VISUALIZATION OF STREAMING INSIDE AND OUTSIDE OF THE LEVITATED DROP
- STERILE DROP DEPLOYMENT AND EXTRACTION

Fig. 1 Processing chamber

1 Basic flange and housing
2 Top flange (cf Fig. 5)
3 Glass cylinder
4 Humidifier (cf Fig. 4)
5 Reflector assembly (cf Fig. 2)
6 Transducer assembly (cf Fig. 3)
7 Humidity sensor
8 Side opening with septum
9 Sample injector
10 Feeding tube/Manipulator



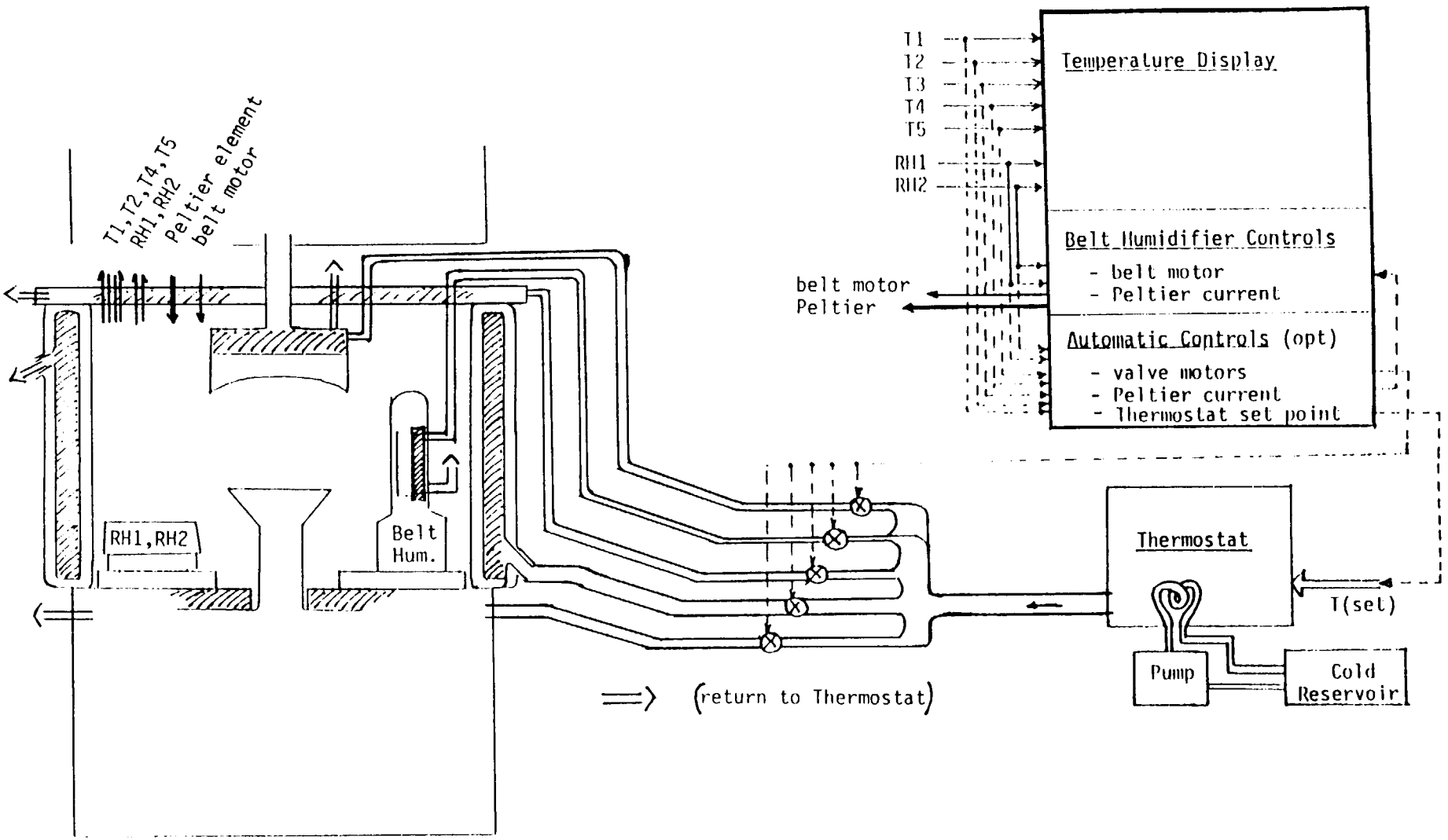


Figure 6. Thermal Control Schematic

DROP EVAPORATION IN A SINGLE-AXIS ACOUSTIC LEVITATOR

EQUATION OF MASS FLOW:

$$\frac{dm}{dt} = -\pi d_s^2 \beta \frac{\rho_v}{R_v T}(1-h_r) \qquad (1)$$

$$\beta = Nu\frac{D}{\delta} = Nu\frac{D}{d_s} = Sh\frac{D}{d_s} \qquad (2)$$

d_s - DROP DIAMETER
ρ_v - VAPOR PRESSURE OF SOLVENT
R_v - GAS CONSTANT
T - TEMPERATURE
h_r - RELATIVE HUMIDITY
D - GAS DIFFUSION CONSTANT
δ - BOUNDARY LAYER THICKNESS
Nu - NUSSELT NUMBER (TOTAL HEAT FLOW)/(CONDUCTIVE HEAT FLOW)
Sh - SHERWOOD NUMBER (TOTAL MASS FLOW)/(DIFFUSIVE MASS FLOW)

BASIC THEORY OF DROP EVAPORATION (IN ANALOGY TO HEAT FLOW)

A FREE DROP IN AN ISOTHERMAL ENVIRONMENT AT A RELATIVE HUMIDITY, $h_r < 1$, HAS SATURATED HUMIDITY ($h_r = 1$) INSIDE THE BOUNDARY LAYER. DENSITY DIFFERENCES BETWEEN THE BOUNDARY LAYER AND THE BACKGROUND RESULT IN NATURAL CONVECTION (Nu = Sh > 2) WHICH IS FURTHER ENHANCED UNDER ACOUSTIC LEVITATION CONDITIONS BY STREAMING. (FIG. 2)

EQUATION (1) LEADS TO A SIMPLE NORMALIZED EQUATION FOR THE DROP DIAMETER AS A FUNCTION OF TIME

$$\overline{d_s^2} = \frac{d_s^2}{d_{s,0}^2} = 1 - 0.37\,\bar{t} \qquad (3)$$

WITH $\bar{t} = t/t_{0.5}$, AND

$$t_{0.5} = \frac{9.25 \times 10^{-2}\, d_{s,0}^2\, R_v\, T\, \rho_s}{D\, p_v\, (1-h_r)\, Nu} \qquad (4)$$

WHERE $t_{0.5}$ IS THE TIME REQUIRED FOR A DROP TO SHRINK TO 50% OF ITS INITIAL VOLUME.

THE REFERENCE TIME, $t_{0.5}$, (EQUATION (4)) CONTAINS KNOWN TEMPERATURE DEPENDENT PROPERTIES OF THE SOLVENT. THE ONLY UNKNOWN IS THE NUSSELT (OR SHERWOOD) NUMBER.

$$t_{0.5}[h_r] \approx \frac{24}{Nu}\frac{d_{s,0}^2}{1-h_r}\left(\frac{T}{T_0}\right)^{-0.94} e^{-19.7\left(1-\frac{T_0}{T}\right)}$$

for H2O drops in air

SOLUTION DROPS

WHEN THE DROP CONTAINS A "SALT" SOLUTION, WITH CONCENTRATION c_a , AND THE PROCESSING CHAMBER CONTAINS A LIQUID RESERVOIR WITH THE SAME SOLUTION BUT A LARGER OR SMALLER CONCENTRATION, c_∞ , THAN EQUATION (1) HAS TO BE MODIFIED BECAUSE THE VAPOR PRESSURE (HUMIDITY) IN THE DROP BOUNDARY LAYER, c_a , AND IN THE ENVIRONMENT ARE REDUCED ACCORDING TO RAOULT'S LAW. (FIG.3)

THE NORMALIZED DROP DIAMETER, $\overline{d_s} = d_s / d_{s,0}$, AS A FUNCTION OF THE NORMALIZED TIME IS GIVEN BY

$$2\frac{\overline{d_s}\, d(\overline{d_s})}{1 - c_a \overline{d_s}^3} = -0.37\, \frac{1 - h_r c_\infty}{1 - h_{r,0}}\, d\bar{t} \qquad (5)$$

WITH $h_r(c_\infty)$ BEING THE RELATIVE HUMIDITY OF THE "SALTY" BACKGROUND. FIG. 5 SHOWS THE SHRINKING CURVES FOR DIFFERENT CONCENTRATION RATIOS, c_a / c_∞, AND INDICATES LIMITED DROP SHRINKING FOR $c_a / c_\infty < 1$ AND LIMITED DROP GROWTH FOR $c_a / c_\infty > 1$ RESULTING FROM LIMITED CONCENTRATION CHANGES INSIDE THE DROP.

$$\frac{\dot{t}_{0.5}}{[h_r]} \approx \frac{24}{Nu} \frac{d_{s,0}^2}{1 - \dot{h}_r} \left(\frac{T}{T_0}\right)^{-0.94} e^{-19.7\left(1 - \frac{T_0}{T}\right)}$$

$$1 - \dot{h}_r \approx c_{s,\infty} \quad \text{cf fig. 3}$$

NUSSELT NUMBER MODEL

BECAUSE OF THE ANALOGY BETWEEN HEAT FLOW AND MASS FLOW OF A LEVITATED SAMPLE, WE CAN REFER TO THE EXTENSIVE THERMAL INVESTIGATIONS BY C.P. LEE AND T. WANG [1] AND BY E. LEUNG [2]. THESE AUTHORS FOUND AN EXPERIMENTALLY PROVEN CONNECTION BETWEEN THE NUSSELT NUMBER, Nu, THE GRASHOF NUMBER, Gr, (THE RATIO OF BUOYANCY AND VISCOSITY EFFECTS) AND THE EFFECTIVE REYNOLDS NUMBER, Re, OF A LEVITATED SAMPLE RESULTING FROM FORCED CONVECTION IN THE ENVIRONMENT ARROUND THE SAMPLE. (FIG. 5)

FOR RELATIVELY LARGE SPL AND RESULTING REYNOLDS NUMBERS, E. LEUNG FOUND AS A GOOD APPROXIMATION FOR THE HEAT FLOW (FIG. 6),
FOR $10 < Re < 50$

$$Nu = 2 + e^A . Gr^B$$

WITH $A = -0.72 + 0.46 \ln(1 + Re)$
AND $B = 0.25 - 0.015 \ln(1 + Re)$.

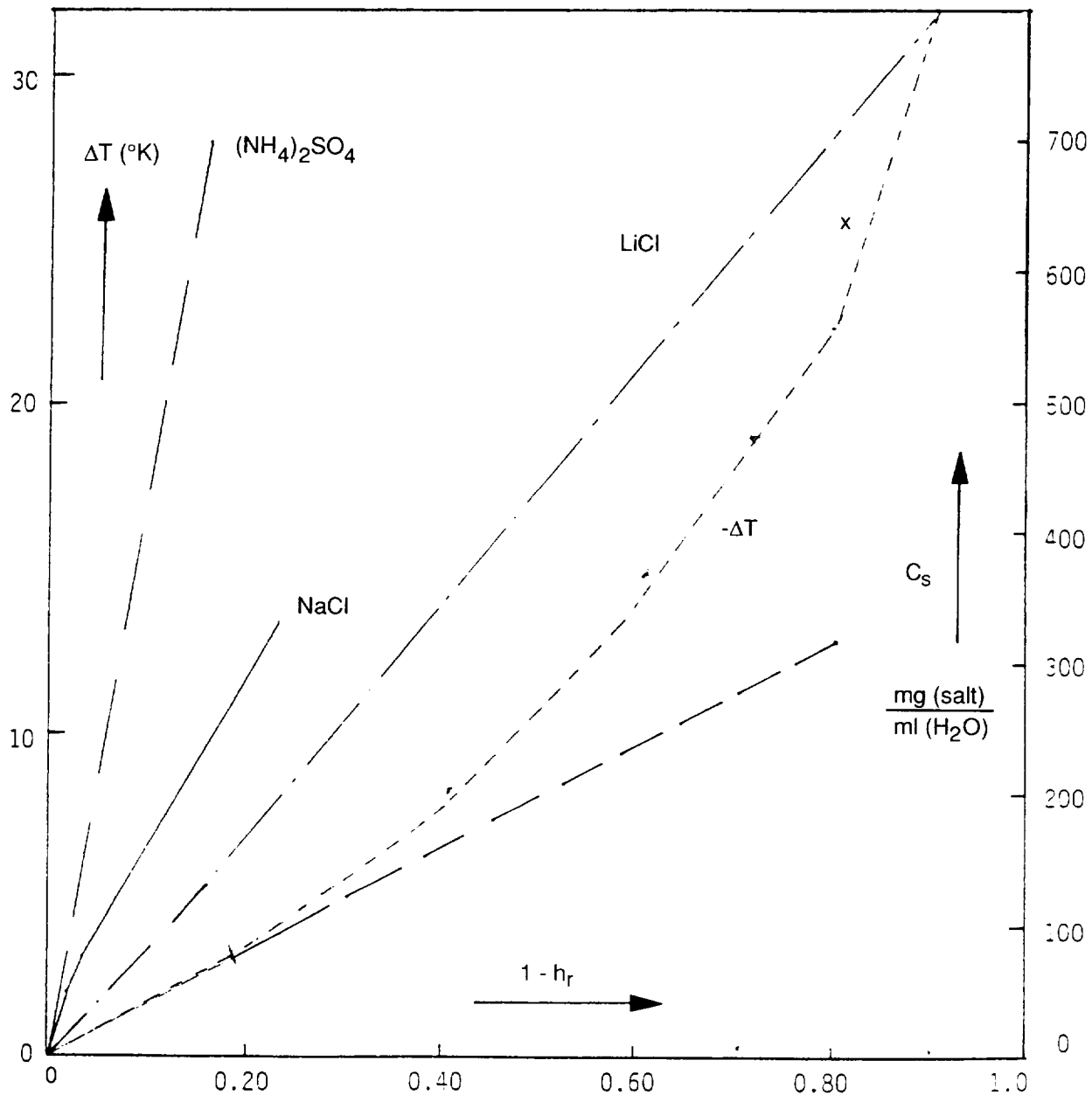


Figure 3 Required undercooling and salinity of the fluid reservoir for subsaturated humidity in the processing chamber at 20°C.

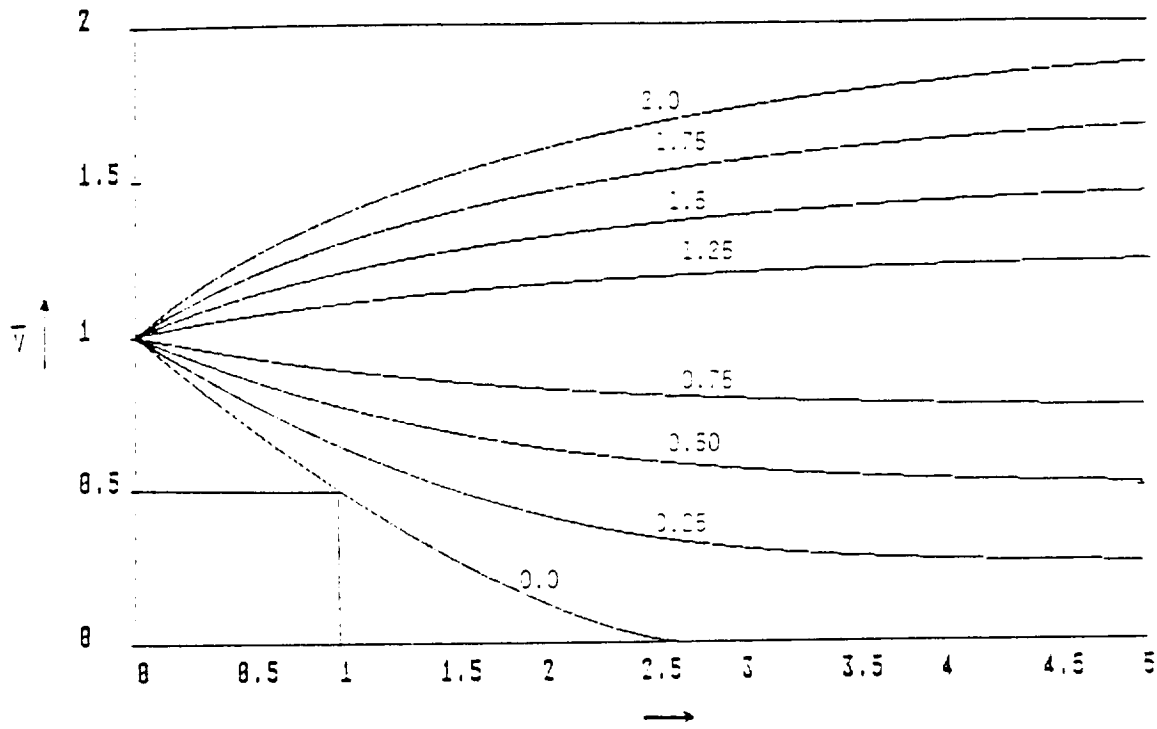


Figure 4. Normalized drop volume, $\overline{V} = V_s / V_{5.03}$ versus normalized time, $\overline{t} = t/t_{1/2}$, for different values of the ratio c_a/c_b. The C_a is the salt concentration in the drop and c_b is its concentration in the fluid reservoir.

GRASHOFF NUMBER MODEL

THE GRASHOFF NUMBER FOR A SOLUTION DROP WITH SATURATED VAPOR IN ITS BOUNDARY LAYER LEVITATED IN AN ISOTHERMAL ENVIRONMENT OF A RELATIVE HUMIDITY OF $h_r < 1$, CAN BE EXPRESSED AS

$$Gr = d_s^2 \frac{\Delta\rho}{\rho_\infty} \frac{1-h_r}{\nu^2} g$$

d_s - SAMPLE DIAMETER
ρ_∞ , $\Delta\rho$ - AMBIENT GAS DENSITY,
EXCESS DENSITY IN BOUNDARY LAYER
h_r - RELATIVE HUMIDITY OF GAS
ν - KINEMATIC VISCOSITY
g - GRAV. ACCELERATION (g = 9.81 m/s)

FOR WATER DROPS IN AIR THE GRASHOF NUMBER CAN BE APPROXIMATED BY

$$Gr = 208 \; d_s^3 \; (1 - h_r) \; e^{\alpha(T).T}$$

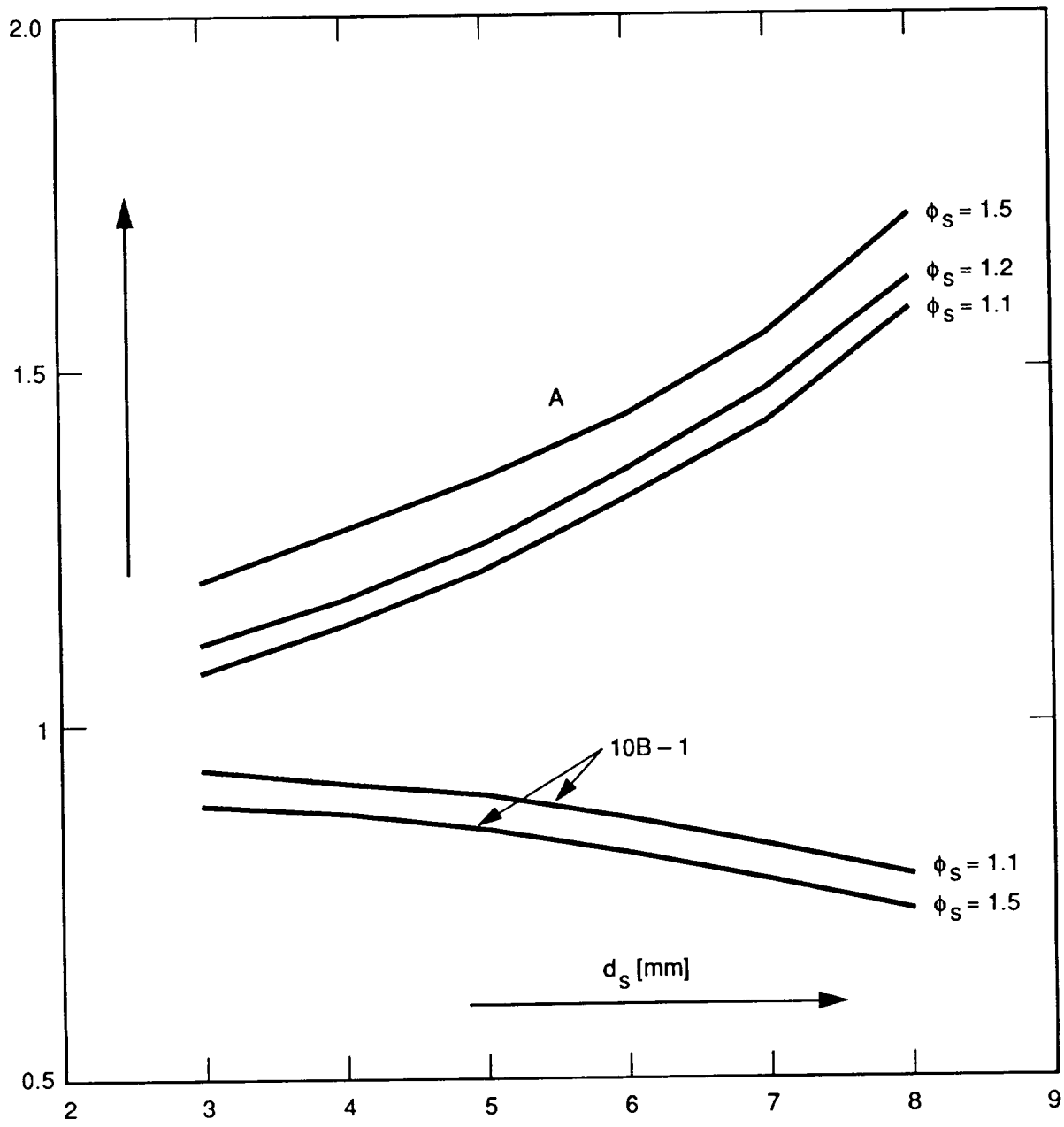
WITH $\alpha = 0.058 \; (1 - 0.0033T)$ AND d_s MEASURED IN cm.

DISCUSSION OF EXPERIMENTAL RESULTS

WHEN INSERTING TYPICAL REYNOLDS NUMBERS, Re, FOR WATER DROPS WITH DIAMETERS BETWEEN 2 AND 6mm, LEVITATED IN AMBIENT AIR AT 20 KHZ (TABLE 1), WE FIND NUSSELT NUMBERS BETWEEN 5 AND 10 DEPENDING ON DROP DIAMETER d , RELATIVE HUMIDITY, h_r , TEMPERATURE, T, AND SPL (OR LEVITATION SAFETY FACTOR, ϕ_s). FOR CONSTANT LEVITATION SAFETY FACTOR, ϕ_s , Nu INCREASES LINEARLY WITH T AND d .

FIGURE 7 SHOWS A TYPICAL DROP SHRINKING CURVE MEASURED AT 20 C AND A RELATIVE HUMIDITY OF ABOUT 80%. IN THE DISCRETE RANGE BETWEEN 2 AND 3 mm, THE CALCULATED NUSSELT NUMBER IS Nu = __ WHICH DIFFERS BY A FACTOR OF 1.3 FROM THE MEASURED VALUE; IT MAY RESULT FROM UNCERTAINTIES IN THE HUMIDITY MEASUREMENT.

WHEN THE DROP DIAMETER, TEMPERATURE, SPL, AND RELATIVE HUMIDITY ARE ONLY SLIGHTLY VARIED DURING A MEASUREMENT THE NUSSELT NUMBER CAN BE ASSUMED CONSTANT. IN THIS CASE IT IS POSSIBLE TO PREDICT THE RELATIVE SHRINKING TIME, $t_{0.5}$, (EQUATION 4), FOR A GIVEN ACCURACY OF THE MEASURED HUMIDITY, h_r , AND TEMPERATURE, T.



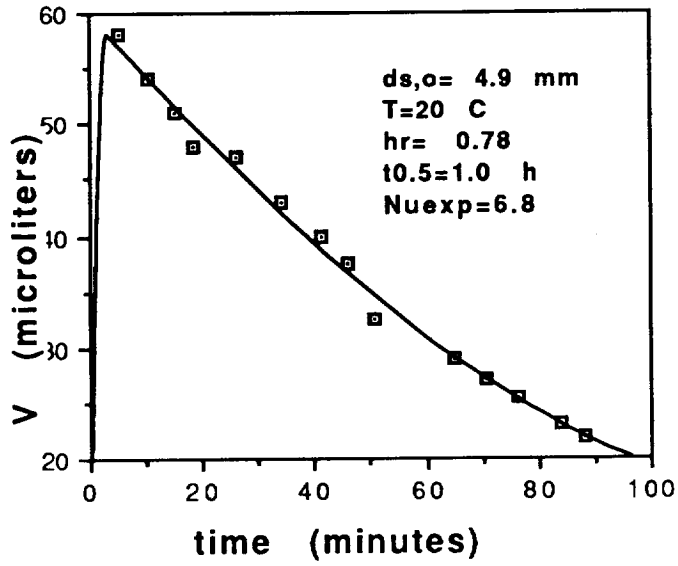
$$Nu \approx 2 + e^{A} (1 + Gr)^{B}$$

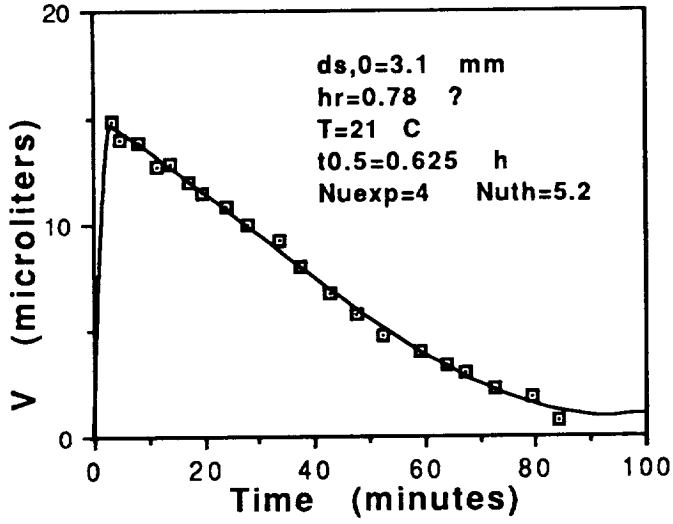
$$Gr \approx 208 \; d_s^3 \; (1 - hr) \; e^{\alpha (T) \, T} \qquad d \text{ [cm]}$$

$$d \approx 0.058 \; (1 - 0.0033T) \qquad T \text{ [°C]}$$

FIG. 6

diameter (mm)	2	3	4	5	6
Re	46	49.6	56	65.4	78.4
e^{A}	2.86	2.96	3.13	3.35	3.63
B	0.192	0.191	0.189	0.187	0.184
Gr (20 C)	4.9	16.5	39	76	132
Nu (h_r = 0)	5.9	7.0	8.3	9.5	10.9
Nu (h_r = 0.8)	4.8	5.7	6.6	7.5	8.5


ds,o= 4.9 mm
T=20 C
hr= 0.78
t0.5=1.0 h
Nuexp=6.8
V (microliters)
time (minutes)
60
50
40
30
20
0
20
40
60
80
100


ds,0=3.1 mm
hr=0.78 ?
T=21 C
t0.5=0.625 h
Nuexp=4 Nuth=5.2
V (microliters)
Time (minutes)
20
10
0
0
20
40
60
80
100

N91-21357

Rheological Properties, Shape Oscillations and Coalescence of Liquid Drops with Surfactants

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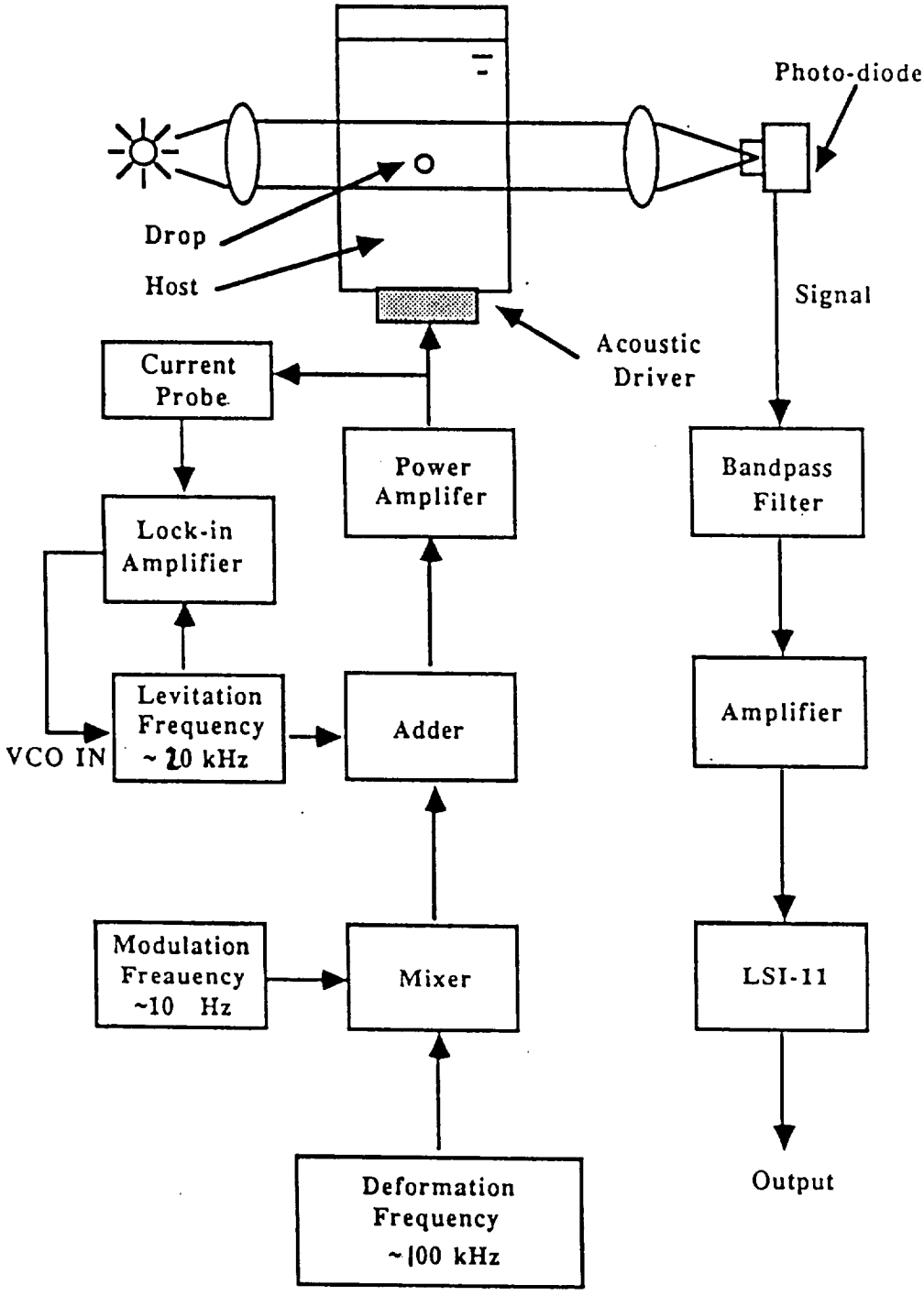
Department of Mechanical Engineering
Yale University

A method has bee developed to deduce dynamic interfacial properties of liquid drops. The method involves measuring the frequency and damping of free quadrupole oscillations of an acoustically levitated drop. Experimental results for pure liquid-liquid systems agree well with theoretical predictions. Additionally, the effects of surfactants is considered. Extension of these results to a proposed microgravity experiment on the DPM in USML-1 will be discussed.

Efforts are also underway to model the time history of the thickness of the fluid layer between two pre-coalescence drops, and to measure the film thickness experimentally. Preliminary results will be reported, along with plans for coalescence experiments proposed for USML-1.



Schematic diagram of the experimental setup.

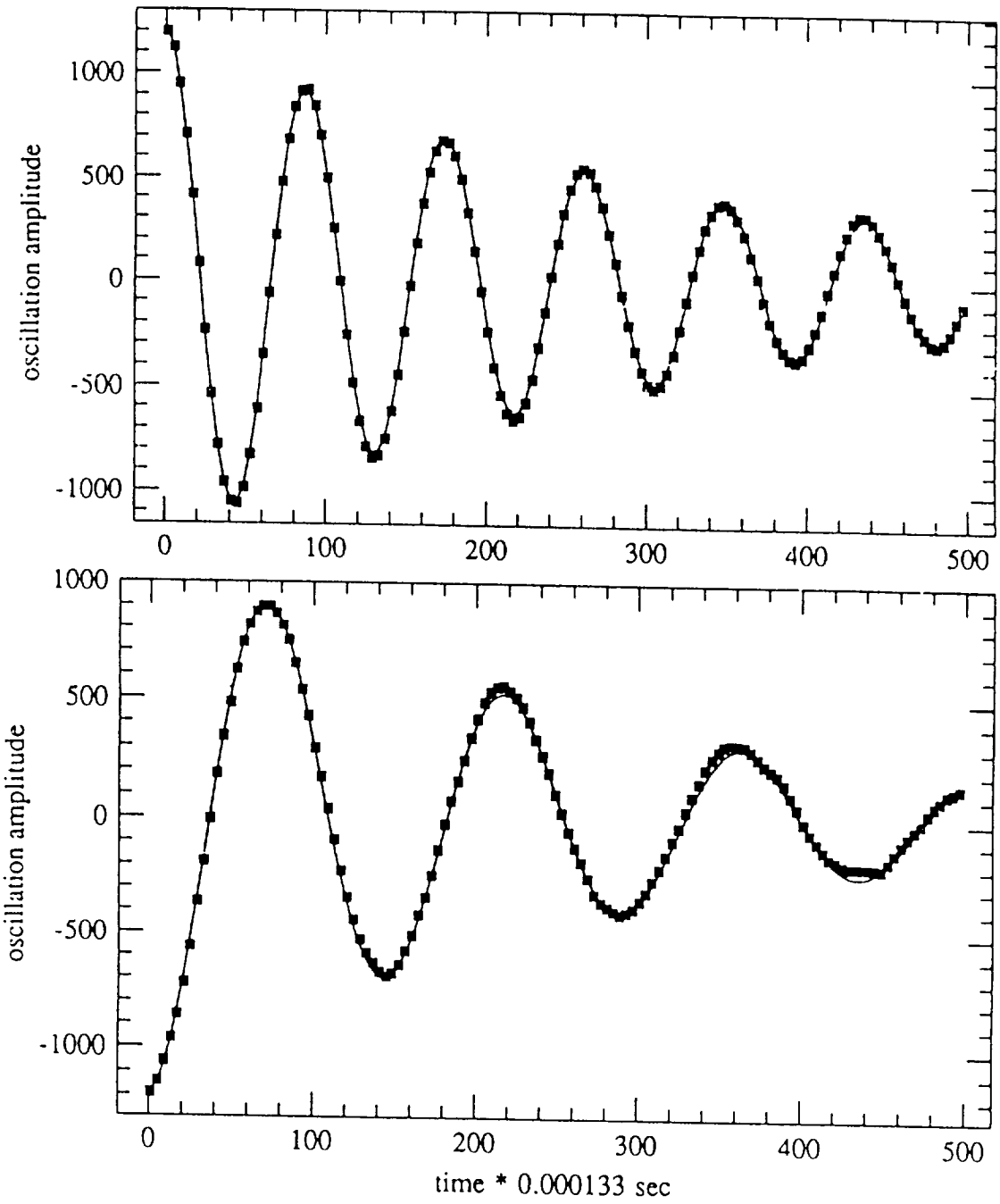


Fig. 2 Signals for a drop undergoing free quadrupole oscillations before and after least squares fitting. For the purpose of clarity, only one fourth of the measured data points are shown in each figure. The upper figure is for a hexane drop of a diameter 0.2002 cm in pure water. The lower figure is for a hexane drop of a diameter 0.1954 cm in 1.75 mM SDS aqueous solution.

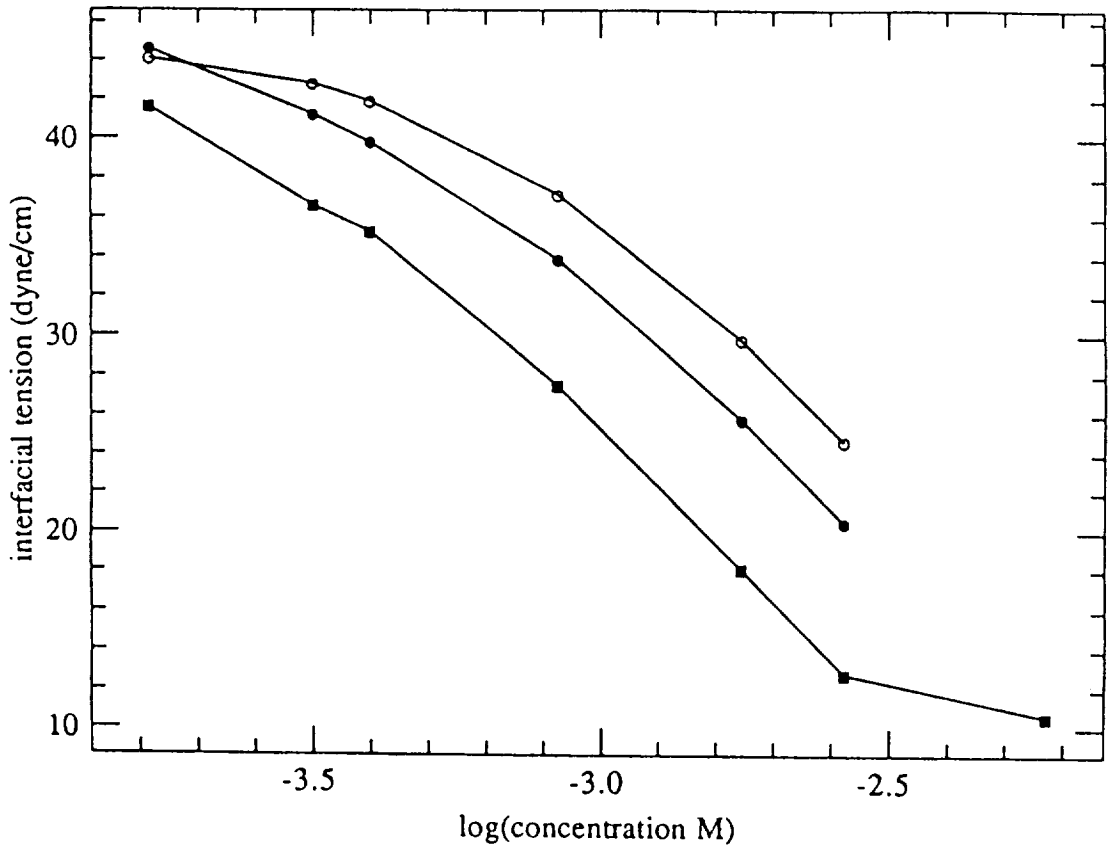


Fig. 14 Interfacial tension at different SDS concentrations. Circles and squares denote results obtained within five minutes and one hour after the drop is introduced into the solution respectively. The open circles represent results measured by Rehfeld using drop's weight method. Rehfeld's data were also taken within five minutes after the drop was formed.

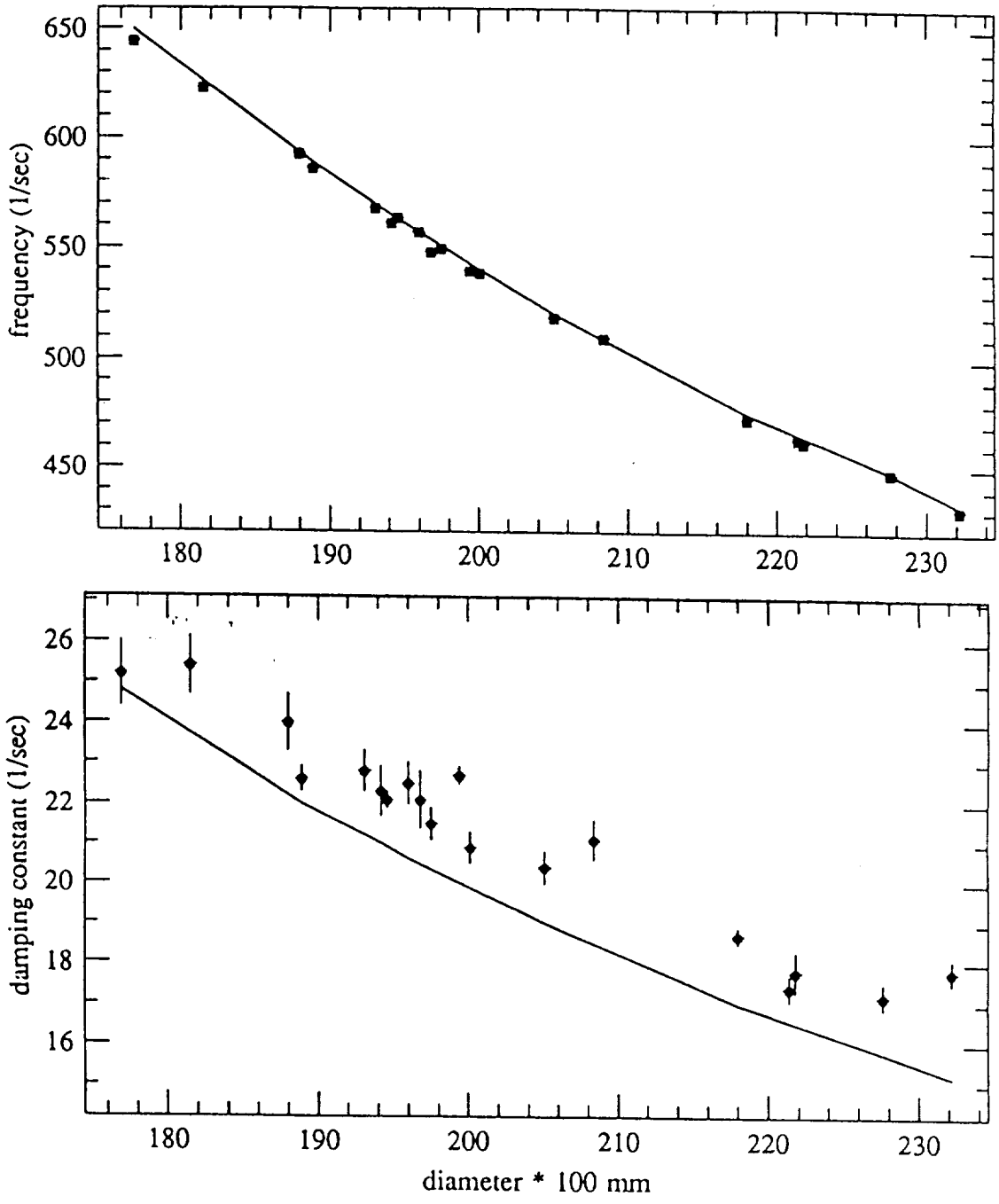


Fig. 4 Frequency and damping constant versus diameter for hexane drops in pure water at 24±2°C. The solid curves are the theoretical predictions based on Eq. [1].

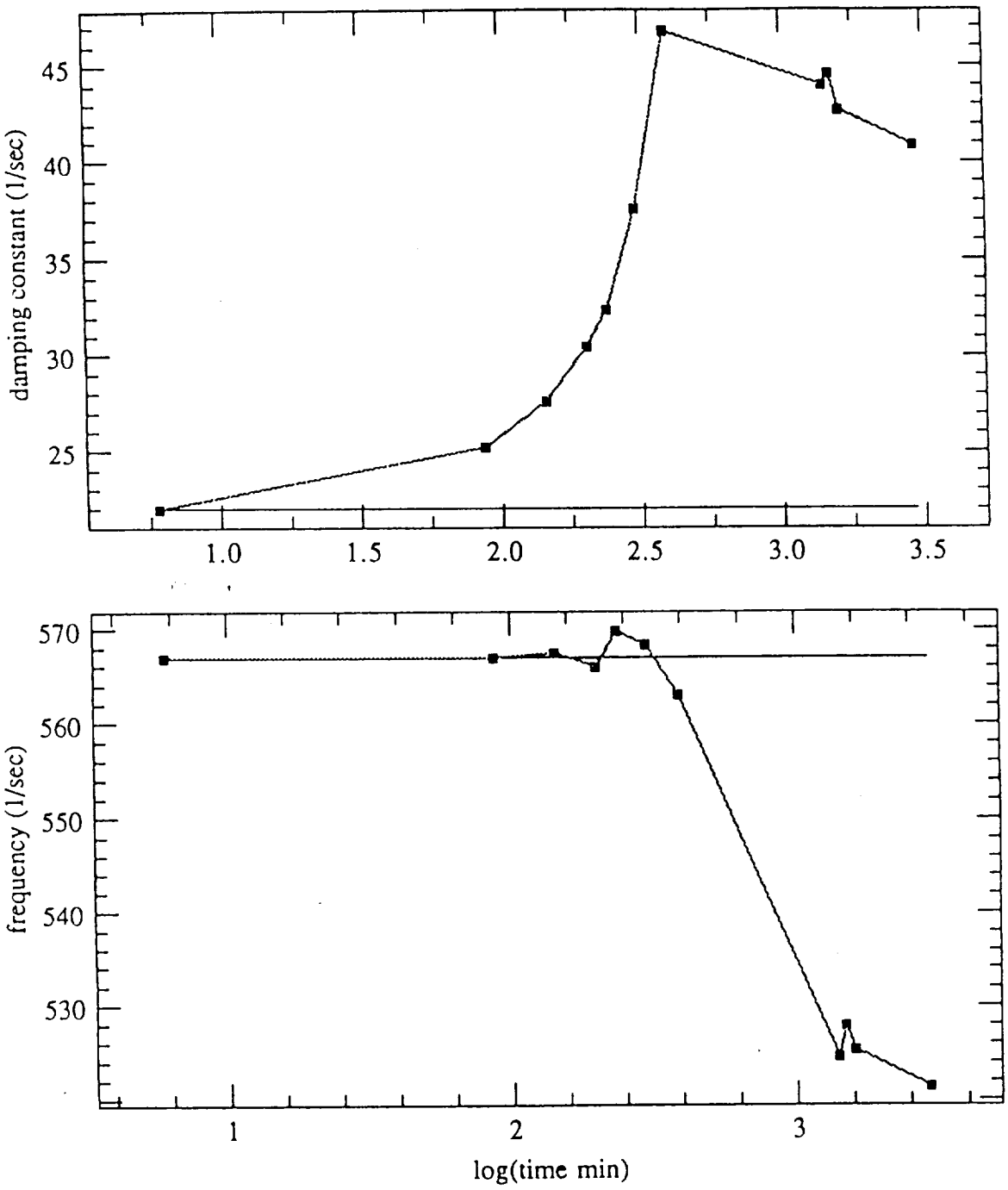


Fig. 3 Time history of the frequency and damping constant for a hexane drop of a diameter 0.954±0.004 mm in pure water at 22°C. The solid curves are the theoretical predictions based on Eq. [1].

N91-21358

Optical Scattering Methods Applicable to Drops and Bubbles

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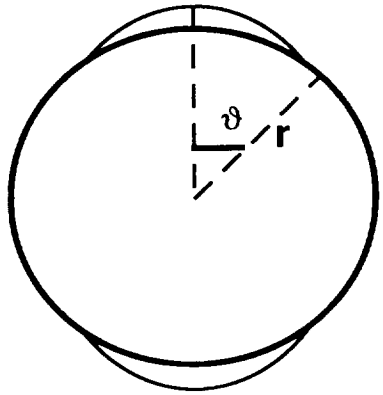
An overview of optical scattering properties of drops and bubbles will be given. The properties lead to unconventional methods for optically monitoring the size or shape of a scatterer and are applicable to acoustically levitated objects. Several of the methods are applicable to the detection and measurement of small amplitude oscillations. Relevant optical phenomena include: (1) rainbows, (2) diffraction catastrophes from spheroids, (3) critical angle scattering, (4) effects of coatings, (5) glory scattering, and (6) optical levitation. [Research partially supported by the Office of Naval Research.]



OPTICAL PSEUDO-EXTINCTION METHOD FOR MEASURING $\varepsilon_2(t)$

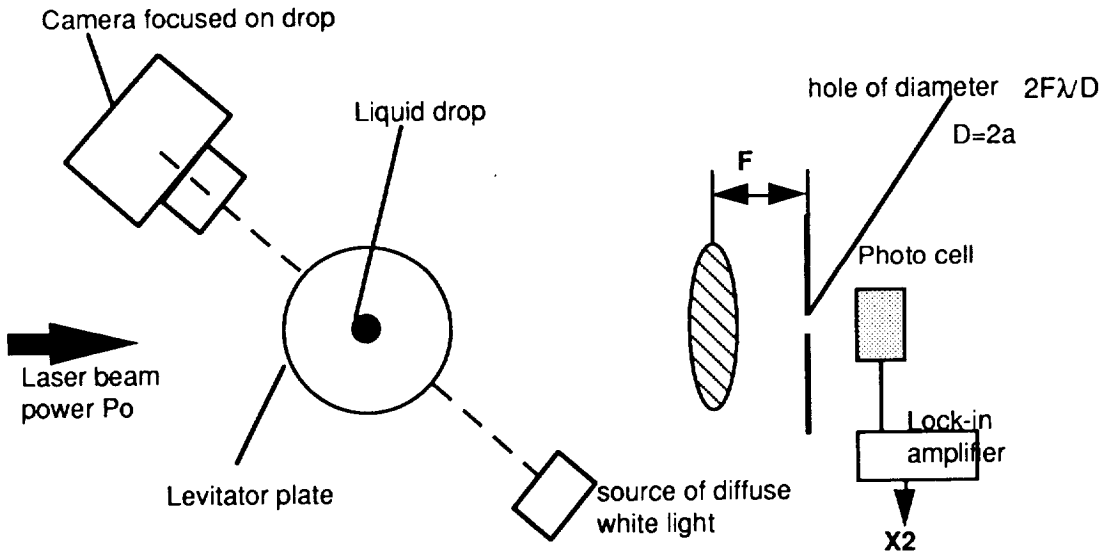
R=a

$$r(\vartheta,t) = R + \varepsilon_1(t)\cos\vartheta + \varepsilon_2(t)(3\cos^2\vartheta - 1)$$

$$A(t) = 1/2\int_0^{2\pi} r^2 d\vartheta = \pi R^2 + \varepsilon_2(t)\pi R + O(\varepsilon^3)$$

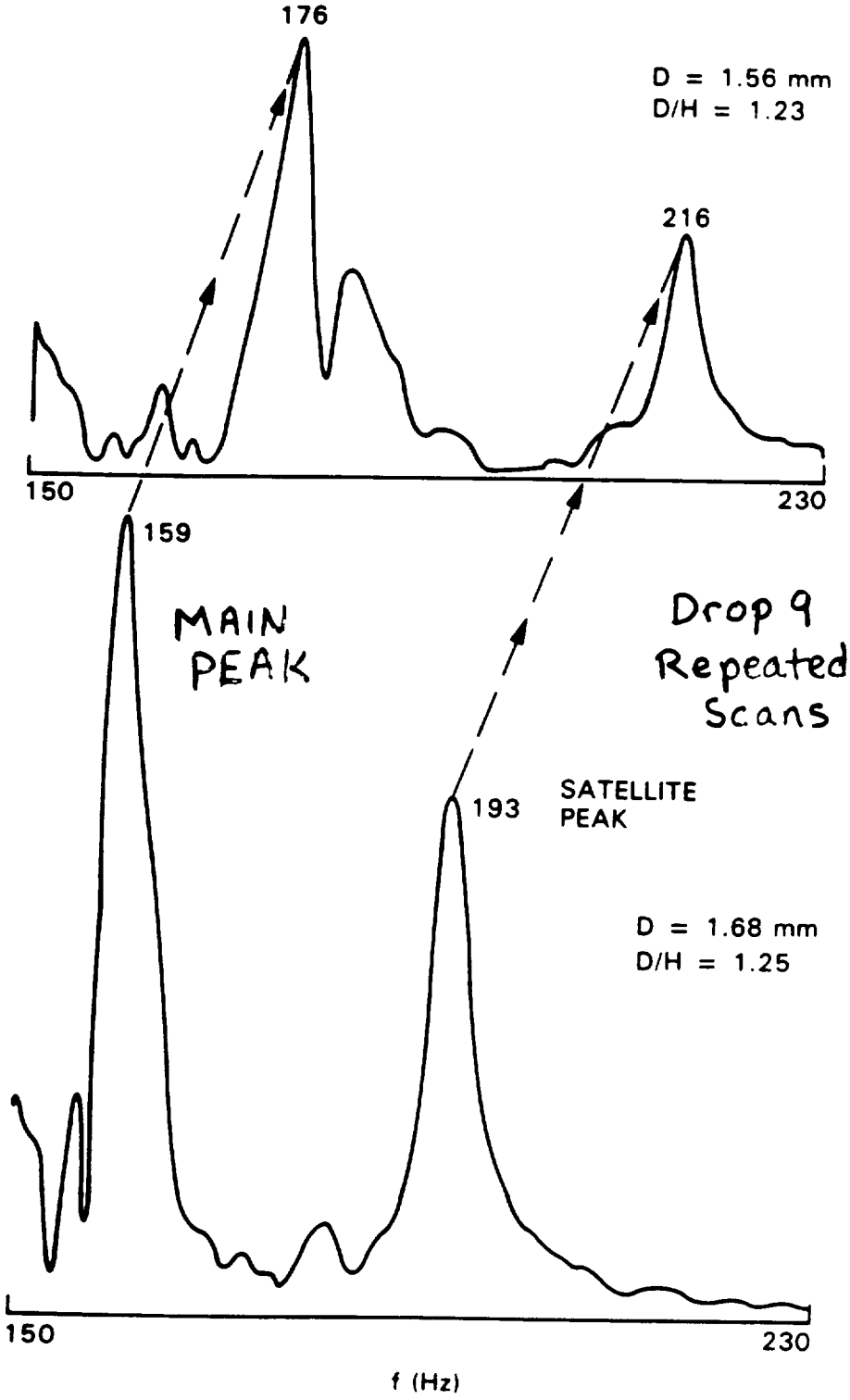
A(t): cross sectional area

POWER TO PHOTOCELL = Po - IA(t)



$$\varepsilon_2(t) = x_2(f)\cos(2\pi ft + \psi) + \varepsilon_2(\text{equilibrium})$$

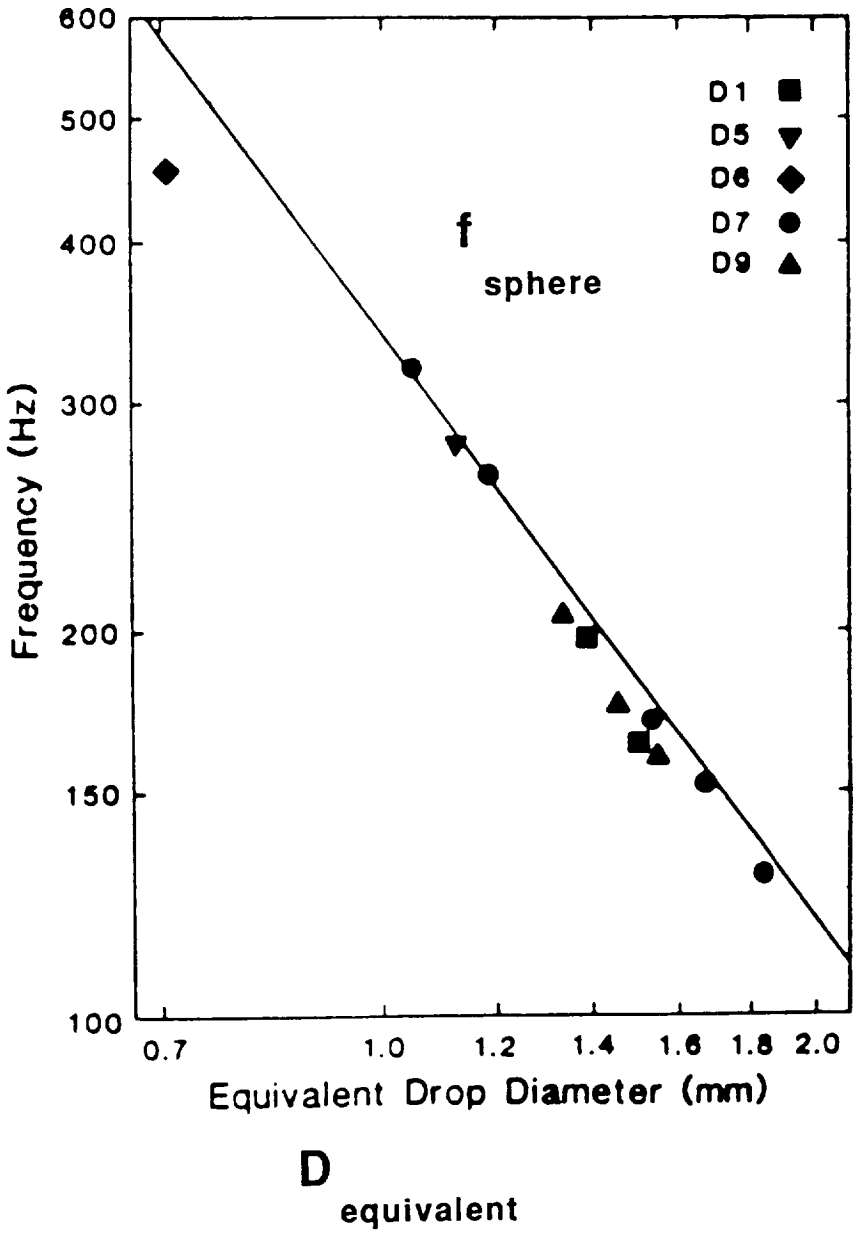
INSENSITIVE TO THE POSITION OF THE DROP

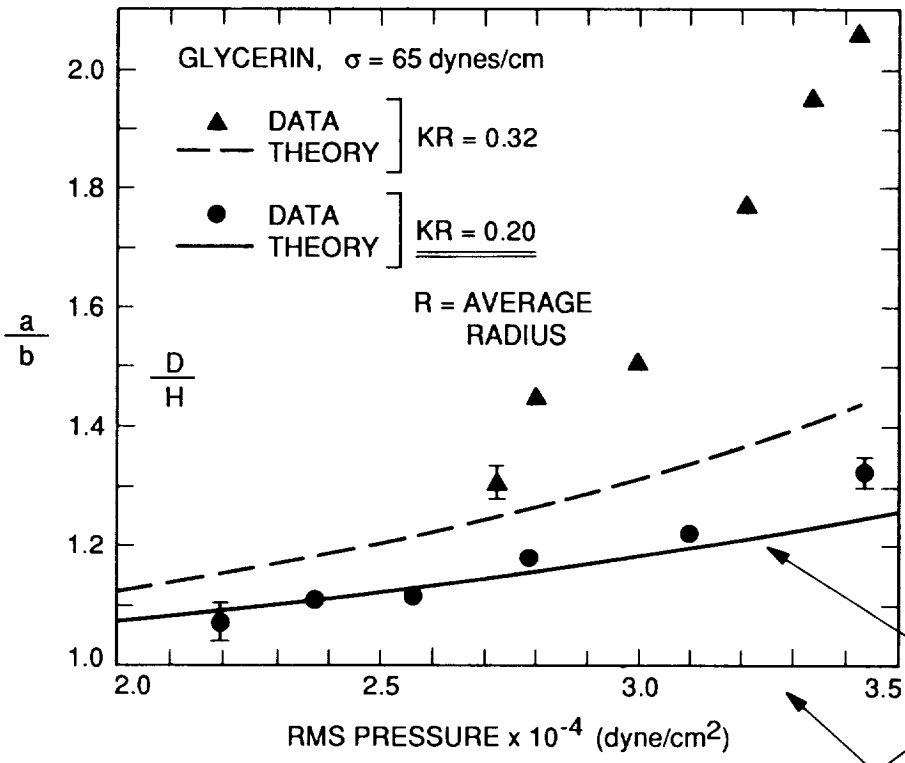

176
D = 1.56 mm
D/H = 1.23
216
150
230
159
MAIN
PEAK
Drop 9
Repeated
Scans
193
SATELLITE
PEAK
D = 1.68 mm
D/H = 1.25
150
230
f (Hz)

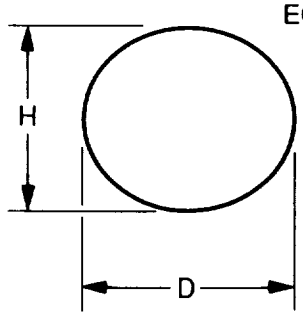
ADJUSTED DATA

D(equivalent) = DIAMETER OF SPHERE HAVING THE SAME VOLUME AS THE DROP:

$$D.D\left(\frac{D}{H}\right)^{\frac{1}{3}}$$

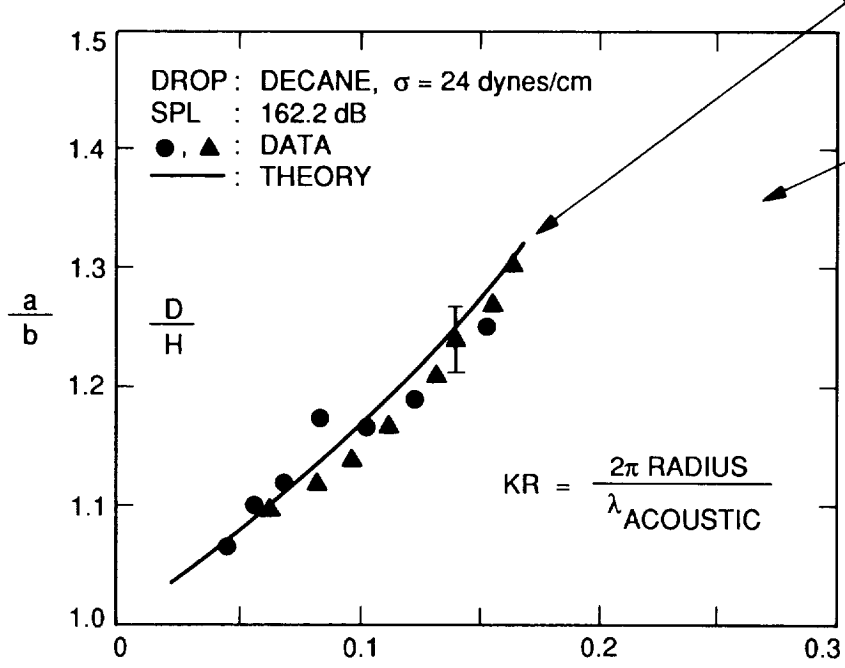






OBLATE EQUILIBRIUM

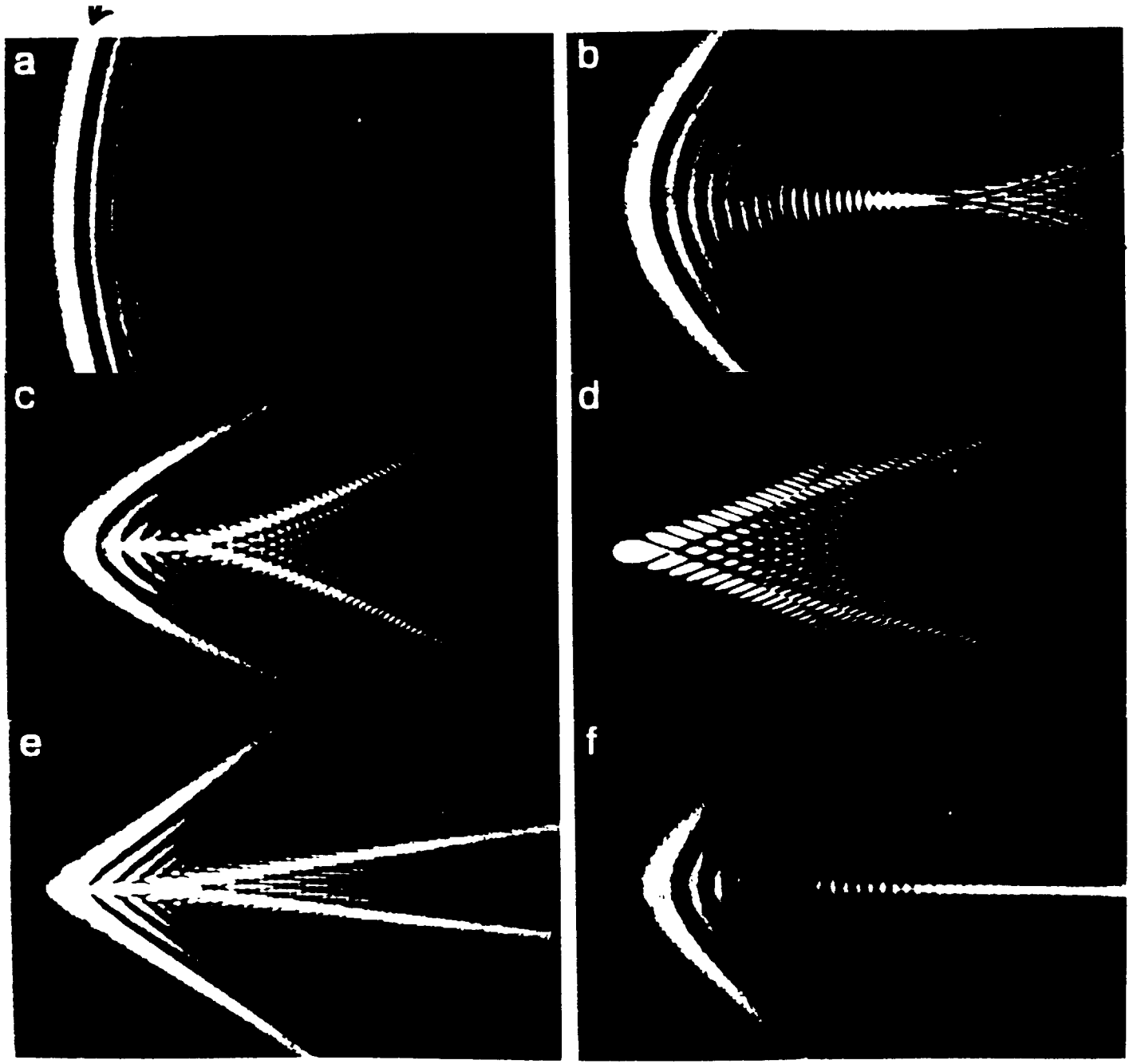
THEORY FOR ASPECT RATIO D/H
MARSTON
J. ACOUST. SOC. AM.
(1980, 1981)



E.H. TRINH & C.J. HSU
J. ACOUST. SOC. AM.
(1986)
MEASUREMENTS
OF THE ASPECT RATIO

$\frac{D}{H} = \frac{a}{b}$ OF A

LEVITATED DROP



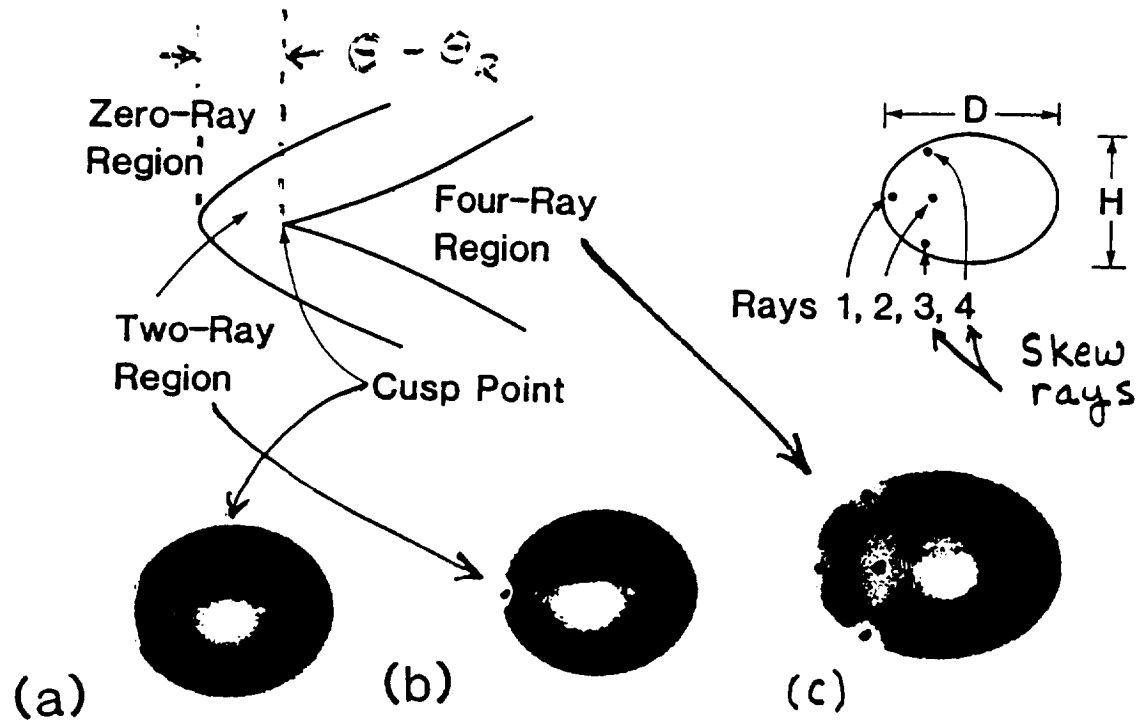
Transverse Cusp

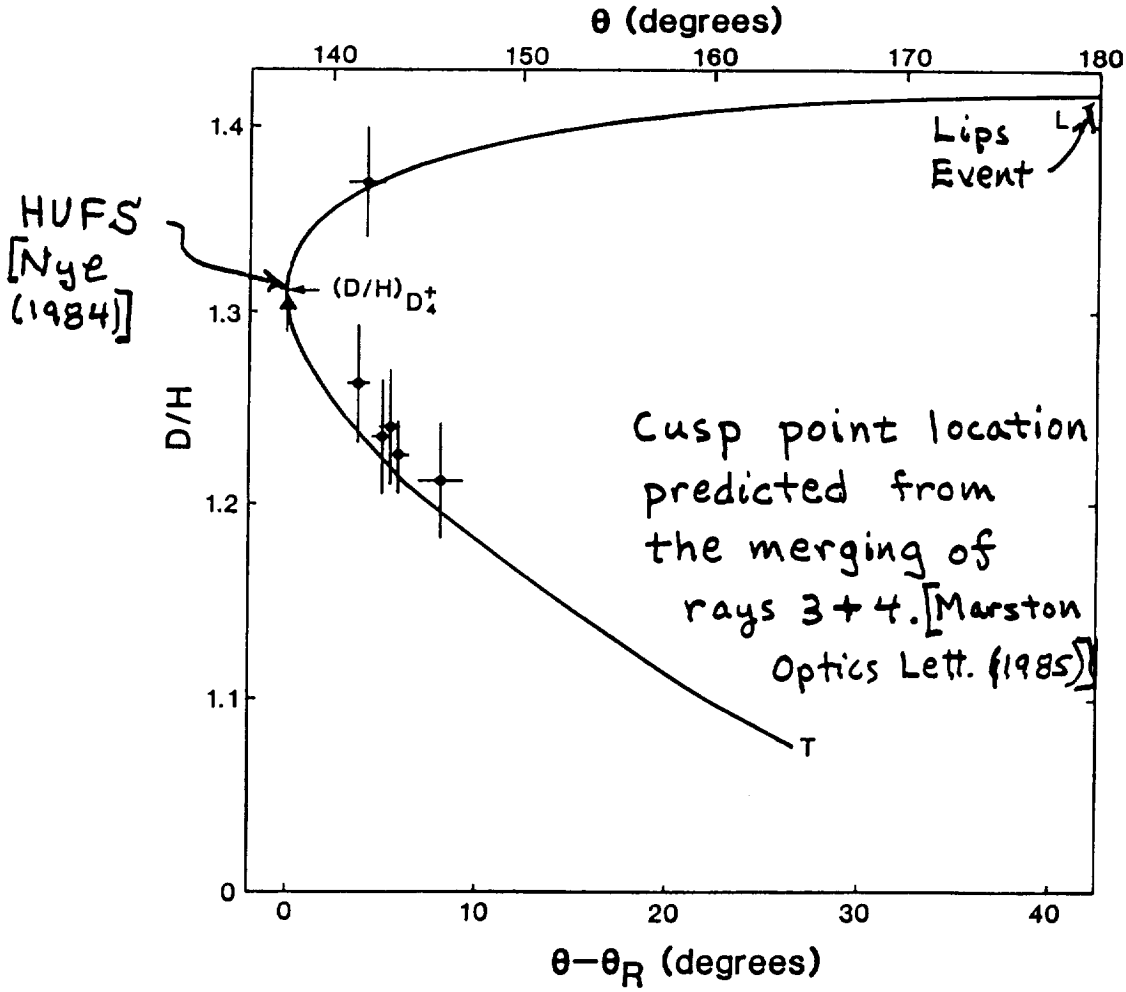
Hyperbolic Umbilic Focal Section

5 Degrees

Marston + Trinh (1984)

Light scattering from oblate drop of water


Zero-Ray Region
Four-Ray Region
Two-Ray Region
Cusp Point
D
H
Rays 1, 2, 3, 4
Skew rays
(a)
(b)
(c)


θ (degrees)
140
150
160
170
180
1.4
1.3
1.2
1.1
0
D/H
Lips Event
L
HUFS [Nye (1984)]
(D/H)D4+
T
Cusp point location predicted from the merging of rays 3+4. [Marston Optics Lett. (1985)]
0
10
20
30
40
θ−θR (degrees)

Rainbow scattering from spheroidal drops—an explanation of the hyperbolic umbilic foci

J. F. Nye

Nature 312, 531 (Dec. 1984)

H. H. Wills Physical Laboratory, Tyndall Avenue,
Bristol BS8 1TL, UK

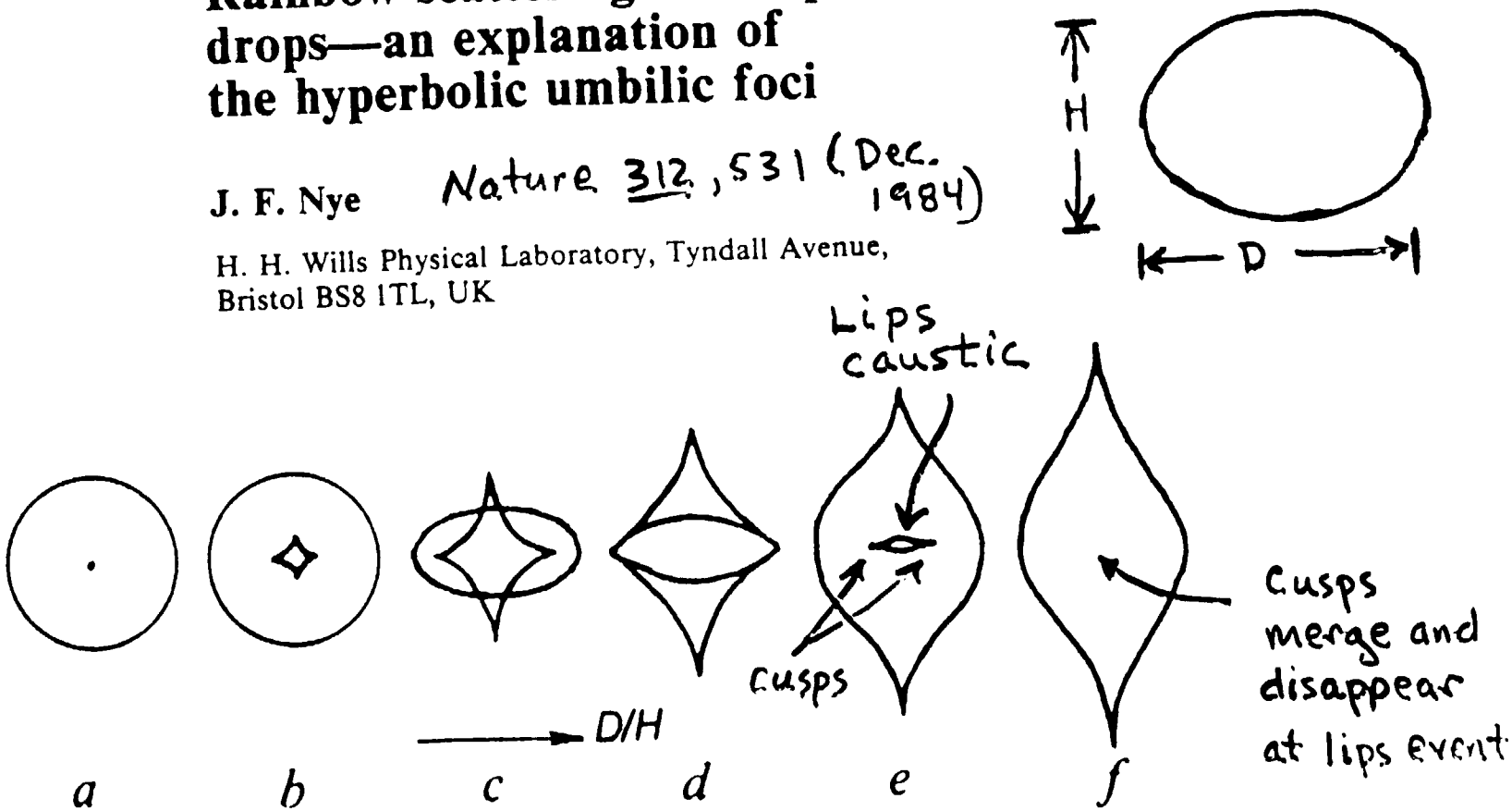
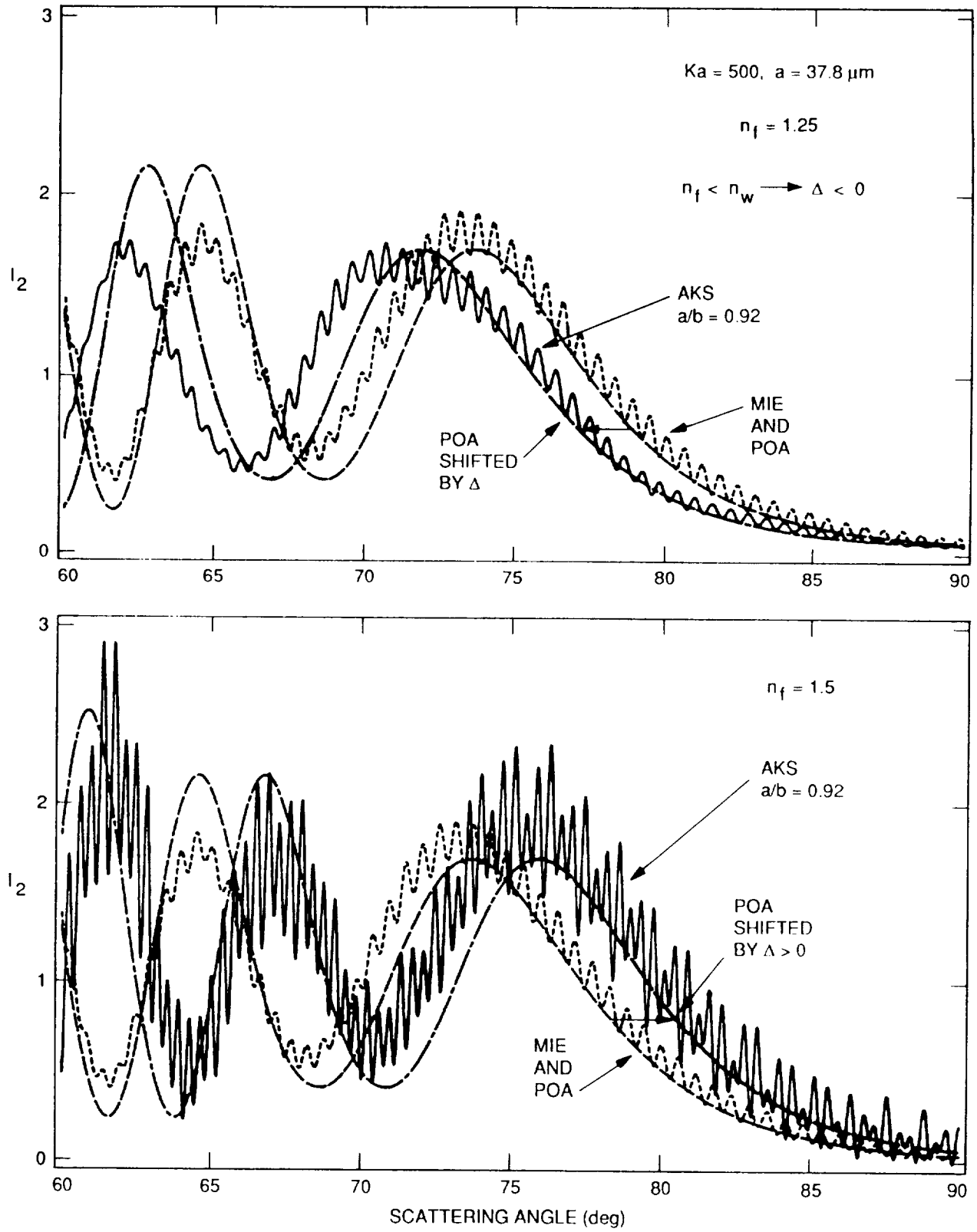


Fig. 2 Sequence of caustics in the far field as D/H increases from 1. *a*, The circular rainbow with a singular point at its centre. On perturbation the point breaks up (*b*, *c*) into an expanding four-cusped figure. At *d*, two hyperbolic umbilic foci occur. On further increase of D/H, the inner figure contracts (*e*), and then disappears (*f*) in a lips event. The angular width of the complete figure is the same throughout.


Ka = 500, a = 37.8 μm
n_f = 1.25
$n_f < n_w \rightarrow \Delta < 0$
AKS
a/b = 0.92
MIE
AND
POA
POA
SHIFTED
BY Δ
n_f = 1.5
AKS
a/b = 0.92
POA
SHIFTED
BY Δ > 0
MIE
AND
POA
I_2
0
1
2
3
60
65
70
75
80
85
90
SCATTERING ANGLE (deg)

N91-21359

UNDERCOOLING OF ACOUSTICALLY LEVITATED MOLTEN DROPS

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and

M. E. GLICKSMAN

Materials Engineering Department
Rensselaer Polytechnic Institute
Troy, New York 12181, USA.

Abstract

We have observed that the acoustically levitated molten SCN drops can generally be undercooled to a degree where the impurities in the drop are responsible for the nucleation of the solid phase. However, we have also observed that ultrasound occasionally terminates undercooling of the levitated drop by initiating the nucleation of the solid at an undercooling level which is lower than that found for the nucleation catalyzed by the impurities in the drop. This premature nucleation can be explained by thermodynamic considerations which predict an increase in effective undercooling of the liquid upon the collapse of cavities. Pre-existing gas microbubbles which grow under the influence of ultrasound are suggested as the source of cavitation. The highly undercooled SCN drops can be utilized to measure the growth velocity of the solid in the deeply undercooled region including the hypercooled region.

Objective

To study the effect of ultrasound on the undercooling of acoustically levitated molten drops.

Experimental Procedure

* Succinonitrile

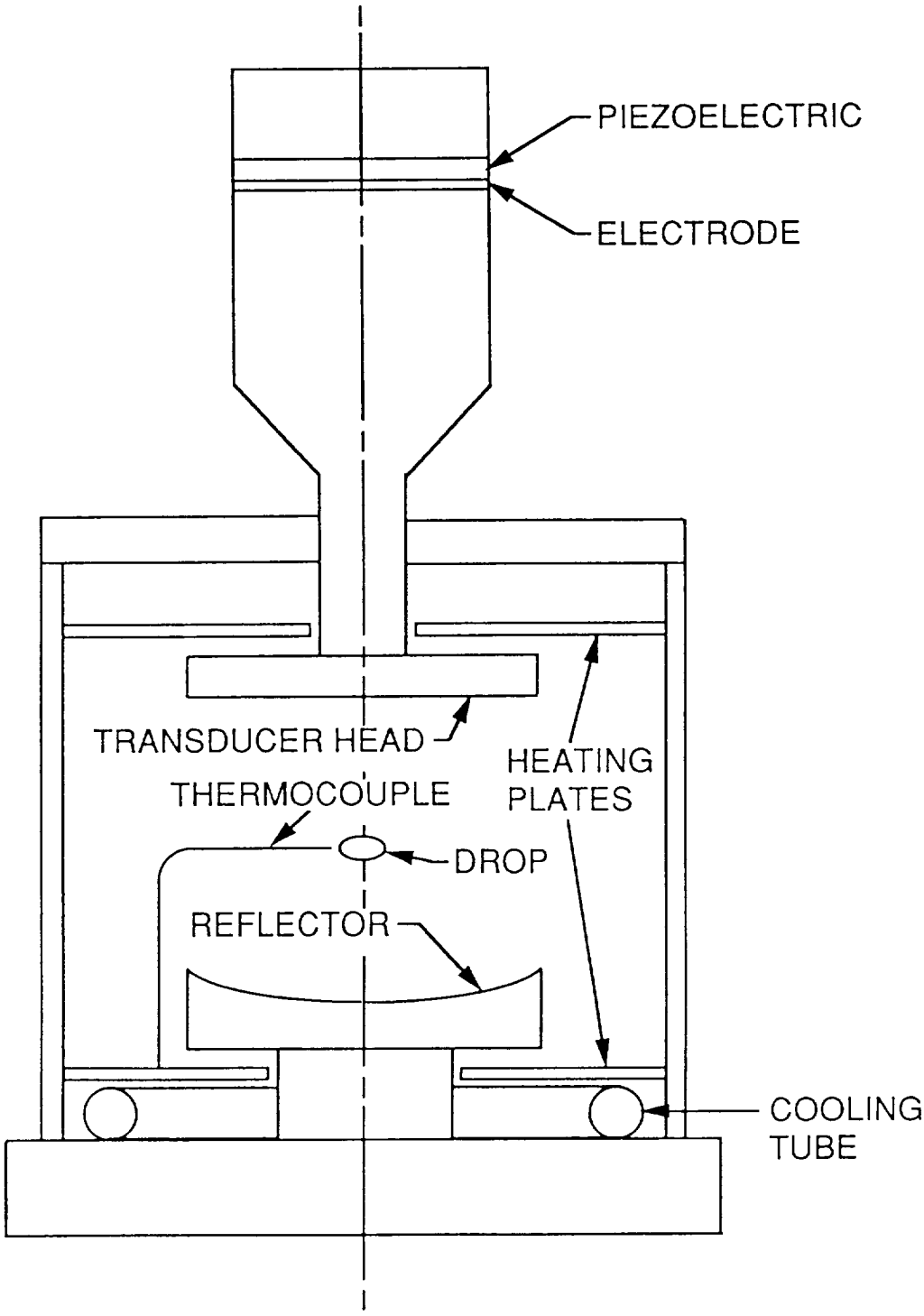
  $CN\text{-}C_2H_2\text{-}NC$

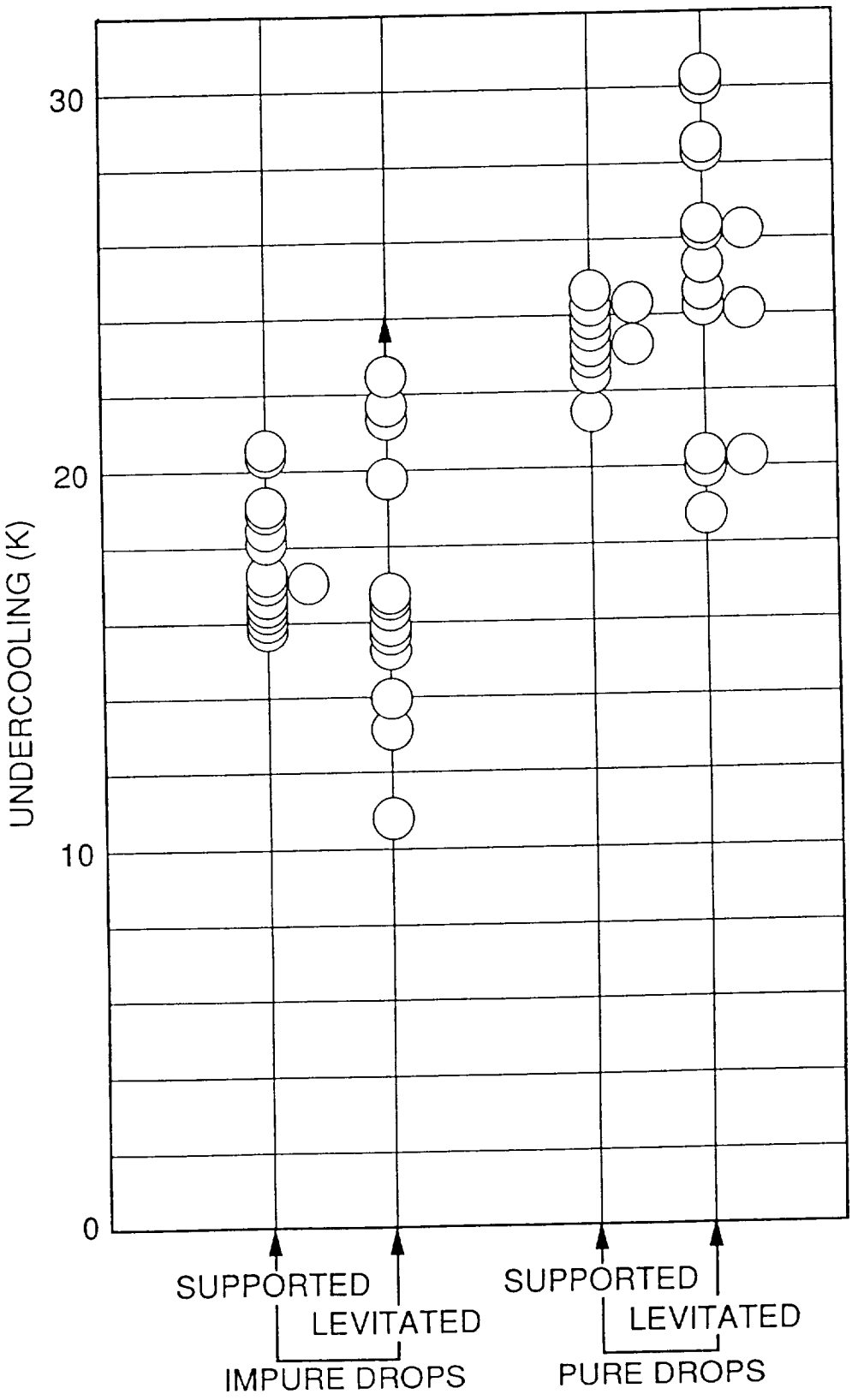
  impure (97 %, 54.0 °C)

  pure (>99.9 %, 58.0 °C)

* Undercooling Level

  1. acoustically levitated drops
  2. mechanically supported drops


PIEZOELECTRIC
ELECTRODE
TRANSDUCER HEAD
HEATING PLATES
THERMOCOUPLE
DROP
REFLECTOR
COOLING TUBE


30
20
10
0
UNDERCOOLING (K)
SUPPORTED
LEVITATED
IMPURE DROPS
SUPPORTED
LEVITATED
PURE DROPS

Means and Standard Deviations of the Undercooling Levels

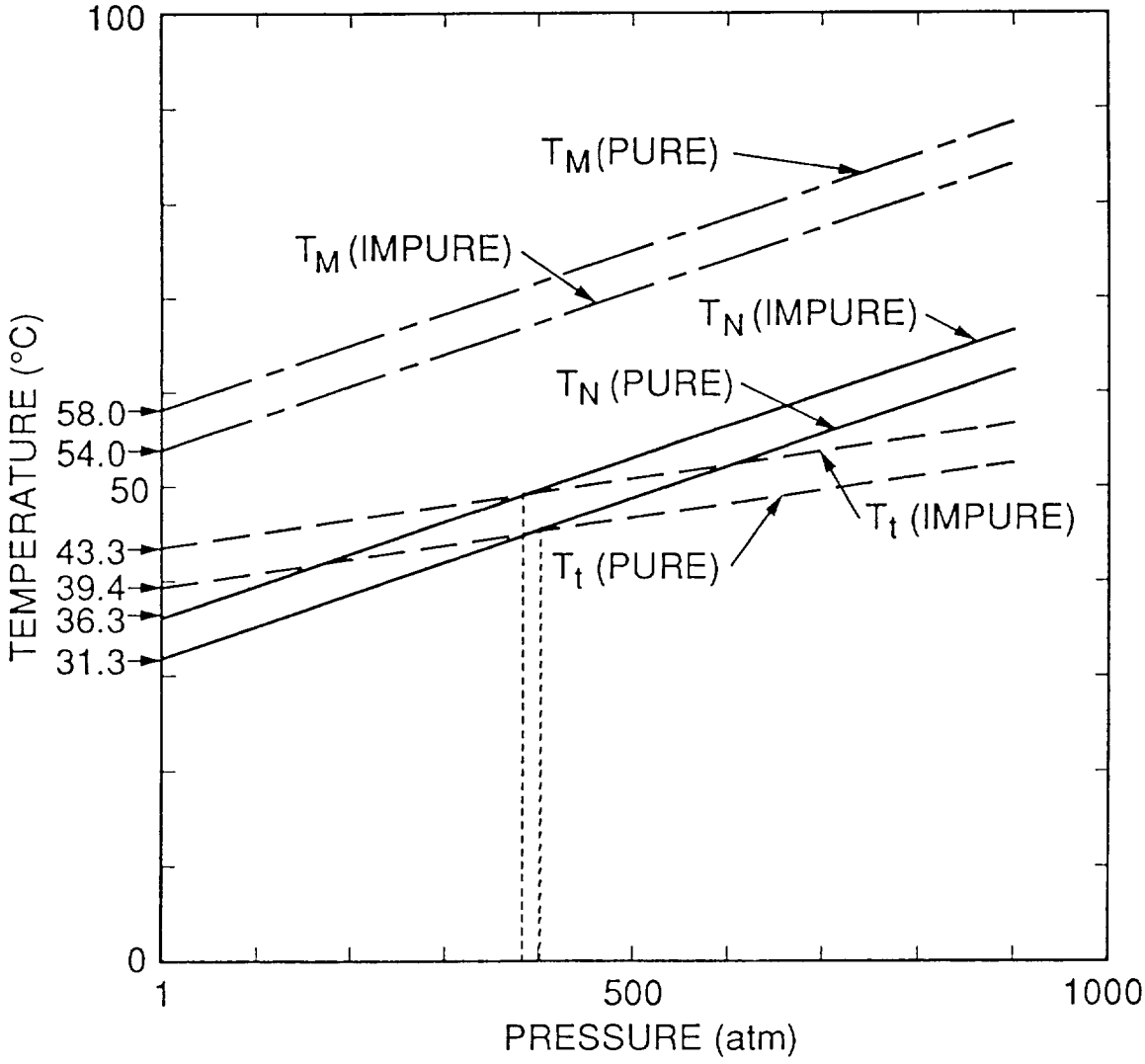
		mean (K)	standard dev.
impure	supported	17.7	1.58
	levitated	16.9 (18.2)	3.64 (3.03)
pure	supported	23.4	0.95
	levitated	24.8 (26.7)	3.72 (2.21)

Possible Causes of Dynamic Nucleation

1. Foreign impurities

2. Collision of subcritical embryos

3. Cavitation


100
50
0
1
500
1000
TEMPERATURE (°C)
PRESSURE (atm)
58.0
54.0
43.3
39.4
36.3
31.3
T_M(PURE)
T_M (IMPURE)
T_N (IMPURE)
T_N (PURE)
T_t (IMPURE)
T_t (PURE)

Conclusions

1. Acoustically levitated drops can generally be undercooled to a degree where the impurities are responsible for the nucleation of the solid.

2. Ultrasound occasionally terminates the nucleation of the solid. This premature nucleation can be explained by thermodynamic considerations which predict an increase in effective undercooling of the liquid upon cavitation.

3. SCN drops can be undercooled into the hypercooled region (>23.1 K). The drops can be utilized to measure the physical properties in the deeply undercooled region.

SPLINTER SESSION 2

Technical Papers

N91-21360

Containerless Synthesis of Ceramic Materials Using Microwave Heating

B. Dunn and S. Crouch - Baker
Department of Materials Science and Engineering,
University of California Los Angeles,
Los Angeles, CA 90024

Recently, it has been demonstrated that microwave heating techniques may be employed for the synthesis of a number of multicomponent ceramic oxide - based materials, e.g. $YBa_2Cu_3O_7$ and $CuFe_2O_4$. A characteristic, and potential extremely useful, feature of such syntheses is that they occur in significantly less time than that required using conventional furnace - based techniques. However, the information obtained to date is necessarily rather empirical, and systematic investigations of the use of microwave heating for the syntheses of ceramic materials are required.

The syntheses of ceramic materials at high temperatures are often affected by unwanted, deleterious reactions of the reactants and/or products with the reaction container. This may severely limit the choice of available container materials.

Consequently, it is of interest to investigate the high temperature synthesis of ceramic materials using microwave heating in a containerless environment. Suitable candidate materials for initial study include, for example, the ultra - hard borides and carbides.

Containerless synthesis of ceramic materials using microwave heating

B. Dunn and S. Crouch - Baker

Department of Materials Science and Engineering,

University of California Los Angeles,

Los Angeles, CA 90024

Preparation of pure oxide - based phases using microwave heating

Product	Starting Materials	Microwave Heating Time*	Conventional Heating Time**
La_2CuO_4	La_2O_3, CuO	10 - 30 min	12 - 24 hr
$CuFe_2O_4$	Fe_2O_3, CuO	30 min	23 hr
$BaWO_4$	BaO, WO_3	30 min	2 hr
KVO_3	K_2CO_3, V_2O_5	7 min	12 hr

* For a power level of 500 W.
** Refers to use of resistive (i.e. furnace - based) heating.

D. R. Baghurst and D. M. P. Mingos, J. Chem. Soc., Chem. Comm. 829 (1988).

D. R. Baghurst, A. M. Chippindale and D. M. P. Mingos, Nature 332, 311 (1988).

Processing of Ceramics in a Microgravitational Environment

1) Sedimentation

- Powder processing
- Particle-size distribution effect on further processing
- Compare with solution-based methods

2) Particle shape

- Ideally spherical particles
- Better powder packing leading to

  shorter solid-state reaction times (surface area/energy effects)

  lack of exaggerated grain growth (increased strength)

3) Containerless Processing

- Impurity control

Other applications:

- Annealing phenomena

  local temperature distribution

- Phase transitions

N91-21361

A High-Speed Spatial (Linear) Scanning Pyrometer: A Tool for Diagnostics, Temperature Mapping, and Property Determinations at High Temperatures

A. Cezairliyan, R.F. Chang, and G.M. Foley
Thermophysics Division
National Institute of Standards and Technology
Gaithersburg, Maryland 20899

Development of a fast spatial scanning pyrometer for temperature measurements above 1500 K is described.

The salient features of the pyrometer are:

1. it measures spectral radiance temperature (at 0.65 μm) at 1024 points along a straight line (25 mm long) on the target,

2. it has no moving parts and uses a self-scanning linear array of silicon photodiodes as the detector,

3. its output is recorded digitally every 1 μs with a full-scale resolution of about 1 part in 4000, permitting performance of a complete cycle of measurements (1024 points) in about 1 ms.

Operational characteristics of the pyrometer are given. Examples of measurements of the temperature along rapidly heated (resistive self-heating) specimens (rod, tube, strip) are presented. Potential use of the pyrometer in experiments, both ground-based and in microgravity, requiring temperature mapping and property determinations of the specimen at high temperatures is discussed.

A HIGH-SPEED SPATIAL (LINEAR) SCANNING PYROMETER: A TOOL FOR DIAGNOSTICS, TEMPERATURE MAPPING, AND PROPERTY DETERMINATIONS AT HIGH TEMPERATURES

A. Cezairliyan, R.F. Chang, and G.M. Foley
Thermophysics Division
National Institute of Standards and Technology
Gaithersburg, Maryland 20899

Abstract

Development of a fast spatial scanning pyrometer for temperature measurements above 1500 K is described.

The salient features of the pyrometer are:

1. it measures spectral radiance temperature (at 0.65 μm) at 1024 points along a straight line (25 mm long) on the target,

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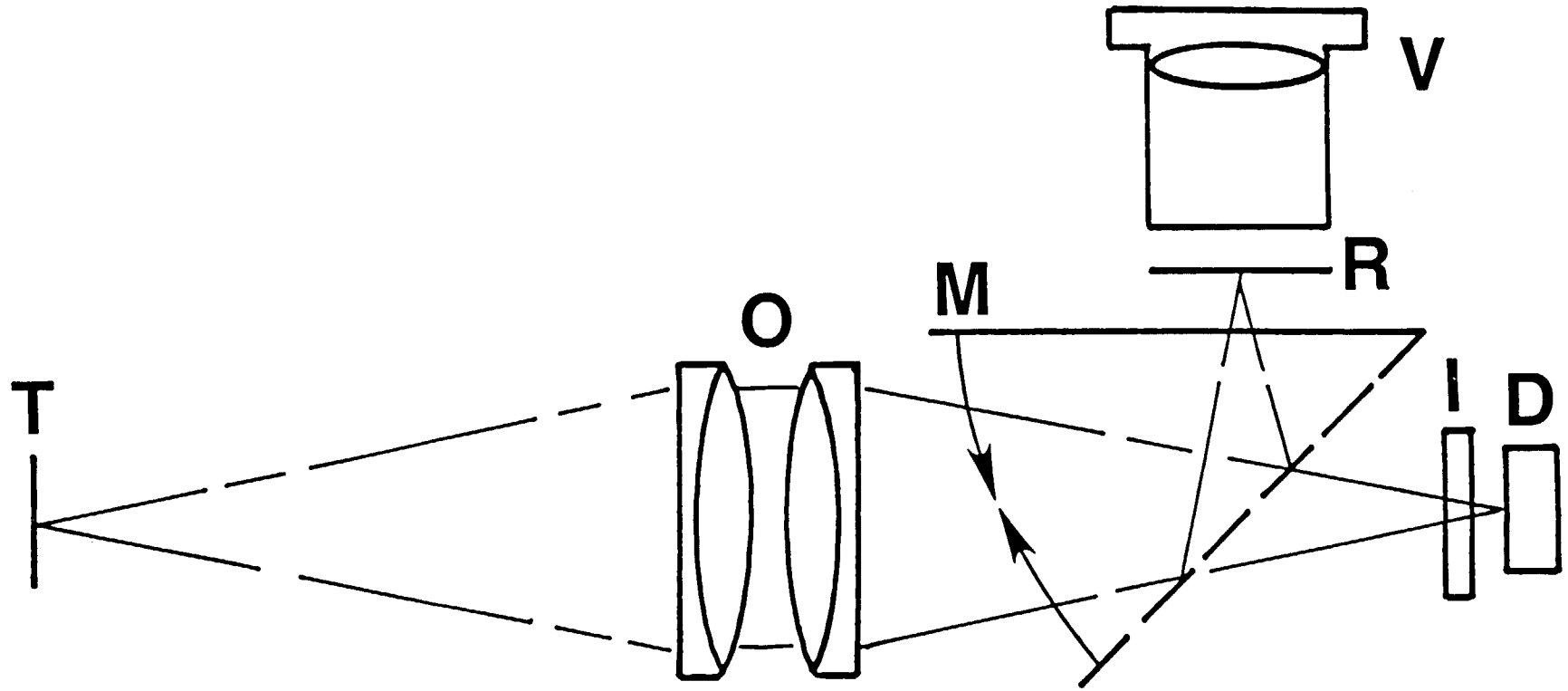


Fig. 1. Schematic diagram of the spatial (linear) scanning pyrometer where T is the target, O is the objective lens (1:1 magnification), I is the interference filter (40 nm bandwidth centered at 650 nm), D is a 1024-element silicon photodiode linear array, V is the viewing eyepiece, R is a reticle and M is a movable mirror.

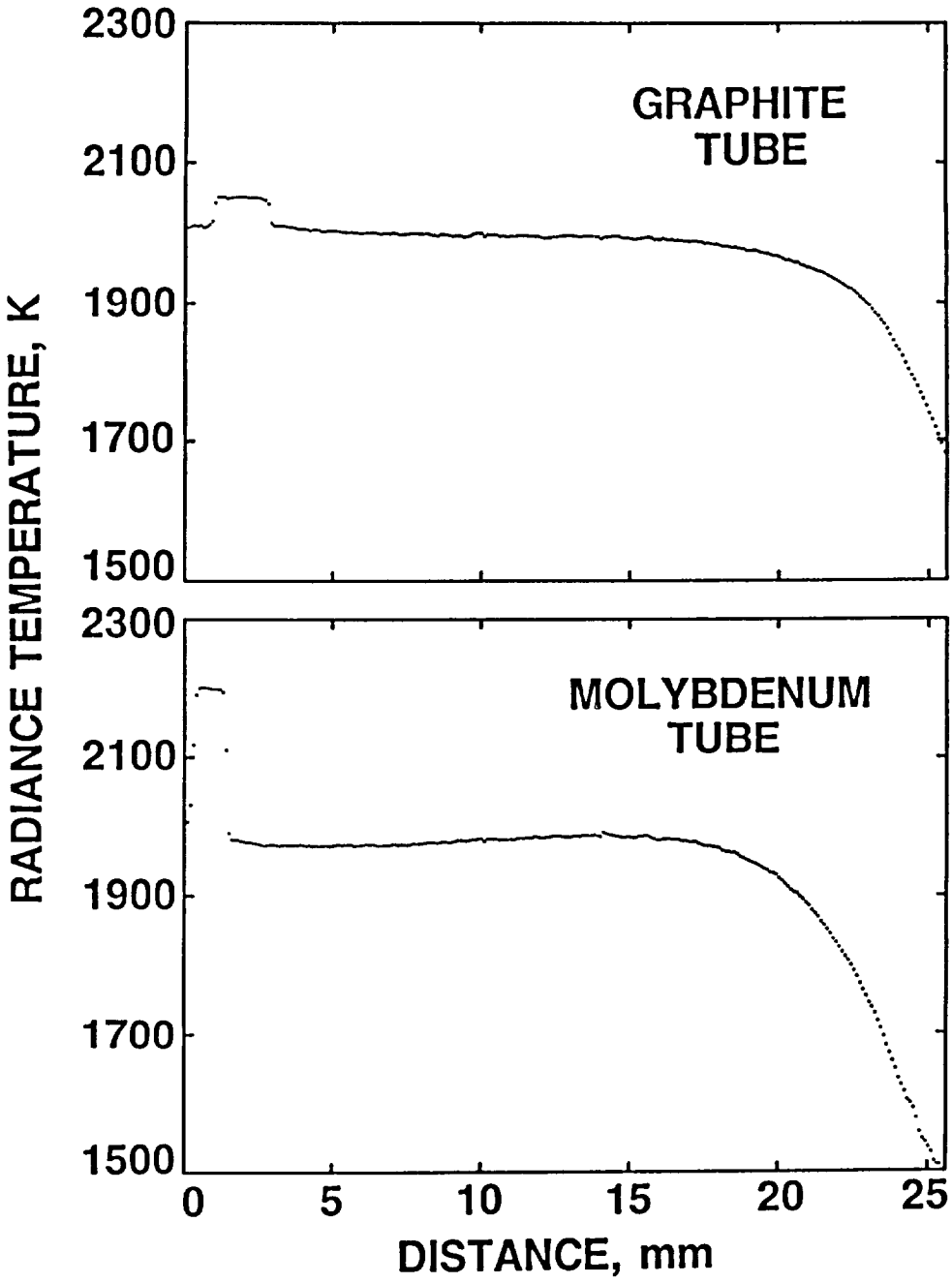


Fig. 2. A profile of the radiance temperature as measured by the pyrometer in a (1 ms) scan along one-half of a graphite tube (upper) and a molybdenum tube (lower) during rapid pulse heating. The region of increased radiance (left) is due to a small "blackbody" hole fabricated through the wall near the middle of the tube. A large temperature gradient (right) is observed along the specimen near the water-cooled clamp.

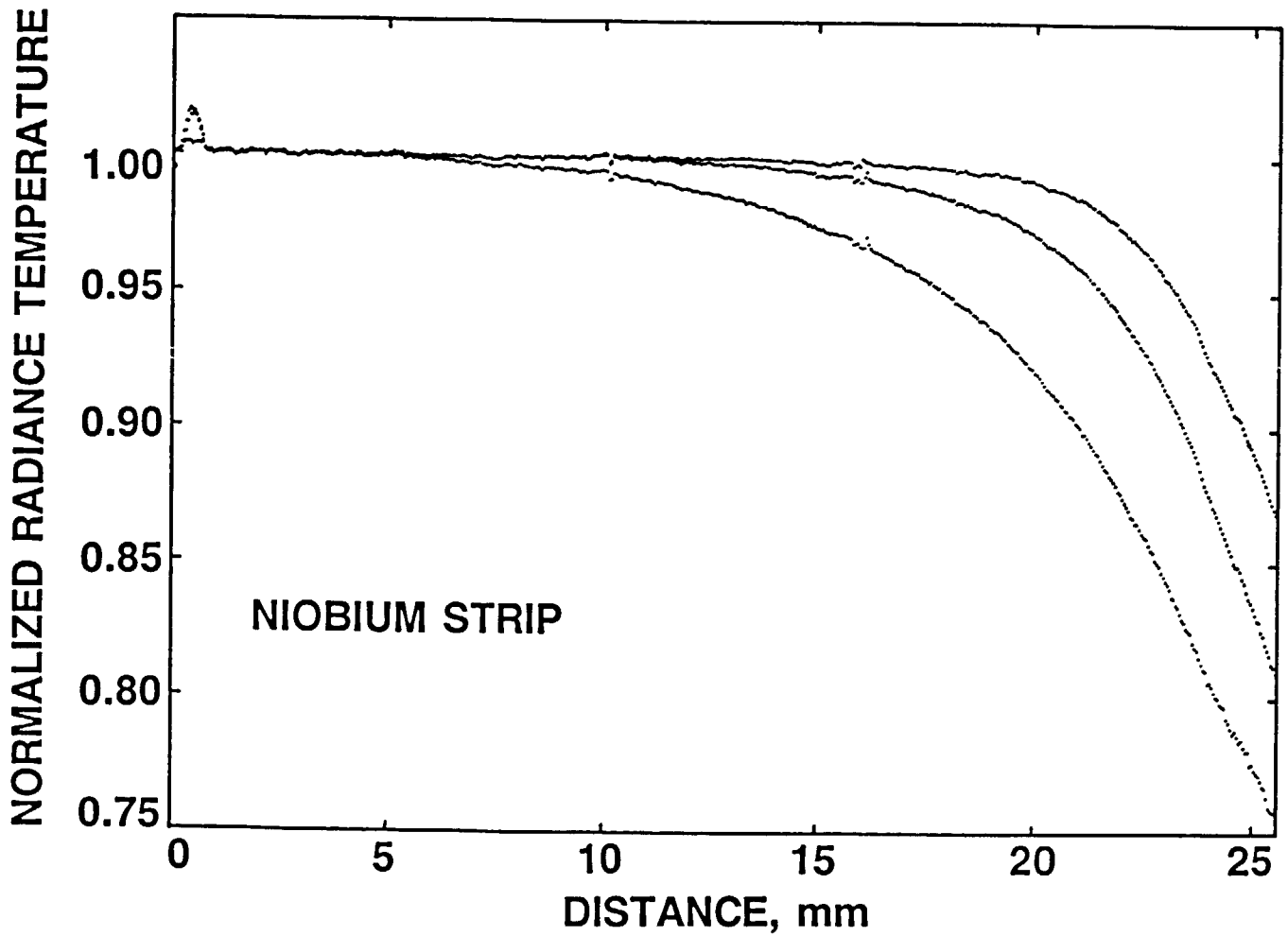


Fig. 3. Profiles of the radiance temperature as measured by the pyrometer in 1 ms-scans along one-half of a niobium strip during pulse heating at three different rates: highest heating rate (uppermost profile), lowest heating rate (lowermost profile).

N91-21362

Multiple Sensor Multifrequency Eddy Current Monitor for Solidification and Growth

J. Wallace
Casting Analysis Corp.

Recently we have developed a compact cylindrical multisensor eddy current measuring system with integral furnace to monitor II-VI crystal growth to provide interfacial information, solutal segregation and conductivities of the grown materials. The use of an array of sensors surrounding the furnace element allows one to monitor the volume of interest. Coupling these data with inverse multifrequency analysis allows radial conductivity profiles to be generated at each sensor position. At present work is going on to incorporate these outputs to control the processes within the melt volume. The standard eddy current system functions with materials whose electrical conductivities are as low as 2E2 Mhos/m. A need was seen to extend the measurement range to poorly conducting media so the unit was modified to allow measurement of materials conductivities 4 orders of magnitude lower and bulk dielectric properties. Typically these have included submicron thick films and semiinsulating GaAs. We have used this system to monitor complex heat transfer in grey bodies as well as semiconductor and metallic solidification studies. The ability to provide a multidimensional monitor of processing will be necessary for useful remote process control and understanding.

EDDY CURRENT MONITORING FOR MATERIALS PROCESSING

MULTISENSOR FURNACE CONTROLLER

1. NONCONTACT MATERIAL SENSING FOR CONTROL OF LOW THERMAL MASS FURNACE

2. LOCATING, POSITIONING FOR SIZE CONTROL, SEEDING, AND GROWTH

3. RECORD TRANSIENT PROPERTIES OF MELTING AND GROWTH

MATERIALS

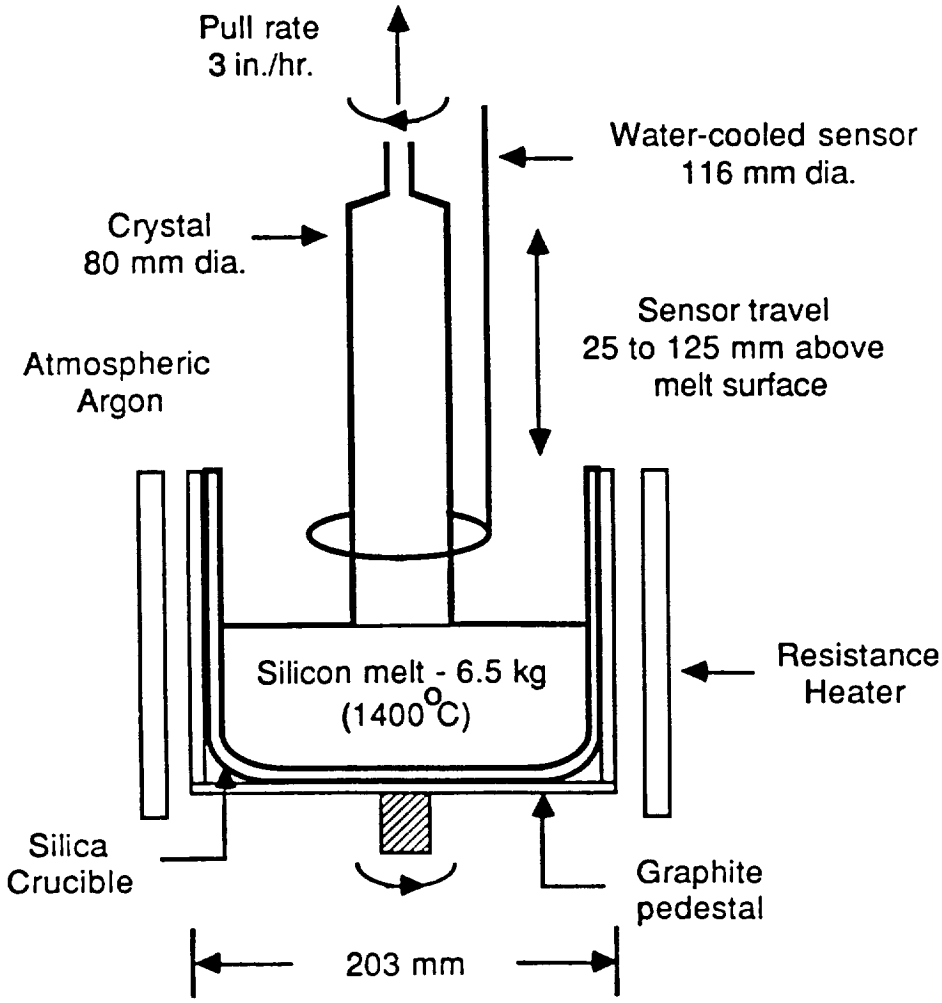
1970	1975	1982	1986	1988	1989
STEEL Induction Pressing Working	Cu and Al Alloys Melting Mixing Phase Sep.	Silicon Temp. Distr. Melt Interface	GaAs Compounding Temp. Distr. Melt Stab.	CdTe HgCdTe Complete Growth Zone Analysis	Aqueous Solutions

HARDWARE

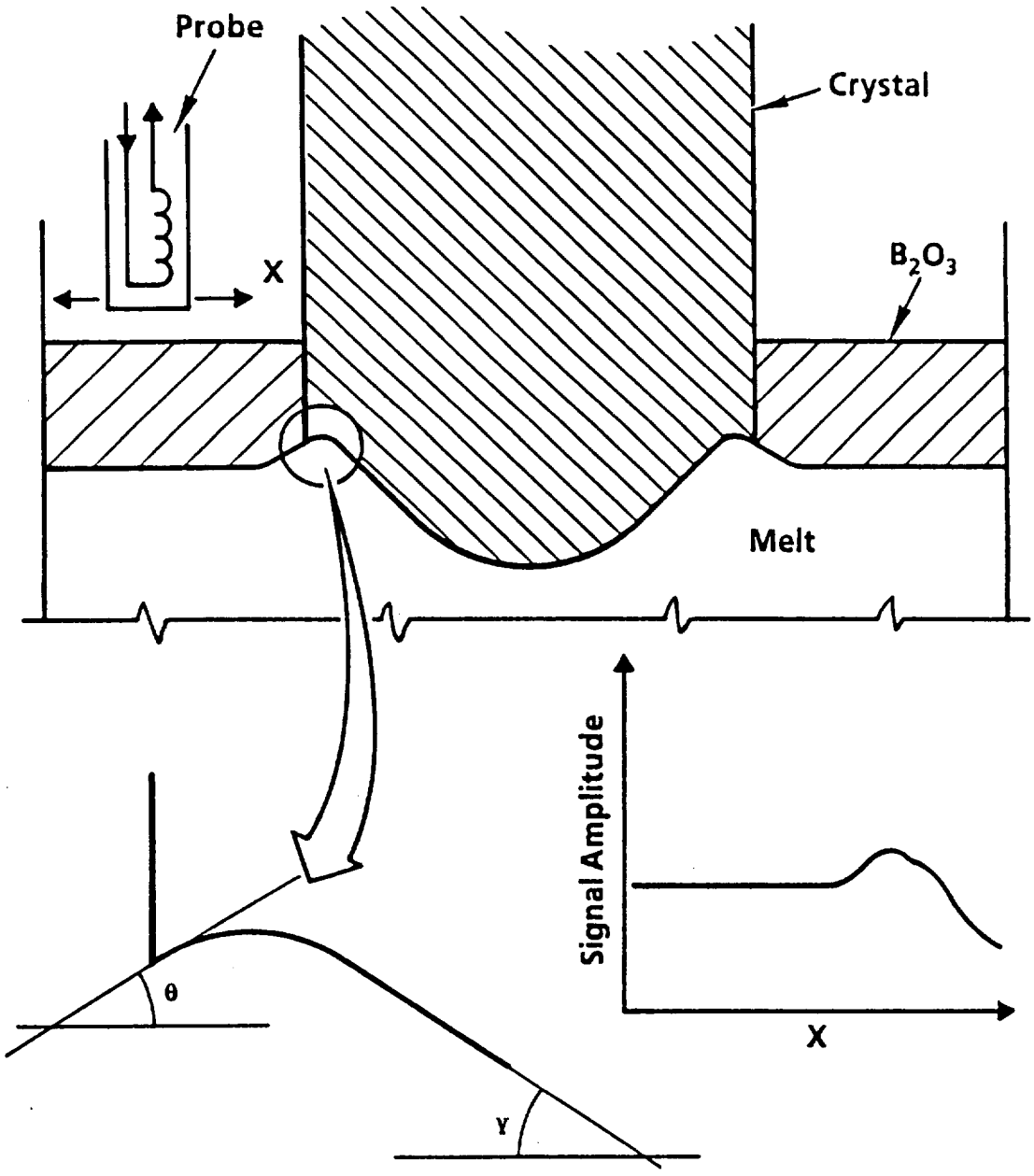
1972	1977-1978	1983	1987	1988
Sensor 1600C	Induction Environments Quadrature Calibration	Simult. Multi- Frequency	Sensor Arrays	Frequncy to 1.2 GHz

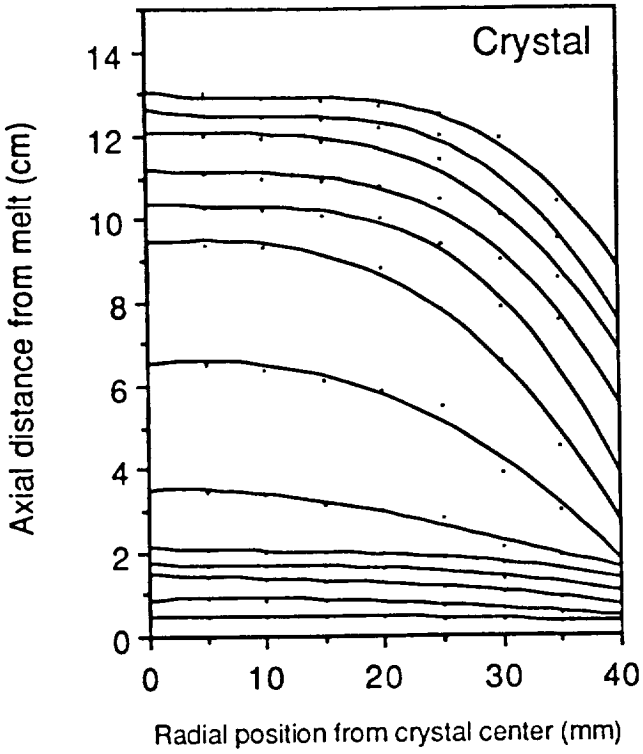
SOFTWARE

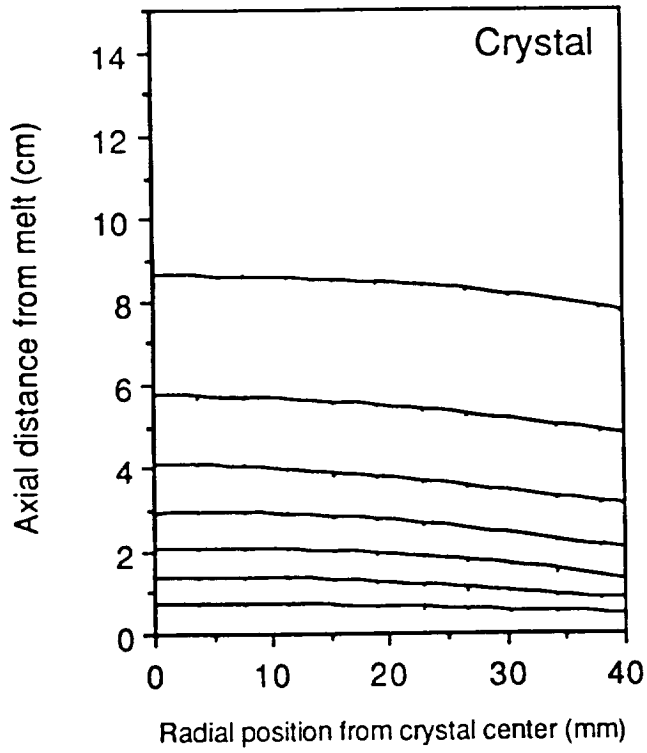
Computer- based data acqu.	Quadrature Relaxation	Inverse Property Analysis	Parallel Processing For Control

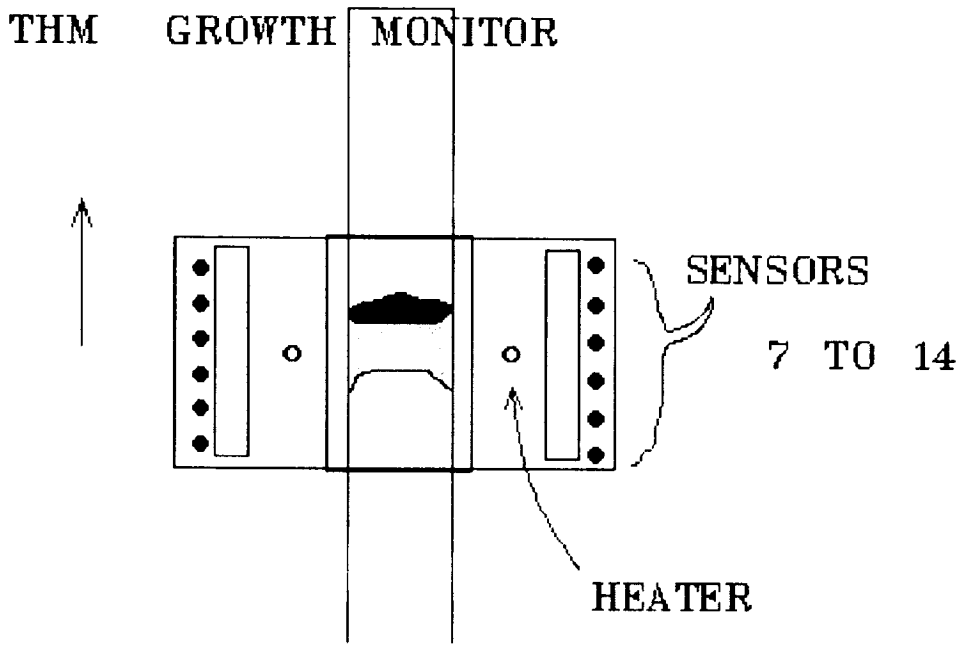

Pull rate
3 in./hr.
Water-cooled sensor
116 mm dia.
Crystal
80 mm dia.
Sensor travel
25 to 125 mm above
melt surface
Atmospheric
Argon
Silicon melt - 6.5 kg
(1400°C)
Resistance
Heater
Silica
Crucible
Graphite
pedestal
203 mm

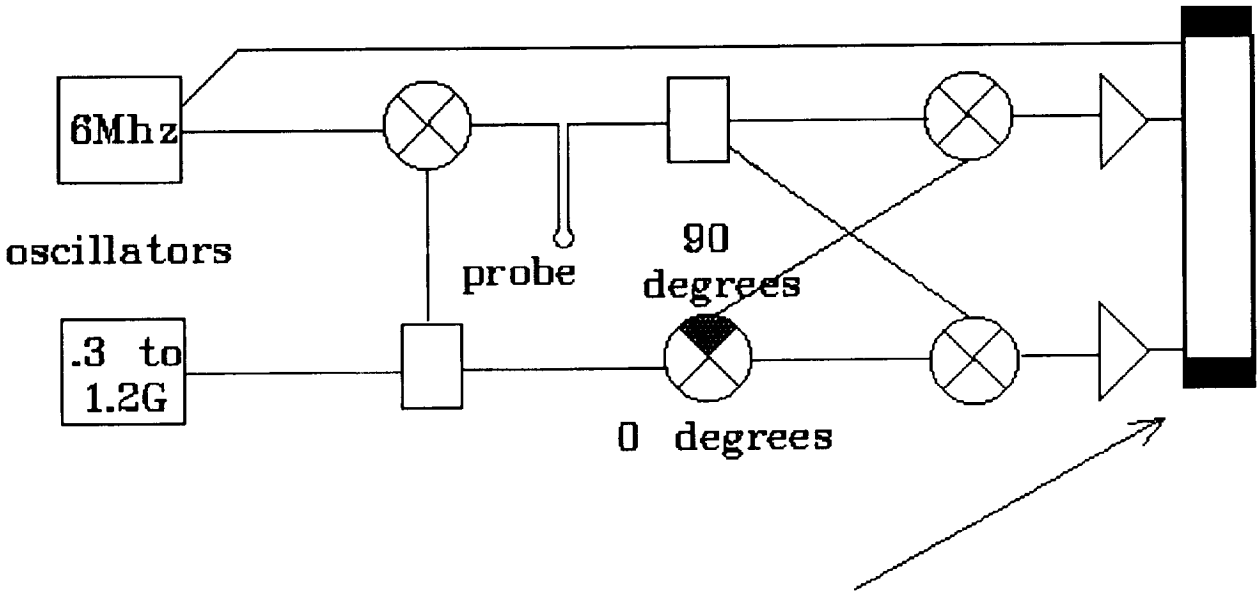
In Situ Eddy Current Analysis of Crystal Interface Shape



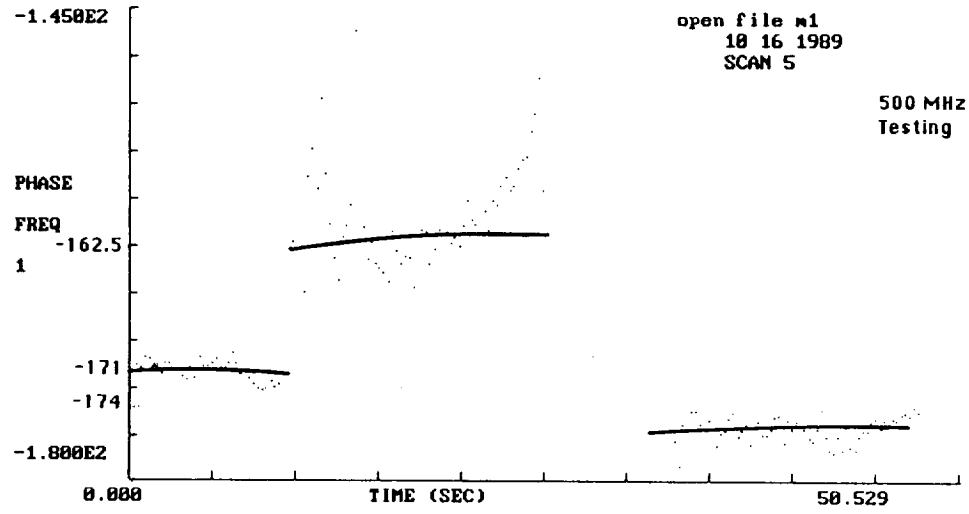

Crystal
Axial distance from melt (cm)
0
2
4
6
8
10
12
14
0
10
20
30
40
Radial position from crystal center (mm)

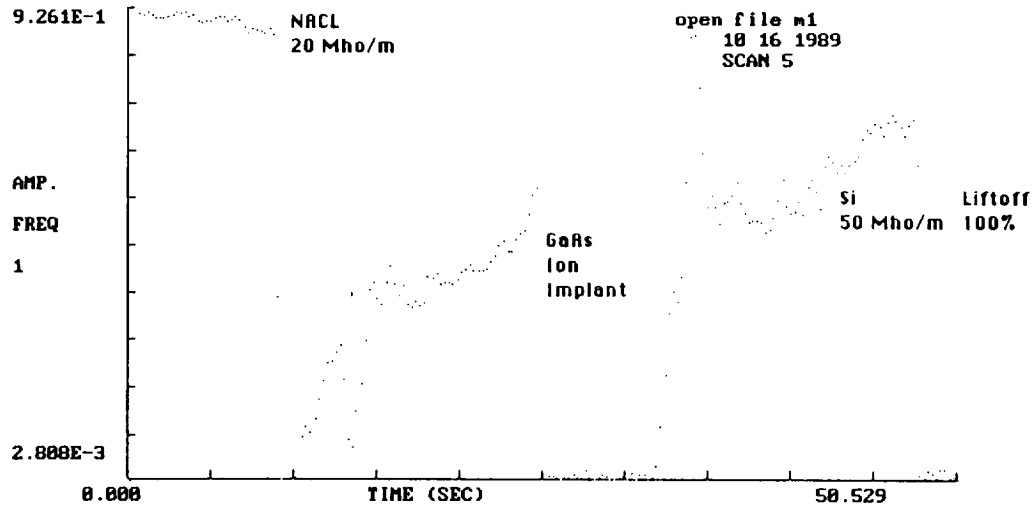

Crystal
Axial distance from melt (cm)
0
2
4
6
8
10
12
14
0
10
20
30
40
Radial position from crystal center (mm)

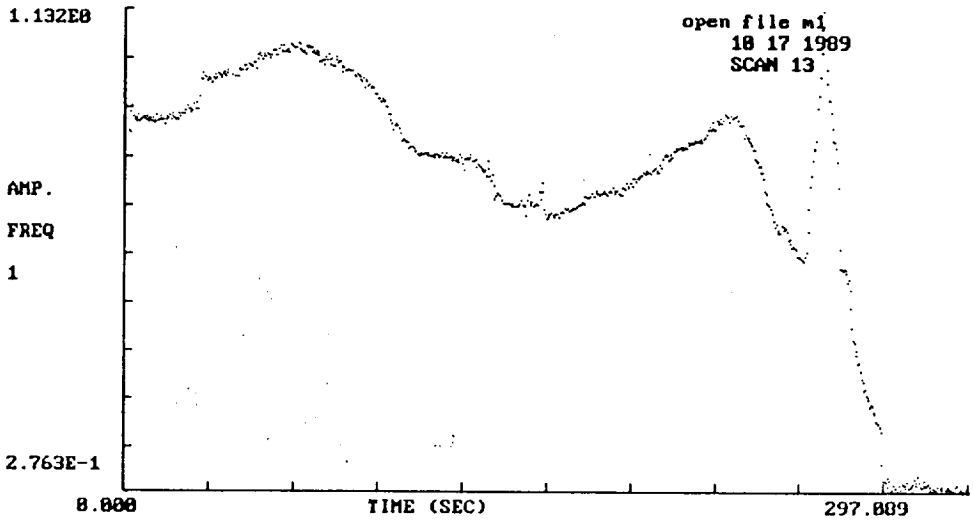

THM GROWTH MONITOR
SENSORS
7 TO 14
HEATER

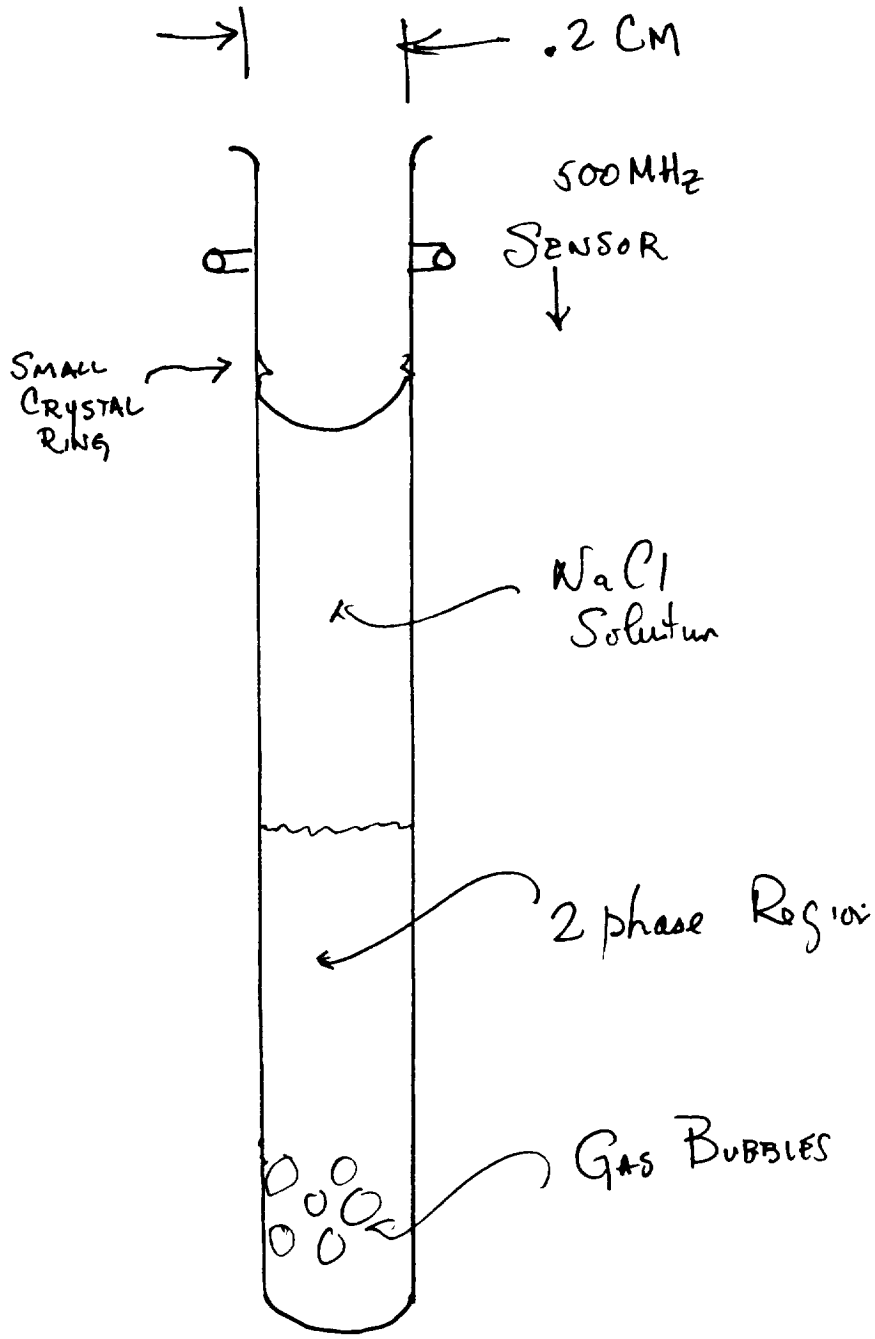


SYCHRONOUS DUAL DETECTOR


-1.450E2
open file m1
10 16 1989
SCAN 5
500 MHz
Testing
PHASE
FREQ
-162.5
1
-171
-174
-1.800E2
0.000
TIME (SEC)
50.529


9.261E-1
NACL
20 Mho/m
open file m1
10 16 1989
SCAN 5
AMP.
FREQ
1
Si
50 Mho/m
Liftoff
100%
GaAs
Ion
Implant
2.000E-3
0.000
TIME (SEC)
50.529


1.132E0
open file m1
10 17 1989
SCAN 13
AMP.
FREQ
1
2.763E-1
0.000
TIME (SEC)
297.089


.2 CM
500 MHz
SENSOR
SMALL CRYSTAL RING
NaCl Solution
2 phase Region
GAS BUBBLES

N91-21363

Workshop on Containerless Experimentation in Microgravity

High-Temperature Metal Purification Using a Compact, Portable rf Heating and Levitation System on the Wake Shield

C. A. Hahs
Oak Ridge National Laboratory*

This paper describes the potential use of a compact, battery-operated rf levitator and heating system to purify high-temperature melting materials in space. The wake shield now being fabricated for the Space Vacuum Epitaxy Center at the university of Texas will provide an Ultra-high vacuum (-10-14 Torr hydrogen, 10-14 Torr helium, 10-30 Torr oxygen,...). This paper illustrates the use of the wake shield to purify niobium, titanium, tungsten, iridium, and other metals to a purity level not achievable on earth.

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* Operator by Martin Marietta Energy Systems, Inc. for the U.S. Department of Energy under Contract No. DE-AC05-840R21400.

NASA

HIGH TEMPERATURE METAL PURIFICATION USING A COMPACT PORTABLE RF HEATING AND LEVITATION SYSTEM ON THE WAKE SHIELD

C. A. Hahs

Instrumentation & Controls Division

Oak Ridge National Laboratory

ornl

NASA

HIGH TEMPERATURE METAL PURIFICATION EXPERIMENTS USING A COMPACT PORTABLE RF HEATING AND LEVITATION SYSTEM ON THE WAKE SHIELD

• Vacuum	10^{-14} Torr
• Sample Heating & Levitation	Liquid-Cooled rf Coils
• Operating Temperature Range	650 - 2600^{o} C
• Battery Power	1500 W
• Process Control	3-point Contact (of sample material)
• Number of Samples	60
• Sample Stability	> 1 rpm
• Sample Access	Via Coil Ends
• Video Camera	Sample Detail
	General Viewing

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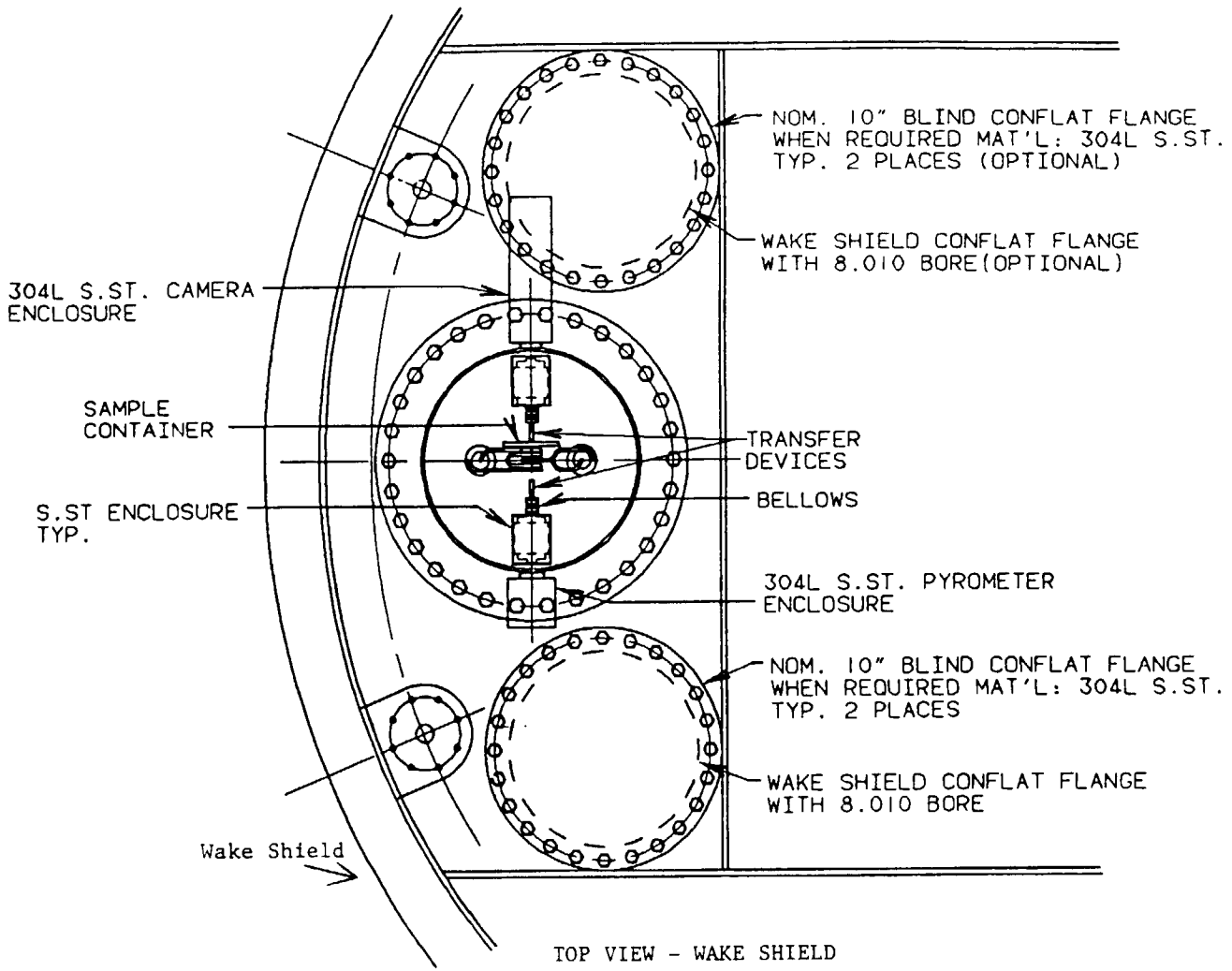
NOM. 10" BLIND CONFLAT FLANGE
WHEN REQUIRED MAT'L: 304L S.ST.
TYP. 2 PLACES (OPTIONAL)
WAKE SHIELD CONFLAT FLANGE
WITH 8.010 BORE(OPTIONAL)
304L S.ST. CAMERA
ENCLOSURE
SAMPLE
CONTAINER
TRANSFER
DEVICES
BELLOWS
S.ST ENCLOSURE
TYP.
304L S.ST. PYROMETER
ENCLOSURE
NOM. 10" BLIND CONFLAT FLANGE
WHEN REQUIRED MAT'L: 304L S.ST.
TYP. 2 PLACES
WAKE SHIELD CONFLAT FLANGE
WITH 8.010 BORE
Wake Shield
TOP VIEW - WAKE SHIELD

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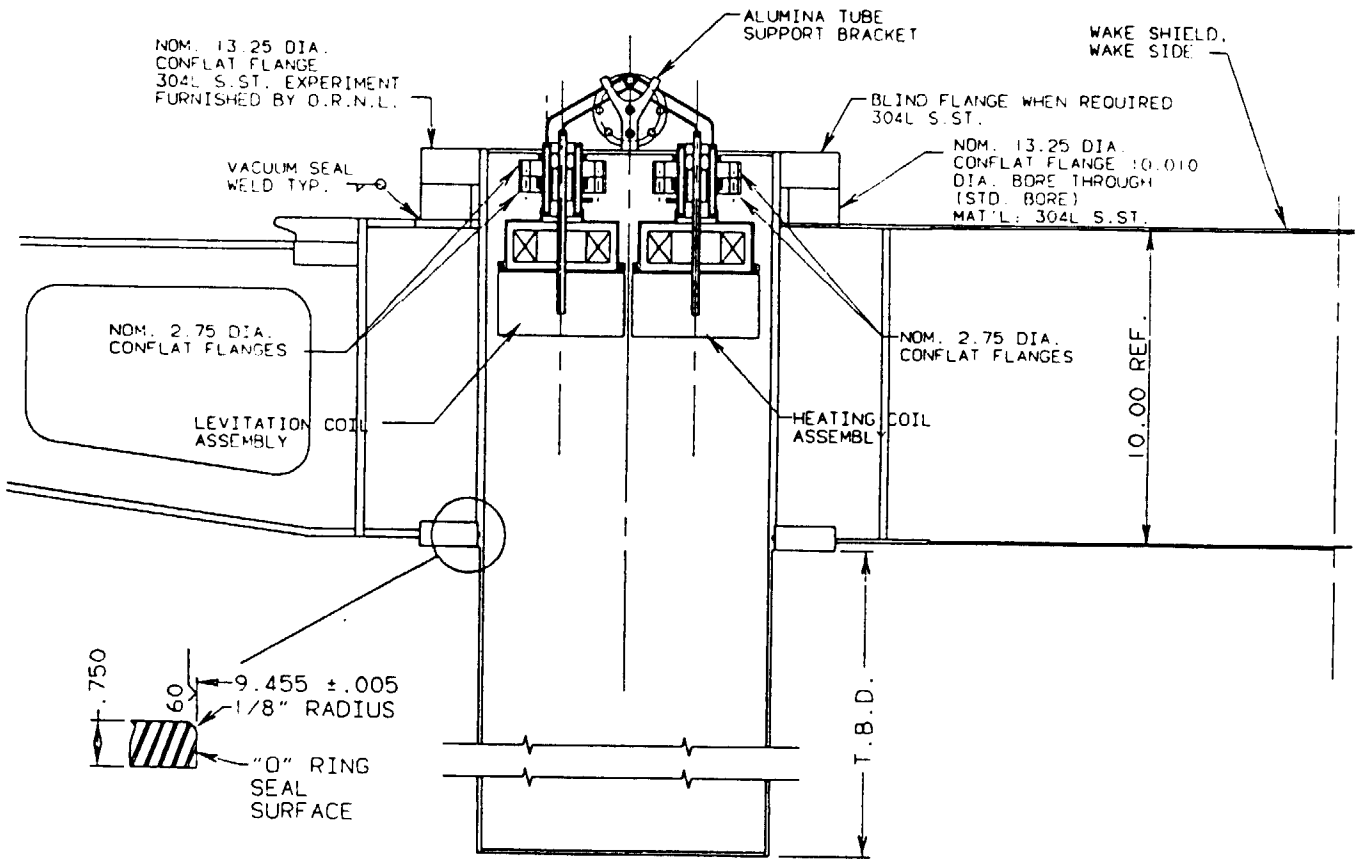

Figure 2 Typical Section

TYPICAL SECTION - WAKE SHIELD

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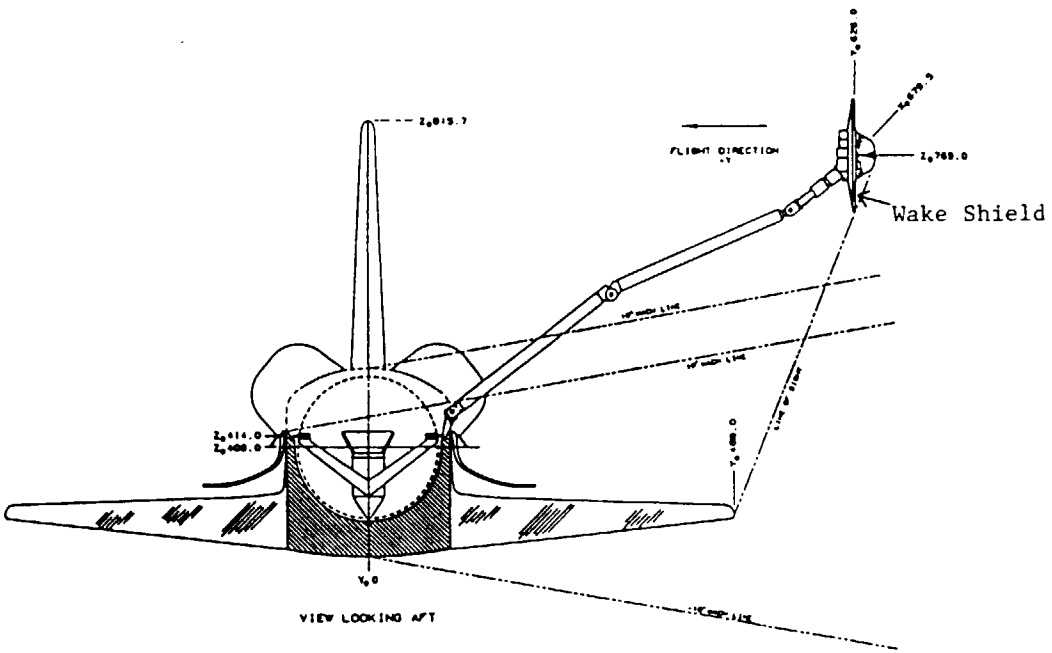
WSF Flight Position (cont)
(GG /0° Roll Attitude)



ornl

N91-21364

Workshop on Containerless Experimentation in Microgravity

Compact rf Heating and Levitation Systems for the NASA Modular Electromagnetic Levitator

R. J. Fox
Oak Ridge National Laboratory*

The levitator demonstrates levitation of a 5mm diam aluminum sphere at 1 G using a small, compact rf levitator operating from a small 12-V battery. This system is designed to levitate and melt niobium in space; however, the small battery limits the power for melting demonstrations. This system was developed for NASA-MSFC in Huntsville, Alabama, as part of the Modular Electromagnetic Levitator development.

* Operator by Martin Marietta Energy Systems, Inc. for the U.S. Department of Energy under Contract No. DE-AC05-840R21400.

Mel Project at ORNAL

R.J. Fox

PROBLEM STATEMENT

The primary goal of the MEL project is containerless induction-melting and processing of metals, e.g., niobium, in the micro-G environment of earth-orbit.

POWER LIMITATIONS

The maximum electrical power available on the shuttle is 2 KW for each MSL user. For the MEL operation, this power must be shared between the induction-heater and all peripheral equipment. A reasonable power allocation for just the induction-heater might be 1 KW.

INDUCTION-HEATER COIL EFFICIENCY

In the laboratory, containerless melting metals often accomplished by inductively heating the sample in a "cusp" coil. Such a coil consists of two coaxial coils connected series-opposed. These coils can levitate as well as heat a metallic sample. However, the heating efficiency of a cusp coil is rather poor because the sample is levitated at a point rather near the zero point in the magnetic flux of the coil. Figure 1 is a computer generated B-field map of a 4-turn cusp coil intended for use in a micro-G environment. Note that, in the plot, the two turns in the center are not activated and are not involved in generating the levitation field. The two center turns serve a different purpose. Connected series-aiding and driven from an independent rf source, this coil heats the sample much more efficiently than the cusp coil because the sample is held near the position of its flux maximum. Thus, we have a three-coil dual-frequency configuration. Although this system is more complex than the simple cusp coil, the added complexity is justified by the improved efficiency.

RF TRANSFORMER

A resonant toroidal current transformer provides the high circulating secondary currents necessary to achieve adequate power transfer to the sample while using a simple rugged heater coil.

POWER INVERTER

A pair of 30 ampere MOSFET switches in a push-pull class D circuit (Fig. 2) are link coupled to the resonant transformer previously discussed. Self-excitation is used to avoid any operation off-resonance that might result from load changes.

INVERTER PERFORMANCE HIGHLIGHTS

With the DC input current set to 28 amperes at nominal bus voltage, the rf current to the heater coil exceeded 300 amperes (Fig. 3). This was sufficient to levitate both tungsten and platinum (at 1-G). Set to a lower input current, 15 amperes, a platinum sample (Fig. 4), was heated to over 2600°C.

SYSTEM EFFICIENCY

An overall heating efficiency measurement was made. The input power was read from the DC inputs and the output power was measured calorimetrically as the thermal power delivered to a water-cooled 5- mm diameter inconel sphere (molten niobium equivalent resistivity).

Input power: 29 V X 17.5 A = 507.5 W
The thermal power to the sample was 178.8 W
Efficiency: 178.8/507.5 = 35.2% from the power bus.

NASA

MODULAR ELECTROMAGNETIC HEATING AND LEVITATION COIL FOR THE MEL

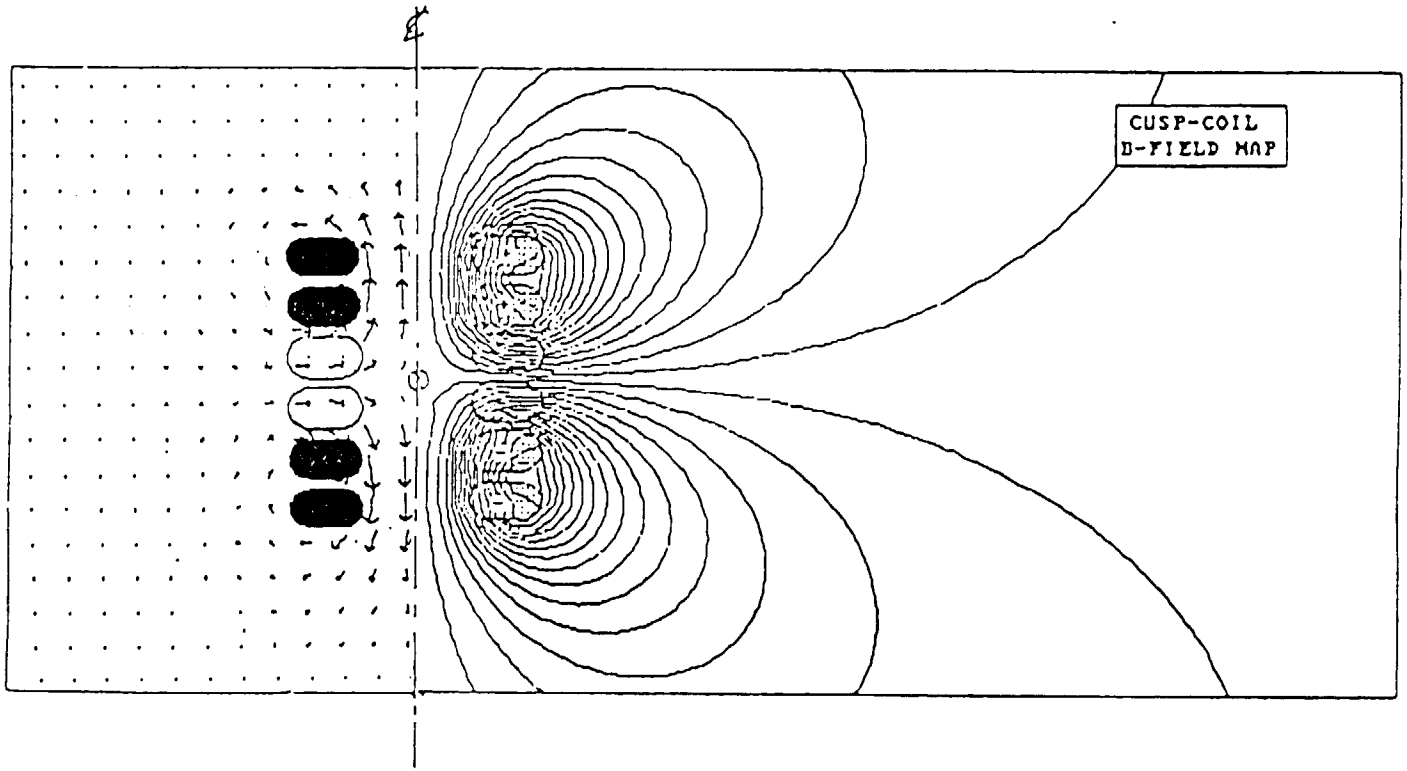


FIG. 1

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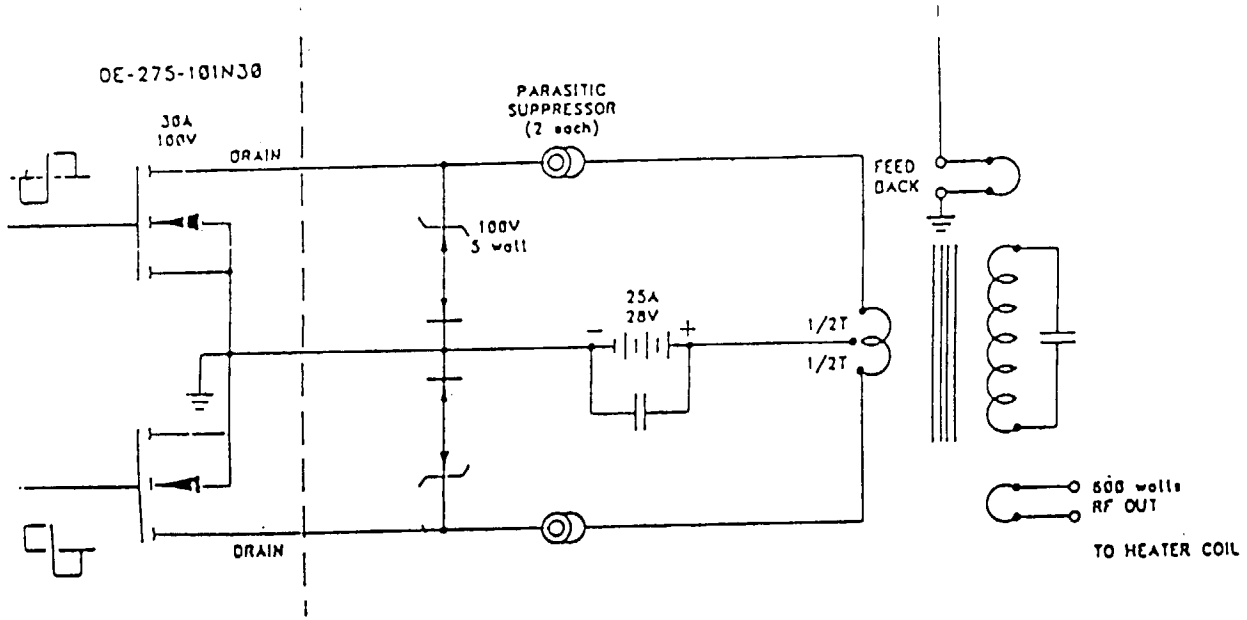

PUSH-PULL RF-POWER
SELF EXCITED

Fig. 2

RJF, 8-4-89

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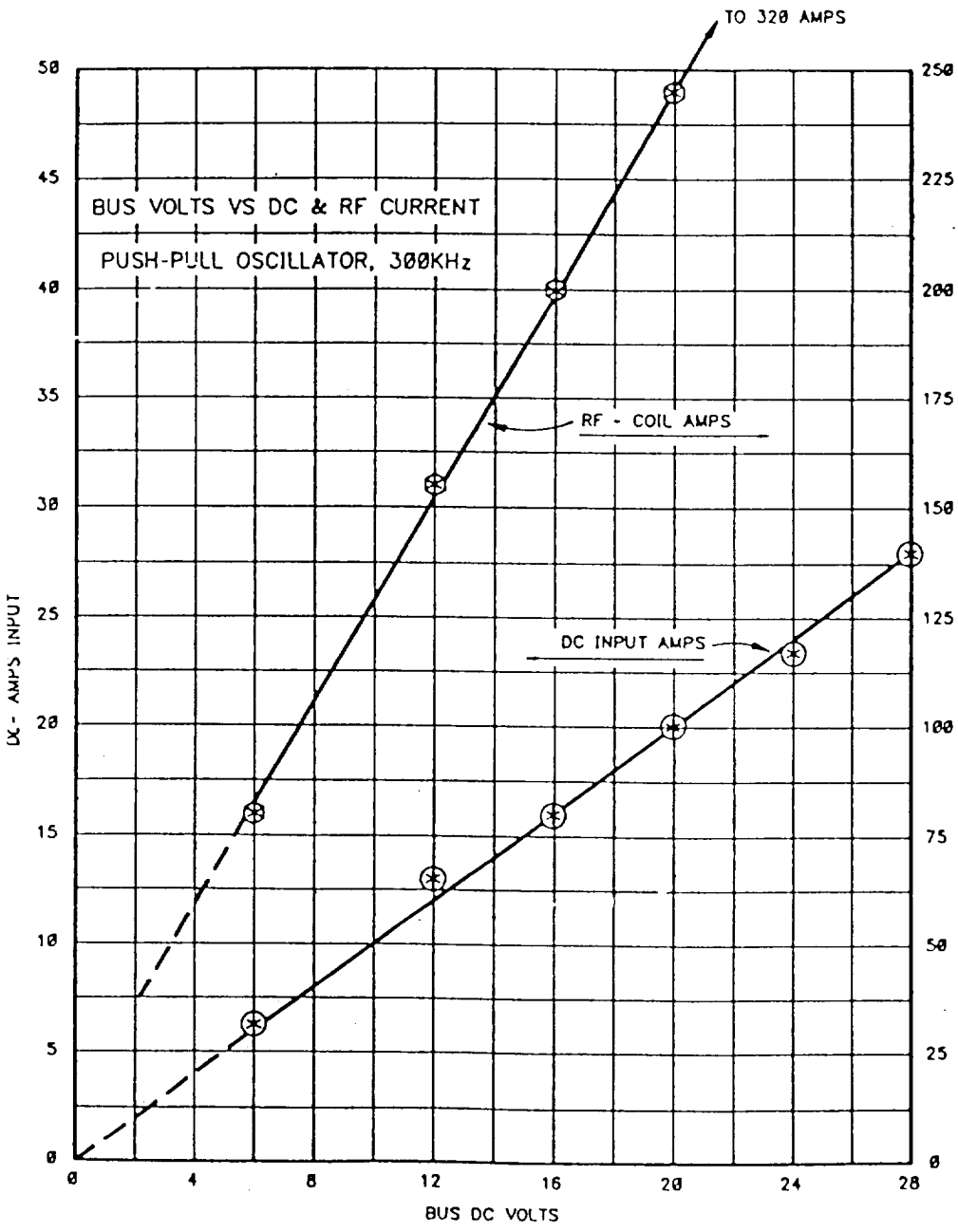
TO 320 AMPS
BUS VOLTS VS DC & RF CURRENT
PUSH-PULL OSCILLATOR, 300KHz
RF - COIL AMPS
DC INPUT AMPS
DC- AMPS INPUT
BUS DC VOLTS
0
4
8
12
16
20
24
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Fig. 3

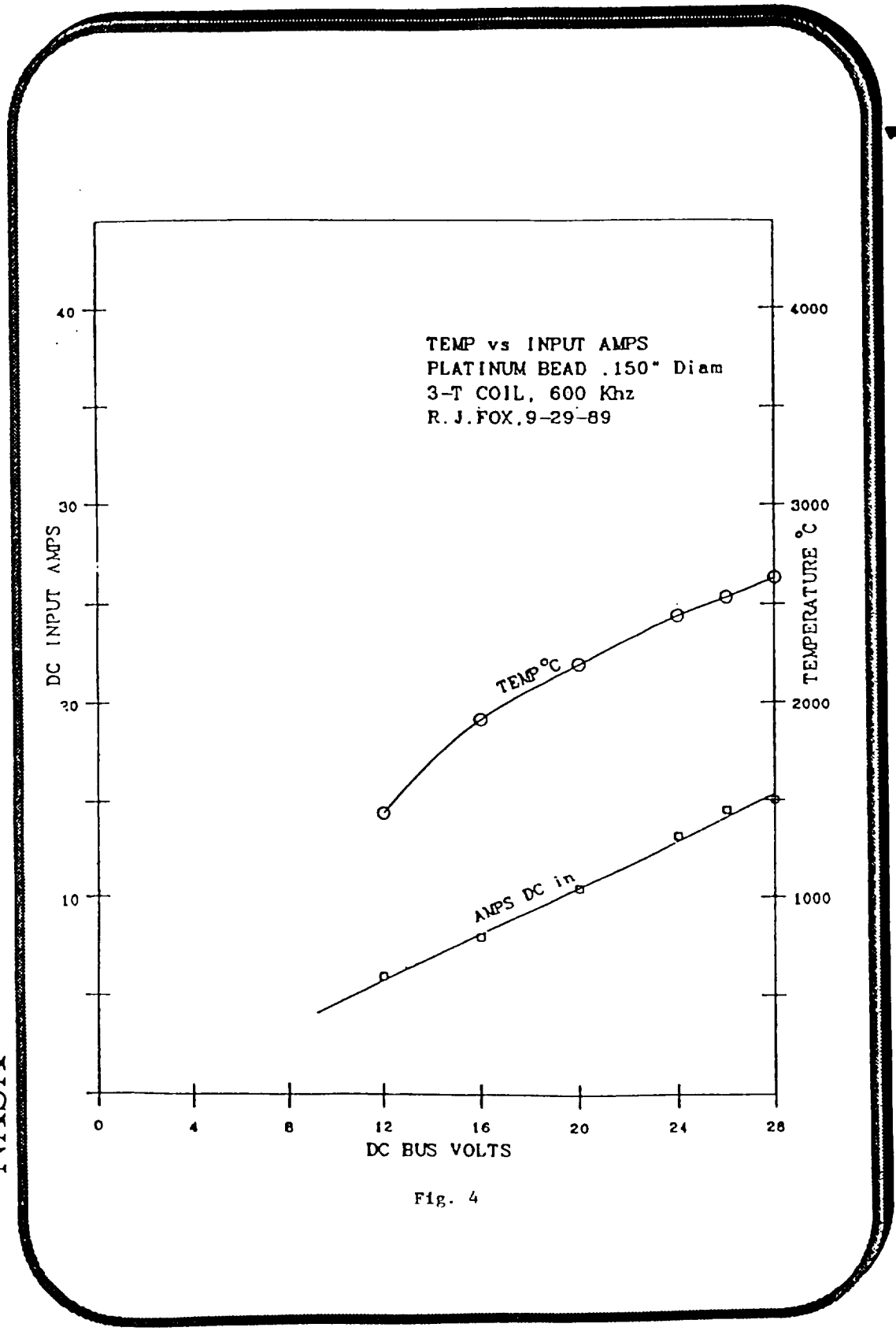


Fig. 4

N91-21365

MEL PROJECT AT ORNL
C. A. HAHS 1/17/90

SUBJECT: Containerless Experimentation in Microgravity Workshop

TITLE: High Temperature Metal Purification Using a Compact Portable Rf Heating and Levitation System on the Wake Shield

INTRODUCTION:

The Wake Shield Facility (WSF), now under construction at the University of Houston, can provided an ideal vacuum environment for the purification of high temperature metals in space. The Modular Electromagnetic Levitator, (MEL), being developed for NASA at the Oak Ridge National Laboratory, will provide principal investigators the opportunity to study undercooling of metals in space and allow them to determine material properties in space. The battery powered rf levitation and heating system developed for the MEL has a demonstrated efficiency of 36%. Sample handling hardware is under development to process multiple samples. This hardware is sufficiently compact to be installed on the WSF. Figures 1 and 2 illustrate how the rf heating and levitation system can be mounted on the wake shield. This system is being considered to purify metals at temperatures below 3000^{o}C.

N91-21366

Optical Property Measurements as a Diagnostic Tool for Control of Materials Processing in Space and on Earth

S. Krishnan, J.K.R. Weber, P.C. Nordine, and R.A. Schiffman,
Intersonics Incorporated, 3453 Commercial Ave, Northbrook, Il.

ABSTRACT

One of the major science justifications for containerless processing of materials in microgravity is the possibility of developing new and exciting materials through high temperature processing via the liquid state. The lack of convection produces quiescent melts enabling undercooling, glass formation and other interesting phenomenon to occur readily. However, methods for following, controlling and measuring processing parameters are not fully developed at this time. In this paper, we describe a new method, including results, to measure, control and follow containerless processing in ground based levitators.

This new technique enables instantaneous optical property measurements from a transient solid or liquid surface concurrent with true temperature measurement. This has been used succesfully as a diagnostic tool to follow processing of aluminum, silicon and titanium during electromagnetic levitation. Experiments on aluminum show the disappearnce of the oxide (emittance 0.33) at ca. 1300 C leaving a liquid surface with an emittance of 0.06. Electromagnetic levitation of silicon shows a liquid with a constant emittance (0.2) but with a solid whose emittance decreases very rapidly with increasing temperature. Consequently, the processing of materials at high temperatures can be controlled quite well through the control of surface optical properties. Candidate materials will be described and science justifications will be presented.

RESULTS: The first figure illustrates the change in polarization that occurs on reflection from a surface and also the design of a laser polarimeter in the case of a levitated droplet. The second figure summarizes the experiments that illustrate the versatility of the laser polarimetric technique as a diagnostic tool in containerless processing. The third figure shows the temperature dependence of the spectral emissivity for clean liquid aluminum while the fourth slide compares the data obtained in our levitation experiments for solid and liquid silicon with those in the liqterature. The last figure shows oxide formation and removal in the case of iridium illustrating that laser polarimetry is sensitive to changes in surface chemistry and physics.

CONCLUSION: It has been clearly demonstrated that laser polarimetry is a fast, reproducible technique that allows process chemistry and physics to be monitored and controlled. Intersonics is currently developing the Division of Amplitude Polarimetric Pyrometer (DAPP) for accurate temperature and emissivity measurement.

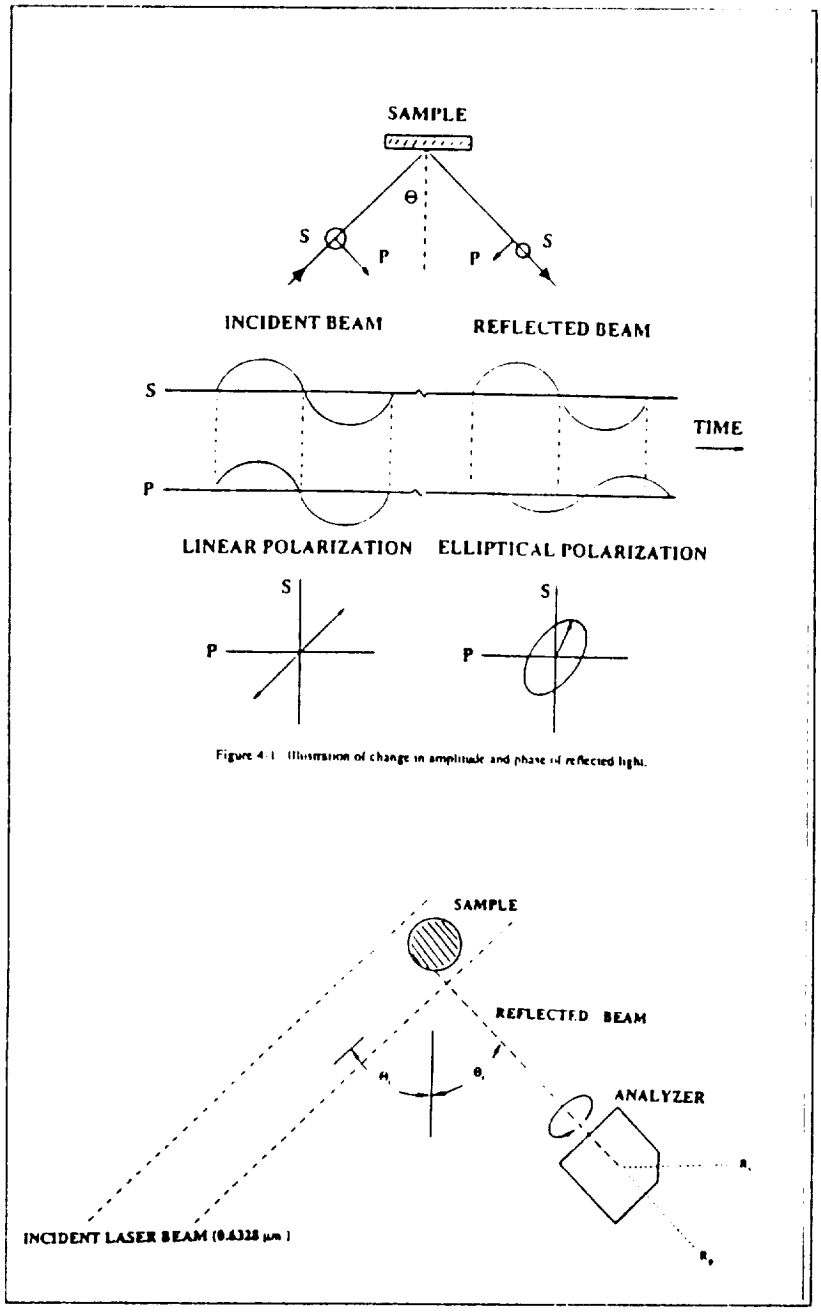


Figure 4-1 Illustration of change in amplitude and phase of reflected light.

Illustration of Rotating Analyzer Ellipsometry

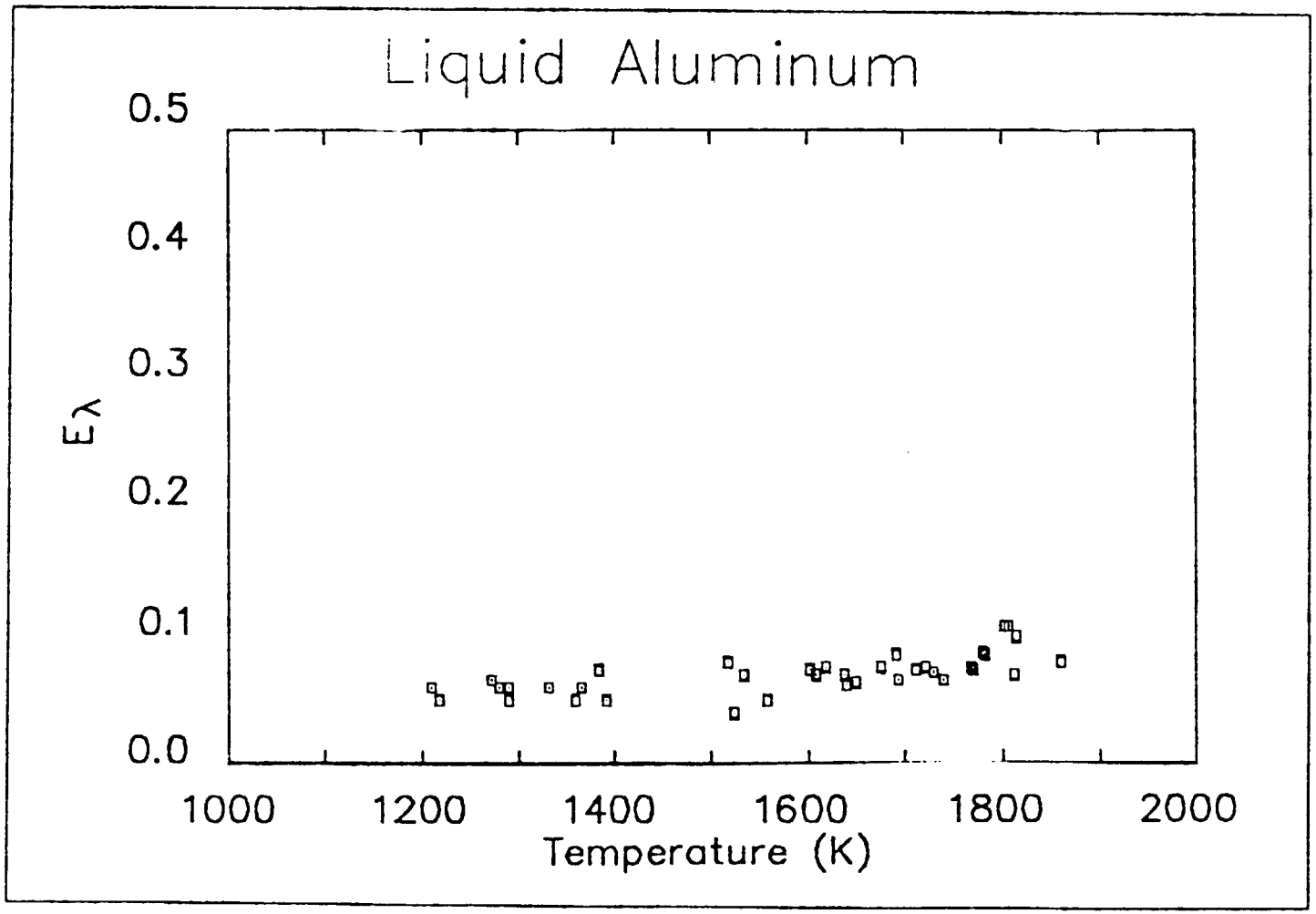
SUMMARY

* LASER POLARIMETRY IS FAST, REPRODUCIBLE, AND EXACT METHOD TO MEASURE THE OPTICAL PROPERTIES OF MATERIALS AT HIGH TEMPERATURES IN CONTAINERLESS EXPERIMENTS.

* IT HAS BEEN USED TO FOLLOW:

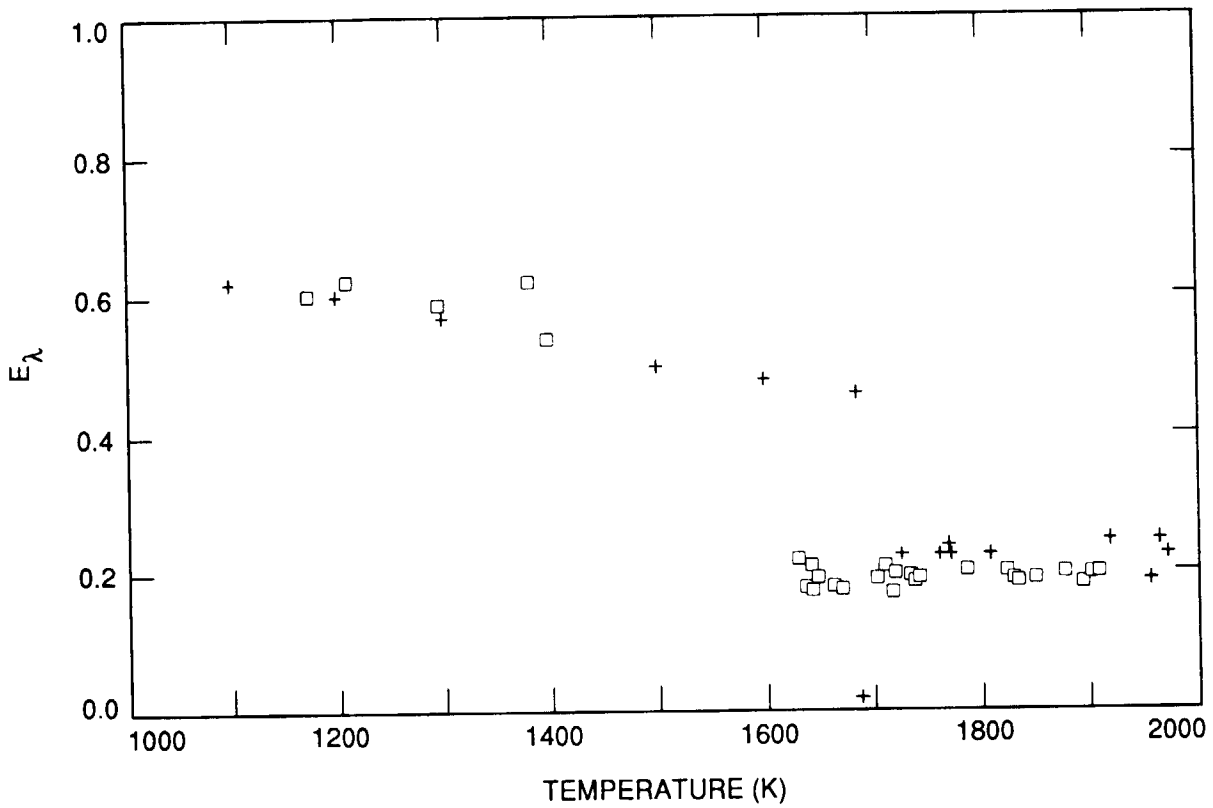
a OXIDATION – Ir, B
b PHASE TRANSFORMATION – Hf
C MELTING, UNDERCOOLING – Nb, Pt, Pd, Si
d BAND STRUCTURE CHANGES – Cu, Au, Pd

* DIVISION OF AMPLITUDE POLARIMETRIC PYRAMETER (DAPP) IS BEING DEVELOPED FOR MICROGRAVITY APPLICATIONS BY INTERSONICS INCORPORATED.

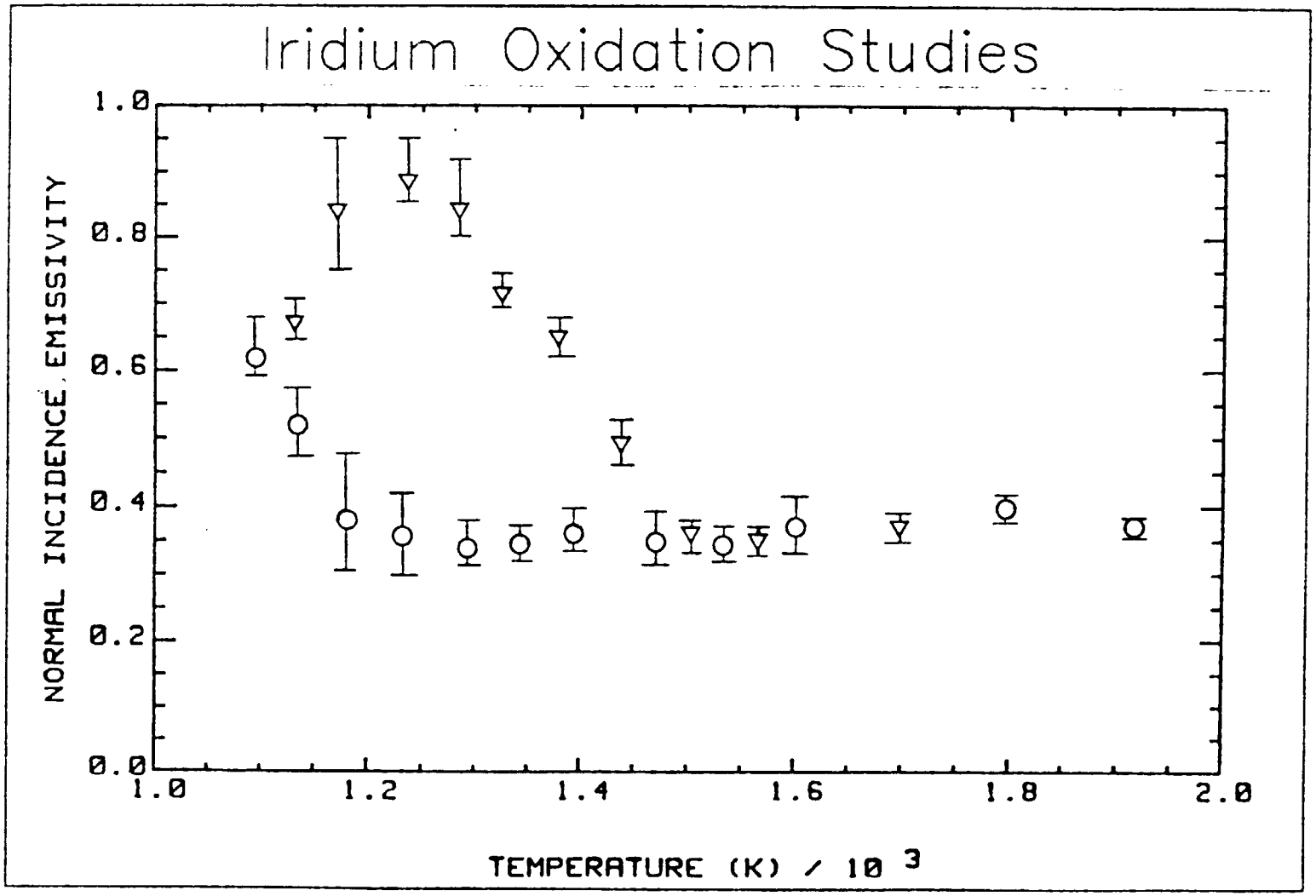


Normal incidence spectral emissivity of liquid aluminum as a function of temperature at 633 nm (□).

SOLID AND LIQUID SILICON



NORMAL INCIDENCE SPECTRAL EMISSIVITY OF SOLID AND LIQUID SILICON AS A FUNCTION OF TEMPERATURE AT 633 nm (□). LITERATURE DATA INDICATED BY (+). MELTING POINT INDICATED BY ARROW.



Oxidation hystersis of iridium reprsenting the average results of experiments in air, 10% O_2 – Ar, and 30% O_2 – Ar taken during a cooling (○) and heating (▽) cycle.

N91-21367

Containerless Processing of Amorphous Ceramics

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ABSTRACT

The absence of gravity allows containerless processing of materials which could not otherwise be processed. High melting point, hard materials such as borides, nitrides and refractory metals are usually brittle in their crystalline form. The absence of dislocations in amorphous materials frequently endows them with flexibility and toughness.

Systematic studies of the properties of many amorphous materials have not been carried out. The requirements for their production is that they can be processed in a controlled way without container interaction. Containerless processing in microgravity could permit the control necessary to produce amorphous forms of hard materials.

INTRODUCTION

Amorphous forms of some ceramic materials are of scientific and technological interest because of their unique properties. Compared to their crystalline counterparts, amorphous materials are frequently tougher, more flexible and offer superior corrosion resistance. It is also possible to transform an amorphous phase to a crystalline one after processing it.

The formation of amorphous ceramics is potentially easier than formation of amorphous metals. This is because ceramics are polyatomic molecules, crystallization then requires the condensation of an appropriate set of atoms in lattice sites. In these circumstances, structure in the liquid phase and the processing route[1-3] can influence the ability to form a particular phase. The effect of convection can be to enhance mixing and therefore make crystallization possible in situations where diffusional limitations would prevent this. Impurities and condensed phases can have a strong effect on the ability to form amorphous materials.

EXPERIMENTAL REQUIREMENTS

Some desirable experimental requirements are given below:

1. Processing of high melting point non-conductors above the liquidus.

2. Control of nucleation. The ability to process in the absence of heterogeneous nucleation sites is important.

3. Control of material purity.

4. Control of processing atmosphere.

5. Quiescent samples, free from convection.

Some of these conditions can be obtained by containerless processing on Earth. The very limited capability for containerless processing of non-conducting, low viscosity liquids presents a significant experimental limitation. While it is possible to process small samples for very short periods[4,5], longer experiments with samples larger than a few hundred milligrams are not possible with presently available ground-based technology.

There is frequently a need to achieve all of the above experimental conditions simultaneously. Microgravity processing can allow this because the positioning forces are small and buoyancy-driven convection is eliminated.

SCIENTIFIC RESEARCH OPPORTUNITIES

1. Elimination of Heterogeneous Nucleation: This would extend the range of materials from which glasses could be made. Work by Day etal[6] in a single-axis acoustic levitator in microgravity has demonstrated a factor of ca. 2.7 reduction in critical cooling rate (Rc) for a silica-based glass. The reduction in Rc was attributed to the absence of heterogeneous nucleation. Many glass systems of scientific or technological interest[7] cannot be formed in the presence of nucleation sites or with presently available hardware.

2. Preparation of Pure, Clean Materials: In most cases, the properties of materials have been measured by contained techniques. The purity of the sample has been assumed equal to that of the starting material. Containerless experiments by the authors[8] indicate that this assumption is not always correct. Containerless processing in a controlled atmosphere at high temperature provides a means to produce clean, pure samples.

Figure 1 shows a plot of pressure vs. inverse temperature for the vapor species over silicon and aluminum with an ambient oxygen pressure of 10^{-6} atm. This illustrates that the liquid elements can be cleaned of oxide by evaporation. Further oxidation is suppressed by cleaning the atmosphere with a blanket of vapor from the specimen.

3. Quiescent Samples Free From Convection: This potentially allows accurate measurement of diffusion coefficients in liquids. Convection effects can mask diffusion effects by stirring of liquids. This makes it difficult to perform studies of the effects of diffusion on nucleation in multi-component liquids.

4. High Temperature Liquid Phase Processing Without Contamination: Impurities can arise from two sources; induced and inherent. Induced impurities occur due to dissolution or reaction with a container. Thermodynamic criteria show that the solubility of the container increases with temperature. While some kinetic inhibition of container dissolution can be expected, at temperatures above about 1500 K, impurity concentrations of tens of parts per million can be expected. Containerless processing completely eliminates this source of contamination.

Inherent impurities are those present in the starting material. The concentration of these can sometimes be decreased by appropriate processing techniques. For example, some impurities can be removed as vapor, volatile oxide or other species.

5. Superheating Above the Liquidus: Nucleation can be caused by sparingly soluble impurities. Materials such as carbides, nitrides or oxides of similar density to the bulk material can remain suspended in the liquid and act as nuclei. If the liquid is superheated, the impurities can be dissolved.

6. Impurity Nucleation Studies: It has been shown that nucleation of gas bubbles in liquid iron can be controlled by adjusting the ambient oxygen pressure[9]. Refractory metal oxides can be formed from impurities at low activity such as aluminum. Once the ambient oxygen pressure is sufficient to form the refractory oxide, this provides a nucleation site.

7. Complete Processing Without Contact: This provides the possibility of repeated heating, melting and solidification without the introduction of impurities from handling.

HARDWARE REQUIREMENTS AND CAPABILITIES

The basic hardware requirements are outlined below. Operation in microgravity is necessary to achieve the requirements of many experiments. The minimum requirement for instrumentation is noncontact temperature measurement.

* Position control of poorly conducting liquid samples.

* High temperature capability, 2000-2500 K.

* Controlled atmosphere operation.

* Instrumentation: NCTM, Emittance, Gas Analysis, Video Imaging.

Following is a brief discussion of some flight hardware. These are described more fully in other papers in these proceedings.[10,11]

Processing furnaces: Figure 2 shows the High Temperature Acoustic Levitator (HAL). This and the Acoustic Levitation Furnace (ALF) operate on a similar principle to the SAAL but with temperature capability to 2000 K or above. They are highly developed versions of the space hardened SAAL and incorporate a

advanced features[12]. Both use six acoustic transducers laid out in opposed orthogonal pairs. This produces spherically symmetric energy wells which can be used to process a liquid sample in a controlled atmosphere. The HAL system has been used to process liquid samples in a program aboard the KC-135[13].

Instrumentation: The Division of Amplitude Polarimetric Pyrometer (DAPP) combines an accurate radiometer and an ellipsometer to provide true noncontact temperature measurement[14,15]. This application of an ellipsometer enables the emittance of the surface to be measured. The DAPP eliminates one of the major sources of uncertainty in noncontact temperature measurement.

CONCLUSIONS

The ability to process materials in microgravity extends the range of materials which can be studied and processed. The addition of microgravity processing experiments to a ground-based research program offers the opportunity to quantify the effects of heterogeneous nucleation, impurities and convection on specific systems.

Appropriate hardware already exists to make high temperature studies of ceramic materials. It is important that this technology is put into use and that flight opportunities become available in the near future. This goal could be achieved by extending the KC-135 program and supplementing this with sounding rocket experiments.

A portion of this work is funded under NASA contracts.

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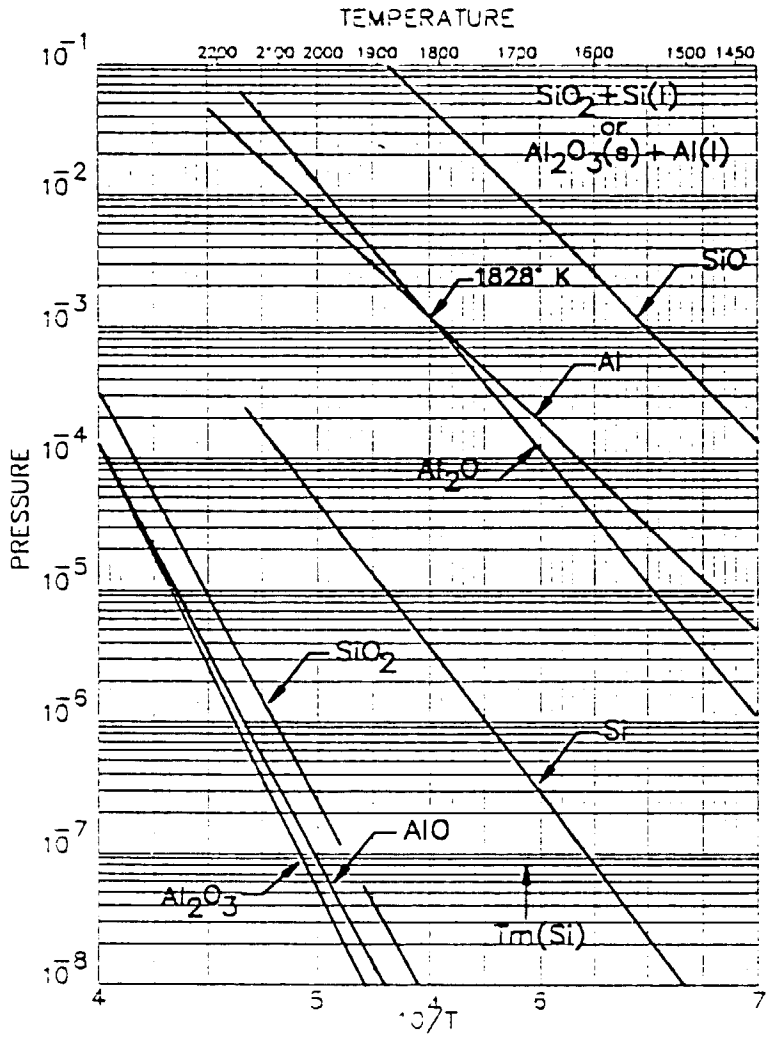


Figure 1

Plot of Vapor Pressure of Species over the Al-O and Si-O Systems as a Function of Inverse Temperature. $pO_2 = 10^{-6}$ Atm.

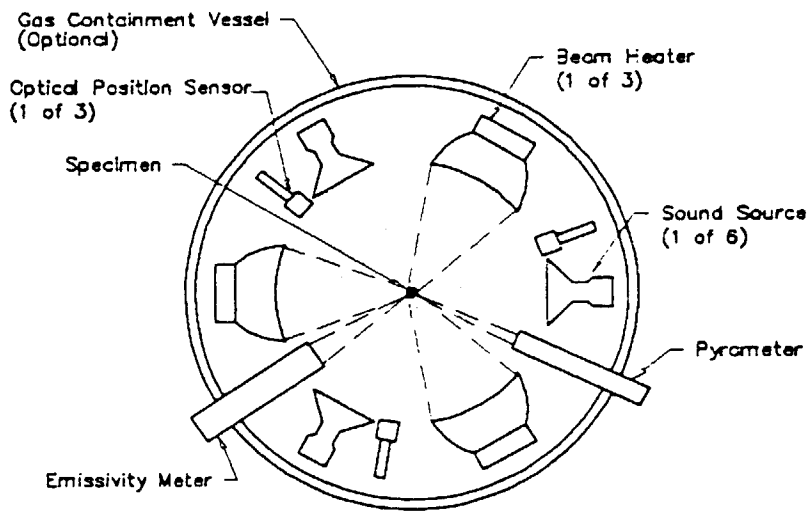


Figure 2

Schematic View of the Intersonics High Temperature Acoustic Levitator.

N91-21368

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY WORKSHOP

ABSTRACT

HIGH TEMPERATURE ACOUSTIC AND HYBRID MICROWAVE/ACOUSTIC LEVITATORS FOR MATERIALS PROCESSING: PROGRESS REPORT

M. Barmatz

Jet Propulsion Laboratory, California Institute of Technology, Pasadena, CA 91109

The physical acoustics group at the Jet Propulsion Laboratory has developed a single mode acoustic levitator technique for advanced containerless materials processing. This technique was successfully demonstrated in ground-based studies to temperatures ≈ 1000 °C in a uniform temperature furnace environment and to temperatures > 1500 °C using laser beams to locally heat the sample. At this time, we are evaluating microwaves as a more efficient means than lasers for locally heating a positioned sample. Recent tests of a prototype single mode hybrid microwave/acoustic levitator successfully demonstrated the feasibility of using microwave power as a heating source. The potential advantages of combining acoustic positioning forces and microwave heating for containerless processing investigations will be discussed and results of ground-based acoustic, microwave, and hybrid microwave/acoustic studies will be presented.

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY WORKSHOP

HIGH TEMPERATURE ACOUSTIC AND HYBRID MICROWAVE/ACOUSTIC LEVITATORS FOR MATERIALS PROCESSING: PROGRESS REPORT

MARTIN BARMATZ

JPL

JANUARY 18, 1990

PRESENTATION OUTLINE

- HIGH TEMPERATURE SINGLE MODE ACOUSTIC LEVITATOR
  - ISOTHERMAL ENVIRONMENT ($\leq$ 1000 °C)
  - LASER BEAM HEATING ($\approx$ 1500 °C)
  - VIDEO TAPE
- HYBRID MICROWAVE/ACOUSTIC LEVITATOR
  - ADVANTAGES OF MICROWAVE HEATING
  - DEVELOPMENT PROGRAM
  - TEMPERATURE FEEDBACK CONTROL
  - PROTOTYPE HYBRID
  - POTENTIAL SCIENCE AREAS

MICROWAVE/ACOUSTIC

RESEARCH TEAM

DR. M. BARMATZ - MICROWAVE/ACOUSTIC PHYSICIST

DR. J. WATKINS - MICROWAVE/ACOUSTIC PHYSICIST

MR. J. STONEBURNER - ACOUSTIC PHYSICIST

DR. H. JACKSON - THEORETICAL PHYSICIST

DR. C. SHIPLEY - SCIENTIFIC PROGRAMMER

MR. G. AVENI - ACOUSTIC SCIENTIST

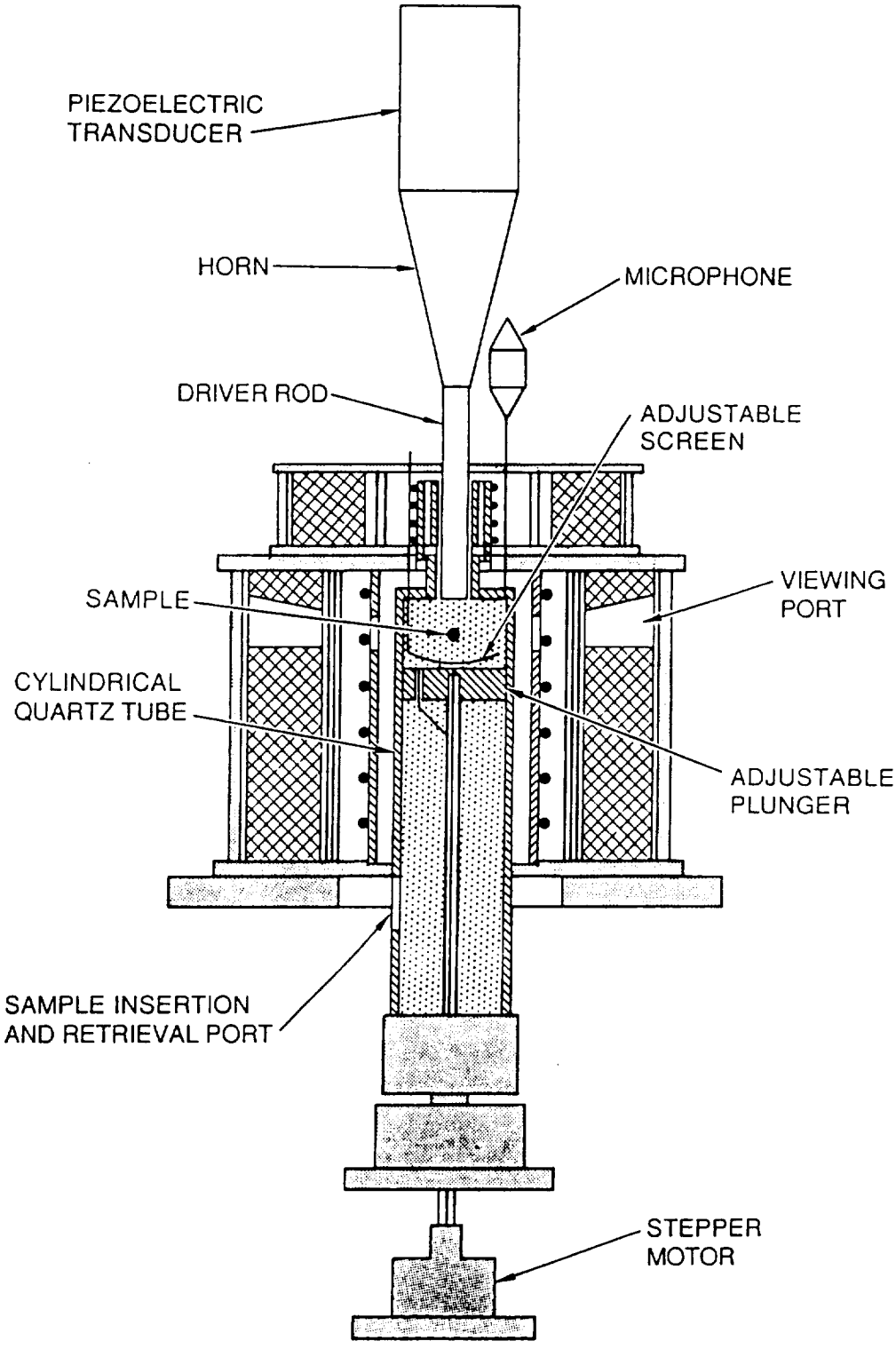
MR. C. HAGENLAGER - PROGRAMMER

MR. R. ZANTESON - MACHINIST

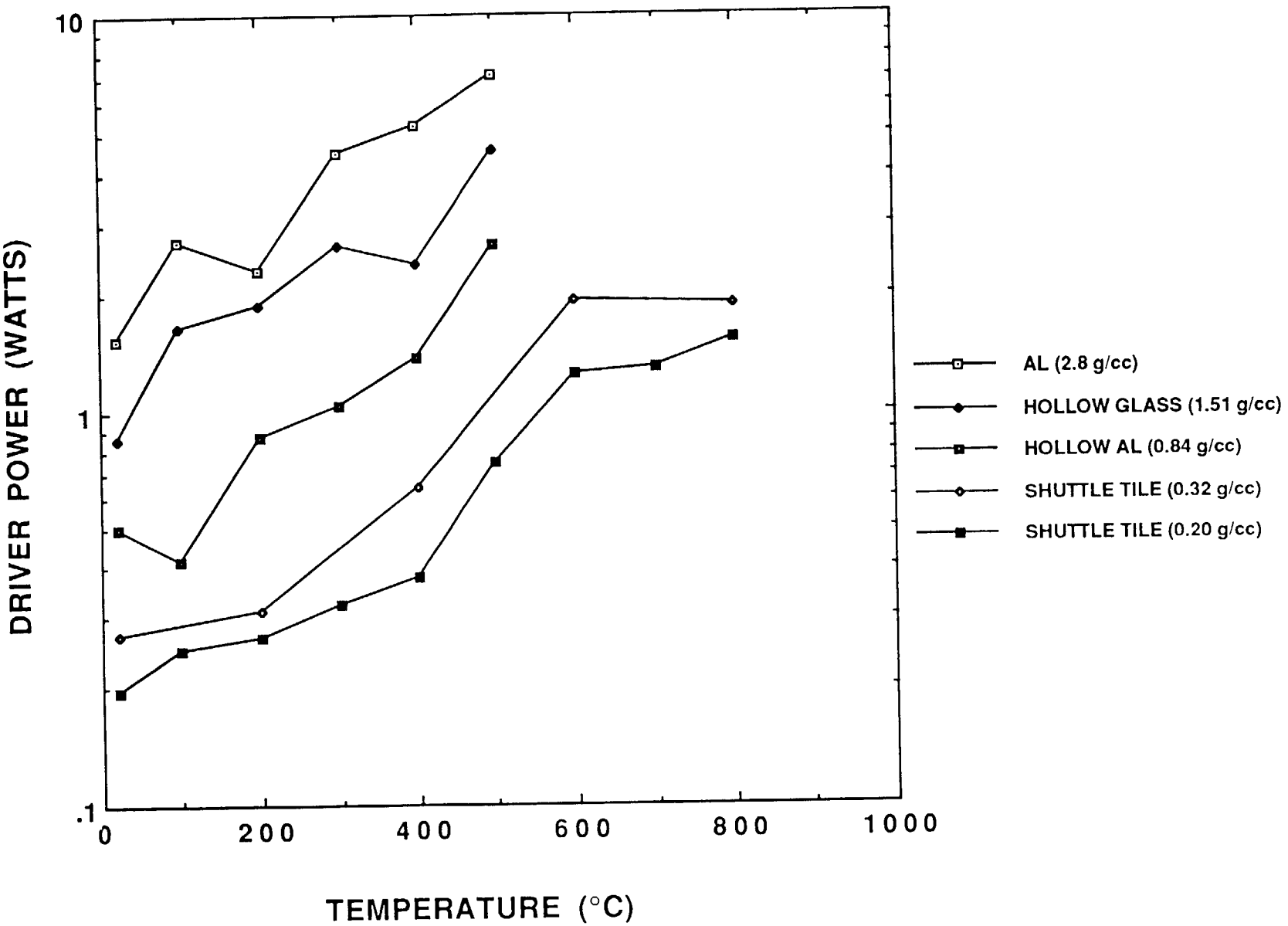
HIGH TEMPERATURE SINGLE MODE ACOUSTIC LEVITATOR

- CYLINDRICAL SINGLE MODE POSITIONER
  - FIXED FREQUENCY 20 KHZ DRIVER
  - (011) MODE EXCITATION
- ISOTHERMAL FURNACE (1000 °C)
- 100 WATT NEODYMIUM-YAG LASER - DUAL BEAM
  - ≈ 3 mm DIAMETER SHUTTLE TILE SAMPLE
- NON-CONTACT TEMPERATURE MEASUREMENT
  - QUANTUM LOGIC LASER PYROMETER

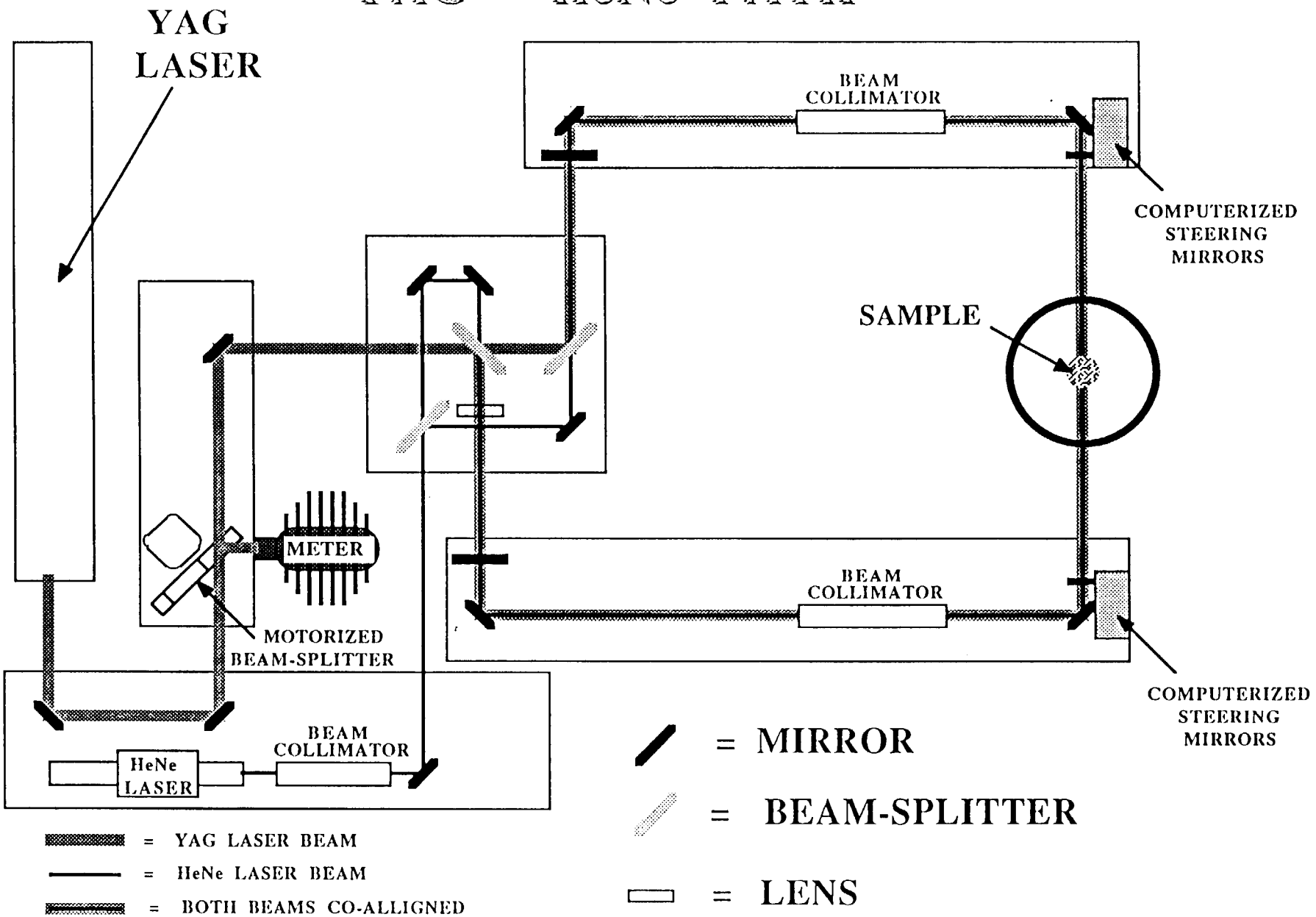
HIGH TEMPERATURE SINGLE MODE ACOUSTIC LEVITATOR

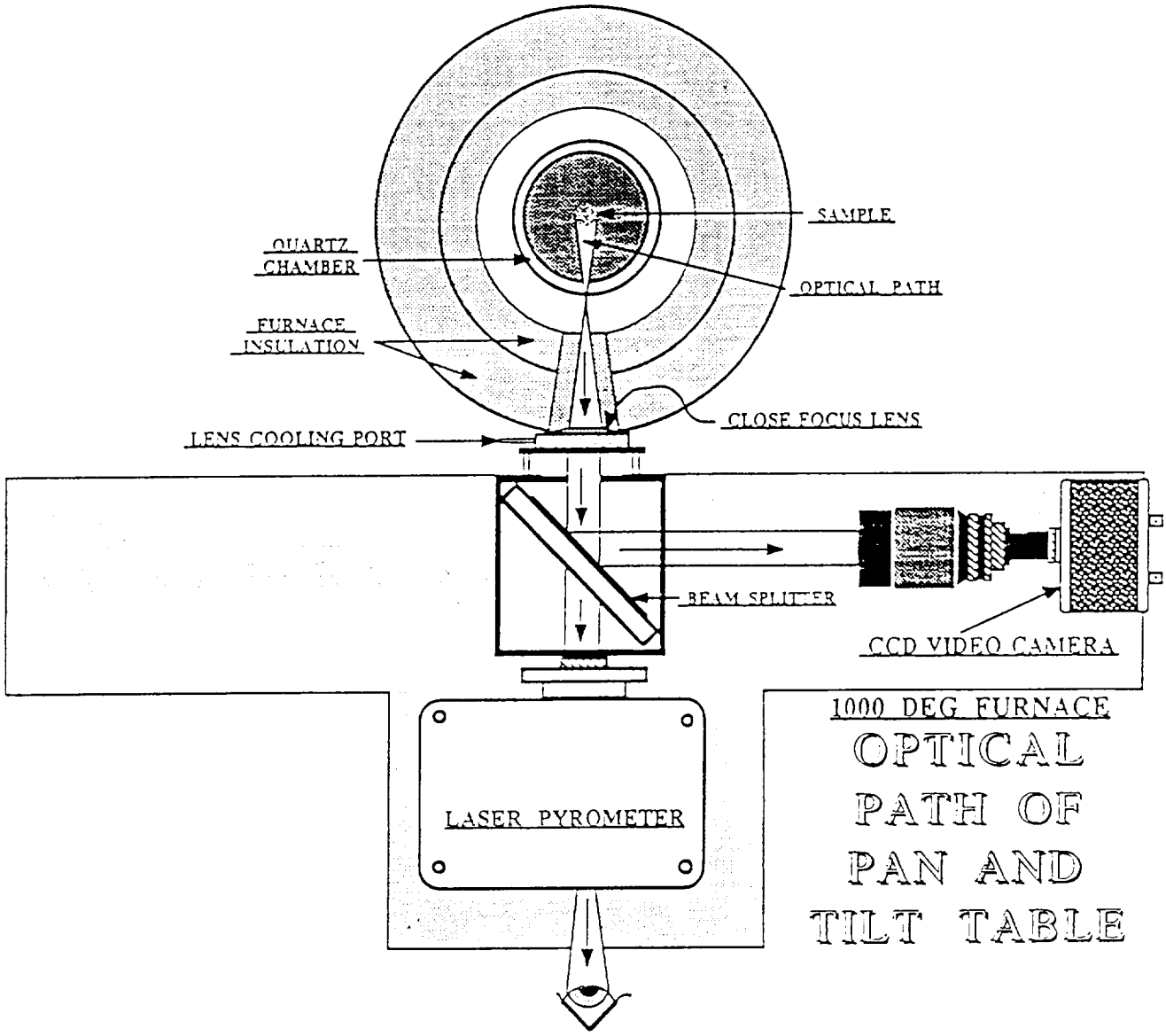


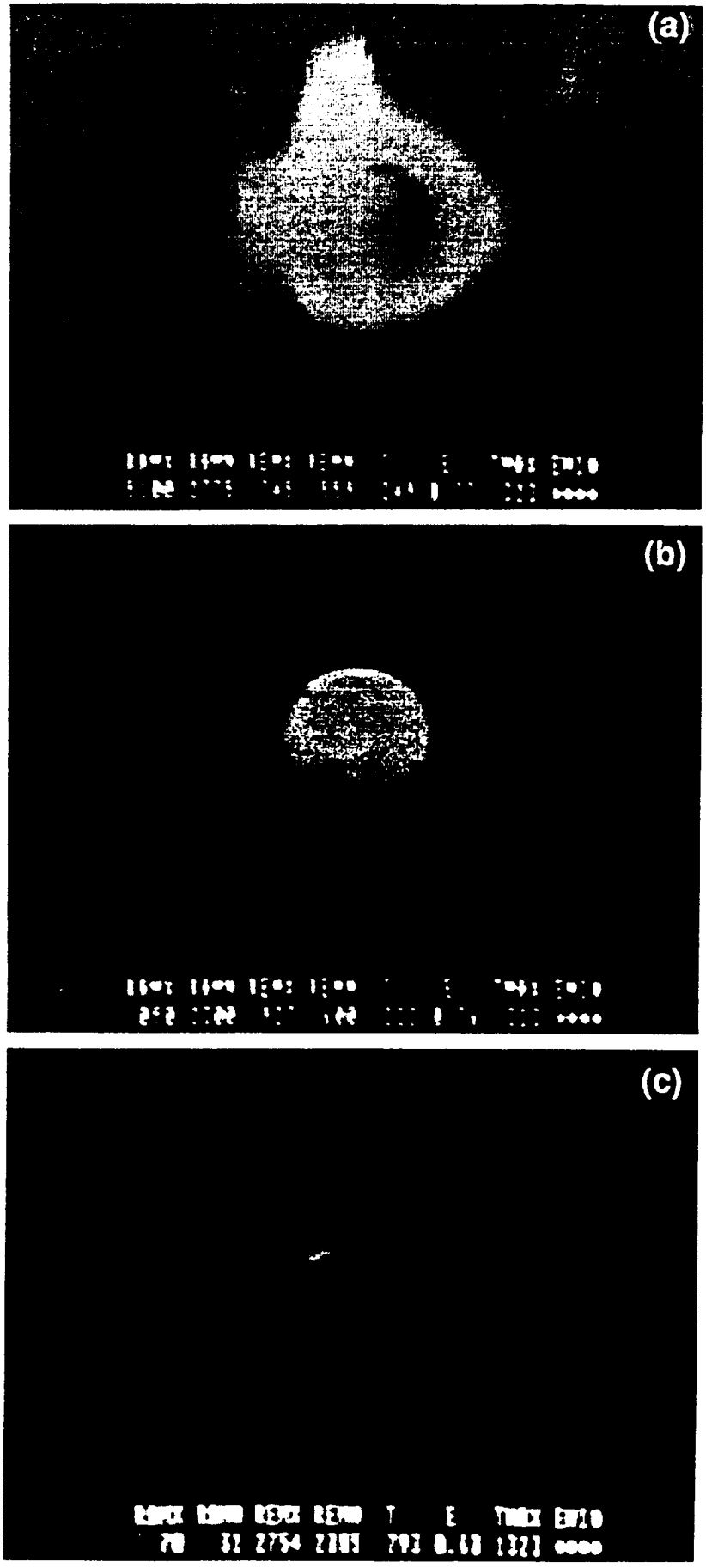
DRIVER POWER VS TEMPERATURE



YAG - HeNe PATH




SAMPLE
QUARTZ CHAMBER
OPTICAL PATH
FURNACE INSULATION
CLOSE FOCUS LENS
LENS COOLING PORT
BEAM SPLITTER
CCD VIDEO CAMERA
1000 DEG FURNACE
LASER PYROMETER
OPTICAL PATH OF PAN AND TILT TABLE



MICROWAVE/ACOUSTIC HYBRID LEVITATOR

MICROWAVE HEATING ADVANTAGES

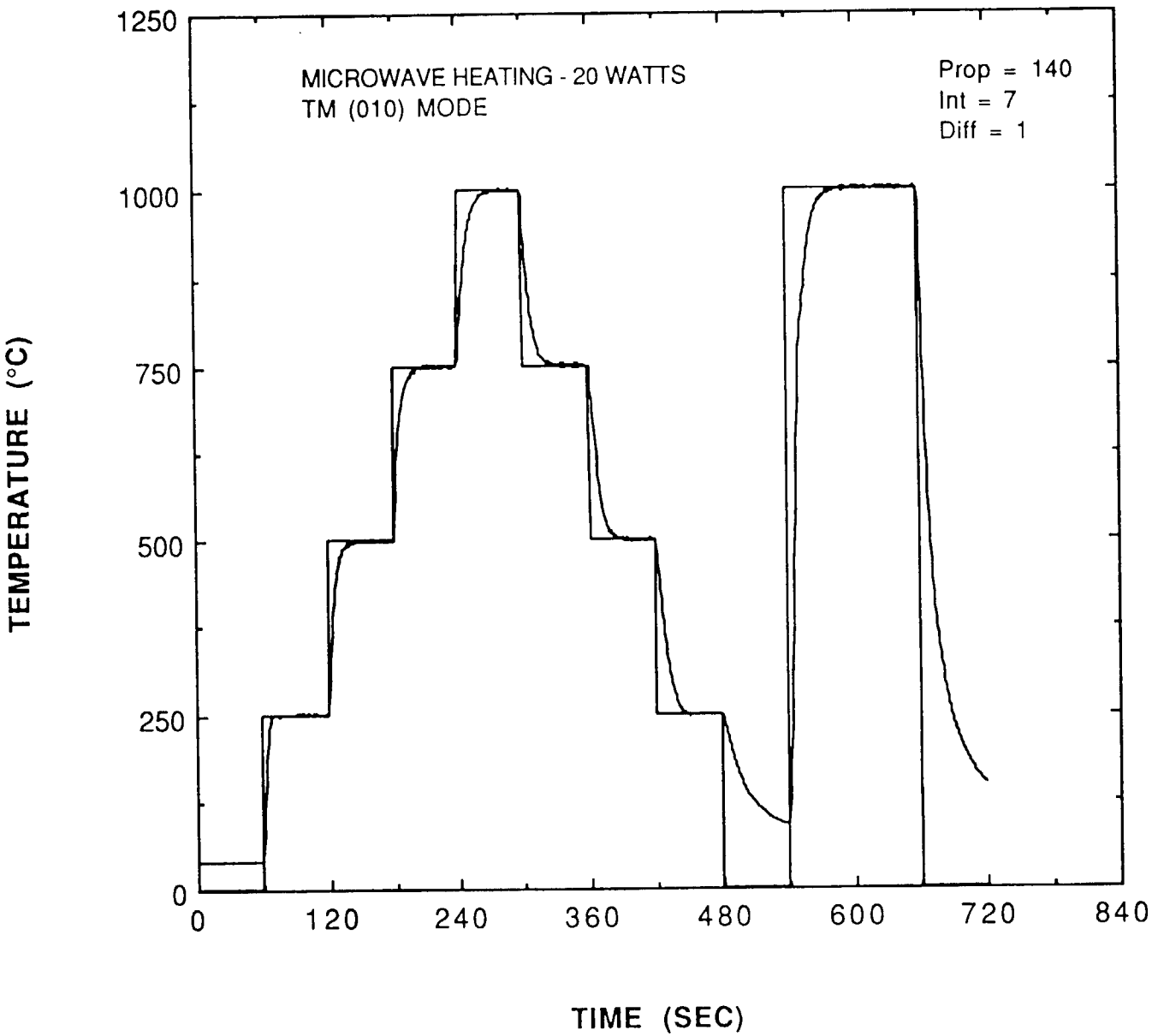
- EFFICIENT POWER CONVERSION COMPARED TO LASERS AND ARC LAMPS
- SMALL, LIGHT WEIGHT POWER SYSTEM
- VOLUMETRIC SAMPLE HEATING IS POSSIBLE
- SAMPLE POSITIONING IS NOT CRITICAL
- FAST CONTROLLABLE HEATING OF SAMPLE
  QUICK RESPONSE TIME
- COLD CHAMBER WALLS ⇒ QUICK CONTROLLABLE COOLING - TEMPERATURE CONTROLLED PROCESSING
- SELECTIVE HEATING OF SAMPLE COMPONENTS
- POSITIONING OF HOT AND COLD SAMPLES SIMULTANEOUSLY (DROP COALESCENCE)

MICROWAVE/ACOUSTIC HYBRID LEVITATOR

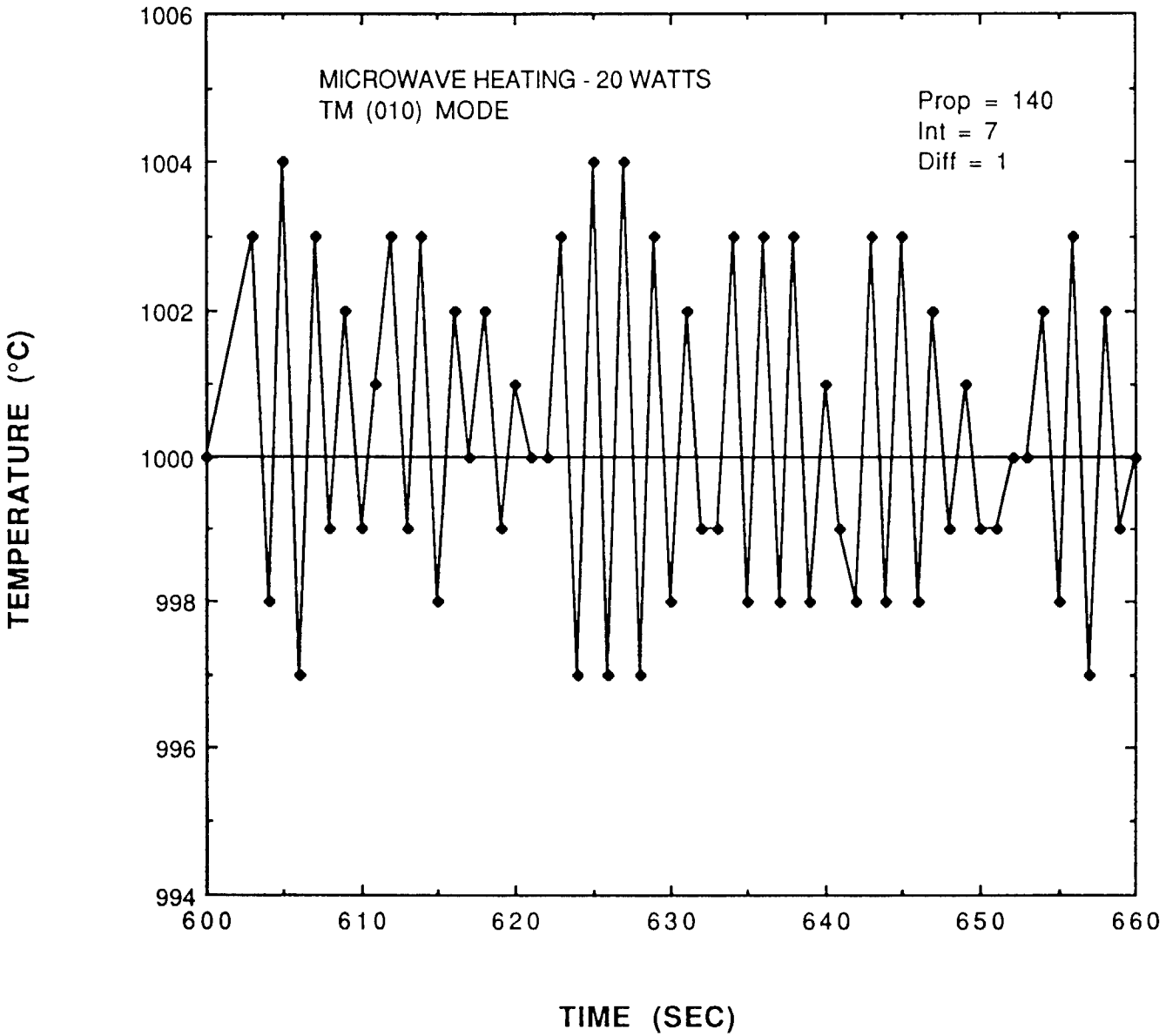
DEVELOPMENT PROGRAM

- EVALUATE MICROWAVE HEATING CONCEPT APPLIED TO CONTAINERLESS PROCESSING
  - MODEL - ABSORPTION OF A SPHERE
  - MODEL - TEMPERATURE PROFILE WITHIN SPHERE - INVERTED TEMPERATURE PROFILE (HOTTEST IN CENTER)
  - TEMPERATURE FEEDBACK CONTROL
  - MATERIALS CHARACTERIZATION - DIELECTRIC CONSTANT
    - GLASSES (LEAD BORATE - 900 °C)
    - CERAMICS (ZEOLITE - > 1100 °C)
  - DEMONSTRATE HYBRID LEVITATOR CONCEPT
    - PROTOTYPE - 20 KHZ (ACOUSTIC) LEVITATION - 10 WATTS (MICROWAVE)
    - HIGH POWER HYBRID - 1KW MICROWAVE SOURCE

TEMPERATURE CONTROL OF ALUMINA SILICATE

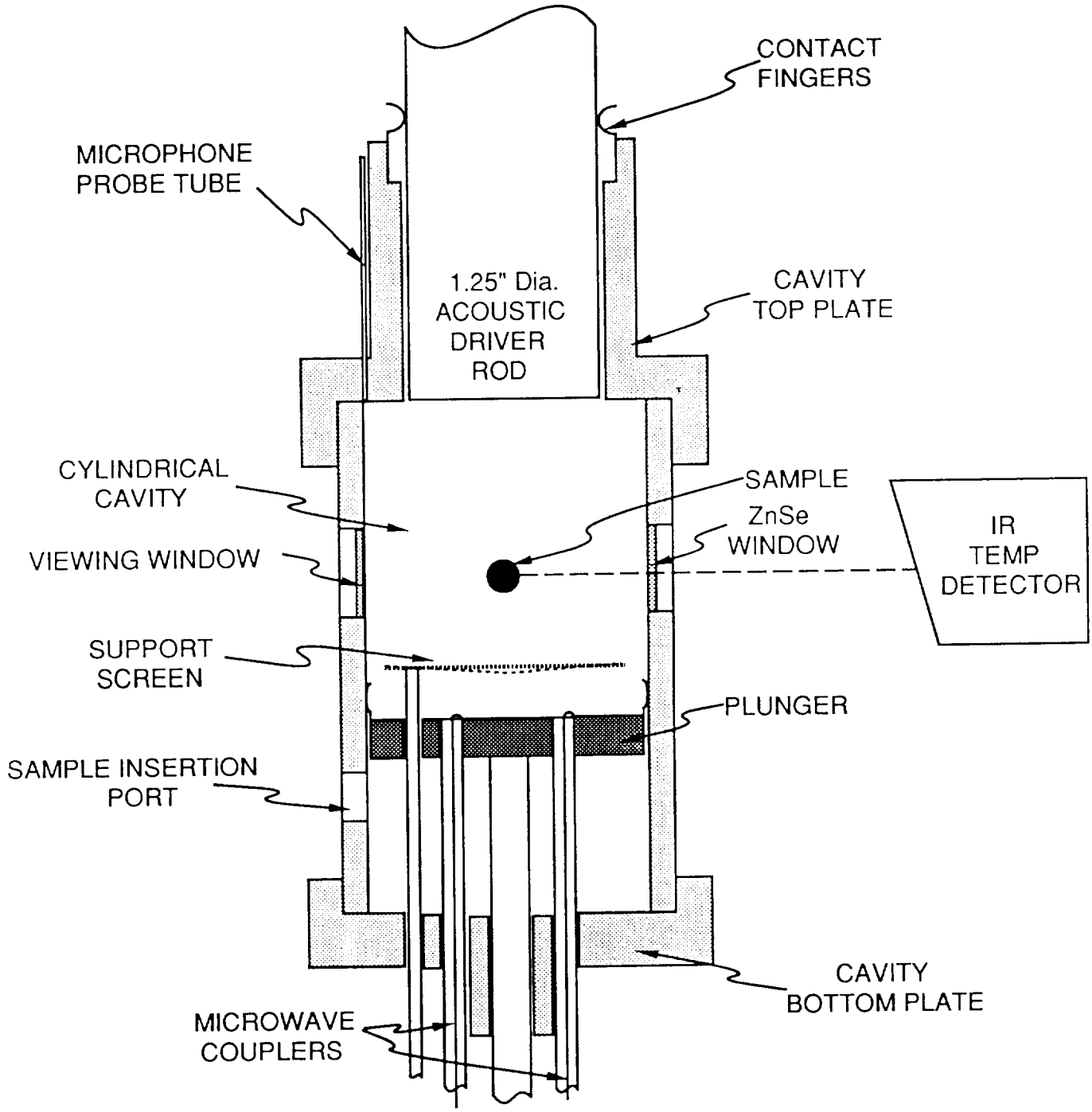


TEMPERATURE CONTROL OF ALUMINA SILICATE



MICROWAVE / ACOUSTIC HYBRID LEVITATOR

PIEZOELECTRIC DRIVER - SINGLE MODE POSITIONER



MICROWAVE/ACOUSTIC HYBRID LEVITATOR

POTENTIAL SCIENCE AREAS

- TEMPERATURE CONTROLLED PROCESSING
  - QUICK HEATING AND COOLING
  - PHASE TRANSFORMATION STUDIES
  - GLASS AND CERAMIC MATERIALS SYNTHESIS
  - TEMPERATURE MODULATION STUDIES
- ENHANCED MATERIALS PROCESSING DUE TO INVERTED TEMPERATURE PROFILE
  - UNIQUE ANNEALING OR ZONE REFINING
- NON-CONTACT THERMOPHYSICAL PROPERTIES MEASUREMENTS
  - SPECIFIC HEAT, DIELECTRIC PROPERTIES

N91-21369

A New, Simple Electrostatic-acoustic Hybrid Levitator

E. G. Lierke, H. Loeb, and D. Gross
Battelle Institute, Frankfurt, W. Germany

Battelle has developed a hybrid levitator by combining the known single-axis acoustic standing wave levitator with a coaxial DC electric field. The acoustic 20kHz driver serves as the ground electrode for the electric field, while a convex electrode - integrated into the acoustic reflector - provides a slightly convergent electric field. The resulting Coulomb forces on the charged liquid or solid sample support its weight and, together with the acoustic force, center the sample.

Liquid samples with volumes $\lesssim$ 100 micro-liters are deployed from a syringe reservoir into the acoustic pressure node. The sample is charged using a miniature high-voltage power supply ($\lesssim$ 20 kV) connected to the syringe needle. As the electric field - generated by a second miniature power supply - is increased, the acoustic intensity is reduced.

The combination of both fields allows stable levitation of samples larger than either single technique could position on the ground. Decreasing the acoustic intensity reduces acoustic convection and sample deformation.

Neither the electrostatic nor the acoustic field requires sample positioning sensing or active control.

The levitator - now used for static and dynamic fluid physics investigations on the ground - can be easily modified for space operations.

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

CONSIDERED AREAS OF INTEREST (on ground and under micro-g conditions)

- FLUID PHYSICS EXPERIMENTS (STATIC AND DYNAMIC)
- CRYSTAL GROWTH FROM SOLUTION DROPS
- (SINGLE-DROP COMBUSTION)

CONSIDERED SAMPLES AND ENVIRONMENTAL CONDITIONS

- WATER AND OTHER SOLUTION DROPS IN NEAR-AMBIENT ENVIRONMENT
  - VOLUME < 100µl
  - AIR AT 1 BAR PRESSURE
  - TEMPERATURE: 0° - 100° C
  - RELATIVE HUMIDITY: 0 - 100%

FEASIBLE LEVITATORS

- ACOUSTIC LEVITATOR
- ELECTROSTATIC LEVITATOR
- ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

FEATURES OF A ONE-AXIAL ACOUSTIC STANDING-WAVE LEVITATOR

1) MULTIAXIAL POSITIONING FORCES RESULT FROM GRADIENTS OF ACOUSTIC RADIATION PRESSURE (AXIAL AND RADIAL, BERNOULLI, PRESSURE) IN A RESONANT STANDING WAVE BETWEEN A PISTON RADIATOR AND A FLAT, TAPERED OR CURVED REFLECTOR. (FIG. 1)

2) THE SAMPLE IS STABLY LEVITATED NEAR THE PRESSURE NODE (VELOCITY ANTINODE) TYPICALLY WITH AXIAL DISPLACEMENTS $< \lambda/8$ AND RADIAL DISPLACEMENTS $< \lambda/4$.

3) AXIAL FORCES ARE STRONGER THAN RADIAL FORCES (4:1).

4) LEVITATION FORCES DECREASE WITH INCREASING ka $(=\pi \frac{d_s}{\lambda})$ AND DIMINISH AT ka > 2.2. (FIG. 2)

5) THE SMALLEST OPTIMAL SOUND PRESSURE LEVEL (SPL) FOR A GIVEN SAMPLE DIAMETER IS REQUIRED AT ka = $\pi/3$ (DIAMETER, $d_s = \lambda/3$).

6) ACOUSTIC LEVITATION FORCES ARE COMBINED WITH CONVECTION CURRENTS IN THE SURROUNDING GAS. (FIG. 3)

7) THESE CONVECTION CURRENTS LEAD TO ENHANCED MASS AND HEAT FLOWS WHICH ARE REPRESENTED BY EFFECTIVE REYNOLDS, SHERWOOD, OR NUSSELT NUMBERS.

8) THE OBLATE DEFORMATION OF ACOUSTICALLY LEVITATED DROPS IN 1-G IS CONSIDERABLY LARGER THAN THE PROLATE DEFORMATIONS IN ELECTROSTATIC LEVITATORS. (FIG. 4)

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

MAIN EQUATIONS AND PARAMETERS OF THE ACOUSTIC LEVITATOR

AXIAL FORCE:

$$F = \frac{5}{6}\pi\rho_o \tilde{v}_{max}^2 ka^3 f_1(2ka)\cdot\sin(2k\Delta z) = \frac{4}{3}\pi a^3 \rho_s g_o$$

$k = \frac{\omega}{c_o} = \frac{2\pi}{\lambda}$ $\quad a = d_s/2$ (d_s is the sample diameter)

$\rho_o c_o^2 = p_o \gamma$ $\quad \rho_s$ = sample density

$g_o = 9.81\ m/s^2$ (sea-level grav. accel.)

$\tilde{v}_{max}$ - antinode velocity amplitude

$\tilde{p}_{max}$ - antinode acoustic pressure amplitude

$M_{ac} = \frac{\tilde{v}_{max}}{c_o}$ - acoustic Mach number

$\phi_s = \sin^{-1}(2k\Delta z)$ - levitation safety factor

Δz - displacement of sample from pressure node

$$f_1(2ka) = \frac{3}{(2ka)^3}\left\{\frac{\sin(2ka)}{2ka} - \cos(2ka)\right\} = \frac{1.31}{2ka} f_2(2ka)$$

$$M_{ac}^2 = \frac{8}{5}\frac{\rho_s g_o}{p_o \gamma k}\frac{\phi_s}{f_1(2ka)} = 1.22\frac{\rho_s g_o d_s}{p_o \gamma}\frac{\phi_s}{f_2(2ka)}$$

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

MAIN EQUATIONS AND PARAMETERS OF THE ACOUSTIC LEVITATOR

CONVECTION FLOW VELOCITY ARROUND SAMPLE:

$$\hat{V} \simeq \frac{\tilde{v}_{max}^2}{\omega a} = \frac{c}{ka} M_{ac}^2$$

REYNOLDS NUMBER FOR ACOUSTIC CONVECTION:

$$Re = \frac{d_s \hat{V}}{\nu} = \frac{\lambda c_0}{\nu k} M_{ac}^2 \qquad (\nu : \text{kinematic viscosity})$$

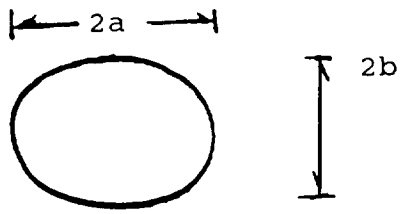
SOUND PRESSURE LEVEL:

$$SPL\ [dB] = 10 \log \frac{\tilde{p}_{max}^2}{2 p^{*2}} \simeq 194 + 10 \log M_{ac}^2$$

BOND NUMBER:

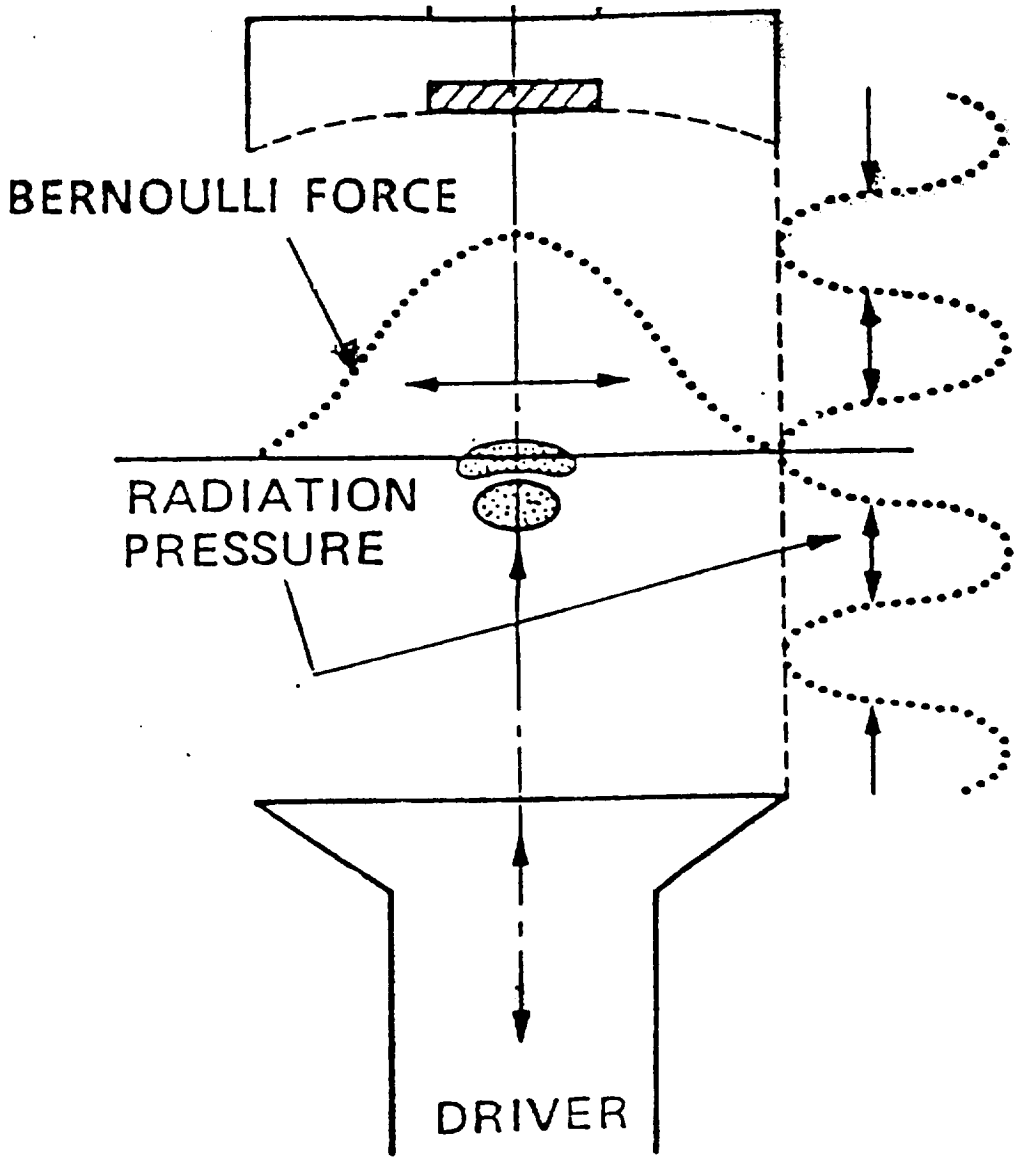
$$B = \frac{\rho_s}{\sigma_s} \frac{d_s^2}{4} g \qquad (\sigma_s : \text{surface tension})$$

ASPECT RATIO: a/b



Characteristic parameters for terrestrial levitation of water drops ambient air at 21 kHz

d [mm]:	2	3	4	5	6
$M_{ac} \cdot 10^2$	1.93	2.0	2.12	2.29	2.53
$\hat{V}$ [cm/s]	32	23	19.4	18	18
Re	46	49.6	56	65.4	78.4
SPL [dB]	159.7	160	160.5	161.2	162.1
a/b	1.18	1.36	1.6	1.9	2.1
f_1 (2ka)	0.94	0.87	0.77	0.66	0.55
f_2 (2ka)	0.56	0.78	0.93	1.00	0.98
Bo	0.136	0.307	0.545	0.85	1.22



$$p_{rad} = \frac{p^2}{2\rho c^2} \cdot \sin^2 k_z z \cdot \cos^2 k_r r$$

$$p_{Bern} = -\rho/2 \cdot v^2 \cos^2 k_z z \cos^2 k_r r$$

Fig. 2: Drop levitation in an one-axial acoustic levitator (schematical)

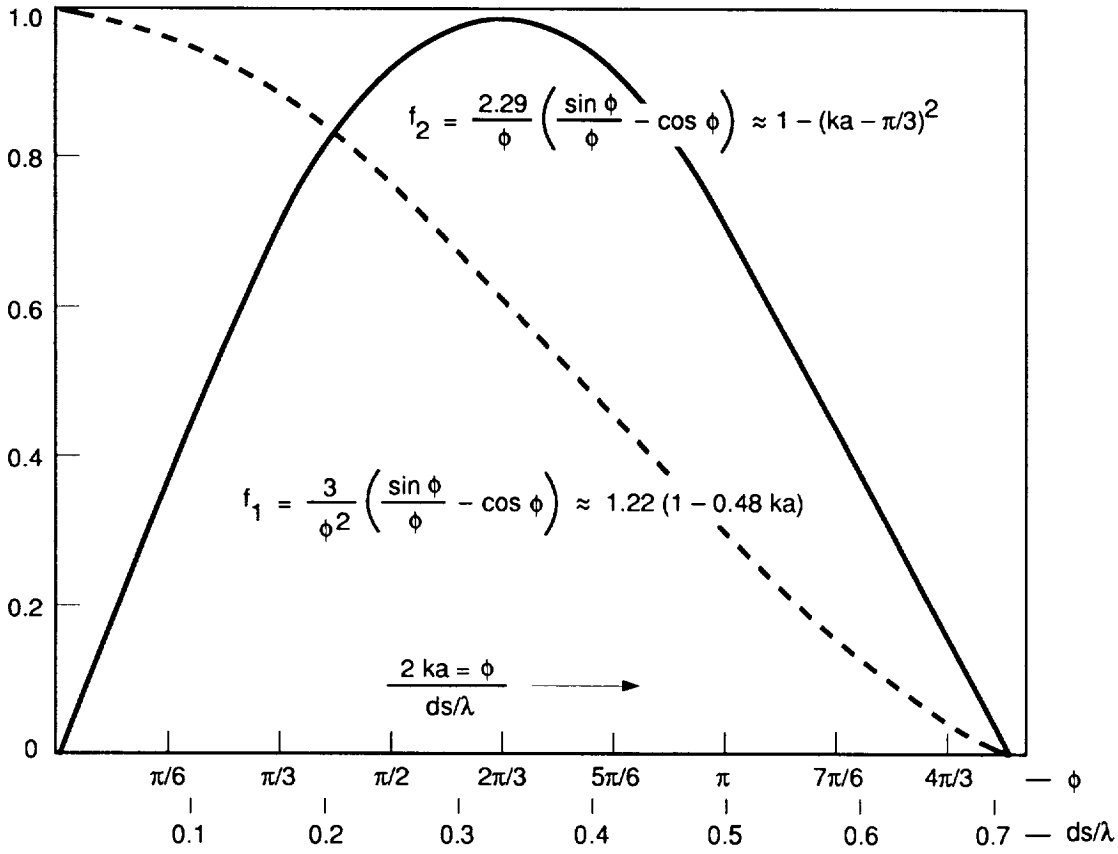
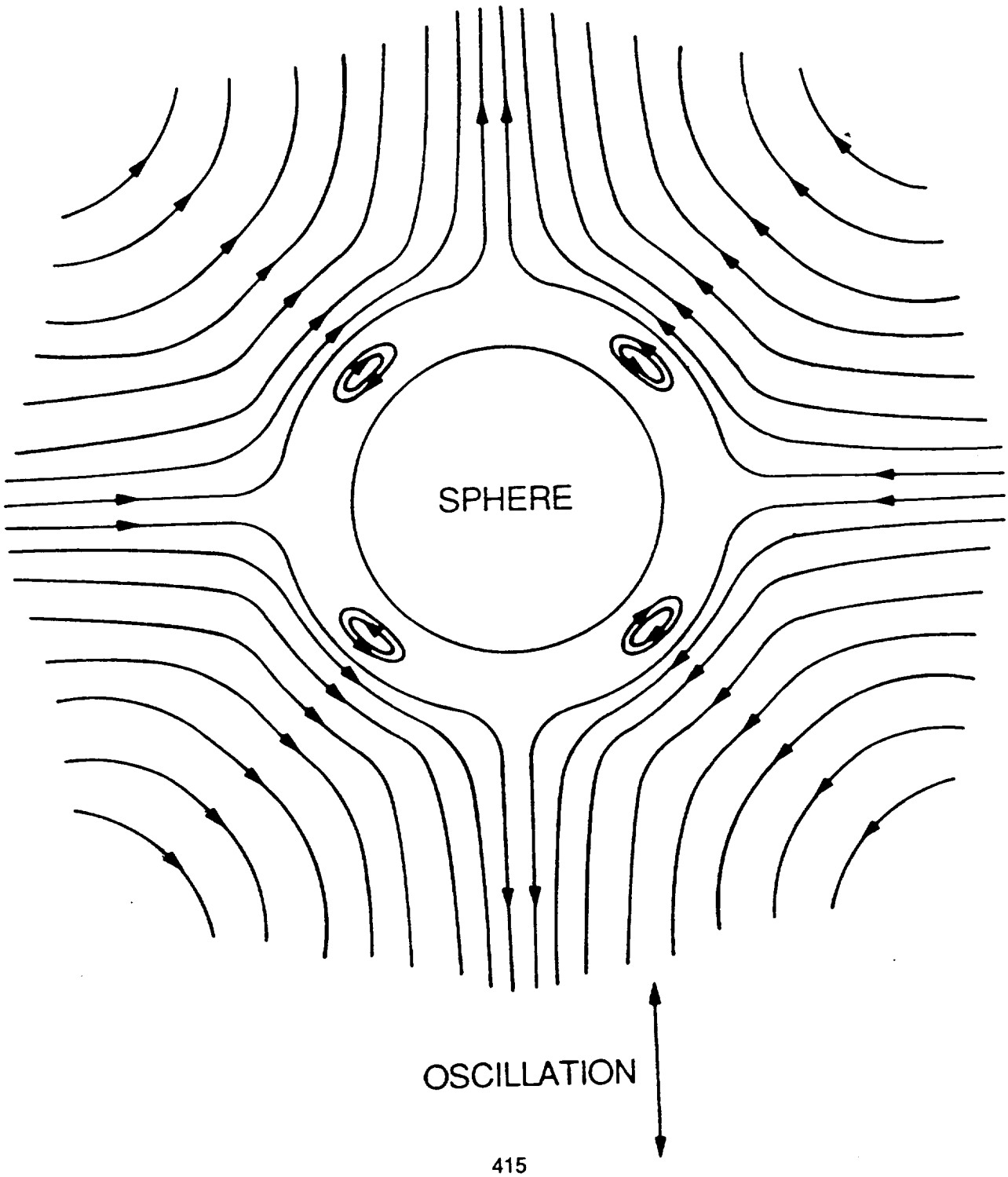
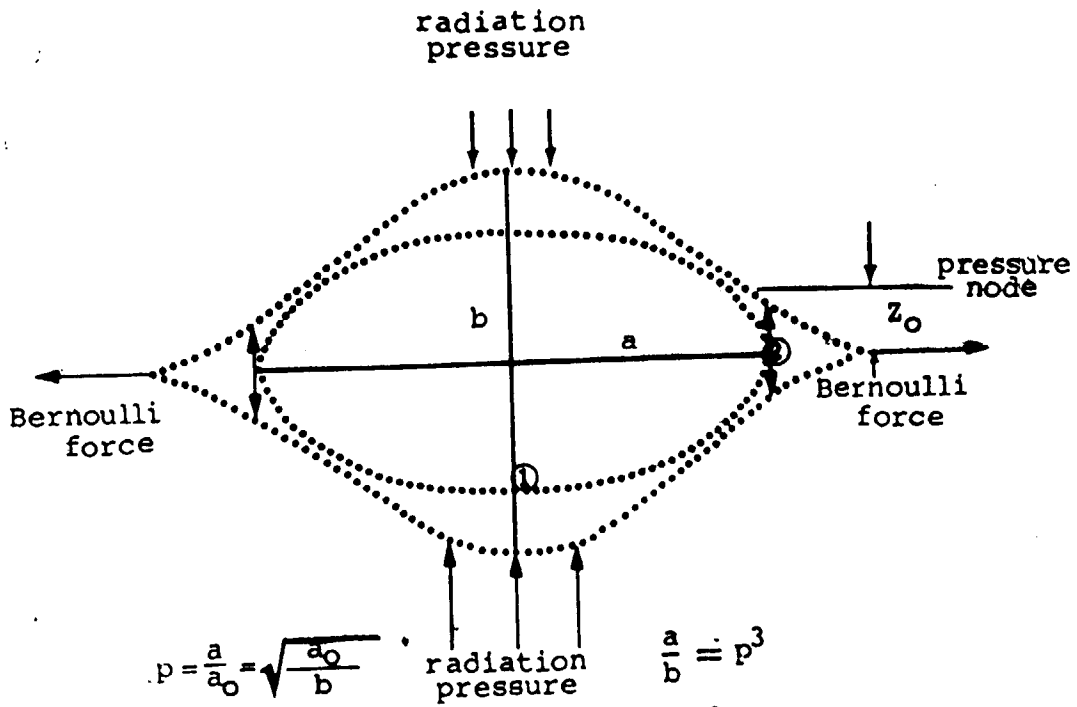

1.0
0.8
0.6
0.4
0.2
0
$f_2 = \frac{2.29}{\phi}\left(\frac{\sin\phi}{\phi} - \cos\phi\right) \approx 1 - (ka - \pi/3)^2$
$f_1 = \frac{3}{\phi^2}\left(\frac{\sin\phi}{\phi} - \cos\phi\right) \approx 1.22\,(1 - 0.48\,ka)$
$\frac{2\,ka = \phi}{ds/\lambda}$
π/6
π/3
π/2
2π/3
5π/6
π
7π/6
4π/3
— φ
0.1
0.2
0.3
0.4
0.5
0.6
0.7
— ds/λ

Figure 2.


SPHERE
OSCILLATION



$$\Phi_s = \frac{F_{ac}}{m_s g_o} - \textit{Safety factor} \; ; \qquad B_o = \frac{\rho_s}{\sigma_s} a_o^2 g_o - \textit{Bond number}$$

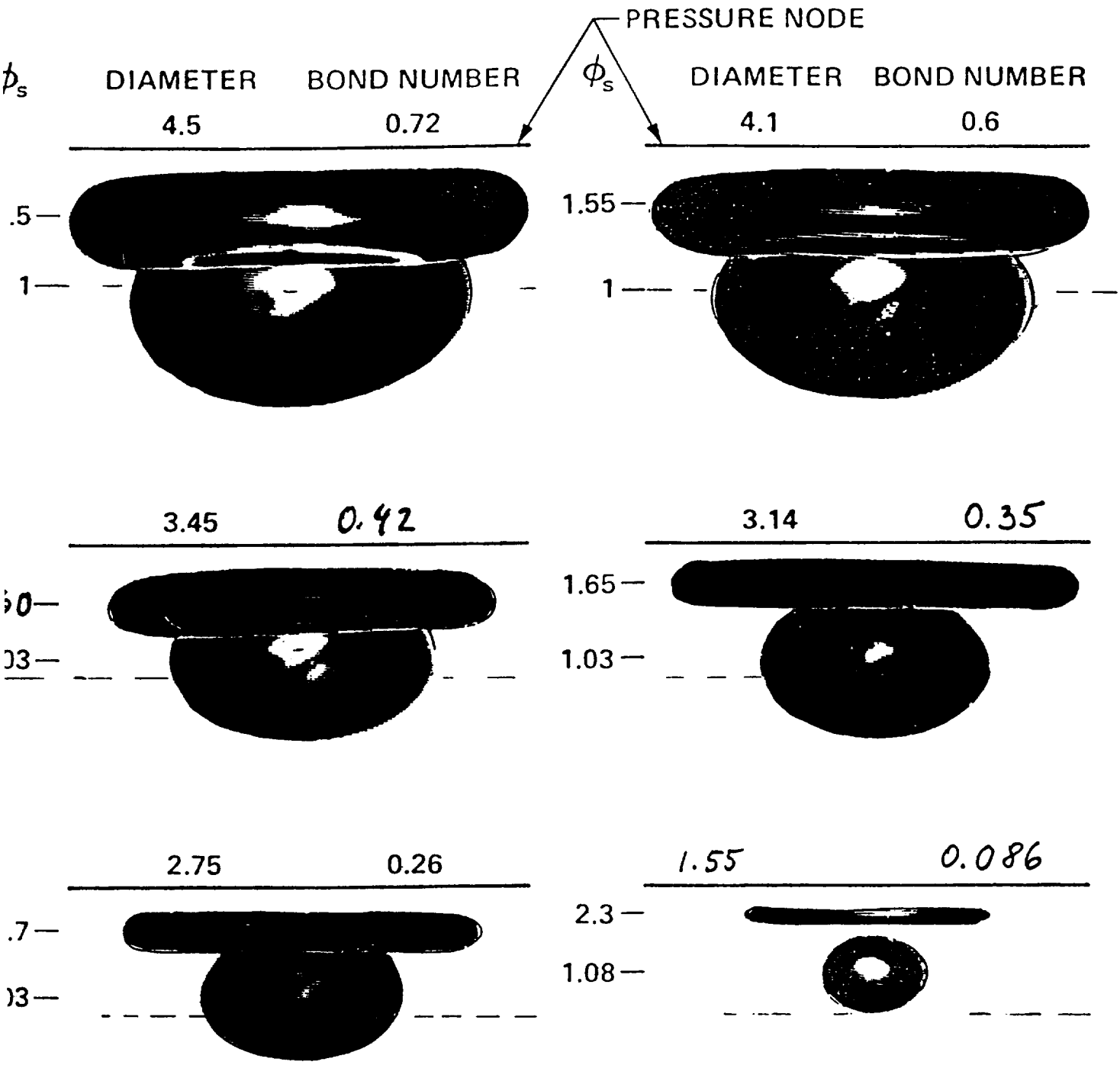


FIGURE 3

YPICAL AXIAL DISPLACEMENT AND DEFORMATION OF WATER DROPS OF DIFFERENT INITIAL RADIUS a_o AT SMALL AND LARGE LEVITATION SAFETY FACTOR Φ_s IN A ONE-AXIAL 20 kHz ACOUSTIC LEVITATOR IN AIR (λ = 17mm)

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

FEATURES OF THE ELECTROSTATIC LEVITATOR

1) AXIAL POSITIONING FORCES RESULT FROM COULOMB FORCES ON A SAMPLE WITH CHARGE, Q, IN A HOMOGENEOUS ELECTRIC FIELD, E (AS KNOWN FROM MILIKAN'S EXPERIMENT). (FIG. 5)

2) THE BALANCE BAETGWEEN SAMPLE WEIGHT AND LEVITATION FORCE IS UNSTABLE AND REQUIRED ACCURATE POSITIONING CONTROL AND VOLTAGE CONTROL WITH AN APPROPRIATE TIME CONSTANT (SERVO-CONTROLLED POWER SUPPLY).

3) SMALL RADIAL FORCES CAN ABE PROVIDED WITH TAPERED OR CURVED ELECTRODES.

4) THE SAMPLE ENVIRONMENT CAN BE ANY GAS OR VACUUM. THERE ARE NO FORCED-CONVECTION CURRENTS.

5) DEPLOYMENT AND EXTRACTION OF A LIQUID SAMPLE ARE MORE DIFFICULT THAN IN ACOUSTIC LEVITATORS.

6) THE PROLATE DEFORMATION OF LIQUID SAMPLES IS SMALL COMPARED TO THEAT RESULTING FROM ACOUSTIC LEVITATION.

7) SAMPLE CHARGE, Q, AND ELECTRIC FIELD, E, MUST BE SLECTED WITH ATTENTION TO THE RAYLEIGH–TAYLOR INSTABILITY LIMITS. (FIG. 6)

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

MAIN EQUATIONS AND PARAMETERS OF THE ELECTROSTATIC LEVITATOR

$$F_{el} = E \cdot Q = \frac{4}{3} \pi a_s^3 \rho_s g$$

$$Q = 4\pi \varepsilon_0 a_s U_2$$

$$E = \frac{U_1}{\ell}$$

NORMALIZATION (RAYLEIGH-TAYLOR LIMIT)

$$\bar{E} = \frac{U_1}{\ell} \sqrt{\frac{4\pi a_s \varepsilon_0}{\sigma_s}} \qquad \bar{Q} = \frac{U_2}{a_s} \sqrt{\frac{4\pi \varepsilon_0 a_s}{\sigma_s}}$$

$$\bar{F}_{el} = \frac{F_{el}}{\frac{4}{3}\pi \sigma_s a_s} = \frac{\bar{E} \cdot \bar{Q}}{\frac{4}{3}\pi} = Bo = \frac{\rho_s}{\sigma_s} a_s g$$

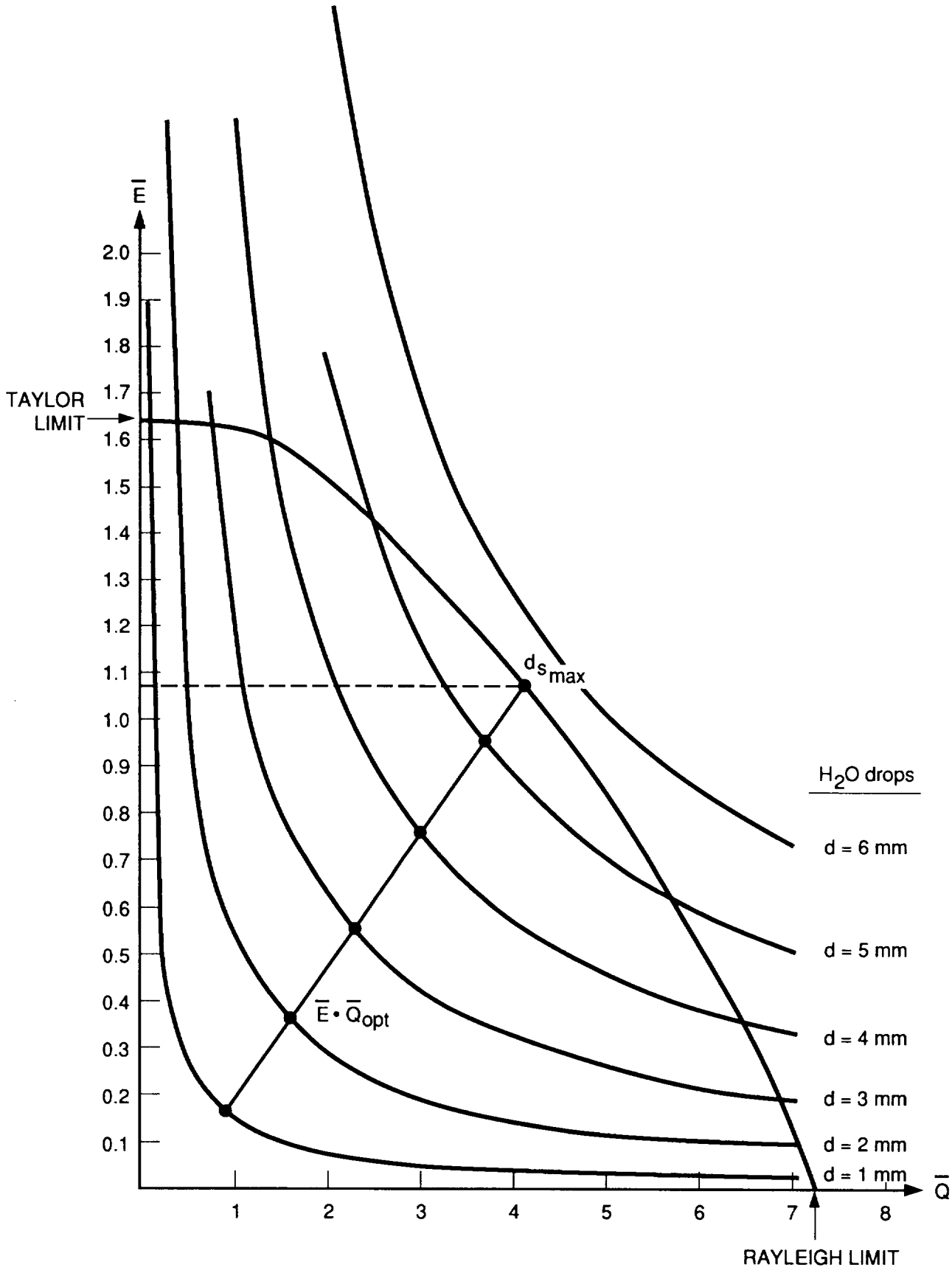
RAYLEIGH-TAYLOR LIMIT FOR VARIOUS BOND NUMBERS

Bo:	0.2	0.4	0.6	0.8	1.0	1.06
Q(min)	0.55	1.05	1.55	2.30	3.45	4.25
Q(max)	6.95	6.65	6.30	5.80	5.00	4.25

$$\bar{Q}_{opt} \simeq 4.2 \qquad \bar{E}_{opt} = Bo$$

$$U_2\,[kV] \simeq 7.5 \sqrt{d_s\,[cm]}$$

$$U_1\,[kV] \simeq 39\, d_s\,[cm] \quad \text{for} \quad = 3.2cm.$$


$\overline{E}$
2.0
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
TAYLOR LIMIT
$d_{s_{max}}$
$\overline{E} \cdot \overline{Q}_{opt}$
H_2O drops
d = 6 mm
d = 5 mm
d = 4 mm
d = 3 mm
d = 2 mm
d = 1 mm
1
2
3
4
5
6
7
8
$\overline{Q}$
RAYLEIGH LIMIT

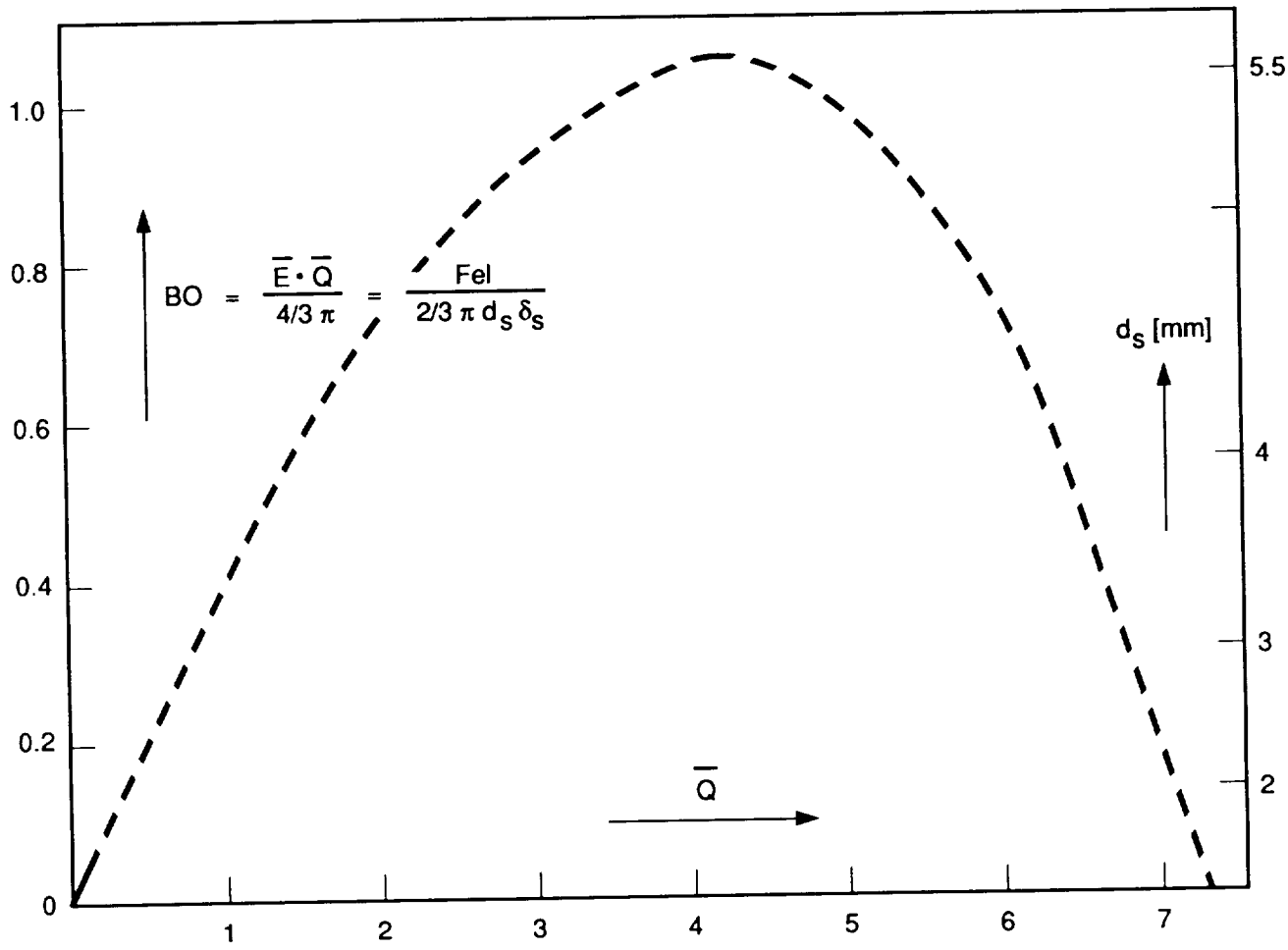

1.0
0.8
0.6
0.4
0.2
0
BO = $\frac{\bar{E} \cdot \bar{Q}}{4/3\,\pi} = \frac{Fel}{2/3\,\pi\, d_s\, \delta_s}$
$\bar{Q}$
1
2
3
4
5
6
7
5.5
4
3
2
d_s [mm]

Figure 6.

Characteristic parameters for the electrostatic levitation of water drops (1-G; with electrode separation of 3.2cm)

d_s [mm]:	1	2	3	4	5	5.5
Bo	0.034	0.136	0.307	0.545	0.85	1.03
$\bar{Q}$(min)	0.1	0.4	0.75	1.4	2.5	3.75
$\bar{Q}$(max)	7.2	7.05	6.8	6.4	5.65	4.75
U_2(min)[kV]	0.11	0.64	1.48	3.19	6.36	10.0
U_2(max)[kV]	8.2	11.4	13.4	14.6	14.4	12.7
U_2(opt)[kV]	2.4	3.4	4.1	4.75	5.3	5.56
U_1(opt)[kV]	1.23	3.5	6.4	9.9	13.8	15.9

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

SPECIFIC FEATURES OF THE ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR

1) THE LEVITATOR CAN OPERATE EITHER AS ELECTROSTATIC, ACOUSTIC, OR HYBRID LEVITATOR. (FIG. 7)

2) HARDWARE COMPONENTS ARE SIMPLY INTEGRATED WITHOUT INTERFERENCE.

3) ELECTRONICS AND MECHANICAL HARDWARE ARE SIMPLE AND NEED NO CONTROL CIRCUITRY FOR POSITIONING, CALIBRATION AND TUNING.

4) LEVITATOR CAN OPERATE ON GROUND AND UNDER MICROGRAVITY CONDITIONS.

5) SINCE THE ACOUSTIC LEVITATOR PROVIDES STABILITY ARROUND THE PRESSURE NODE SUCH THAT

$$-1 < \sin(2k\Delta z) < 1,$$

THE ELECTROSTATIC FORCE (FIELD VOLTAGE, U_1) CAN BE VARIED WITHIN

$$U_{1,o}(1 - \phi_{s,ac}) < U_1 < U_{1,o}(1 + \phi_{s,ac})$$

WHERE $\phi_{s,ac}$ IS THE ACOUSTIC SAFETY FACTOR. (FIG. 8)

6) THE ACOUSTIC LEVITATION FORCE CAN BE REDUCED TO LESS THAN 5% OF ITS 1-G LEVEL RESULTING IN REDUCED CONVECTION FLOW (AS WELL AS A MASS AND HEAT-FLOW REDUCTION).

7) FLUID SAMPLE DEFORMATION BY ACOUSTIC FORCES (OBLATE) AND ELECTROSTATIC FORCES (PROLATE) CAN CANCEL OUT RESULTING IN SPHERICAL DROPS.

8) DROP OSCILLATIONS (TRANSLATIONAL AND SHAPE) CAN BE EXCITED BY MODULATION OF EITHER THE ELECTROSTATIC OR ACOUSTIC FIELD.

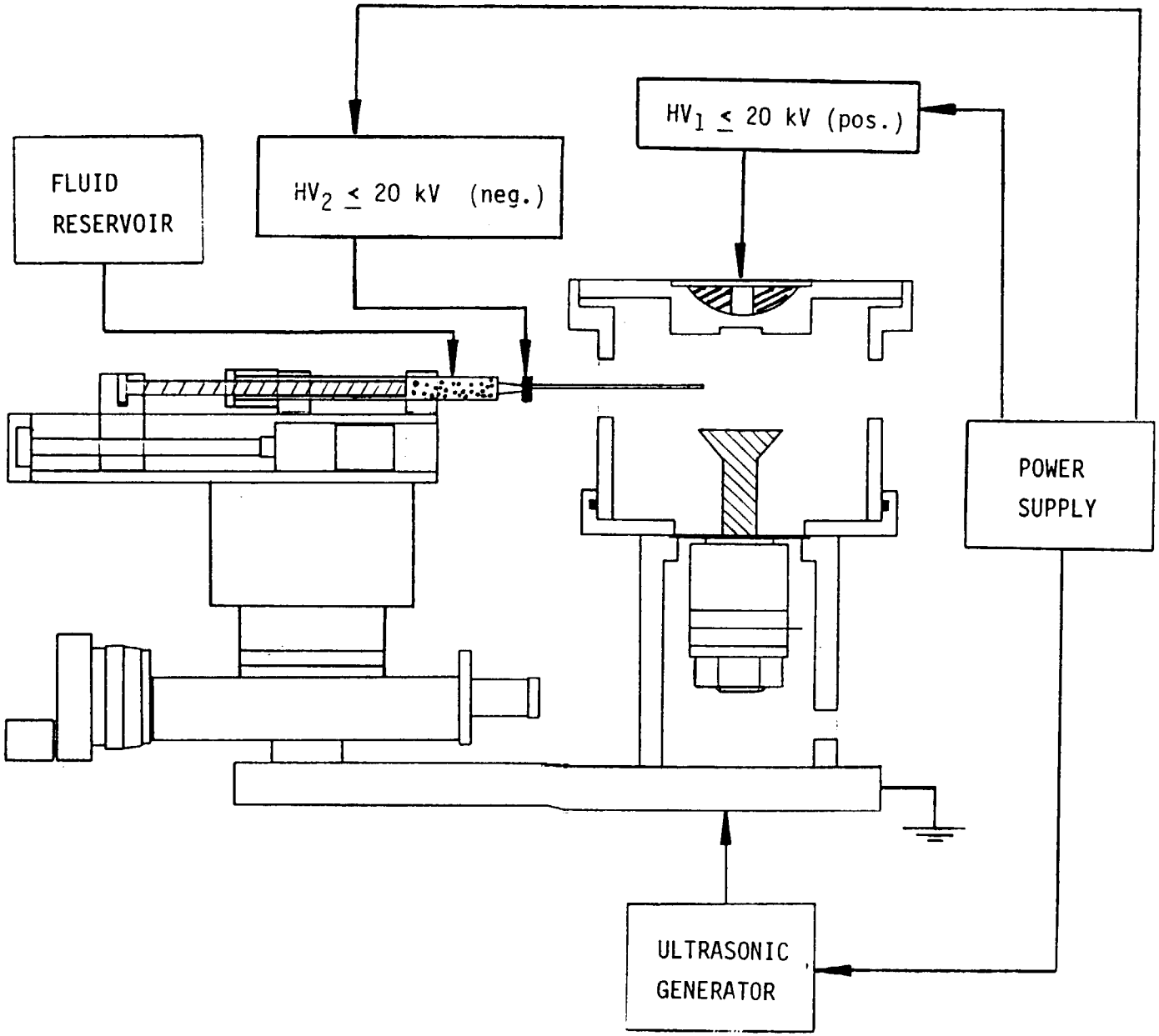


Fig. 7 : Block diagram of the Electrostatic-Acoustic-Hybrid Levitator

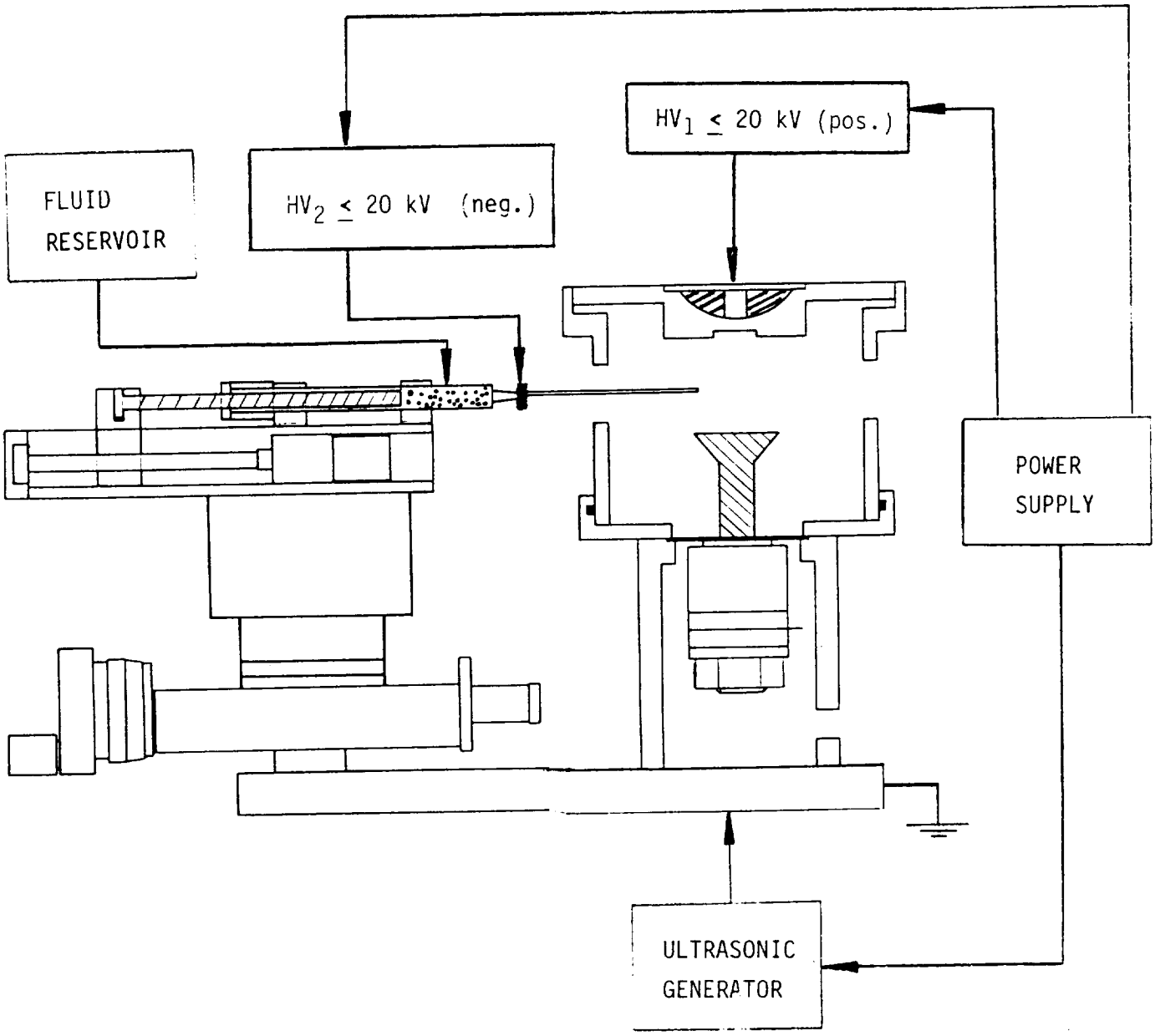


Fig. 7 : Block diagram of the Electrostatic-Acoustic-Hybrid Levitator

ADVANTAGES OF THE ELECTROSTATIC-ACOUSTIC HYBRID

- SIMPLE HARDWARE
- SPHERICAL DROPS
- FEASIBLE AT REDUCED GAS PRESSURE IN 1 G
- FEASIBLE FOR MOLTEN METALS (HIGH PRESSURE) IN 1 G
- EXCELLENT MICROGRAVITY SIMULATOR FOR ACOUSTIC LEVITATION
- GOOD FOR DISCHARGE MEASUREMENTS
- REDUCED FORCED CONVECTION, HEAT AND MASS TRANSFER
- MEASUREMENT OF SURFACE TENSION AND VISCOSITY IN 1 G
- MEASUREMENT OF THERMOPHYSICAL PROPERTIES IN 1 G
- EXTRAPOLATION OF G LEVELS EFFECTS OVER SEVERAL ORDERS OF MAGNITUDE
- SHAPE EFFECTS STUDIES ON SURFACE TENSION AND VISCOSITY

N91-21370

Feedback Controlled Electrostatic and Electromagnetic Sample Positioners

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Four different sample positioners will be discussed in this presentation. Three of them are electrostatic systems each of which operates at the different operational condition. The fourth positioner is the electromagnetic system which positions conducting samples. However, these four systems share a common operating principle in that the sample positioning is achieved by feedback controlled forces which can be electrostatic, dielectrophoretic, or electromagnetic. The first system is the electrostatic liquid drop positioner which operates at the near ambient condition. Containerless protein crystal growth and cell culturing experiments require the liquid drop positioner with temperature/humidity control and appropriate diagnostic capabilities, while the experiments on charged drop dynamics may require the electrostatic-acoustic hybrid system in which drop oscillation or rotation can be induced acoustically. The multi-drop positioning system developed for the protein crystal growth and biological applications will be described in a separate presentation, and a detailed description will be focused on the electrostatic-acoustic hybrid system for its capabilities and limitations for the drop dynamics experiments.

The second system is the tetrahedral electrostatic positioner which is being developed fo, the high temperature materials processing in vacuum. Tetrahedral system is capable of three dimensional position control and damping. Being a microprocessor controlled positioner, various modes operation can be generated all through software programming. In the tight control mode, the positioner keeps the sample at a fixed position and damps any transient movement about this position. In the soft control mode, the system allows the sample to freely float within a preassigned region so that the sample can be isolated from most of the oscillatory disturbances generated by the spacecraft. The presentation will include sample charge behavior as different materials were heated up to 1250°C, sample charging by electron guns, and a method of inducing sample rotation. This section will be concluded with the prospect of this system as a flight module.

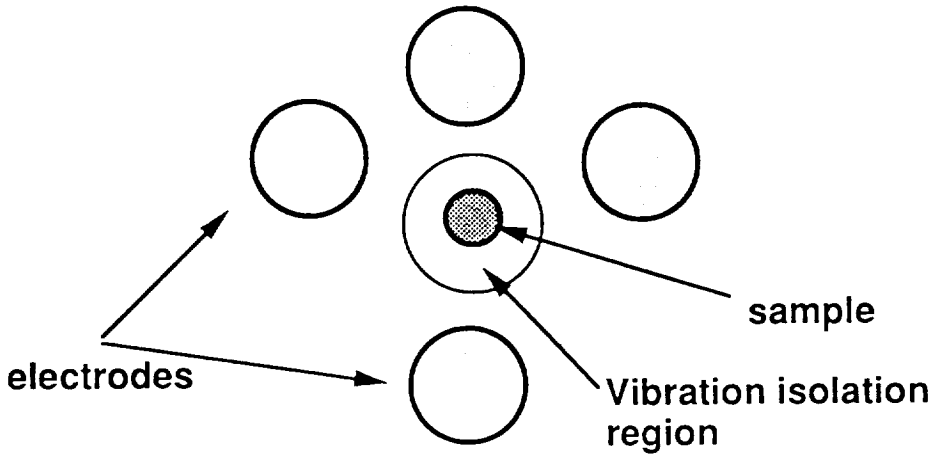
The third system is essentially the same tetrahedral system described above except that, in this mode of operation, the position control is achieved by dielectrophoretic forces in the pressurized gas environment. This system is being developed for those materials which contain volatile components.

Finally, the feasibility of a feedback controlled electromagnetic positioner will be presented. This approach is based on the same principle as the tetrahedral dielectrophoretic positioner as far as the basic principle of operation is concerned. Four coils are arranged in a tetrahedral configuration and positioning forces are generated by the same positioning algorithm used in the tetrahedral electrostatic systems. This system will have the same capabilities of positioning, damping, and vibration isolation as electrostatic systems. As long as the sample has appreciable electric conductivity, this system will be operable both in gaseous and vacuum environments. An additional advantage of this system will be that the sample rotation can be induced or damped in a controlled way which is not possible in the conventional electromagnetic positioners known today.

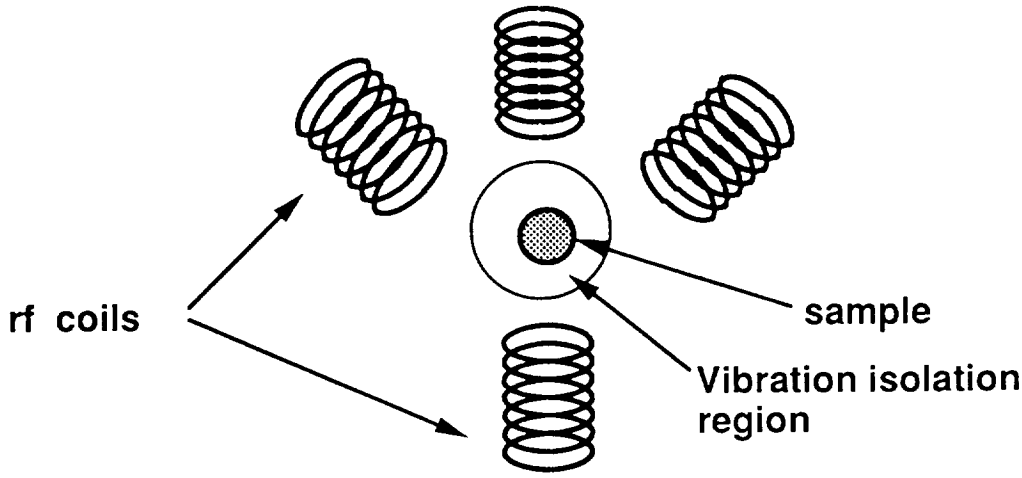
Tetrahedral Electrostatic/Electromagnetic Sample Positioners

- Electrostatic positioning
  - charged samples
  - feedback controlled DC electric field
  - conducting or nonconducting materials
  - solid or liquid phase
  - vacuum or controlled gaseous environment
- Dielectrophoretic Positioning
  - no sample charges
  - feedback controlled DC (or AC) inhomogeneous electric field
  - conducting or nonconducting materials
  - solid or liquid phase
  - vacuum or controlled gaseous environment
- Electromagnetic Positioning
  - feedback controlled rf magnetic field
  - conducting sample materials
  - vacuum or controlled gaseous environment

TETRAHEDRAL ELECTROSTATIC POSITIONER
with vibration isolation



TETRAHEDRAL ELECTROMAGNETIC POSITIONER
with vibration isolation



Tetrahedral Eletrode/Coil Arrangement

- Open structure
- Clear sample viewing
- Easy access to diagnostic instruments
- Decoupled positioning and heating
- Enough space for vibration isolation

Feedback Controlled sample positioning

- Minimum control force in response to external perturbation
  - Positioning: with stability better than 20 microns
  - Damping: controlled damping factor
  - No internal/external flow
  - No rotational instability
- Perturbation free region: programmable
  - absolute quiescence in the region (~10 sec)
- Selection of sample position: +/- 5 mm
- Controlled sample velocity: TBD

Electrostatic-Acoustic Hybrids

- Utilization of feedback controlled electrostatic sample positioning capabilities
- Utilization of acoustic sample manipulation capabilities (oscillation and rotation)
- Charged drop dynamics, crystal growth, cell culturing

N91-21371

A Computer Model of the Electrostatic Positioning System

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Positioning systems based upon electrostatic forces are being developed for the containerless processing of materials that cannot use acoustic or electromagnetic positioning techniques. Currently, electrode configurations for these electrostatic systems are designed on the basis of approximate analytical calculations and past experience. A three-dimensional computer model is being developed that will predict the electrostatic fields and forces for a given electrode configuration and will allow for a more rapid evaluation of proposed designs. Early results of this model will be presented.

THREE-DIMENSIONAL, NUMERICAL MODELING
OF ELECTROSTATIC POSITIONING SYSTEMS

PURPOSE OF THE MODEL

- TO DETERMINE THE ELECTRIC FIELD AND CHARGE DISTRIBUTIONS WITHIN THE CHAMBER AND ON THE SURFACES OF SAMPLES, SO AS TO AID THE SCIENTIFIC UNDERSTANDING OF THE EFFECTS OF ELECTROSTATIC POSITIONING ON THE SAMPLE

- TO PROVIDE AN ENGINEERING TOOL FOR THE EFFICIENT DESIGN OF FUTURE ELECTROSTATIC POSITIONING SYSTEMS

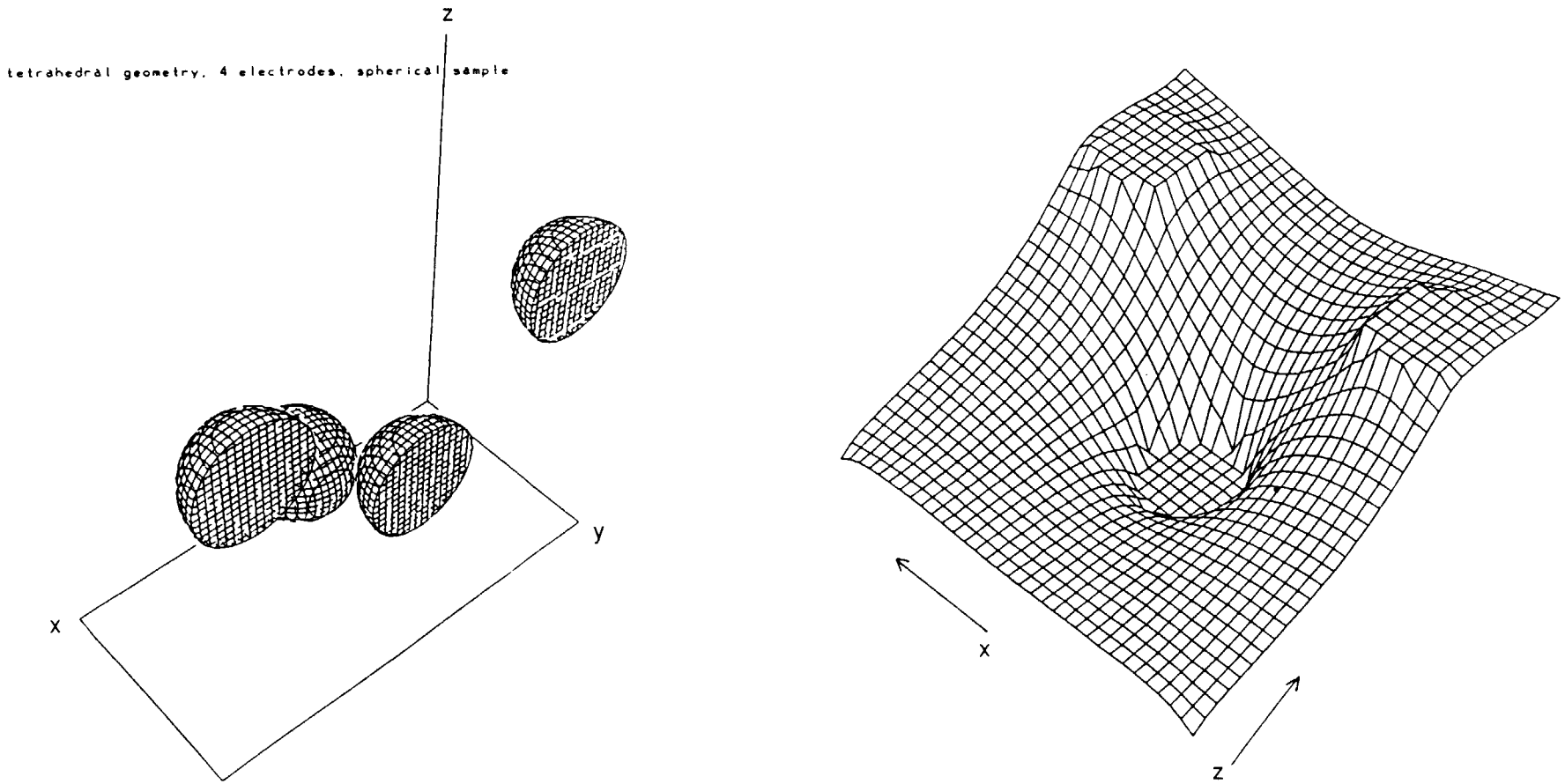

Figure 2: Cross-sectional view of a tetrahedral positioner, and plot of electric potential at the surface of the cross section.

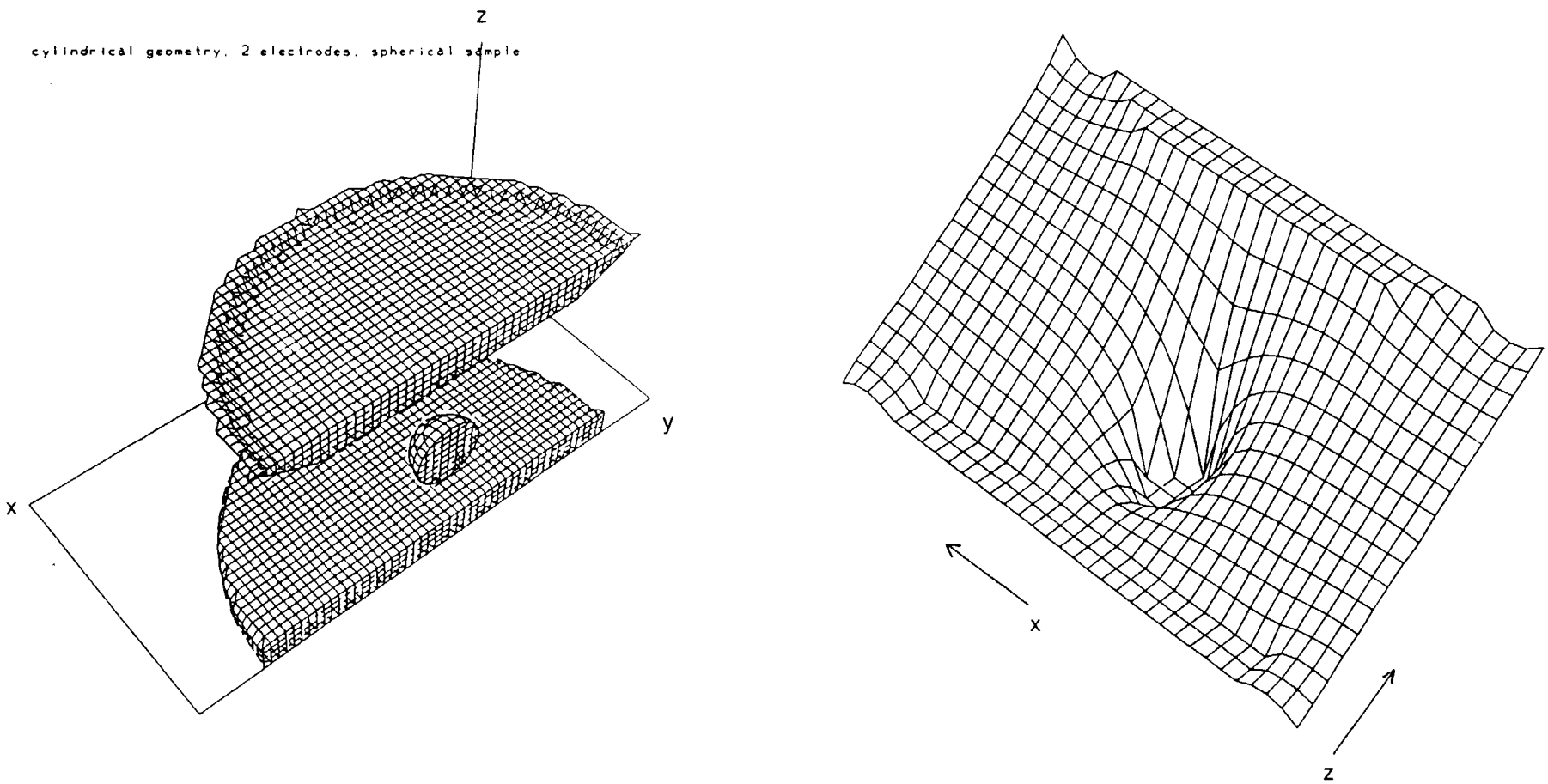

Figure 1: Cross-sectional view of single-axis positioner, and plot of electric potential at the surface of the cross section.

THREE-DIMENSIONAL, NUMERICAL MODELING
OF ELECTROSTATIC POSITIONING SYSTEMS

THE NUMERICAL MODEL

- A THREE-DIMENSIONAL, FINITE-DIFFERENCE MODEL IS USED
- INITIALLY, IT MODELS THE CASE OF SOLID CONDUCTORS IN A STATIC ELECTRIC FIELD
- FUTURE IMPROVEMENTS WILL INCLUDE DIELECTRICS, ELECTRON BEAMS, STATIC MAGNETIC FIELDS, THERMIONIC EMISSION, AND LIQUID SAMPLES
- IT ALLOWS FOR A VARIETY OF ELECTRODE SHAPES AND CONFIGURATIONS
- IT USES A MULTIGRID METHOD TO SOLVE FOR THE POTENTIAL FIELD
- IN FUTURE, IT WILL USE AN ADAPTIVE GRID REFINEMENT TECHNIQUE TO MORE ACCURATELY HANDLE HIGH FIELD REGIONS

N91-21372

SOME CONSIDERATIONS FOR VARIOUS POSITIONING SYSTEMS AND THEIR SCIENCE CAPABILITIES

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Northbrook, Illinois

Abstract

Containerless processing of materials at elevated temperatures is discussed with emphasis on 1) high temperature chemistry and thermophysical properties and 2) materials science and materials processing. Acoustic and electromagnetic positioning of high temperature melts are discussed. Results from recent ground-based experiments, including KC-135 testing of an acoustic levitator, are presented. Some current positioning technologies and the potential for enhancing them are considered. Further, a summary of these technologies and their science capabilities for the development of future experiments is given in this paper.

Need for Containerless Processing

The capability of conducting materials research in a containerless state at high temperatures is of significant interest to the scientific community. In a typical materials process, contaminants from the crucible can alter or even invalidate results of the investigation. As the processing temperature increases, there is an increase in the probability and the rate of unwanted reactions occurring between the crucible and its contents. Containerless processing eliminates the crucible, thus eliminating the dominant source of contamination. In acoustic levitation, the materials being investigated come in contact only with the medium in which the acoustic waves propagate. This medium typically may be dry air or an inert gas, such as argon, or even a special gas for specific reactions. In electromagnetic levitation the specimen investigated can be processed in either vacuum or some suitable gas, but the specimen must have sufficient electrical conductivity.

Nonresonant Acoustic Levitator Systems

Fig. 1A illustrates the basic concept central to a single-axis resonant system.[1] The nonresonant system shown in Fig. 1B uses an acoustic transducer at the bottom, which produces a primary, nearly plane wave, which impinges on a small reflector.[2] The approximately spherical reflected wave interferes with the primary wave and the resulting acoustic field produces continuous restoring forces on a specimen. The magnitude of these forces is quite small. While sufficient to levitate small objects at ambient temperatures, it is not strong enough when the supporting gas is at a high temperature. However, in the microgravity of space these restoring forces are more than adequate to position a research specimen.[3]

One configuration of the Single Axis Acoustic Levitator, called SAAL, is shown in cross section in Fig. 1B. Three levitation experiments using the SAAL on an October, 1985 Space Shuttle flight successfully demonstrated for the first time containerless processing of glass specimens at high temperatures and attained some scientific and engineering objectives.[4] Positioning of both solids and liquids without a container at temperatures from 800 to 1500°C was achieved.

In each case a specimen was injected and captured in the acoustic potential energy well. Specimens were positioned in the containerless state while being heated, melted, cooled, and solidified.

Acoustic Levitation Furnace (ALF)

The acoustic levitation furnace (ALF) facility under development will use a hotwall furnace and acoustic positioning for containerless processing of materials up to 1750°C. The facility is capable of processing a substantial number of experimental specimens under a variety of conditions thus enabling multiple research efforts. The ALF is designed with six opposed acoustic sources in three orthogonal axes to provide acoustic control of both solid and liquid specimens.[5] Fig. 1C shows the schematic arrangement of these acoustic transducers. The resulting acoustic field produces an energy well that is symmetric and stable, providing excellent translational stability for the specimen. These features can ensure that the containerless state is maintained throughout processing and that the specimen remains both centered and spherical when melted.

High Temperature Acoustic Levitator (HAL)

The high temperature acoustic levitator (HAL) is a facility designed for containerless processing of materials at temperatures up to 2000°C or above. The facility employs an array of six high power acoustic transducers which produce a very symmetric acoustic field promoting a stable levitating environment. A beam heating technique is used to obtain temperatures in excess of 2000°C. In one version of this approach radiation emitted from compact xenon arc lamps are focused onto a levitated sample using ellipsoidal reflectors. Fig. 2 shows a brassboard version of HAL which has been designed to be flown aboard the KC-135 microgravity test facility.

Fig. 3A shows sound positioning versus time data for a density 2.2 gm/cm_3 sample. Good damping of residual motion is apparent within a time constant of about 2 seconds. Figure 3B shows a similar plot for a specimen density of 8.9 heated to over 1400K.

Stabilized Electromagnetic Levitator (SEL)

The Stabilized Electromagnetic Levitator is a highly stable multi-coil levitator for melting and undercooling studies in a microgravity environment. The module design is shown in Fig. 2. SEL is characterized by independent control of heating and positioning. Both highly or poorly conductive materials, metallic or non-metallic, may be levitated. High frequency induction heating of samples to 2700°C or greater will be possible. Heating and cooling rates of at least 200°C/sec. would be available. By varying the signals between coils, sample stability and oscillation can be controlled. Independent heating will allow undercooling studies without sample instability.

The basic design and development work for a SEL test module has been completed at Intersonics under a SBIR grant from NASA. The test module is a single-axis system powered by modified, commercially available, high efficiency, solid-state, radio frequency amplifiers. Techniques for coil construction, transmission line fabrication and insulation are under development at Intersonics.

Summary and Conclusion

Acoustic levitation in space is providing the experimenter and eventually the manufacturer with new techniques for high-temperature containerless materials processing. In acoustic levitation the technology has evolved from a single-axis resonance tube levitator in 1971 to the nonresonant, three-axis, opposed source system now under development in ALF and HAL. An electromagnetic system is being developed which may have greater flexibility than previous designs. A summary of the science capabilities for these systems is given in Table 1.

With the availability of these new techniques for high-temperature containerless materials processing, a vastly expanded range of materials from processing up to (and perhaps beyond) 2700°C are envisioned. It is reasonable to anticipate that significant advances in the production of new materials, new ceramics, alloys, and optical and electronic materials will result.

Acknowledgment

This work was supported by the NASA, George C. Marshall Space Flight Center.

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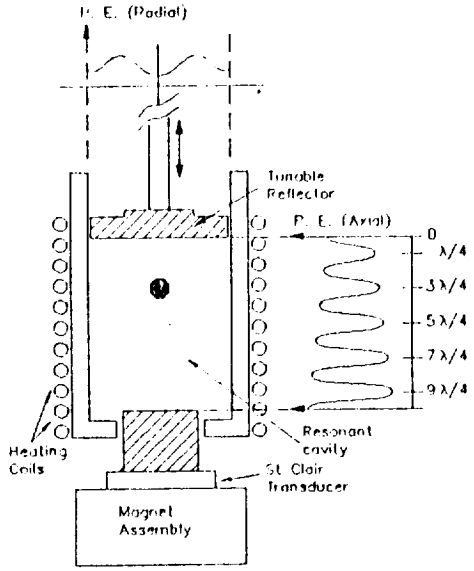
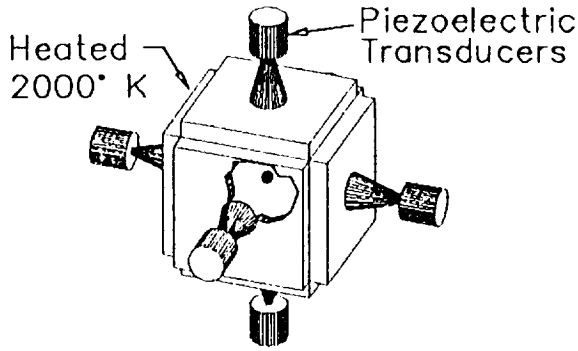
5. Merkley, D. R., Rey, C. A., Hammarlund, G. R., and Danley, T. J., "Specimen Translational Control Capabilities Using an Opposed Radiator Acoustic Levitation System," 114th Meeting of the Acoustical Society of America, Miami Fl, Nov. 20, 1987.

INTERSONICS INCORPORATED

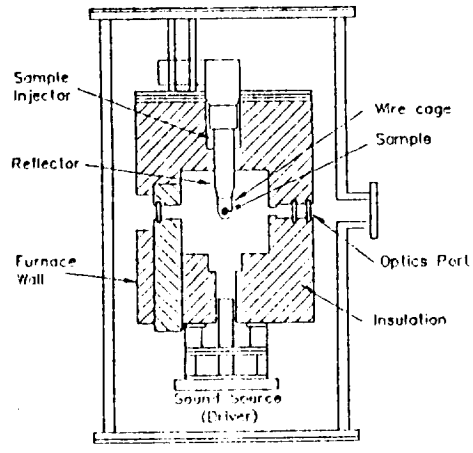
ACOUSTIC LEVITATORS



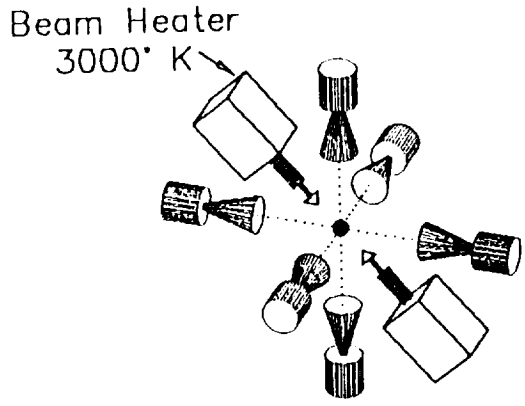
SINGLE AXIS RESONANT LEVITATOR
Figure A



ACOUSTIC LEVITATION FURNACE (ALF)
Figure C

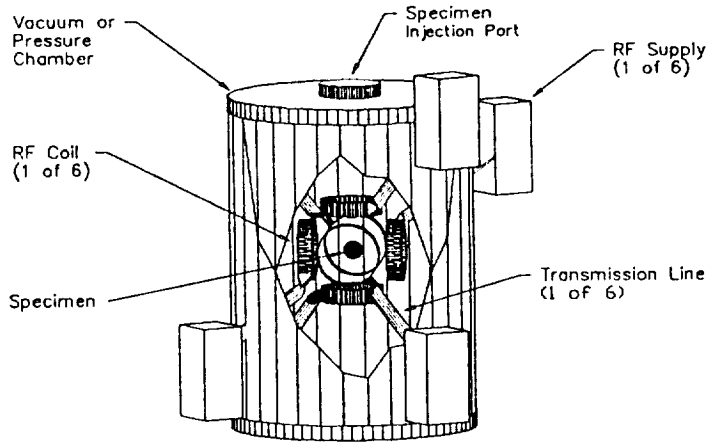


SINGLE AXIS ACOUSTIC LEVITATOR (SAAL)
Figure B

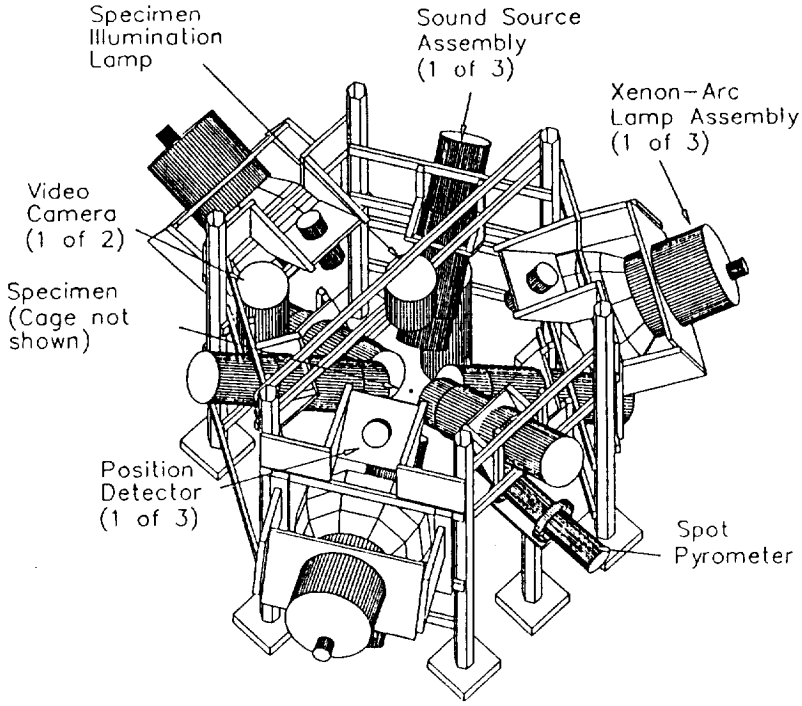


HIGH TEMPERATURE ACOUSTIC LEVITATOR (HAL)
Figure D

FIGURE 1



STABILIZED ELECTROMAGNETIC LEVITATOR (SEL)



HIGH TEMPERATURE ACOUSTIC LEVITATOR GENERAL ASSEMBLY

FIGURE 2

C-6

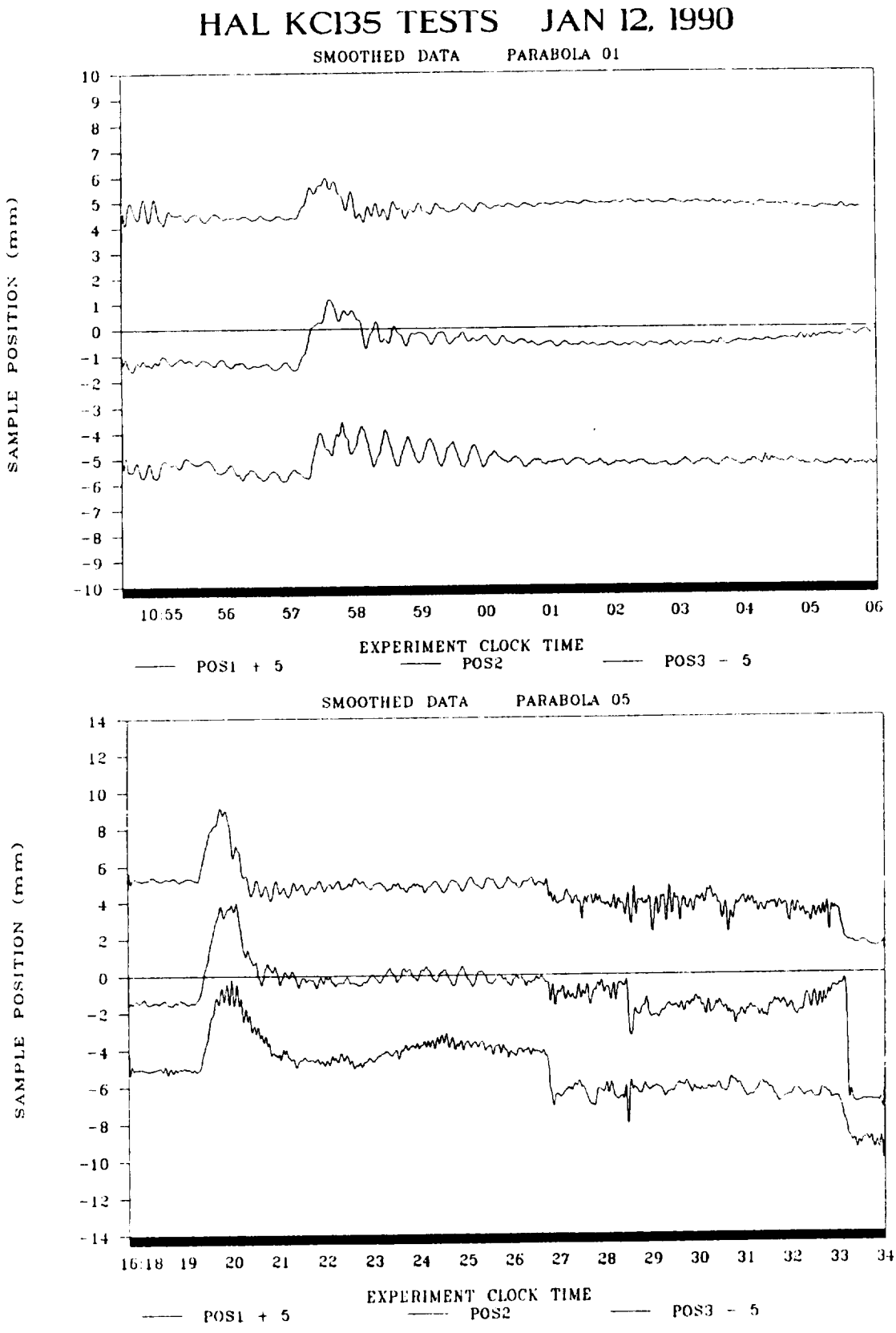


FIGURE 3

MICROGRAVITY SCIENCE CAPABILITIES SUMMARY FOR

HIGH TEMPERATURE CONTAINERLESS PROCESSING MODULE (HTCPM)

A High Temperature Containerless Processing Module (HTCPM) could be one of the modules supported by the DPM package for Spacelab, and could be a precursor module for the Modular Containerless Processing Facility (MCPF) for the space station. Depending on the science requirements, it may utilize one or more of the following technologies:

PARAMETER	ACOUSTIC (HOT WALL) (ALF)	ACOUSTIC (BEAM HEATED) (HAL)	STABILIZED ELECTRO-MAGNETIC (SEL)
Temperatures	800-1750 °C	30-2000 °C or higher	30 to 2700 °C or higher
Specimen Isothermality	Very Good (± 2 °C/cm)	Good	Good
Processing Gas	Inert, Reducing, Oxidizing	Inert, Reducing, Oxidizing	Inert, Reducing, Oxidizing, Vacuum
Gas Purity Particulate Contamination	Good Class 1000 or Better	Excellent	Excellent
Heating and Cooling Rates	Slow to Moderate (< 2 °C/sec.)	Very Fast (-200 °C/sec.)	Very Fast (-200 °C/sec.)
Conductive Specimen Required	No	No	Yes
Liquid or Solid Processed	Yes	Yes	Yes
Specimen Size	2 - 10mm	2 - 6mm	2 - 6mm

SOME POSSIBLE APPLICATIONS FOR CONTAINERLESS PROCESSING

AT HIGH TEMPERATURES IN MICROGRAVITY

HIGH TEMPERATURE CHEMISTRY

- Cp VS TEMPERATURE, UNDERCOOLED LIQUIDS
- OPTICAL PROPERTY MEASUREMENT - n, k, e, ETC
- SURFACE FILM BEHAVIOR, GROWTH RATES, ETC
- THERMODYNAMICS AND KINETICS (OF OXIDE AND NITRIDES)
- VAPOR PRESSURES BY LIF, ETC
- PASSIVATION/CORROSION EFFECTS

MATERIALS PROCESSING

- PURIFY, REMOVE OXIDES, IN METALS/ALLOYS
- HIGH PURITY SEMICONDUCTORS
- MICRO ALLOYING, SUPER ALLOYS
- CONTROLLED MICRO STRUCTURES
- HIGH Tc SUPERCONDUCTORS
- DEEP UNDERCOOLING, AMORPHOUS MATERIALS
- NON-EQUILIBRIUM STUDIES
- OXIDE DISPERSION STRENGTHENING
- OPTICAL MATERIALS, BENCHMARK MATERIALS

TABLE 1

N91-21373

RADIOGRAPHIC INSTRUMENTATION FOR DPM EXPERIMENTS

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R.T. Simchick
Lockheed Corporation, Hampton, VA
and

P.G. Barber
Longwood College, Farmville, VA

Radiography has been successfully used to monitor both the shape and position of the melt-solid interface in Bridgman growth and has been used, by others, to observe fluid flow. The image recording medium is either film or image enhanced real time VCR recording.

The presented paper discussed the new developments in x-ray radiography that may be applicable to containerless experimentation. The two features discussed were the use of radiography to determine the position and shape of the solid-liquid interface and, with the aid of appropriate markers, the flow patterns in either the surface or bulk of the liquid state. In addition, both surface energy and fluid viscosity measurements can be made with the aid of the described radiographic system.

The experimental techniques presented were developed under MSAD-ATD support and are part of an ongoing research effort at Langley Research Center.

CONTAINERLESS EXPERIMENTATION
IN
MICROGRAVITY WORKSHOP

17-19 JANUARY, 1990

RADIOGRAPHIC INSTRUMENTATION FOR DPM EXPERIMENTS

Archie Fripp and W.J. Debnam
Langley Research Center, Hampton, Va.

R.T. Simchick
Lockheed Corporation, Hampton, Va.

and
P.G. Barber
Longwood College, Farmville, Va.

OBJECTIVE OF TALK

TO INTRODUCE A MEASUREMENT TECHNIQUE DEVELOPED FOR BRIDGMAN CRYSTAL GROWTH WHICH MAY, IF PROPERLY DEVELOPED, BE USEFUL TO THE CONTAINERLESS PROCESSING EXPERIMENTS FOR THE MEASUREMENT OF THE LIQUID-SOLID INTERFACE AND BOTH SURFACE AND BULK FLUID FLOW.

* RADIOGRAPHIC INSTRUMENTATION IN BRIDGMAN GROWTH

I. INTERFACE MEASUREMENTS
- IMPORTANCE OF INTERFACE
- INSTRUMENTATION
- FILM RESULTS
- REAL TIME MEASUREMENTS

II. FLUID FLOW
- MARKER DEVELOPMENT
- BUBBLE MOVEMENT

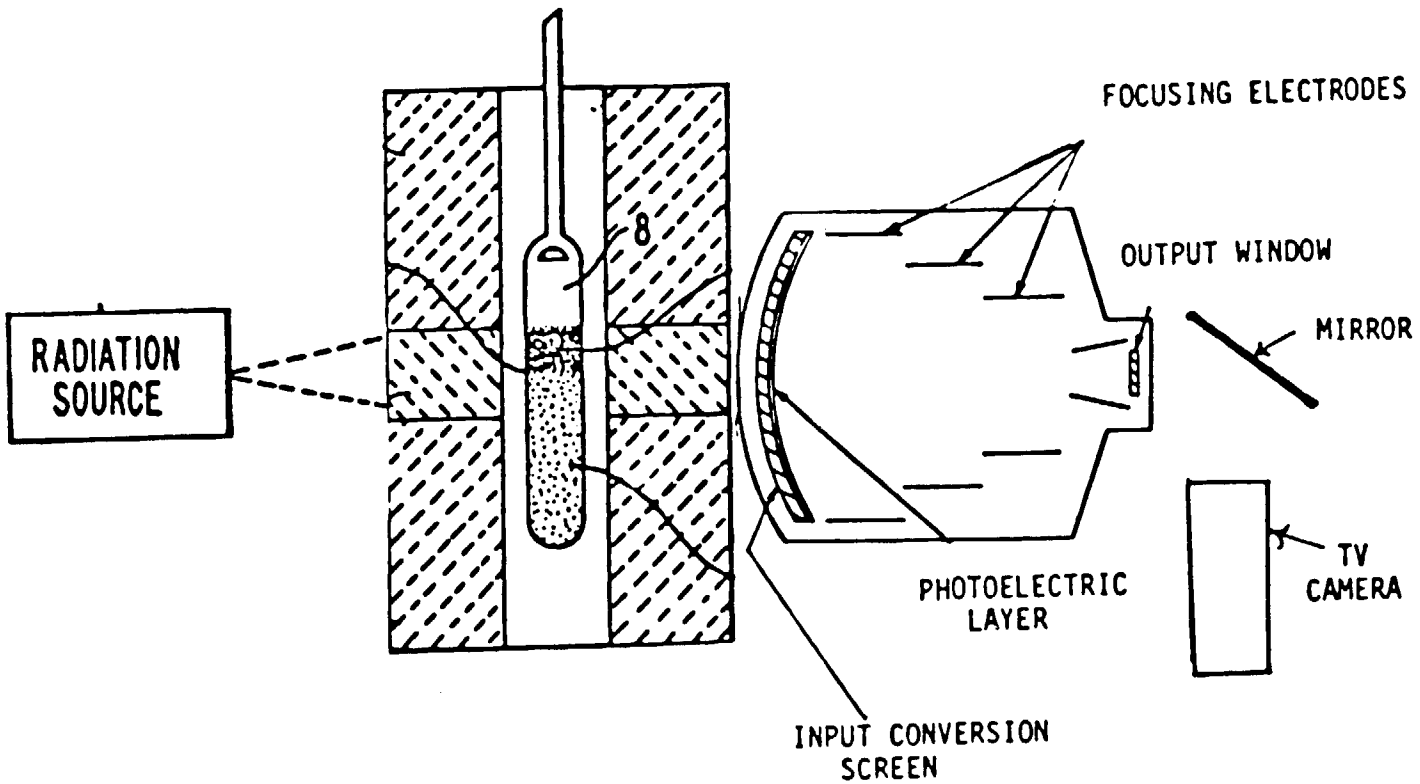
* APPLICATIONS TO CONTAINERLESS PROCESSING

SURFACE AND BULK FLOW MEASUREMENTS

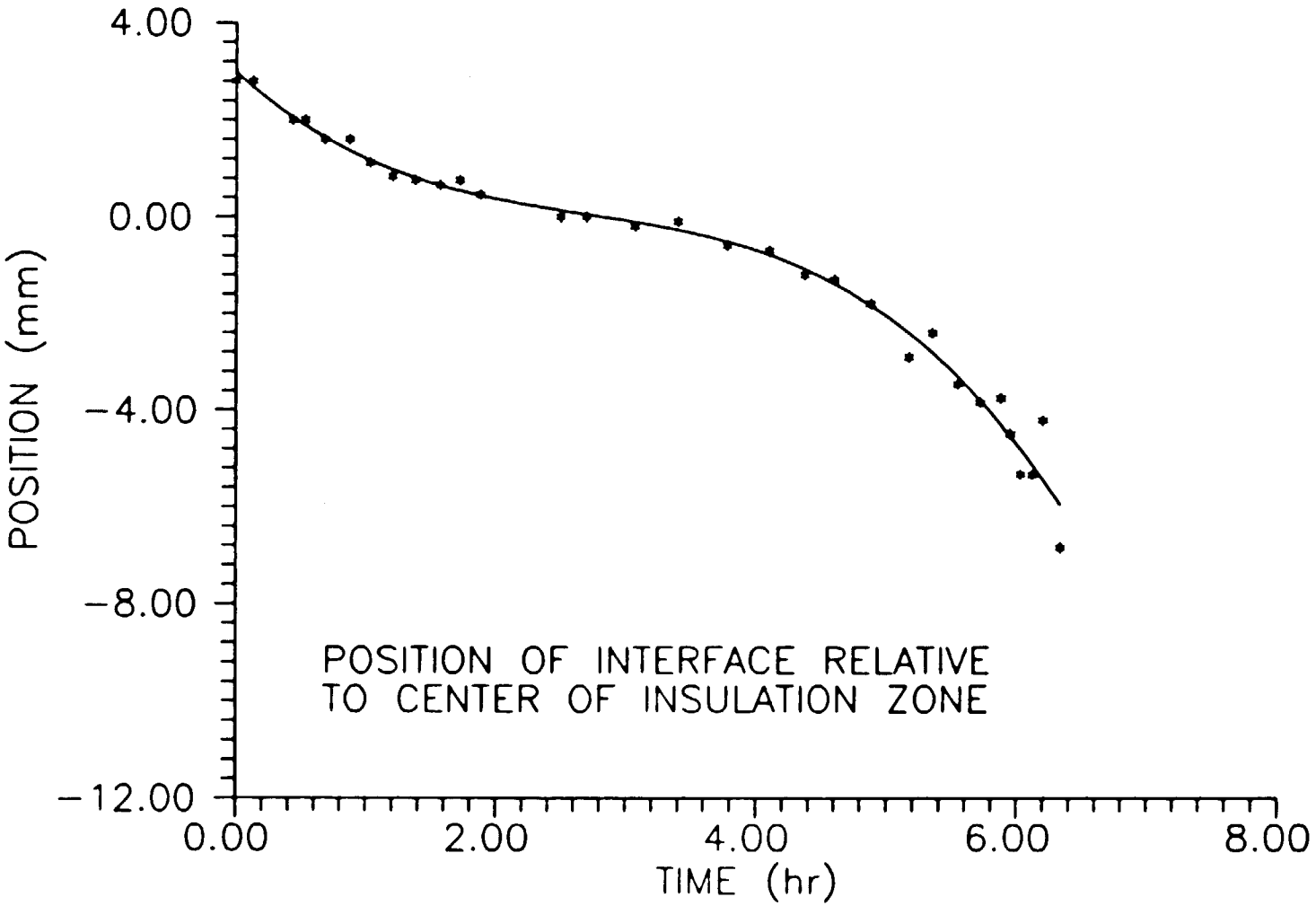
* APPLICATIONS TO OTHER MEASUREMENTS

VISCOSITY AND SURFACE ENERGY

* CONCLUSIONS



REAL TIME RADIOGRAPHY


4.00
0.00
-4.00
-8.00
-12.00
POSITION (mm)
POSITION OF INTERFACE RELATIVE
TO CENTER OF INSULATION ZONE
0.00
2.00
4.00
6.00
8.00
TIME (hr)

REQUIREMENTS FOR FLUID FLOW MARKERS

WET BY FLUID

IMPERVIOUS TO FLUID

MATCHING FLUID DENSITY

LARGE X-RAY DENSITY

SMALL SIZE

NON-NUCLEATING SURFACE

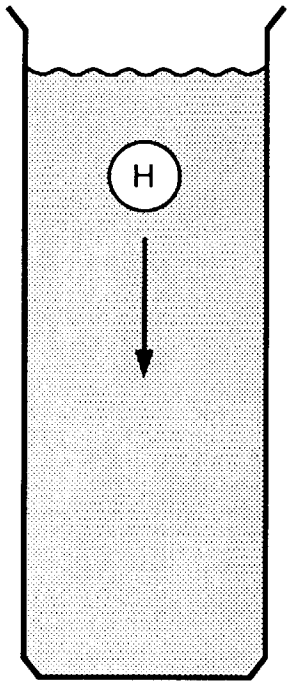
VISCOSITY MEASUREMENTS

STOKES LAW, V_t

$V_t = gD(\rho_L - \rho_S) / 18\mu$

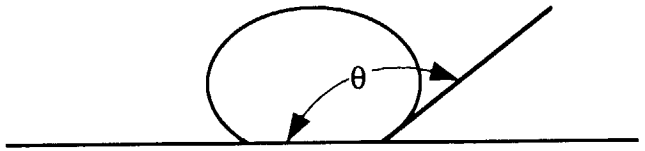
DROP SPHERE OF KNOWN DIAMETER & DENSITY

MEASURE DROP TIME

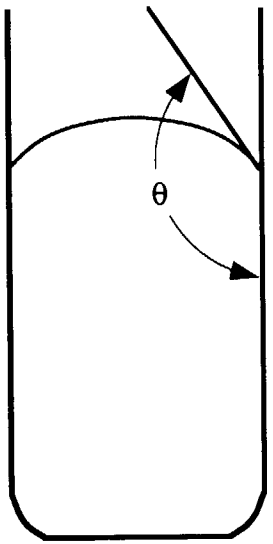


SURFACE TENSION MEASUREMENTS

YOUNG & DUPRE EQUATION



$$\cos\theta = \frac{\sigma_{SV} - \sigma_{SL}}{\sigma_{LV}}$$



SUMMARY

TECHNIQUE PROVEN USEFUL IN BRIDGMAN GROWTH INTERFACE MEASUREMENTS

FLUID FLOW AS YET UNPROVEN

CAN IT BE USEFUL FOR MCPF TYPE EXPERIMENTS?

APPLICATIONS TO OTHER THERMOPHYSICAL MEASUREMENTS?

N91-21374

THE METROLOGY OF SPHERICAL SHELLS USING SYNCHROTRON X-RAY MICROTOMOGRAPHY

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Vanderbilt University

Kevin L. D'Amico
EXXON Research and Engineering Company

Sandia National Laboratories has a need for large, high quality spherical shells for its Inertial Confinement Fusion project . The Jet Propulsion Laboratory has developed the technique to produce these spherical shells and Vanderbilt University is continuing to refine the process for producing larger, higher quality shells. In order to assess the usefulness of the product shells, it is necessary to characterize them using a non-destructive technique.

With recent advances in solid state imaging technology, and the increasing availability of synchrotron x-ray radiation sources, synchrotron x-ray microtomography is emerging as a nondestructive technique for the evaluation of the structure and composition of small specimens with spatial resolution in the micron range. Synchrotron radiation offers the following advantages over conventional x-ray sources: 1) high brightness; 2) continuous emission which is tunable over a large energy range; 3) faster data collection rates; 4) highly collimated beam of large cross-section permitting the illumination of large specimens. Synchrotron x-ray microtomography enables the structure of individual spheres to be evaluated in order to reveal the concentricity and sphericity of the internal void and the uniformity of the shell wall.

The Center for Microgravity Research and Applications has been utilizing the unique capabilities of the EXXON X-2 X-ray beamline at National Synchrotron Light Source (NSLS) to evaluate the application of microtomography to characterize spherical shells. Currently the work is performed on a collaborative basis with EXXON. This work will be illustrated with reconstructions of some of the recent shell product.

Materials characterization techniques are used to document the effect of processing materials in a microgravity environment. Generally, these specimens are so valuable that one wants to obtain as much information as possible from as few as a single specimen that may be returned from space. Preferably we want to use techniques which preserve the specimen for subsequent analysis.

Microtomography provides a nondestructive means to map the x-ray attenuation coefficient of a specimen in three dimensions. Local variations in the mass attenuation coefficient may be due to changes in density or composition within the specimen. These variations are interpretable in terms of the microstructure.

This paper presents the results of a nondestructive evaluation of the structure of aluminum spherical shells using synchrotron x-ray microtomography. These structures were solidified from the molten state under microgravity conditions in the JPL cryogenic drop tube facility. We are specifically interested in obtaining information on the structure of the internal void, and the uniformity of the shell wall thickness.

In the figure we show a reconstruction of two shells which have collided during the free fall in the drop tube before they were completely solidified. We have presented the data in terms of an isosurface generation, marking the transition in attenuation at the outer surface of the sphere and the inner surface of the shell wall where the density changes from gas to aluminum.

The shells are 2mm in diameter with a 200 micron wall thickness. The point resolution in this data set is 6 microns. Each shell has been mathematically "sectioned" to reveal the interior surface of the void. Despite the irregular shape of some of the shells, it is noteworthy that the voids themselves are so spherical.

There is a need for 'on-orbit' characterization capabilities aboard space station. Though it is impractical to propose a tomography facility, at a minimum, a simple radiography system would be very useful for documenting the 'as-grown' condition of some fragile materials which may be damaged by decelerations upon their return to earth, or simply as a diagnostic tool for monitoring various solidification experiments.

Strategic planners should note that several presentations at this workshop have been devoted to the need for materials characterization.

OSSI FLIGHT INSTRUMENT PROJECT REVIEW

DROP PHYSICS MODULE

APRIL 3, 1990

GARY HILL

Drop Coalescence Studies

N91-21375

A. V. Anikumar and T. G. Wang
Vanderbilt University

The objective of this experimental study is to understand the detailed mechanics of the coalescence of liquid drops. The experiments are being conducted in an immiscible acoustic levitator with degassed water as the most medium.

Typically, a quasineutrally buoyant drop to silicone oil mixed with bromobenzene is levitated close to the velocity node of the levitator. A second drop of the same liquid is introduced, and as it slowly seeks the same levitation position, the drops coalesce. Coalescence is delayed until the host film between drops is completely drained. Following coalescence, the excess surface energy in the coalesced drop is dissipated through shape oscillations.

The final events of film rupture followed by drop coalescence are rapid, and are photographically studied with high-speed video (1000 fps.). Laser-induced fluorescence technique is used to visualize the dynamics of host film drainage. The details of the coalescence mechanics will be presented.

DROP COALESCENCE STUDIES

A.V. Anilkumar and T.G. Wang
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Nashville, Tennessee

Introduction

The objective of this experimental study is to understand the detailed mechanics of drop coalescence. The main aspects of interest are: surface tension controlled coalescence dynamics, the dynamics of host film drainage, and the role of relative drop size. The experiments are being conducted in an immiscible acoustic levitator with degassed water as the host medium.

The coalescence studies are part of a more general investigation of the problem of collision and coalescence of drops being conducted at the Center for Microgravity Research and Applications (MRA) at Vanderbilt University. These experiments form a core ground-based support program for collision - coalescence and materials processing experiments being planned for future space flights. In addition, the ground-based work should provide an understanding of such basic issues as interactions of rain drops and the evolution and size distribution of natural and industrial aerosols.

Experimental Technique

Figure 1 is a schematic of the set up for conducting coalescence experiments. The acoustic cell design and the levitation technique resemble that of Trinh and Wang (1982). The acoustic cavity is 13.4 cm X 13.4 cm in inside cross section and the driver transducer vibrating surface is 12.7 cm in diameter. The transducer is driven in its fundamental longitudinal resonance at 19.14 kHz, and the height of the water column is 11.8 cm.

To initiate the experiments, a quasi-neutrally buoyant drop of silicone oil (Dow Corning 510/100 cs) mixed with bromobenzene is first levitated near the pressure maximum closest to the top surface of the water column. A second drop of the same liquid is then carefully introduced, and as it slowly seeks the same levitation position, the drops coalesce. Coalescence, one initiated, proceeds rapidly and is photographically studied with high-speed video (Kodak Ektapro 1000) at 1000 frames per second.

Results

The presence of the host film between drops is a barrier to coalescence. Coalescence is delayed till the film is completely drained. since very low acoustic force is used in drop levitation, the film drainage is slow. At a certain critical film thickness, the film suddenly ruptures and the two drops contact and coalesce. The mechanism of film rupture is yet to be understood.

Equal size drops. Figure 2 (a-h) depicts the coalescence, initiated just following film rupture, of two drops of almost the same size (one drop seeded with aluminum tracers). As the two drops contact (Figure 2a), the lost surface energy appears as kinetic energy driving the coalescence rapidly. The coalescence front is planar (Figure 2 b,c,d) and there is no mixing induced during

the coalescence process. The excess surface energy in the coalesced drop is dissipated through shape oscillations (Figure 2 e-h). Marston and Goosby (1985) have analyzed the damped shape oscillations of a drop in an immiscible system and, using their derivation for drop oscillation frequency, one can determine the surface tension of the coalesced drop.

Unequal size drops. Figure 3 (a-h) depicts the coalescence of two drops of diameter ratio 1.7:1.These pictures depict the cross section of the coalescing drops which is made visible by seeding the drops with a fluorescent dye and illuminating with a thin Argon ion laser sheet. The smaller drop is partially engulfed (Figure 3 a-e) by the larger drop and the shape oscillations of the coalesced drop are dominated by mode 2 oscillations (Figure 3 g,h). For higher diameter ratios, the engulfing is more a local shape perturbation on the larger drop.

Future Work

Currently, efforts are being directed towards using laser-induced fluorescence technique to study the host film drainage and rupture. It is not clear whether films drainage is uniform and that coalescing surfaces remain parallel up to rupture as assumed by Foote (1971) and others in their analyses. The film could very well be unstable to random surface perturbations; this issue needs to be resolved. If necessary, interference techniques will be employed to make film thickness measurements.

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Marston, P.L. and Goosby, S.G., Physics of Fluids, vol. 28 (5), pp. 1233-1242, 1985.

Trinh, E. and Wang, T.G., Journal of Fluid Mechanics, Vol. 115, pp. 453-474, 1982.

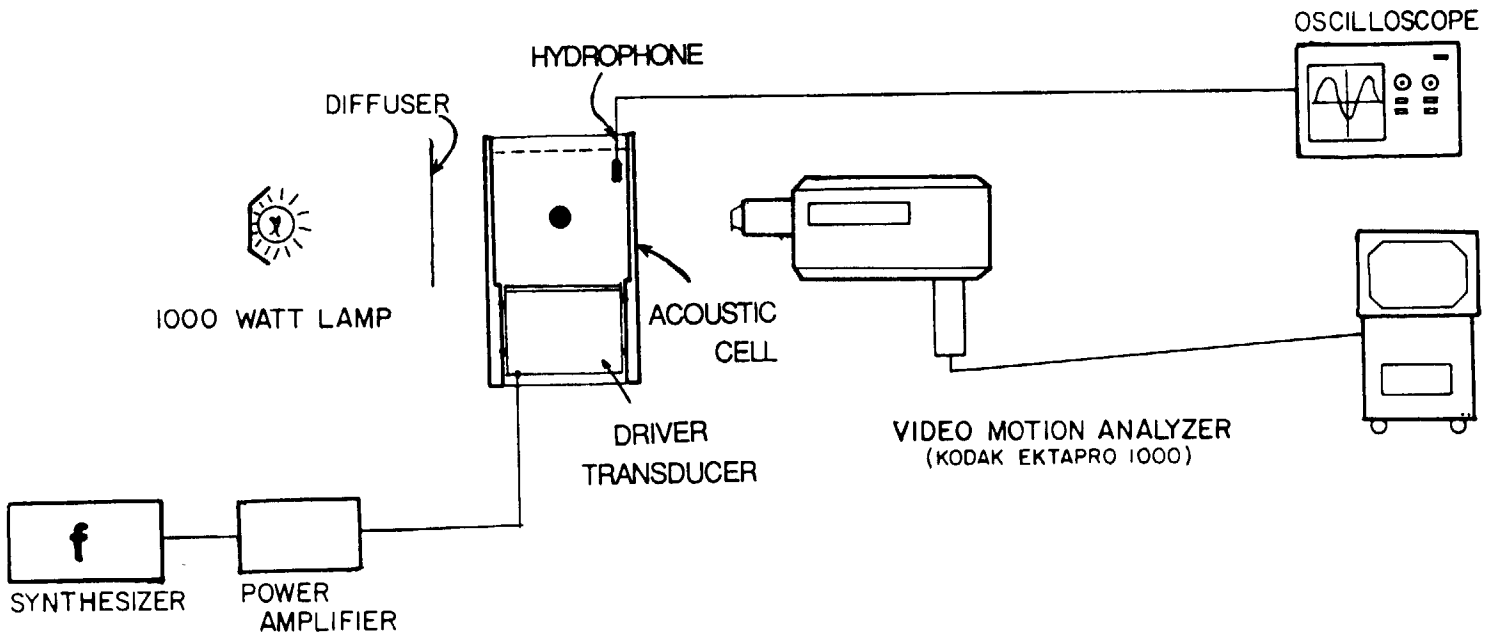

FIG. 1: EXPERIMENTAL SET UP (Schematic)

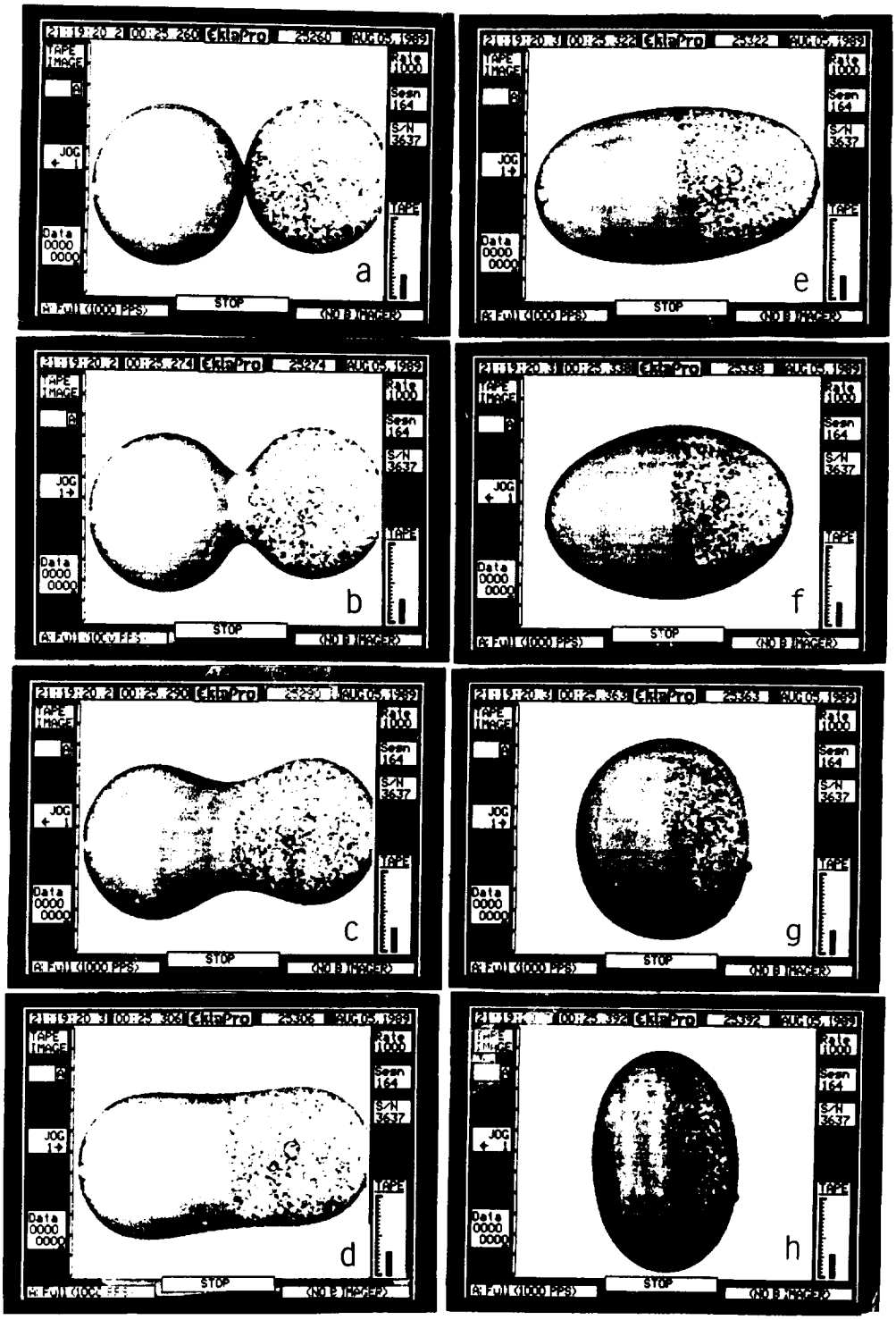

FIG. 2: COALESCENCE AND OSCILLATION

(Equal Size Drops)

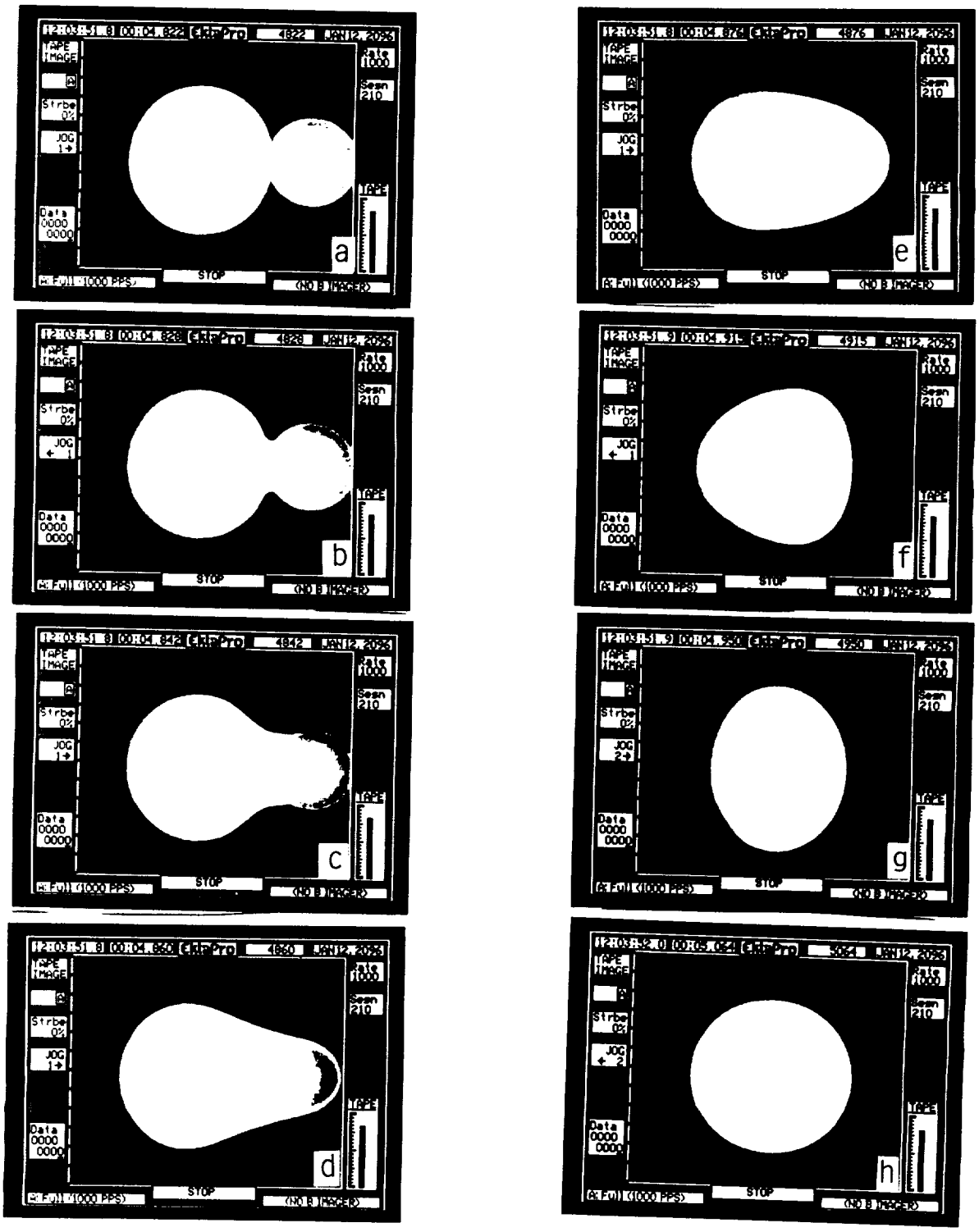

FIG. 3: COALESCENCE AND OSCILLATION
(Unequal Size Drops)

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N91-21376

Oscillational Instabilities in Single Mode Acoustics Levitators

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ABSTRACT

An extension of standard results for the acoustic force on an object in a single-mode resonant chamber yields predictions for the onset of oscillational instabilities when objects are levitated or positioned in these chambers. Our results are consistent with experimental investigators. The present approach accounts for the effect of time delays on the response of a cavity to the motion of an object inside it. Quantitative features of the instabilities are investigated. We discuss the experimental conditions required for sample stability, saturation of sample oscillations, hysteretic effects, and the loss of ability to levitate.

I would like to discuss some progress that has been made in the study of a phenomenon that is both a technological challenge to the designers of acoustic positioners and an exciting research topic with relevance to fundamental issues in the mechanics of solid bodies and continuous media. This phenomenon is illustrated in the behavior of a sample levitated in an ACES module in Space Shuttle Flight STS31B, January 1984. The sample began to oscillate, and those oscillations ultimately resulted in the loss of positioning of the object. While the oscillations were initially orderly and periodic, or at least approximately so, they later became random, or in nonlinear dynamics terminology, chaotic. Oscillations have also been observed in ground-based levitators, specifically a triple-axis levitator. Here one sees two kinds of behavior. One can observe oscillations that decay away. They are clearly a transient feature of the motion of the levitated object, which eventually settles into a stable position. This position represents a "dynamically stable fixed point." As a second alternative, there are oscillations that do not decay. Rather they persist at a fixed amplitude for as long as the acoustic field in the chamber is excited. This kind of oscillation is called a dynamically stable limit cycle. A striking feature of this behavior is that it develops *spontaneously*. That is, the levitated object can start out essentially stationary, and then it will begin to oscillate with a steadily growing amplitude until the amplitude of the oscillations saturates at its limiting value.

It is possible to respond to this interesting phenomenon in a variety of ways (Figure 1). One can regard it as a problem to be overcome by appropriate modifications in the design of the positioning apparatus — for example, by utilizing some form of feedback to respond to changes in the position of the object by changing some aspect of the acoustic drive. Alternatively, one might attempt to understand the physical mechanism underlying the oscillations and utilize this knowledge to design more stable cavities, or operate them in such a way as to avoid the parameter range in which such oscillations occur.

I strongly feel that the superior approach is the one that is based on a clear, physical understanding of the mechanism for oscillational instabilities. A positioner designed in such a way as to avoid oscillations will ultimately prove more practical than one that relies on an

external mechanism to detect and damp out oscillations via electronic feedback. I think that there is an even more fruitful approach to take to this phenomenon. Regardless of its possible negative, or even potentially positive, impact on the application of acoustic positioning technology, the dynamical instability that occurs in acoustic positioner represents a fertile research topic. One stands to gain a significantly improved understanding of a variety of important dynamical phenomena.

In the remainder of this article, I will touch on some of those topics, discussing what has already been accomplished and what remains to be done.

Figure 2 - Adiabatic invariance. The principle of adiabatic invariance as applied to the single mode levitator allows one to establish a connection between the object's effect on the resonant frequency of an isolated mode in a high Q cavity and the acoustic forces and torques that this mode exerts on the object. The principle of adiabatic invariance, as discovered by Boltzman and Ehrenfest, asserts the following proportionality:

$$\vec{F}_{acoustic} \quad \text{å} \quad \vec{\nabla}_{\vec{r}_0}\omega(\vec{r}_0)$$

Here ω is the resonant frequency of the cavity and r_0 is the position of the object of interest. The constant of proportionality can be calculated in a variety of interesting cases (Figure 3). It depends on quantities like the intensity of the acoustic field. This relationship has been derived and it has been tested experimentally. It provides a useful way of calibrating single mode levitators.

Figures 4 and 5 - The limits of adiabatic invariance. The above relationship applies when all changes in the position and orientation of the object in the chamber are *quasistatic*. Furthermore, it assumes that the acoustic mode is both undriven and undamped. For example, assume that we are in the real world in which acoustic modes are damped and must be driven. What if changes that occur in a system occur at a finite rather than infinitesimal rate? In the case of an acoustic positioner, we have the beginning of an answer to those questions. Garrett

and Barmatz have shown that in an analogue system (the damped, driven harmonic oscillator), parametric changes occurring at a finite rate can lead to instabilities that are strikingly similar to the oscillational instabilities that are observed in acoustic positioners. Dr. Barmatz and I have verified the results of Garrett and Barmatz by performing a highly nonlinear calculation of the forces on a small spherical object that is moving inside a single mode chamber. We have begun to explore in a greater generality the effects of time delays and mechanical feedback on the motion of an object of arbitrary shape and size in a single mode chamber. Here we are investigating virgin territory in dynamics, in that we are attempting to understand in a systematic way the limits of adiabatic invariance and the novel consequences of the corrections to that important dynamical principle that must be introduced to describe the behavior of real, physical objects.

As an example of the progress achieved so far, Figure 6 shows curves that we have calculated for the threshold and saturated amplitudes of oscillations in a single mode acoustic levitator operating in a gravitation-free environment. The amplitudes normalized to the dimension, L_z, of the chamber are plotted against the frequency of the drive minus the resonant frequency of the chamber, normalized to the half-width of the resonance.

There are other modifications to adiabatic invariance that merit study (Figure 7). The investigations described above have been, or will be, carried out on cavities in which the relevant acoustic mode is isolated. The question of what happens when the positioning is accomplished by the excitation of degenerate, or nearly degenerate, modes also deserves our attention. The triple axis levitator utilizes three exactly degenerate plane wave modes in order to levitate and position the object inside. This levitator can rotate the object inside at a controllable rate. A triple-axis levitator was used in the Drop Dynamics Module (DDM) on Shuttle Flight. The rate of rotation is controlled by the relative phase of two of the plane wave modes. There is, as yet, no entirely satisfactory explanation for this phenomenon. A proper description will involve a detailed discussion of the energy exchange that occurs between two degenerate modes, most probably in violation of the principle of adiabatic invariance. It is

interesting that a recent issue of *Physical Review Letters* contains an article describing anti-adiabatic behavior in a quantum mechanical system at the point of level crossing (or degeneracy).

Figure 8. Another aspect of the oscillational instability that deserves study is the possibility of chaotic motion by the moving object. Recall that the object in the videotaped ACES module executed motion that was far from regular. It is altogether likely that this motion fits the now-accepted definition of chaos — in that it is locally deterministic but unpredictable in the long run. However, any attempt to understand that motion will be complicated by the fact that one must take into account the fact that the acoustic drive was constantly being electronically adjusted to home-in on the resonant frequency of the cavity. This "external" feedback gives rise to a more complicated set of equations. We are currently simulating the motion of a small sphere in a single mode cavity using equations that properly describe the motion of the atmosphere in a cavity subject to a fixed-acoustic drive. We hope to be able to construct a full dynamical phase diagram of the object's motion, and, in the process, to explore the interplay between chaotic motion of a solid object and the concomitant behavior of the medium whose acoustic field is levitating it.

Figure 9. Finally, it is worth pointing out why it is a good idea to perform experiments on the motion of levitated object in space rather than in an earth-bound laboratory. The microgravity environment possesses three distinct advantages. First, the relatively low acoustic intensities required to position an object in microgravity (as opposed to the 155–160 dB that is needed in 1 g) make it possible to eliminate unwanted effects, such as rotational instabilities, and also to control nonlinearities in the system. Second, it is possible to utilize a less dense levitating medium in microgravity, so the system becomes more nearly Hamiltonian, because of the reduced viscous drag. Finally, in microgravity it is possible to position a sample at a velocity antinode and exploit the full symmetry of the levitating system.

FIGURE 1 TWO ASPECTS OF OSCILLATIONAL INSTABILITIES

- TECHNOLOGICAL
  - HOW DOES ONE AVOID OR EXPLOIT INSTABILITIES?
    - REACTIVE APPROACH
      - DESIGN THE ACOUSTIC DRIVE WITH FEEDBACK THAT ALLOWS IT TO RESPOND TO EVENTS IN THE POSITIONER
    - BASIC SCIENCE
      - APPROACH: UNDERSTAND THE MECHANISMS LEADING TO INSTABILITY
      - DESIGN AND OPERATE THE POSITIONER IN ACCORD WITH THE INSIGHT GAINED

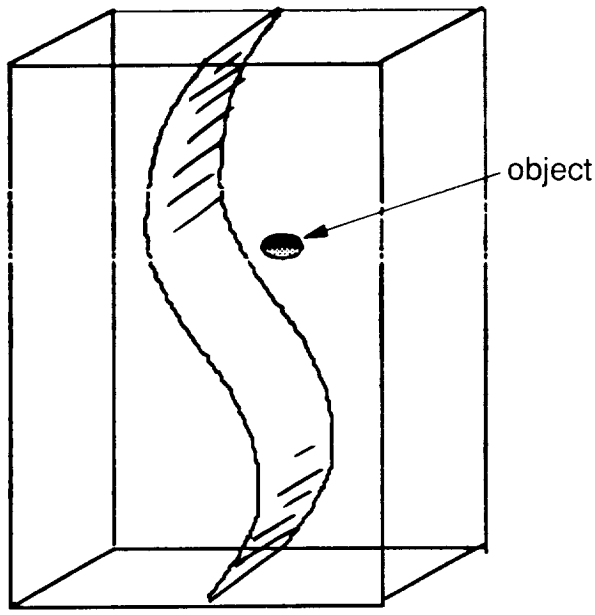
FIGURE 2 SCIENTIFIC IMPLICATIONS OF OSCILLATIONAL INSTABILITIES

- **ADIABATIC INVARIANCE**
  - BOLTZMANN - EHRENFEST PRINCIPLE

    APPLIES WHEN

    - THE OBJECT IS STILL OR MOVING ASYMPTOTICALLY SLOWLY ("QUASISTATIC")
    - THE POSITIONING MODE IS ISOLATED (NO ENERGY EXCHANGE WITH NEARBY ACOUSTIC MODES)
    - DELAY EFFECTS ARE UNIMPORTANT
    - THE POSITIONING MODE IS UNDAMPED AND UNDRIVEN

FIGURE 3



$U(\vec{r}_0, \vec{\theta}, s_1, s_2 \ldots.)$ å $\Delta\omega_0(\vec{r}_0, \vec{\theta}, s_1, \ldots)$

- POSITIONING POTENTIAL — PERTURBATION OF THE MODE'S NATURAL FREQUENCY
- $\vec{r}_0$ = POSITION OF OBJECT — $\left(\vec{F} = -\vec{\nabla}_{r_0} U\right)$ POSITIONING FORCE
- $\vec{\theta}$ = ORIENTATION OF OBJECT — $\left(\vec{\tau} = -\vec{\nabla}_{\theta} U\right)$
- $s_1, \ldots$ = SHAPE PARAMETERS (CORRESPONDING RELATIONSHIPS FOR SHAPING FORCES)
- GENERALIZES RESULTS OF KING & GOR'KOV
- ALLOWS FOR CALIBRATION OF LEVITATING MODULES

FIGURE 4 THE LIMITS OF ADIABATIC INVARIANCE

- SUPPOSE THE MODE IS DAMPED & DRIVEN DELAY EFFECTS ARE NON-NEGLIGIBLE AND THE POSITIONED OBJECT IS MOVING AT A FINITE RATE.
  - WHAT HAPPENS THEN?
    - S. GARRETT AND M. BARMATZ:
    - $m\frac{d^2}{dt^2} + \gamma\frac{dx}{dt} + k(t)x = Fe^{i\omega t} - Fe^{-i\omega t}$
  - RATE AT WHICH WORK MUST BE PERFORMED TO CHANGE THE SPRING CONSTANT, k (t), is
    - $= \frac{1}{2}\langle x^2 \rangle \frac{dk}{dt}$

FIGURE 5

SOLVING FOR x (t)

$$\frac{1}{2}\langle x(t)^2\rangle \frac{dk}{dt} = \frac{1}{2}\frac{F_0^2}{4\Omega^2 m^2}\frac{1}{\left(\frac{\gamma}{2m}\right)^2 + (\Omega - \omega)^2}\frac{dk}{dt}$$

Resonant frequency of the oscillator

$$+\frac{4F_0^2}{[2\Omega m]^2}\frac{\frac{\gamma}{2m}(\Omega - \omega)}{\left[\left(\frac{\gamma}{2m}\right) + (\Omega - \omega)^2\right]^3}\left(\frac{dk}{dt}\right)^2$$

A velocity dependent force

- ENERGY IS FED INTO THE SYSTEM IF $\omega < \Omega$
- ENERGY IS EXTRACTED FROM THE SYSTEM IF $\omega < \Omega$
- J. RUDNICK AND M. BARMATZ
  - VERIFIED BY EXPLICIT CALCULATION THAT THIS APPROACH HOLDS FOR SMALL SPHERES IN A LEVITATING OR POSITIONING CHAMBER

FIGURE 6

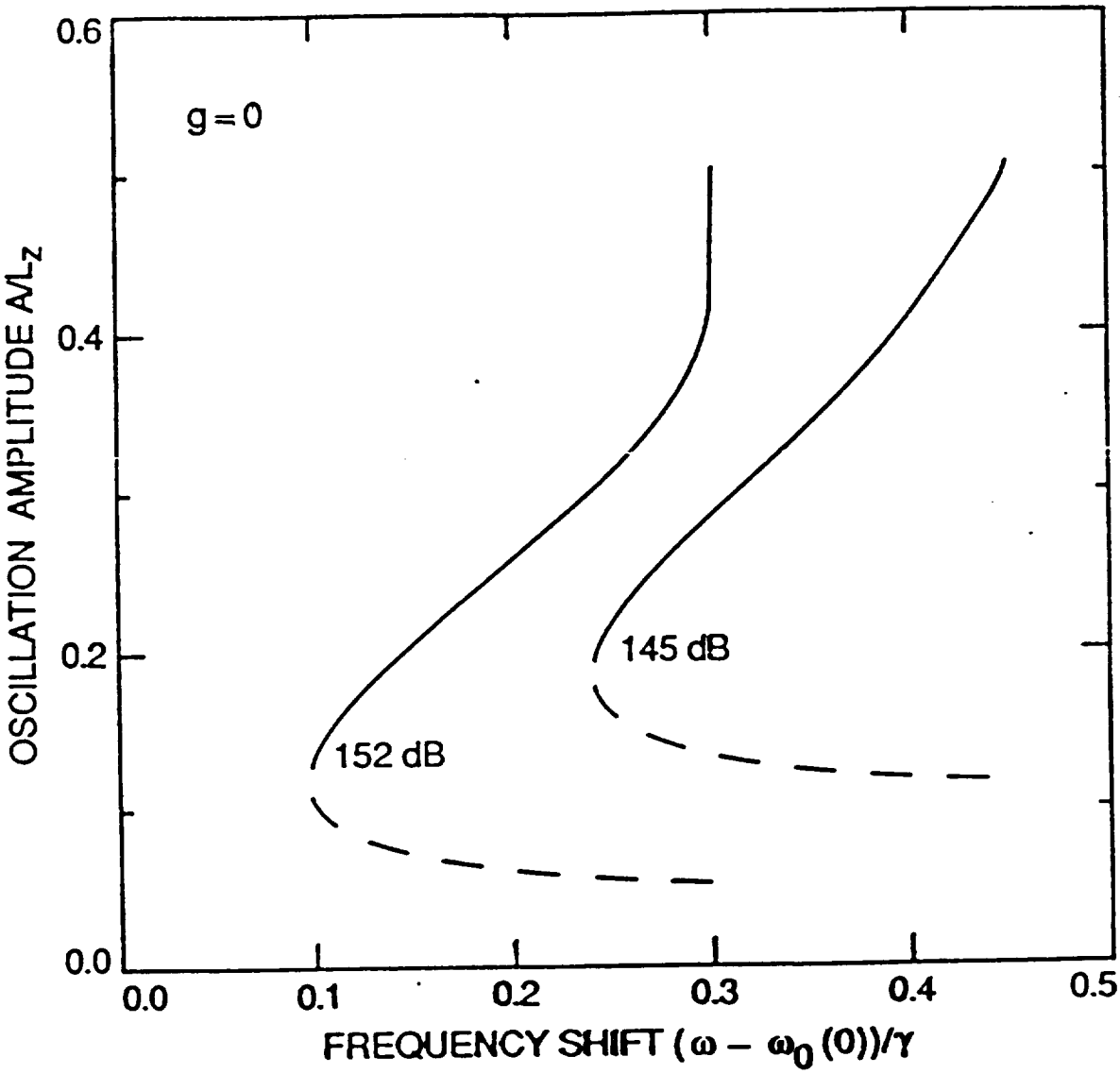

g = 0
152 dB
145 dB
OSCILLATION AMPLITUDE A/L_z
FREQUENCY SHIFT (ω − ω_0 (0))/γ
0.0
0.2
0.4
0.6
0.1
0.3
0.5

FIGURE 7

- UNANSWERED QUESTIONS:
  - DOES THE APPROACH OF GARRETT AND BARMATZ HOLD FOR AN ARBITRARILY SHAPED SAMPLE OF ARBITRARY SIZE?
  - CAN ONE PRODUCE A GENERALIZED PRINCIPLE LIKE THE ONE FOLLOWING FROM BOLTZMANN-EHRENFEST ADIABATIC INVARIANCE TO DESCRIBE OSCILLATIONAL, ROTATIONAL, AND DISTORTIONAL INSTABILITIES FOR LEVITATED OBJECTS?
  - WHAT HAPPENS WHEN THERE ARE TWO (OR MORE) DEGENERATE MODES
    - TRIPLE AXIS LEVITATOR
    - SOURCE OF ROTATIONAL CONTROL
    - ANTI ADIABATICITY

FIGURE 8

- MOTION OF A LEVITATED (OR POSITIONED) OBJECT
  - FIXED POINT (STABLE LEVITATION)
  - LIMIT CYCLE (STABILIZED OSCILLATION)
  - CHAOS (???)
    INTERPLAY BETWEEN MOTION OF A COMPACT OBJECT AND THE MODE(S) THAT SUPPORT IT

FIGURE 9 WHY WORK IN SPACE

- INTENSITY OF POSITIONING FIELD CAN BE REDUCED.
  155-160dB THE MINIMUM REQUIRED AT GROUND LEVEL. UNDESIRABLE EFFECTS ARE ELIMINATED (I.E., ROTATIONAL INSTABILITIES).

- ENVIRONMENT CAN BE MORE FULLY CONTROLLED (LESS DENSE MEDIUM - LESS DRAG)

- FULL SYMMETRY OF THE CAVITY CAN BE EXPLOITED (OBJECTS ARE POSITIONED AT CENTER)

N91-21377

A HIGH TEMPERATURE THERMAL DIFFUSIVITY DETERMINATION PROCEDURE FOR SOLIDS AND LIQUIDS

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A new method for measuring the thermal diffusivity of materials at high temperatures is presented. The method is applicable to solids on earth, and liquids in the reduced gravity environment of space. It is especially suited to levitated liquid metals at elevated temperatures, where thermal diffusivity data is not available. The method is applied in two parts, such that lumped analysis is valid in the first part, and Fourier's law of conduction in the second. In both parts, the spherical specimen is assumed to have been heated to a desired temperature, and cooled. An inverse conduction problem is then formulated, and solved using Laplace transformation techniques. Using this solution, the sample sizes, and experimentally obtained surface temperature history, the thermal diffusivity is determined by minimizing a function that satisfies the heat balance at the surface. Minimization is performed using a modified quasilinearization alogrithm. The method is demonstrated using theoretical cooling curves for three materials, nickel, niobium, and palladium, (for Nr=4 and 10), and is shown to be accurate. Accuracy is very sensitive to error in the temperature data, and increases with better curve fits to the temperature data. An error analysis is also performed, and the effect of errors in the various parameters on the evaluated thermal diffusivity is determined. An experimental study for solids on earth is suggested, before development for implementation in space.

* Professor

+ Graduate Students

REPORTS OF THE SPLINTER SESSIONS CHAIRMEN

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY
IMPORTANT ISSUES FOR RESOLUTION

1. WHAT ARE THE PRIMARY SCIENTIFIC JUSTIFICATIONS FOR CONDUCTING CONTAINERLESS EXPERIMENTS IN MICROGRAVITY?

2. GIVEN THE AVAILABLE FLIGHT EQUIPMENT (SUCH AS DPM AND TEMPUS), WHAT ARE THE MOST IMPORTANT MODIFICATIONS NEEDED TO ENHANCE THESE INSTRUMENTS' CAPABILITIES FOR NEAR-TERM FLIGHT EXPERIMENTS?

3. WHAT CONTAINERLESS FLIGHT INSTRUMENTS SHOULD NASA DEVELOP ACCORDING TO THE SCIENTIFIC JUSTIFICATIONS?

MATERIALS PROCESSING

Scientific Justifications for Containerless Processing:

* Minimize reactions.
* No heterogeneous nucleation due to crucible.
* Clean melt. Self-cleaning of some metals.
* Higher temperatures.
* Avoid vibration effects / controlled "dynamic" effects.

-----> Undercooled melts
  refine structure
  suppress segregation
  "new" phases - metastability, novel glasses
  controlled phase selection.

* Surface modification and control.
* Surface phenomena related to crystallization.
* Phase separation studies.
* Melt purification - via gas phase.
* Simplification of process-modelling experiments (thermophysical property measurements).

-----> Nucleation and growth kinetics
  microstructure evolution and scaling laws.

* Crystal growth from supersaturated solutions, e.g., protein crystals.
* Novel processing techniques, melt "shaping."

Conclusion -
Relevance of Microgravity to Containerless Processing

In μg, the material will not always be contained due to the nature of the environment. Hence, containerless processing is inherent in space processing.

+ Many single effects which can be achieved on Earth are achieved simultaneously.

* Larger samples -
  size-related effects and more accurate measurements.
* Longer times.
* Reduced convection
  - forced
  - buoyancy.
* Reduced sedimentation.
* Processing high-temperature conducting and non-conducting liquids
* Decoupling of heating/positioning in e.m.
* Vacuum/infinite pumping speed.
* Continuous, real-time, non-contact diagnostics.
* Controlled environments.

Recommendations:

CP in μg offers unique opportunities to understand and control structure and properties in materials.

It is recommended that high-quality scientific experiments be carried out in low gravity. This will lead to an increase in the understanding of fundamental materials behavior.

What are desirable modifications?

* Fast and accurate temperature measurement.
* Controlled environment.
* Monitoring of environment (residual gas analyzer).
* High-speed image acquisition.
* Isothermality.
* Large number of different samples/cycles.
* Controlled heating/cooling rates.
* Protection of equipment from evaporation and condensation.
* Clear sample views - diagnostic access.
* Sample stability - controlled shape, position and rotation.
* Flexibility to accommodate a variety of samples.
* Telescience
  - remote operation - PI interactive
  - real-time data transmission.
* Capability to treat multiple specimens simultaneously.

What instruments should be developed ?

* Maintain ground-based support for development/evaluation of equipment.
* Facility for processing high-temperature (> 2000 K) non-conducting liquid samples.
* Facility for processing high-temperature (ca. 3000 K) conducting liquid samples - refractory metals, alloys, semiconductors.
* Facility with capability for simultaneous multi-specimen processing.
* Solution/melt crystal growth facility.
* Facility for net-shaped processing.
* Thermophysical property measurement.

Report of the "Thermophysical Properties and Very High Temperature" Splinter Group

January 17–19, 1990

Panel Members

R. Hauge
W. Hofmeister
W. Johnson
A. Miller
P. Nordine

This report responds to three questions put forward at the workshop on "Containerless Experimentation in Microgravity" held on Jan. 17-19, 1990 at the Pasadena Hilton Hotel. The report is divided into three sections dealing with each of the respective questions. In addition, a final section containing some general remarks and overall recommendations is included.

I. Question - What are the primary science justifications for conducting containerless experiments in microgravity?

A. Findings

There are broad classes of high-temperature materials with great technological and scientific importance. To exploit these materials, their properties must be well understood. It is generally accepted that thermophysical and thermochemical property measurements at high temperature (from 1500 to 4000°C) must necessarily be carried out in a containerless environment. A list of the properties that have broad scientific and technological importance is given in Table I. Accurate measurements of these properties are an essential part of our efforts to understand the fundamental thermodynamic functions and physical properties of liquids and solids at elevated temperatures. Knowledge of these properties also forms the basis for determining the validity of theoretical models and the application of these models to actual material process design. In some instances, such data can be directly transferred to improve existing terrestrial processes.

In recent years, earth-based containerless processing has progressed substantially. This has helped to define its limitations. The key problem areas in ground-based containerless property determination are

- Gravity-induced sample asymmetry.
- Gravity-induced convection.
- Dynamic shape changes and internal fluid flows arising from the large forces necessary to levitate samples in earth gravity.

Earth-based, low-gravity experiments have limited duration that often makes it difficult to achieve the desired sample symmetry and quiescence. Furthermore, sufficient time is frequently not available to carry out particular types of property measurements.

B. Conclusion

The progress in earth-based, containerless processing has provided a strong justification for the extension of these scientific studies to space and for continuation and further development of ground-based containers-processing activities themselves.

C. Recommendation

This scientific advancement will only be realized through a strong commitment by NASA to provide advanced facilities for containerless studies of materials at high temperatures.

II. Question - Given the available flight equipment (such as DPM, TEMPUS, etc.), what are the most important modifications needed to enhance these instruments' capabilities for near-term flight experiments?

A. Findings

1. Non-contact temperature measurements in TEMPUS and DPM are currently inadequate. Silicon-based pyrometry at the standard 650 nm wavelength is a necessity in high-temperature facilities.

2. Accurate measurement of the temperature and wavelength dependence of the emissivity are required for true temperature determination.

3. The temperature range of the DPM is less than 800°C. This is very inadequate. The temperature range of TEMPUS (< 2500°C) should be expanded to accommodate very high melting-point materials.

4. Sample viewing in TEMPUS is inadequate.

5. Fast-quenching capabilities are needed.

6. Various controlled gas environments are required for many measurements, such as gas jets, for inducing rotation and providing forced-convective flows.

7. The ability to measure total radiance is necessary for such measurements as heat capacity and thermal conductivity.

8. Accurate time resolved shape measurements (better that 5 μm spatial resolution, and submillisecond time resolution) are required.

9. Ability to induce controlled-droplet vibrations, oscillations, and rotations is required.

10. A method of sample heating that does not disturb drop symmetry and quiescence is required. Beam heating with a CO_2 laser, microwave source, or electron beam are examples of such heating techniques.

11. Fluid flow visualization capability should be investigated.

12. Spatial thermal mapping capability is very desirable.

13. Eddy current measurement techniques should be developed.

B. Conclusions and Recommendations

The above-mentioned deficiencies limit the usefulness of existing facilities for carrying out the variety of property measurements listed in Table I.

III. Question - What containerless flight instruments should NASA develop according to the scientific justifications?

A. Findings

1. Development of a plane-polarized ellipsometer is necessary for true temperature measurements.

2. A mass spectrometer for detailed analysis of residual gas (in high vacuum) and accurate analysis of controlled gas atmospheres and volatile species would be very desirable.

3. The relative unavailability of orbiter flight time restricts the development of this program. Safety restrictions, costs, and infrequency of manned flights create further difficulties. The development of a relatively inexpensive, shuttle-independent and retrievable, free-flier (low orbit) capability would enhance the evolution of the overall program.

4. A new hybrid facility combining electromagnetic positioning with beam-heating methods that do not disturb the quiescence and symmetry to the extent greater than that dictated by positioning alone would be desirable.

B. Conclusions and Recommendations

The above-mentioned findings should be implemented with the development of a new facility (referred to in item 4).

IV. General Remarks and Overall Recommendations

A. The success of the "Containerless Processing in Microgravity" program is dependent on close interaction among the scientific user community, NASA staff, and hardware developers. In the past, the absence of such interactions has often led to less than optimum facility planning.

B. We strongly encourage the continued development of earth-based, electromagnetic positioning facilities, sample heating techniques, noncontact temperature measurement techniques, and property measurement facilities. Furthermore, we strongly recommend continued ground-based acquisition of a data base for the properties listed in Table I. Space research should be viewed as complementary to ground-based research.

C. Methods for beam heating for use with nonconducting materials (which cannot be electromagnetically levitated) and with conducting materials for which electromagnetic heating creates unacceptable disturbances should be studied in ground-based laboratories such as drop tubes.

TABLE I

LIST OF IMPORTANT THERMOPHYSICAL AND THERMOCHEMICAL PROPERTIES

THERMODYNAMIC QUANTITIES

p, density measurements
C_p, heat capacity in liquids and solids (superheated and undercooled)
$\hat{I}C_p$, difference in liquid/solid heat capacities
$\hat{I}H$, heat of melting and other phase transformations
T_m, melting points and other critical temperatures
∫, thermal expansion coefficients
ß, surface tension of melts
$ß_{sl}$, solid/liquid interfacial free energy (indirectly)

TRANSPORT PROPERTIES

K, thermal diffusivity
K, thermal conductivity (need C_p, k, and ®)
D, atomic diffusion constants
$®_e$, electrical resistivity/conductivity measurements
n, liquid viscosity/fluidity measurements

OPTICAL PROPERTIES

´(¬,T), spectral emissivity as a function of temperature and wavelength
$´_h$, total hemispherical emissivity
n/k, index of refraction (real and imaginary parts)

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY WORKSHOP

19 January 1990

FLUID PHYSICS AND INTERFACIAL PHENOMENA SPLINTER SESSIONS REPORT

A limited assessment of the advantages and opportunities offered by containerless experimentation in microgravity was carried out during the workshop splinter session. Areas of scientific interest in the Physics of Fluids were examined in the framework of three basic questions: the identification of current potentially fruitful areas of research, the specific consideration of the scientific capabilities of currently planned flight equipment, and the evaluation of future instrumentation needs for space experiments.

GENERAL OBSERVATIONS ON CONTAINERLESS EXPERIMENTS IN MICROGRAVITY:

- TOTALLY FREE SURFACES AVAILABLE FOR LONG-DURATION INVESTIGATIONS
- ELIMINATION OF BOUNDARY EFFECTS
- REDUCTION IN BUOYANCY AND SEDIMENTATION
- LARGER SAMPLE SIZES OBTAINABLE (APPROXIMATELY 1–5 cm RANGE)
- HIGHER Q OBTAINABLE FOR RESONANT PHENOMENA
- ABSENCE OF ORDINARY THERMAL AND SOLUTAL CONVECTION
- LIQUID SAMPLE PERFECT-SPHERICITY POTENTIALLY ACHIEVABLE
- WIDER PARAMETER SPACE (PHASES) AVAILABLE FOR MEASUREMENTS
- MORE ACCURATE MEASUREMENTS POTENTIALLY POSSIBLE
- DECOUPLING OF INTERFERING LEVITATION EFFECTS POSSIBLE
- POTENTIAL FOR OPTIMAL VIBRATION ISOLATION

In general, it might be observed that relatively lower temperature experiments will allow easier but tighter control of the experimental environment by using available state-of-the-art, quantitative diagnostics instrumentation. This advantage should be used to obtain rigorous experi-

mental results on near-term space flights. In addition to offering the potential for significant contribution to the advancement of existing scientific and technological problems, containerless experimentation on fluids at moderate temperature also allows the possibility of modelling phenomena occurring at more extreme thermal and environmental conditions.

1. SCIENTIFIC JUSTIFICATIONS

- **FREE SURFACE PHENOMENA**

- ***LINEAR AND NONLINEAR CAPILLARY WAVES DYNAMICS***

(Wave turbulence, chaotic phenomena, statistical mechanical theory of surface tension, nonlinear dynamics)

- ***RHEOLOGY OF SURFACTANT-LADEN SURFACES***

(Verification of the theories of surface viscosity and elasticity for gas-liquid and liquid-liquid interfaces and spherical samples, non-Newtonian liquids)

- ***THERMOCAPILLARY AND ELECTROCAPILLARY PHENOMENA (MARANGONI CONVECTION AND CHARGE EFFECTS)***

(Simple free spherical, initially isothermal simple drops and liquid shells)

- ***LARGE BUBBLE DYNAMICS AND EQUILIBRIUM SHAPE***

(Nonlinear dynamics cavitation problems, nonthermocapillarity-driven bubble manipulation)

- ***AEROSOL BEHAVIOR, AND GAS-PARTICLE DISPERSION DIFFUSIONAL INTERACTIONS***

(Atmospheric, planetary, and intersteller media physics)

- **GRAVITY-FREE TRANSPORT PHENOMENA**

- ***LIQUID-GAS AND LIQUID-LIQUID MASS TRANSPORT PARAMETERS MEASUREMENT***

(Coupling to Marangoni convection, Benard cells stability)

- ***MACROSCOPIC SPHERICAL DROPLET VAPORIZATION/COMBUSTION***

(Supercritical processes in combustion)

- **MEASUREMENT OF LIQUID PROPERTIES**

- ***SURFACE PROPERTIES***

(Long-duration experiments on exposed spherically free surfaces in controlled environments)

- ***BULK THERMODYNAMIC PROPERTIES FOR PURIFIED SAMPLES***

- ***OPTICAL PROPERTIES OF SPHERICAL AND NON-SPHERICAL DROPLETS***

(Remote sensing, visible wavelength and microwave)

- **PHASE TRANSITIONS AND METASTABLE LIQUID PHASES**

- ***SUPERCOOLED AND SUPERHEATED LIQUID BEHAVIOR***

(Thermodynamic, mechanical, and optical properties measurements)

- ***SOLIDIFICATION PROCESS IN FREE MELTS***

(Surface crystallization, mass and heat transport)

- ***HETEROGENEOUS NUCLEATION EFFECTS***

(Bulk and surface nucleation, "dynamic" effects)

- ***CRITICAL AND SUPERCRITICAL PHENOMENA, COOPERATIVE EFFECTS***

- ***CRYSTAL GROWTH FROM SOLUTION***

(Slow growth processes and fluid transport mechanism around growing crystals, optical diagnostics)

CONCLUSION:

Well controlled quantitative experimental investigations may be carried out with care in well controlled systems with well calibrated, up-to-date instrumentation adapted to the space laboratory environment. Demonstration of reliable measurements must be carried out in space using proven instruments.

RECOMMENDATION:

Areas that are most likely to yield reliable quantitative results using existing modified flight instruments' capabilities for near-term flights should be emphasized. Other fields of research requiring more detailed definition should be designated for future investigations. Periodical review of the

progress of ground-based tasks progress and of the advances in relevant disciplines should be carried out in order to reassess the status of flight experiments.

2. NEAR-TERM FLIGHT INSTRUMENTS' CAPABILITIES MODIFICATIONS

DROP PHYSICS MODULE

FINDINGS:

The current facility is strictly dedicated to present selected flight Principal Investigators and is designed around drop dynamics experiments. Additional investigations will require additions in scientific capabilities.

CONCLUSION:

The current apparatus must be considered a starting point for further enhancements. Significant additions to these basic capabilities must be made if this facility is to carry out investigations that are identified in the previous section.

RECOMMENDATION:

- MAKE BOTH CHAMBERS REPLACEABLE FOR EASIER CONTAMINATION CONTROL

- TIGHTEN PRESENT ENVIRONMENT CONTROLS
    - CHAMBER AND SAMPLE TEMPERATURE MEASUREMENTS
    - POSSIBILITY OF CONTROLLING SAMPLE TEMPERATURE DIRECTLY
    - FLEXIBILITY IN CONTROL OF GAS ENVIRONMENT
    - PRESSURIZATION CAPABILITY
    - TIGHTER HUMIDITY CONTROL AND MEASUREMENT

- ADD CAPABILITY FOR INTRODUCING ELECTRIC FIELD

- ADD HIGH-SPEED VIDEO-IMAGING CAPABILITY

- ADD INTEGRATED OPTICAL DIAGNOSTIC CAPABILITIES:
  - LASER SCATTERING
  - SCHLIEREN PHOTOGRAPHY
  - LASER PSEUDO-EXTINCTION TECHNIQUE
  - CAPABILITY FOR INSERTING A LIQUID CHAMBER
  - LOW TEMPERATURE CAPABILITY (-40 °C)
  - PURGING, BAKING, AND ENVIRONMENT COMPOSITION-MONITORING INSTRUMENT

3. OTHER CONTAINERLESS FLIGHT INSTRUMENTS

FINDINGS:

DPM appears to satisfy currently identified temperature range for fluid physics investigations (30 to 800 to 1000 °C).

CONCLUSION:

Acoustic techniques appear sufficient for the majority of sample manipulation requirements for fluid physics experiments. Desired operation under vacuum may be accomodated by an Acoustic-Electrostatic hybrid system having the capability for independent and complementary operations.

RECOMMENDATION:

To implement the addition of the suggested diagnostics instrumentation, and to develop an Acoustic-Electrostatic hybrid system.

ADDITIONAL COMMENT:

A DESIRABLE CAPABILITY TO BE ADDED: AN ORBIT SAMPLE CHARACTERIZATION WITH RADIOGRAPHIC METHODS (X-RAYS, GAMMA RAYS) FOR SPACE STATION OPERATION.

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY WORKSHOP

DESCRIPTION OF EXPERIMENTAL FACILITIES

AND SCIENCE CAPABILITIES

17-19 JANUARY, 1990

JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

CONTAINERLESS EXPERIMENTATION FACILITIES

This document describes the Containerless Experimentation Facilities and their associated science capabilities. The devices and capabilities described are currently or potentially available for conducting materials sciences research in microgravity aboard the NASA Spacelab and Space Station. In addition, potential and existing facilities for ground-based and low-gravity experimentation as a precursor to microgravity experiments are also described.

Development priorities for future facilities will be based on the science requirements identified during the workshop in January 1990, and responses to subsequent related Announcements of Opportunity (AO's) and NASA Research Announcements (NRA's).

MICROGRAVITY FACILITIES

The Drop Physics Module (DPM) Experimental Facility, currently under development at JPL, is the containerless processing facility scheduled for the United States Microgravity Laboratory (USML) series of flights. The other apparatus devoted to containerless processing and manifested for the second International Microgravity Laboratory (IML-2) flight is TEMPUS, an electromagnetic positioning apparatus developed by DLR in Europe and offered to American, as well as European, investigators. In the current NASA plans, the USML-1 (1992) and USML-2 (1994) flights will carry a DPM facility equipped with the near-ambient temperature acoustic positioner and, in a second bay, the dual-temperature zone acoustic positioner.

For USML-3 (1997) and USML-4 (2000), current planning is to replace one or both of these modules with one or more of either the currently available flight devices or apparatuses under development. The final choice will rest on the science requirements identified during science workshops and the survey of the scientific and industrial communities. The Space Station Modular Containerless Processing Facility (MCPF) will evolve from the experience gained in the Spacelab flights.

GROUND-BASED FACILITIES

Ground-based facilities will be sponsored by NASA primarily to support initial stages of experimentation leading to microgravity work. The development of ground-based full-gravity and low-gravity (KC-135 and Sounding Rocket) facilities will depend on the definition of science requirements by experimenters and potential Principal Investigators who will control the evolution into valid microgravity experiments. Ground-based experiments can provide initial results with quicker turnaround times at a much lower cost than fully qualified manned Spacelab experiments.

NONCONTACT TEMPERATURE MEASUREMENT

A key technology area for high temperature containerless processing is the development of accurate thermodynamic temperature measurement without the need for contact with the specimen. This involves continuous dynamic measurement of both emissivity and radiance of the specimen, in both liquid and solid states. A developing technology, the Division of Amplitude Polarimetric Pyrometer (DAPP), is described. This could be adapted to any of the high-temperature facilities described above. Other devices, commercially available or prototype units adaptable to containerless experimentation, are also described in this document.

CONTAINERLESS EXPERIMENTATION HARDWARE STATUS

EQUIPMENT	EXISTING OR IN CONSTRUCTION	PROTOTYPE	CONCEPTUAL DESIGN
I. AMBIENT- & MEDIUM-TEMPERATURE MICROGRAVITY LEVITATORS			
A) NEAR-AMBIENT ACOUSTIC LEVITATOR (DPM SCHEDULED FOR FLIGHT)	X		
B) DUAL-ZONE ACOUSTIC LEVITATOR (DPM) (SCHEDULED FOR FLIGHT)	X		
C) ACES (FLOWN DEVICE)	X		
D) DDM (FLOWN DEVICE)	X		
E) 3AAL (FLOWN DEVICE)	X		
F) ELECTROSTATIC-ACOUSTIC HYBRID		X	
II. HIGH-TEMPERATURE CONTAINERLESS PROCESSING MODULE (HTCPM) MICROGRAVITY MODULE DPM/MCPF			
A) ELECTROMAGNETIC LEVITATOR (TEMPUS) (SCHEDULED FOR FLIGHT)	X		
B) ACOUSTIC LEVITATION FURNACE (HOT WALL)		X	
C) HIGH-TEMPERATURE ACOUSTIC LEVITATOR (BEAM-HEATED)		X	
D) MODULAR ELECTROMAGNETIC LEVITATOR			X
E) STABILIZED ELECTROMAGNETIC LEVITATOR			X
F) ELECTROSTATIC LEVITATOR			X
G) GAS FILM LEVITATOR (ESA SPONSOR)		X	
H) SAAL (FLOWN DEVICE)	X		
I) EML (FLOWN DEVICE)	X		

EQUIPMENT	EXISTING OR IN CONSTRUCTION	PROTOTYPE	CONCEPTUAL DESIGN
III. HIGH-TEMPERATURE GROUND-BASED LEVITATORS FOR PRECURSOR EXPERIMENTS			
A) HIGH-PRESSURE ACOUSTIC LEVITATOR (HOT OR COLD WALL)		X	X
B) ELECTROMAGNETIC LEVITATOR (BEAM-HEATED)	X		
C) ACOUSTIC LEVITATOR BEAM-HEATED FOR KC-135 OR SOUNDING ROCKETS	X	X	
D) ELECTROSTATIC LEVITATORS			X
IV. NONCONTACT TEMPERATURE MEASUREMENT EQUIPMENT			
A) LASER PYROMETER	X		
B) MULTI-COLOR IMAGING PYROMETER	X		
C) POLARIMETRIC PYROMETER (DAPP)			X
D) NEAR-AMBIENT TEMPERATURE IMAGER	X		

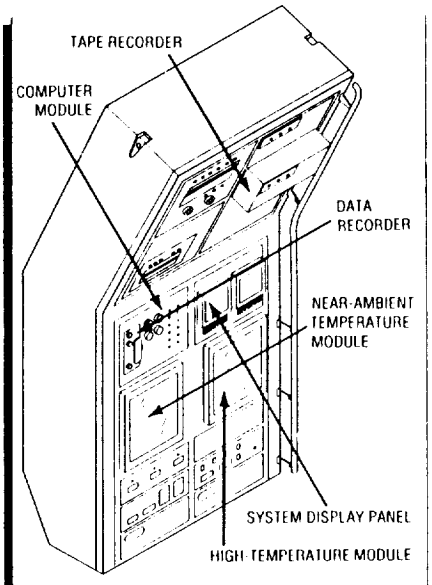
EXISTING MICROGRAVITY EQUIPMENT

CURRENT AND PAST EXPERIMENTAL FACILITIES

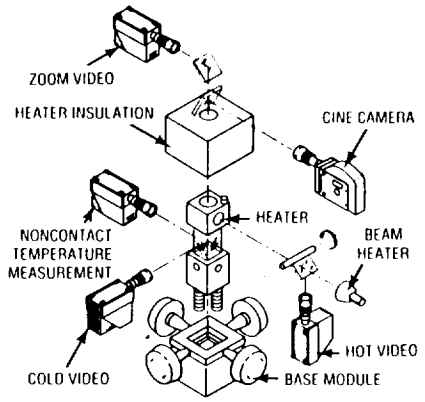
AVAILABLE DEVICES:

DROP PHYSICS MODULE
TEMPUS

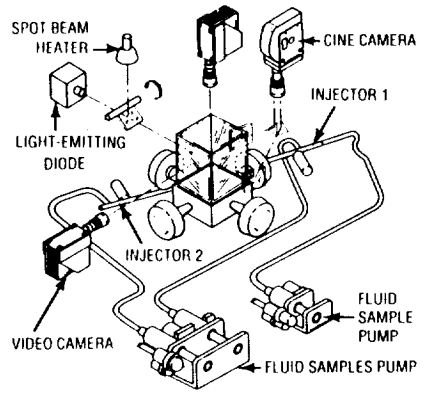
Drop Physics Module (DPM)



Drop Physics Module



High-Temperature Dual-Zone Chamber



Near-Ambient Chamber

Importance

The DPM will be a multipurpose acoustic positioning device, designed to accommodate ambient- and elevated-temperature (around 1000 °C) experiments. Isolation from containers or other mechanical holding materials enhances the ability to undercool a sample, allows the study of highly reactive materials, minimizes specimen contamination, and reveals the subtle interaction between capillary and thermo-inertial forces. The DPM is the first of a series of modules being developed for an integrated containerless processing facility for the space station.

Method

Two acoustic positioning chamber bays will provide the capability to carry out independent near-ambient and high-temperature investigations. Several sealed chambers will accommodate different processing environments. Experimenters will be able to control temperature, hydrostatic pressure, composition of the host gas, humidity, lighting, and acoustic positioning force. Liquid samples will be injected into the near-ambient chamber and accurately positioned with acoustic forces. Sample rotation, positional and shape oscillation, and translation within the processing chamber will be induced when necessary. Liquid behavior and shape will be recorded through video and cinefilm camera systems. Solid samples will be deployed into the cool zone of the dual-temperature experiment cell, translated into the hot zone for processing, and moved back into the cool zone for faster cooling. High sample temperatures will be attained by both resistance and beam heating.

Carrier

Spacelab

Sample Summary

- Samples: Liquid drops, liquid shells, solid samples
- Diameter: 0.5- to 2.7-cm drops (near-ambient chamber); 0.5- to 1-cm melts (high-temperature chamber)
- Temperature range: Ambient to 100°C (near-ambient chamber); ambient to 800°C (resistive furnace); above 800°C (beam heater)

Physical Characteristics

- Near-ambient chamber: 15.25 cm x 15.25 cm x 30 cm
- High-temperature cell: 6.35 cm x 6.35 cm x 30.5 cm
- The DPM fits inside a Spacelab double rack.

Operational Parameters

- Video imaging
  - 30 frames/sec RS-170 format with 1/60- and 1/500-sec shuttering
  - Resolution: 20 μm with 7.5-mm field of view (FOV) to 200 μm with 78-mm FOV
- Cinefilm imaging
  - 16-mm monochrome or color
  - Variable frame rate: 10 to 400 frames/sec
- Infrared (IR) thermal imaging
  - Spectral response: 0.4 to 5 μm
  - Frame rate: 10/sec (minimum)
- Thermocouple measurements
  - Fixed wall thermocouples: ± 1°C accuracy
  - Varying position probe: ± 5°C accuracy
- Acoustic drive
  - Carrier frequencies: 1 to 8 kHz
  - Force modulation: 1 to 30 Hz (minimum)
  - Sound pressure level: 130 to 155 dB (re 0.0002 μBar)
  - Torque: 1.0 dyne/cm (maximum) at ambient temperature
- Beam heater
  - Lamp color temperature: 2500 K
  - Power: 250 W with 1.25-cm beam diameter
- Peak power: 1.8 kW

Instrumentation

- Cathode ray tube and system parameter display panels
- Hydrostatic pressure, humidity, and temperature sensors
- Sheet lighting illumination
- Video and cinefilm camera systems
- IR thermal imager
- Acoustic microphones
- Process Control and Data Acquisition (PCDA) microprocessor

Data Acquisition

PCDA microprocessor controls all automatic functions and records engineering data. Video data are either recorded on the DPM high data rate recorder or on Spacelab/orbiter recorders or downlinked through the Spacelab video analog switch. Cinefilm data are recorded on 16-mm, 400- and 1200-ft magazines. Unused electrical interfaces for power and data acquisition ports will be made available for additional experiment-peculiar instruments.

Facility Integration

The DPM is integrated in a Spacelab double rack, which will accommodate some special stowage items. Other special hardware items will be stored in Spacelab stowage.

- Special Interface Requirements: Avionic air cooling is required in addition to the standard power and mechanical interfaces.
- Integration Options: None.

Additional Notes

The DPM design has been influenced by those of previous flight instruments: the Drop Dynamics Module, the Three-Axis Acoustic Levitator, and the Acoustic Containerless Experiment System.

Development Center

NASA/Jet Propulsion Laboratory
Modular Containerless Processing Facility Project
4800 Oak Grove Drive
Pasadena, CA 91109
(818) 354-5738

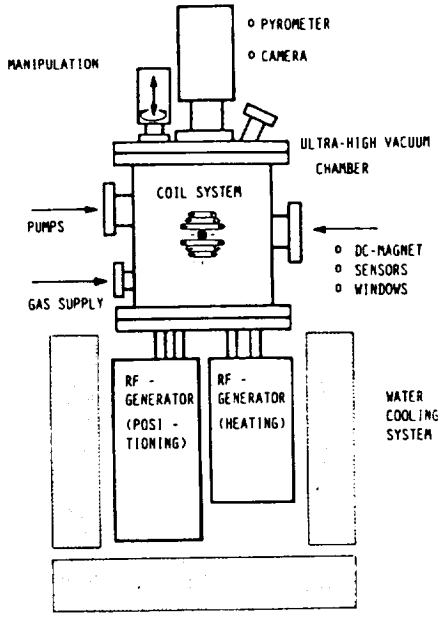
TEMPUS
ELECTROMAGNETIC CONTAINERLESS PROCESSING FACILITY

Tempus is a German Spacelab facility for containerless processing of metallic samples using electromagnetic positioning and inductive heating under ultra-high-vacuum conditions.

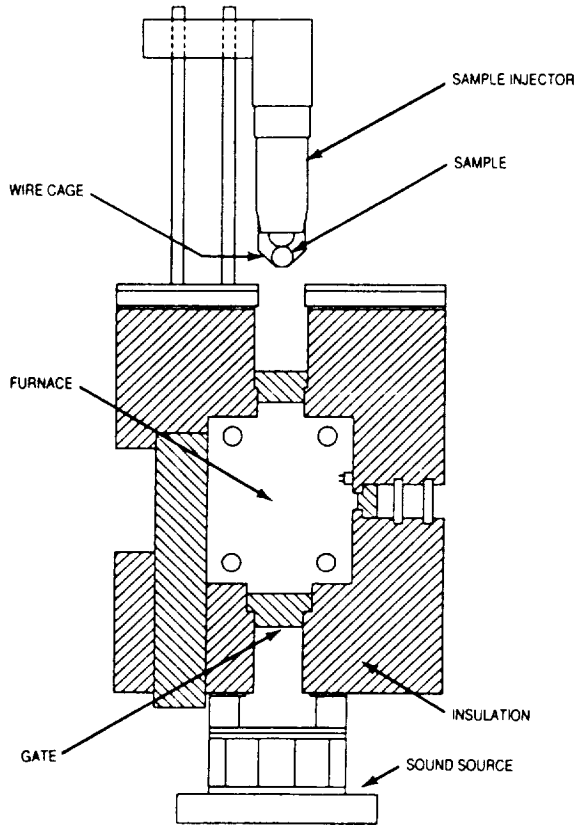
Heating and positioning are generated by two independent RF systems operating at 400 kHz in a dipole mode (for heating) and at 100 kHz (quadrupole mode for positioning). Sample temperature up to 2500 C can be reached by the use of a free oscillating RF generator having high efficiency. The vacuum chamber is monitored by a video camera, and temperature measurements can be performed by a two-color pyrometer. High speed measurements to observe the recalescence peak of undercooled samples can be made.

TECHNICAL SPECIFICATIONS

Sample Diameter	up to 10 mm
Maximum power consumption	1700 W
Combined RF power	1500 W
Sample temperature	up to 2500 C
Number of stored samples	18
Vacuum range	10^{-9} Torr
Temperature measurement range	300 to 2400 C
Observation:	Visual or CCD TV camera



Single Axis Acoustic Levitator (SAAL)



Single Axis Acoustic Levitator

Importance

Containerless processing may make possible the preparation of ultrapure glasses used in optical and electrical applications. The chemical composition of some glasses requires processing at temperatures of 2,500 to 3,000 °C and greater; at these temperatures, no unreactive containers are available. On Earth, melts react with their containment crucibles causing impurities to form in the glass; in microgravity, containerless melting may eliminate this contamination. Additionally, acoustic processing at high temperatures on Earth is impossible because the sound waves cannot overcome gravity. The SAAL apparatus was the first space instrument to levitate, melt (at temperatures up to 1,500 °C), and resolidify glass samples acoustically. Experiments conducted in the SAAL have increased understanding of the role of nucleation in glass formation.

Method

Eight glass samples can be processed sequentially and automatically in the SAAL. Each sample is injected into the furnace cavity from a storage carousel attached to the hot-wall furnace. The sample is positioned without wall contact in the furnace cavity by acoustic energy nodes, which are formed when incident sound waves interfere with reflected sound waves in the furnace. During the melt phase, four silicon/carbide heating rods surround the levitated sample. After this phase, power is switched off the heating rods, allowing radiative cooling and solidification of the sample. Upon completion of the cooling phase, the solidified sample is retrieved by a retracting wire cage, and another sample is automatically inserted. A quartz window serves as a port for photographing the specimen during the experiment process.

Carrier

Materials Science Laboratory (MSL)

Sample Summary

- Capacity/flight: 8 samples/flight
- Sample translation: Caged injector
- Spherical diameter: 4 to 10 mm

Physical Characteristics

- Furnace height: 93.50 cm
- Furnace diameter: 40.50 cm
- Furnace weight: 81.60 kg
- Injector cage: 2.50 cm x 2.50 cm x 2.85 cm
- Processing chamber: 10.20 cm x 10.20 cm x 11.40 cm

Operational Parameters

- Power: 3,100 W (peak); 2,600 W (average)
- Voltage: 32 ±4 Vdc
- Operating temperature: Up to 1,600 °C
- Pressure: 1 atm
- Atmosphere: Air or inert gas
- Levitator frequency: 15 kHz
- Cooling rate: 200 °C/min (sample dependent)

Instrumentation

- Pyrometer
- Thermocouples (2)
- Movie camera

Data Acquisition

Sample and furnace temperatures are recorded by the pyrometer and thermocouples, and a movie camera provides a photographic record of the processing during melts and solidifications. Data management is provided by the MSL Data Acquisition and Control system.

Facility Integration

The SAAL apparatus is housed in an Experiment Apparatus Container and is limited to use on the MSL carrier in the Shuttle orbiter payload bay.

- Special Interface Requirements: The SAAL requires electrical power, data management, cooling fluid, and air pressure interfaces.
- Integration Options: None.

Additional Notes

The SAAL flew on the Materials Experiment Assembly (MEA-A1) as part of the OSTA-2 payload/STS-7, June 1983, and on MEA-A2 as part of the Spacelab D-1 mission, October 1985.

Development Center

NASA/Marshall Space Flight Center
Microgravity Projects/JA61
Marshall Space Flight Center, AL 35812
(205) 544-1979

ACOUSTIC CONTAINERLESS EXPERIMENT SYSTEM

EXPERIMENT CAPABILITY

The Acoustic Containerless Experiment System (ACES) is an unattended flight facility that melts and acoustically manipulates a single sample of glass, ceramic, or metal while it is acoustically centered within a resistance-heated rectangular furnace filled with an inert gas. The experiment is monitored by thermal sensors and a video-recording system that provide 120 minutes of image and date recording. Sample rotation, oscillation, and static deformation are available by means of acoustic forces. Front and back lighting can be selected for viewing sample surface features, or the silhouette of the sample.

OPERATIONAL PARAMETERS OF FLIGHT ON STS 41-B

Voltage	28v dc
Power	360 watts
Operating temperature	700 °C capability
Heating rate	10 °C/min
Cooling rate	4.5 °C/min
Furnace gas environment	Dry N_2 @ 14.7 psi
Video and data recording	120 minutes max.

SAMPLE PARAMETERS

Capacity	1 sample/flight
Size	1 cm diam. max.

PHYSICAL CHARACTERISTICS

Two similar Experiment Apparatus Containers (EAC) are used; one for the furnace system and one for the electronic control and data recording system.

Inside diameter	42.16 cm
Inside length	44.07 cm
Total furnace system weight	59.51 kg
Total electronic system weight	50.62 kg

INTEGRAL INSTRUMENTATION

Single axis video system. A micro-g accelerometer can be externally mounted.

DATA ACQUISITION CAPABILITIES

Sample shape and activity are recorded on videotape. Data for temperature, electrical and acoustic performance are recorded on audio portion of videotape.

FACILITY INTEGRATION OPTIONS

On the first flight, both canisters (EAC) were mounted in the forward locker array of the Orbiter on flight STS-41B.

DROP DYNAMICS MODULE (DDM)

EXPERIMENTAL CAPABILITY

This flight facility uses sound pressure within an acoustic chamber to constrain and manipulate liquid drops at room temperature in a manned microgravity environment. Typical sequences involve drop rotation and induced-shape oscillations and can be controlled by the operator or through pre-recorded programs.

OPERATIONAL PARAMETERS

Voltage	28v dc and 120v ac @ 400 Hz
Power	550 watts
Operating temperature	Room temperature
Rotation	Maximum torque at 155 dB SPL Nulling to less than 1 revolution in 5 minutes
Oscillation	Modulation frequency range is 0.5 to 10 Hz
Image resolution	0.02 cm (from film)
Frequency resolution	0.01 Hz for oscillation and rotation
Gas environment	Spacelab ambient air supply

SAMPLE PARAMETERS

Sample material	Water-glycerine mixtures for a selection of viscosities, and silicone oil for a low-surface tension fluid
Sample capacity	600 cc total of several mixtures
Sample viscosity range	1 to 500 cSt at ambient temperature
Sample size range	0.1 to 10 cc

PHYSICAL CHARACTERISTICS

Overall dimensions	The facility fills one double flight rack
Depth	120 cm
Width	110 cm
Height	330 cm
Weight	360 kg

INTEGRAL INSTRUMENTATION

16-mm camera with magazines of 400 ft and 1200 ft capacity. Frame rates can be 10,16, 24, 30, or 50 frames per second.

DATA ACQUISITION CAPABILITIES

Magnetic tape for engineering and science data. 16-mm film. External video for downlinking to ground-support team along with verbal communications. FACILITY INTEGRATION OPTIONS. This facility is housed in a double flight rack and mounted in Spacelab.

THREE AXIS ACOUSTIC LEVITATOR (3AAL)

EXPERIMENTAL CAPABILITY

This Three Axis Acoustic Levitator (3AAL) apparatus is a flight facility that permits a liquid specimen to be positioned and manipulated by a three-dimensional acoustic energy well. Manipulations include rotation, oscillation and drop fission. The oscillation and rotation can be used to stir the liquid as well as to center gas bubbles in the liquid drop. Consecutive samples can be injected for the fluid supply and then controlled while black-and-white video pictures are obtained at 30 frames per second. By means of a mirror system, on each frame there is recorded one direct view and two reflected views on orthogonal axes. Temperature, acoustic pressure, and acoustic drover power are also recorded. The apparatus permits the investigation of a variety of fluid-dynamic properties and bubble interactions using up to four different fluids or viscosities from four fluid syringes and one gas syringe reservoir. A light source can be programmed to focus on the sample to induce surface tension driven flow.

OPERATIONAL PARAMETERS

Voltage	28v dc
Power	126 watts
Acoustic pressure	145 dB typical
Disturbance acceleration tolerance	10-4 g
Estimated acoustic pressure (fundamental mode)	1 dyne/cm^2

SAMPLE PARAMETERS

Sample diameter	up to 2.5 cm
Sample volume	up to 8.0 cm^3
Maximum sample translation	3 cm

PHYSICAL CHARACTERISTICS

Apparatus length	97.5 cm
Apparatus diameter	38.74 cm
Apparatus weight	93.4 kg
Chamber size	11.5 x 11.5 x 12.7 cm

INTEGRAL INSTRUMENTATION

Temperature sensors and a black-and-white video system.

DATA ACQUISITION CAPABILITIES

Black-and-white videotape with a 120-minute limit due to tape length. Temperature data is recorded on the audio portion of the videotape. All data recording is retained within the Experiment apparatus Container.

FACILITY INTEGRATION OPTIONS

The apparatus is housed in an Experiment Apparatus Container and mounted under thermal shields on the Materials Science Laboratory (MSL-2) in the Orbiter payload bay. Thermal control utilizes a cold plate integral to the mounting on the MSL support structure.

FACILITIES UNDER DEVELOPMENT

POTENTIAL FUTURE MICROGRAVITY FLIGHT FACILITIES

HIGH-TEMPERATURE TETRAHEDRAL POSITIONERS

Tetrahedral positioner is a three-dimensional feedback-controlled positioner in which sample positioning, stabilization, and vibration isolation are achieved by exerting appropriate forces to a sample. Either electrodes or coils are positioned in a tetrahedral arrangement so that control forces (such as electrostatic, electrophoretic or electromagnetic) can be efficiently transmitted to the sample. Tetrahedral structure also leaves widely open space through which various diagnostic instruments (such as NCTM) or heating instruments (such as Xenon lamps or lasers) can be directed. These positioners are being developed for the high-temperature processing of various materials in the different environments. Depending on the physical properties of sample materials and required processing environments, these systems can divide these into three separate subsystems. However, common to all these subsystems are basic positioning algorithm, control units, heating systems, and diagnostic instruments. The basic principle of a tetrahedral positioner has been demonstrated both on ground-based laboratory and in the reduced gravity environment on board KC-135 aircraft.

A) HIGH-TEMPERATURE TETRAHEDRAL ELECTROSTATIC POSITIONER (HTESP) is being developed for the materials processing in high vacuum. Four spherical electrodes are arranged in a tetrahedral configuration and electrostatic forces are transmitted to a charged sample. Requirement on sample charges is ensured by an electron beam at the lower temperature region, and thermionic emission ensures the sample charge at the higher temperature region. Conductors, semiconductors, ceramics, and glasses can be processed in this positioner. Xenon arc lamp or laser can be used to heat the samples.

Projected Science Capabilities	
Max. control force	200 dynes
Positioning accuracy	50 microns
Vibration isolation	possible
Heating method	Xenon arc or laser beam heating
Sample temperature	up to 1700 °C
Cooling	radiation cooling
Vacuum	$< 10^{-7}$ torr
Sample size	1 cm dia.
Sample density	up to 18 g/cc

B) HIGH-TEMPERATURE TETRAHEDRAL ELECTROPHORETIC POSITIONER (HTEPP) is being developed for the processing of volatile materials (conducting or nonconducting), for which controlled gaseous environment is required. Nonuniform alternating electric field produced by tetrahedrally arranged spherical electrodes polarizes the sample and exerts electrophoretic forces for the position control. Therefore, this positioner purely relies on the dipolar forces ignoring any net sample charges which might be difficult to ensure in the gaseous environment during high-temperature processing.

Projected Science Capabilities	
Max. control force	20 dynes
Positioning accuracy	50 microns
Vibration isolation	possible
Heating method	isothermal or beam heating
Sample temperature	up to 1700 °C
Cooling	radiation and forced convection
Chamber pressure	ambient or high pressure
Sample size	0.6 cm dia.
Sample density	up to 18 g/cc

C) HIGH TEMPERATURE TETRAHEDRAL ELECTROMAGNETIC POSITIONER (HTEMP) is being developed for the processing of conducting materials both in vacuum and in the controlled gaseous environment. Four separate coils arranged in a tetrahedral configuration exert electromagnetic forces for the position control. Advantage of this system is that inductive sample heating and positioning can be operable simultaneously. Furthermore, by adjusting relative rf phases, sample rotation can either be induced or damped in a controlled way so that it makes various drop dynamics experiments possible using molten drops.

Projected Science Capabilities	
Max. control force	500 dynes
Positioning accuracy	50 microns
Vibration isolation	possible
Heating method	Inductive and/or beam
Sample temperature	>2000 °C
Cooling	radiation and forced cooling
Chamber pressure	either vacuum or pressurized
Sample size	1 cm dia.
Sample density	up to 18 g/cc

Note: Further information may be obtained from Dr. Won-Kyu Rhim, Jet Propulsion Laboratory, 4800 Oak Grove Dr., Pasadena, CA 91109

EQUIPMENT DESCRIPTION

HIGH-TEMPERATURE ACOUSTIC LEVITATION FURNACE

The high-temperature acoustic levitator uses xenon arc or laser beam heating for containerless melting and undercooling studies of high-temperature glass, ceramic, metal, and alloy samples in microgravity. This facility has a temperature range up to 2000°C, a design goal of 2700°C. It is capable of processing, heating, melting, soaking, cooling, and solidifying samples without the physical contact of a container. Extremely stable sample positioning utilizing optical feedback is provided. Very fast heating and cooling rates of up to 200°C/sec are possible. The samples may be processed in a very-high-purity, particle-free, inert, oxidizing or reducing gaseous environment. The facility accepts nominally spherical samples 2-6 mm in diameter and can perform a large number of experiments sequentially.

The sound pressure level is electronically monitored and controlled by a computer. This can be used to modulate a liquid sample shape to possibly study drop fluid dynamics or to measure physical properties, such as surface tension and viscosity. By properly phasing the acoustic signals between transducers, controlled spin of the specimen can be produced.

During all stages of processing, there can be continuous video and thermal imaging at orthogonal views of the specimen. Thermal imaging can be carried out at a number of wavelengths to provide noncontact temperature measurement. The open architecture of the levitator allows excellent access for diagnostic equipment.

SCIENCE CAPABILITIES

HIGH-TEMPERATURE ACOUSTIC LEVITATION FURNACE

Temperatures *	
Design Goal	30 to 2000°C up to 2700°C
Isothermality *	Good
Temperature Control Precision	TBD (Depends on NCTM)
Design Goal	1°C
Gas Purity	
Particulate Contamination Excellent	(as good as process gas)
Process Gas	Oxidizing, Reducing, or Inert
Specimen Size	2 - 6 mm
Specimen Motion	<1 mm (Electronically Controlled)
Specimen Density	0 to 22 gm/cm^3
Position Accuracy	Very Good, < + 1 mm
Heat Rates *	Very Fast, 0 to 200°C/sec or Higher
Cooling Rates *	Very Fast, 0 to 200°C/sec or Higher
Spin Control	Good (No or Very Low Spin)
Optical Access	Very Convenient
Heaters	Xenon arc, Laser Beam, RF, Microwave

* Dependent on sample properties and size.

EQUIPMENT DESCRIPTION

ACOUSTIC LEVITATION FURNACE

This furnace will be a containerless, processing, hot-wall furnace for melting and undercooling studies of high-temperature glass, ceramic, metal, and alloy specimens in a microgravity environment. The furnace has a temperature range up to 1750°C with cooling rates up to 20°C/sec. A high degree of specimen isothermality (2°C/cm) is achieved. Samples are heated, melted, soaked, cooled, and solidified without the physical contact of a container. This facility accepts nominally spherical samples 2-6 mm in diameter and can perform a large number of experiments sequentially.

The samples are stored in individual cells in the specimen magazine. They are injected into the acoustic energy well of the furnace for processing. A mesh cage surrounds the sample to allow recovery of the sample in the event of loss from the energy well. The overall sound pressure is electronically adjustable by computer control and can be used to modulate the liquid sample shape. The latter can be used to study drop fluid dynamics or to measure physical properties, such as surface tension and viscosity. By properly phasing the acoustic signals between the transducers, controlled spin of the sample can be produced. After sample processing and solidification, the sample is removed from the furnace through the specimen-handling system.

During processing, simultaneous video imaging and thermal imaging is provided. Orthogonal video images of the specimen are acquired. Thermal imaging is possible at various optical wavelengths, thus enabling user-calibrated temperature measurement.

SCIENCE CAPABILITIES

ACOUSTIC LEVITATION FURNACE

Temperatures	800 to 1750°C
Specimen Isothermality	Very Good (+ 2°C/cm)
Temperature Control Precision	Very Precise & Stable, Repeatable +/- 3°C
Gas Purity Particulate Contamination	Laminar Flow Gas Purge, Better than Class 1000 Clean Room
Process Gas	Inert, Reducing, or Oxidizing
Specimen Size	2 - 10 mm
Specimen Density	0 to 22 gm/cm^3
Position Accuracy	Good <+ 2 mm
Spin Control	Good, No, or Very Low Spin
Heat Rates	Programmable, Slow (<2°C/sec)
Cooling Rates	Programmable, Slow, or Moderate with Gas Quench (<20°C/sec)
Optical Access	Ports through Insulation and Windows
Heaters	Very Reliable, Simple Resistive
Heating Duration	Nominally Minutes to 2 Hours
Carrier	Spacelab, Space Station

MODULAR ELECTROMAGNETIC LEVITATOR

The Modular Electromagnetic Levitator under development at Oak Ridge National Laboratory will be designed to optimize flexibility in the accomodation of reconfigurations required by changing scientific capabilities. The basic function of the apparatus will still be to process high melting point metals and alloys in a very controlled, contaminant-free environment. The emphasis will be on the versatility of the processing environmental conditions and parameters, as well as the addition of more complex diagnostics instrumentation. Splat cooling capability for rapid sample quenching is a planned development effort.

TECHNICAL DESCRIPTION

Sample Size	5-10 mm
Maximum Power Consumption	about 500 W
Dual RF Frequencies	450 kHz for positioning 450 kHz to 1.0 MHz for sample heating 35 % efficiency
Sample Temperature	up to 2500°C
Number of Samples	Unlimited or timeline-limited Samples are stored in cannisters to be loaded on orbit
Vacuum Range	10^{-9} Torr Continuous pumping during operation
Temperature Measurement	Single-Color Pyrometer and Recalescence Detector
Observation	2 orthogonal views with video cameras

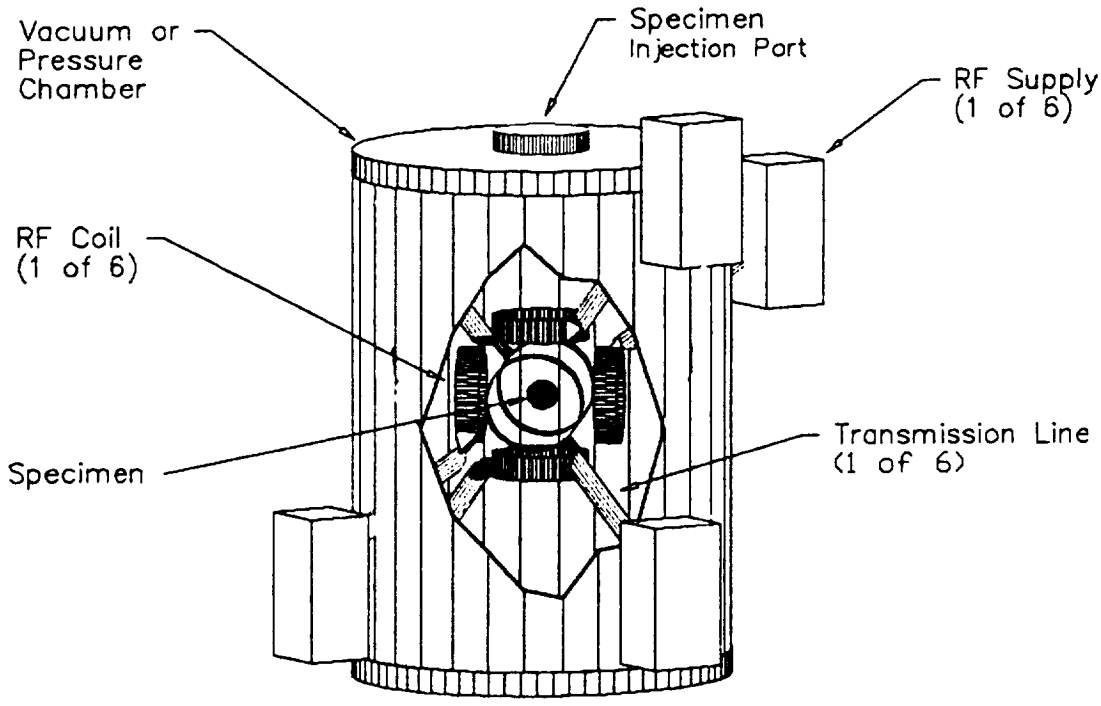
EQUIPMENT DESCRIPTION

STABILIZED ELECTROMAGNETIC LEVITATOR (SEL)

The Stabilized Electromagnetic Levitator is a highly stable multicoil levitator for melting and undercooling studies in microgravity environment. It is characterized by independent control of heating and positioning. Both good or poor conductive materials, metallic or non-metallic, may be levitated. By varying the signals between coils, sample stability and oscillation can be controlled. Independent heating will allow undercooling studies without sample instability.

High frequency induction heating of samples to 2700°C or greater will be possible. Heating and cooling rates up to 200°C/sec will be available. Samples may be processed at low pressures or in controlled atmospheres.

The open architecture of the device will allow ample access for diagnostic and process control equipment, such as NCTM or DAPP. Precise temperature, surface tension, and viscosity measurements will be possible in the stable quiescent sample.



STABILIZED ELECTROMAGNETIC LEVITATOR (SEL)

SCIENCE CAPABILITIES

STABILIZED ELECTROMAGNETIC LEVITATOR (SEL)

Temperatures *	0 to 2700°C or Higher
Isothermality *	Good
Temperature Control Precision	TBD, (Depends on NCTM)
Design Goal	+/- 5°C
Gas Purity Particulate Contamination	Excellent (as Good as Process Gas)
Process Gas	Vacuum, Reactive, or Inert
Specimen Size	2 - 6 mm
Specimen Motion	+ /-1 mm
Position Accuracy	Very Good, <+/- 1 mm
Heat Rates *	Very Fast, 0 to 200°C/sec or Higher
Cooling Rates *	Very Fast, 0 to 200°C/sec or Higher
Spin Control	Good (No or Very Low Spin)
Optical Access	Very Versatile
Heaters	Inductive Heating
Supplemental Beam Heating Possible	

* Dependent on sample properties and size.

GAS LAYER LEVITATOR

The Gas Layer Levitator is under development by the European Space Agency for the containerless processing of glasses and ceramics. Initial ground-based tests have demonstrated the ability of levitating liquid samples at ambient room temperature as well as up to 1500°C. Sample volumes up to 40 cc can be used for containerless processing. Three-dimensional extension of the technique is under investigation for microgravity applications.

Reference: J. Granier and C. Potard, Proceedings 6th European Symposium on Materials Science under Microgravity Conditions, Bordeaux, France, 2-5 December 1986, ESA Publication SP-256, February 1987.

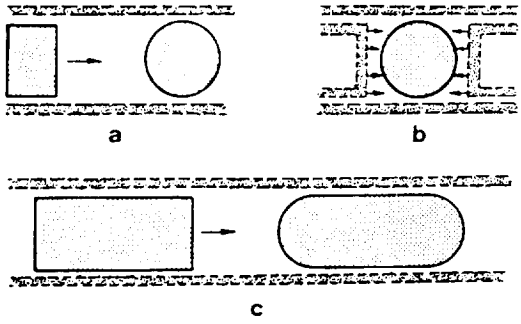
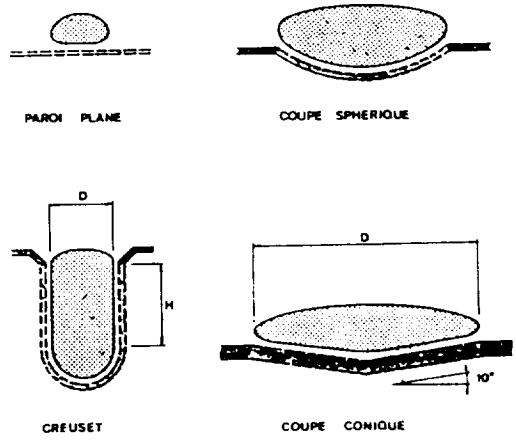


Figure 1. Positionnement et moulage sans contact en microgravité.



CONFIGURATION	DIAMETRE (MM)	HAUTEUR (MM)	VOLUME (CM^3)	DEBIT He (L/MN)
PLANE	–	–	1.4	0.2
SPHERIQUE	60	–	30	0.55
CREUSET	25	30	18.5	0.03
CONIQUE	80	–	40	0.2

Figure 2. Configurations à axe vertical

GROUND-BASED FACILITIES

LABORATORY, KC-135, AND SOUNDING ROCKET APPARATUSES

ANTICIPATED SCIENCE CAPABILITIES SUMMARY

GROUND-BASED LEVITATORS

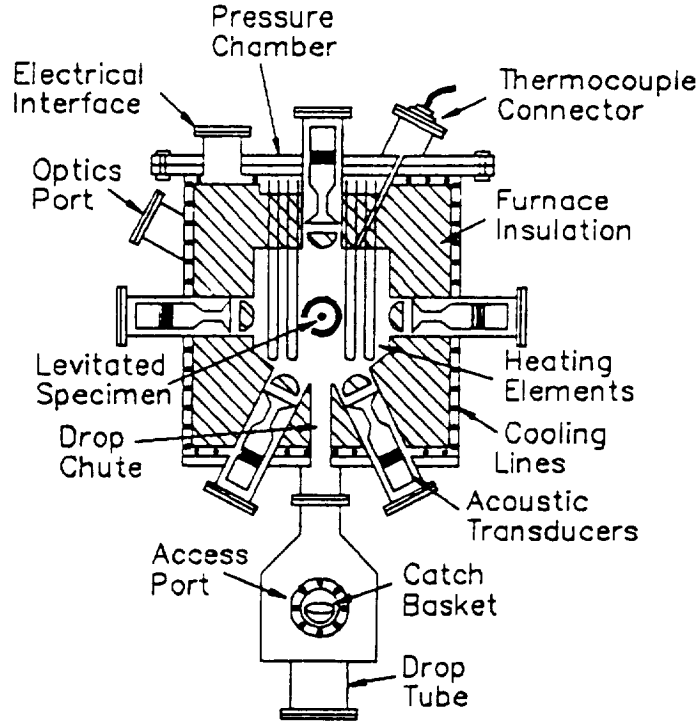
The ground-based full-gravity and low-gravity levitators allow the investigator to conduct initial precursor experiments leading to microgravity work. Ground-based facilities can provide initial quick turnaround experimental results at a reduced cost.

	Electromagnetic Levitator	High Pressure Acoustic Levitator	Acoustic Levitators for KC-135 or Rockets
Temperature Range	to 2200°C	to 1400°C	to 2000°C and Higher
Process Gas	Inert, Reducing, Oxidizing Vacuum	Same	Same
Processing Pressure	0 to 2 Bars	5 to 20 Bars	1 Bar
Heating	Induction or Laser	Furnace or Laser	Arc Lamp
Cooling Rate (°C/sec)	0 to 300 or Higher	0 to 5	0 to 200
Position Control	None	+/- 1 mm	+/- 1 mm
Sample rotation control	Possible	Acoustic Torque Control	Ac. Torque Control
Sample size	2 to 5 mm	2 to 10 mm	2 to 10 mm
Sample density	Unlimited	up to 7 g/cc	Unlimited
Conductive Sample	Yes	No	No

EQUIPMENT DESCRIPTION

GROUND-BASED HIGH-PRESSURE ACOUSTIC LEVITATOR

This facility will be a ground-based high-pressure acoustic levitator for precursor flight experiments. Under increased ambient pressure, samples of a higher density may be levitated in 1g. Samples with densities of 5 have been levitated at a temperature of 1000 °C under 9 atmospheres of pressure. Higher densities and temperatures are possible by increasing the pressure. Samples can be heated by beam or hot-wall methods. Processing in controlled atmospheres is possible. Spin control of the specimen is achieved by phasing of the acoustic signals. Sample temperature will be measured with appropriate noncontact techniques. Thermocouples can also be used in the isothermal hot-wall furnace.



GROUND-BASED
HIGH-PRESSURE ACOUSTIC LEVITATOR

ELECTROSTATIC-ACOUSTIC HYBRID LEVITATOR (EAHL)

This is a general purpose room temperature levitator in which a charged liquid drop is levitated by feedback-controlled electrostatic forces while drop oscillation, rotation, and fission can be induced acoustically. Existing system is particularly suitable for charged drop dynamics and cloud physic experiments in the ground-based laboratories, and with some minor modification it can become a flight module. Charged water or aqueous drops 3 to 4 mm in diameter can be levitated in 1 g and its capabilities for various drop dynamics experiments have been demonstrated. This system can be readily adapted to a wind tunnel, and drop dynamics under wide-flow velocity can be studied.

A charge drop is levitated between a pair of circular electrodes which are mounted at the centers of top and bottom surfaces of an acoustic resonance chamber. A ring electrode may be inserted between the electrodes if three dimensional position control becomes necessary. Two acoustic transducers mounted on two orthogonal side faces of the chamber generate appropriate acoustic waves and induce drop vibration or rotation. The feedback system maintains the drop position at a preassigned point and damps any transient motion around it. Using a synchronized strobe light, frequency of oscillation or rotation is measured and evolving drop shape is recorded on a videotape.

OPERATIONAL PARAMETERS	
Max. levitation voltage	20 kv_dc
Max. levitation force	200 dynes
Max. acoustic pressure	150 dB
SAMPLE PARAMETERS	
Sample phases	solids or liquids
Sample diameter	1 - 4 mm in 1-g virtually no limit in mg
Typical sample charge	5×10^{-10} Coulomb
Max. sample rotation rate	50 revolutions/sec

DATA ACQUISITION Two fast video cameras directed to record both top and side views of the drop.

Further information can be obtained from Dr. Won-Kyu Rhim, Jet Propulsion Laboratory, California Institute of Technology, Pasadena California, 4800 Oak Grove Dr., Pasadena, CA 91109

ELECTROSTATIC MULTI-DROP LEVIATOR (EMDL)

Existing electrostatic four-drop levitator is a ground-based model of the multi-drop positioner which is being developed for the containerless protein crystal growth or cell-culturing experiments in space. Space module will be able to position more than ten large drops each of which serviced by various diagnostic instruments. Sample positioning is accomplished by feedback-controlled electrostatic forces which can be readily modified according to the user's need. This system provides 'simple' and 'clean' experimental environment. It is simple in the sense that freely floating liquid drops form spherical shapes contained by their own surface tension and exposing themselves to isotropic thermal transfer and vapor diffusion fields. Spherical drop also allows us accurate determination of its size which in turn relates to the protein saturation level. It is clean in two other senses. First, the sample is not in physical contact with container walls which might induce uncontrollable nucleation. Secondly, through a software programming on control forces, sample drop can be isolated from much of the oscillatory and impulsive forces which are known to reside in space laboratories. Thus, being free from undesirable container walls and disturbing forces, parameters recorded in the course of experiments will find good correlations with final results and such capability will even allow investigators to interactively dictate the courses of experiments toward the growth of large high-quality protein crystals.

OPERATIONAL PARAMETERS IN 1 G

Max. levitation voltage	15 kv_dc
Max. levitation force	100 dynes
Temperature Control*	4 - 35 $^{o}C \pm 0.1\ ^{o}C$

SAMPLE PARAMETERS

Sample phases	liquids or solids
Sample diameter	1 - 4 mm in 1 g, virtually no limit in mg
Typical sample charge	5×10^{-10} Coulomb
No. of drops	4
Drop size measurement	better than 0.03 $\mu\ell$
Drop size control	programmable control

DATA ACQUISITION

High-resolution video camera records the growth rate.

Further information can be obtained from Dr. Won-Kyu Rhim, Jet Propulsion Laboratory, California Institute of Technology, Pasadena California, 4800 Oak Grove Dr., Pasadena, CA 91109

* being developed

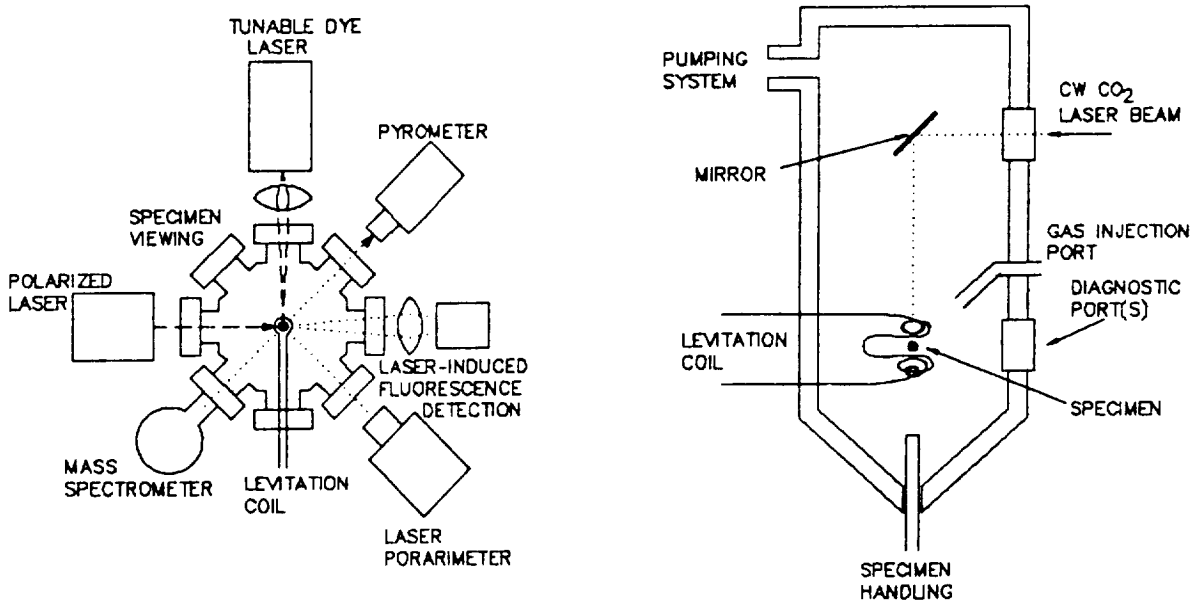
EQUIPMENT DESCRIPTION

GROUND-BASED ELECTROMAGNETIC LEVITATOR (BEAM-HEATED)

This facility allows ground-based containerless evaluation of materials prior to experiments in microgravity. The levitator consists of a dual frequency electromagnetic generator coupled to a coil. The coil is located in a stainless steel vacuum chamber. There are ports radial to the position where the specimen is levitated. Independent control of specimen temperature above that obtained by electromagnetic heating is achieved by CW CO_2 laser beam heating. This allows optimum levitation conditions to be used over a wide range of temperatures.

The chamber can be operated under low pressure or with a variety of controlled atmospheres. The apparent temperature of the specimen is measured with a pyrometer. Feedback control of the laser power is used to maintain stable specimen temperature. The chamber can be equipped with a laser polarimeter to measure the optical properties of the sample. The latter allows accurate, true thermodynamic temperatures to be calculated.

Specimen temperatures at least to 2500 °C can be attained. Rapid cooling can be achieved by helium quenching. The chamber is instrumented with laser-induced fluorescence analysis and a mass spectrometer. These can be used to analyze vapor emanating from hot specimens.



GROUND-BASED ELECTROMAGNETIC LEVITATOR

EQUIPMENT DESCRIPTION

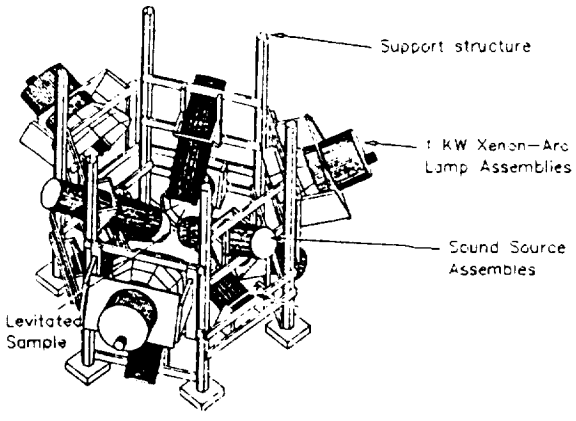
GROUND-BASED ACOUSTIC LEVITATION (BEAM-HEATED)

FOR KC-135 PARABOLIC FLIGHTS OR SOUNDING ROCKETS

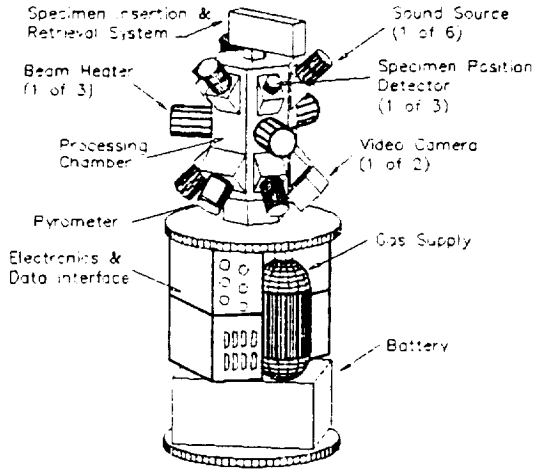
This facility will be a high-temperature acoustic levitator for precursor experiments in low gravity. It will be flown aboard the KC-135 aircraft, and/or in sounding rockets. The KC-135 aircraft provides approximately 20 seconds of weightlessness by following a parabolic flight trajectory. Forty trajectories are typically flown per day. A sounding rocket can provide 5 to 15 minutes of weightlessness with samples and payload being recovered by a parachute.

Beam heating by xenon arc or laser will provide rapid sample heating to temperatures exceeding 2000 °C, depending on sample characteristics. Sample sizes ranging from 2-6 mm can be accommodated. Control of sample rotation and oscillation will be possible. The samples may be processed in various inert or reactive environments.

Video and thermal imaging will be available in orthogonal directions. Thermal imaging at several different optical wavelengths will provide the user with noncontact temperature measurement capabilities.



KC-135 TEST FACILITY
HIGH-TEMPERATURE ACOUSTIC LEVITATOR



SOUNDING ROCKET PROCESSING MODULE
HIGH-TEMPERATURE ACOUSTIC LEVITATOR

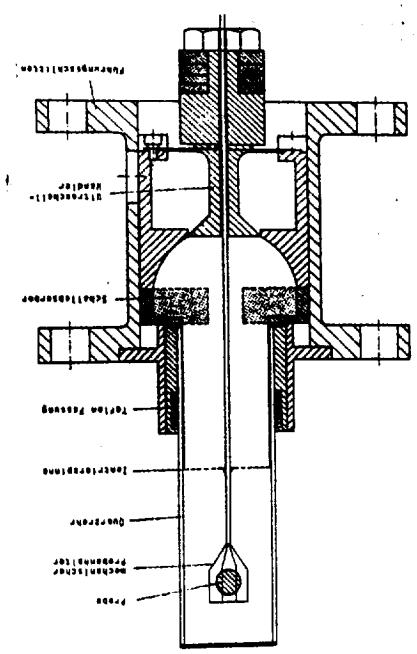
ACOUSTIC POSITIONER AND ELLIPSOIDAL MIRROR FURNACE

ESA SOUNDING ROCKET FLIGHTS APPARATUS

Containerless processing samples at the focus of a monoellipsoidal mirror furnace can be accomplished with a resonant ultrasonic positioner using a closed cylindrical quartz tube. An internal control loop monitors and stabilizes the sample at the focus of the mirror. The sample to be processed is initially contained in a wire basket at the lower focus of the mirror.

TECHNICAL SPECIFICATIONS

Ultrasonic transducer	22 kHz, 30 mm diameter
Quartz tube	34 mm inner diameter, 150 mm long
Sample diameter	2 to 8 mm
Lamp power	450 W maximum
Atmosphere standard	Ar-He (90-10) at 1 atmosphere
Sample temperature	700°C for long duration 1000°C for less than 150 secs
TV monitoring	320 x 244 pixels CCD TV camera



EQUIPMENT DESCRIPTION

NONCONTACT TEMPERATURE MEASUREMENT (NCTM):

DIVISION OF AMPLITUDE POLARIMETRIC PYROMETER (DAPP)

The DAPP is a device for the measurement of surface emissivity and thermodynamic temperature of specular and partially specular surfaces. It employs laser reflection techniques based on the principles of polarized light reflection. The emissivities, indices of refraction and extinction coefficients, are obtained at the laser wavelength. Concurrently, the spectral radiance of the surface is measured. The thermodynamic temperature is then determined to a high level of accuracy.

The device being developed employs a laser operating at 0.6328 um and can measure true surface temperatures in the range 1000-2500 K. The detection system consists of a special polarimeter capable of measuring all four Stokes vectors of reflected and emitted light instantaneously. The error in the emittance measurement is about 1%, the maximum error in temperature measurement is + 5 K at 2000 K. The temperature resolution is about 1 K at 1000 K and the instrument response time is typically 0.1 sec.

The DAPP technology can be adapted to any of the containerless processing systems previously described for the highest accuracy temperature measurements.

DAPP CAPABILITIES

Operating Temperature Range	1000-2500 K
Surfaces Which Can Be Analyzed	Specular or Partially Specular
Design Wavelength	0.6328 um
Response Time	100 milliseconds
Resolution	1 K at 1000 K
Error in Emittance	+/- 1%
Error in Temperature	+/- 5 K at 2000 K

EMISSIVITY AND RADIANCE MEASUREMENTS

LASER PYROMETER

The key element in performing radiative temperature measurements is in the relationship between surface radiance and blackbody radiance, that is, the emissivity of the surface. Since this number is not always known precisely, or may change during the course of an experiment, large errors in estimated temperatures may occur.

The laser pyrometer has the capability to reduce these errors in certain applications by making a measurement of both the target radiance and the target surface emissivity. The latter is achieved with an onboard laser transceiver that performs an in situ measurement of the surface reflectance from which emissivity can be obtained (= 1.0 - reflectance). With calibration of the reflectance channel on diffuse surfaces, measurement errors on similarly diffuse surfaces can be reduced by as much as an order in magnitude. Under certain circumstances, calibration for non-diffuse surfaces can also be performed.

Operational wavelength:	904 nm
Temperature range (LO setting):	1100 K - 1550 K
Temperature range (HI setting):	1400 K - 3000 K
Temperature accuracy:	+/- 3 K
Data rate:	10 per sec.

MULTIPLE-COLOR PYROMETRY

Because of the distribution of the Planck radiative law, optimized pyrometry requires that the operational wavelength be as short as possible, compared to the maximum in the black body spectrum. In this region, radiance increases faster than exponentially with temperature such that target emissivity plays a relatively small role in determining emitted power. The short wavelengths therefore provide more accurate measurement of the temperature than longer wavelengths assuming the same uncertainty in emissivity. However, because of the huge dynamic range that any realistic temperature range will provide at one wavelength, it is preferable to operate at a variety of different wavelengths.

The multiple-color pyrometer manufactured by PSI operates on this principle and uses six different wavelengths each imaged on to a CCD detector contained within an optical head. The pyrometer is also provided with electronics to control the detector, computerized frame aquisition, digitization and analysis software.

Operational wavelengths:	370 nm 420 nm 480 nm 590 nm 750 nm 930 nm
Temperature range:	1000 K - 2500 K
Temperature accuracy:	< 0.2 %

Appendix A

Microgravity Containerless Experimentation
Requirements Survey

Microgravity Containerless Experimentation Requirements Survey
Jet Propulsion Laboratory

The success of scientific and technological experimental investigations in microgravity is crucially dependent on the capabilities and reliability of the flight facilities available to carry out these studies. In order to plan new scientific equipment and to refine existing flight instruments, guidelines must be followed to optimize their applicability to the relevant technical disciplines and to allow the implementation of the more recent and effective technologies. These guidelines must be based on the informed assessment of past experience, the increased knowledge of current development, and the reasonable projection into future needs. The long lead time and high cost of development and deployment of flight experiment facilities require that these guidelines reflect as many of the technical requirements as possible. The first step toward this goal involves the gathering of a wide range of information on the current and future requirement needs of the scientific and industrial research communities that are working with containerless experimentation and materials processing.

Containerless investigations have been conducted by various disciplines in Earth-based laboratories for a long time. The rationale for performing experiments on free-falling or freely suspended samples varies according to the different areas of interest. The availability of long-duration orbital laboratories would appear at first to provide the ideal home for the performance of such investigations. The reality, however, is that each investigator cannot carry out his or her work in such a facility using a custom-developed and individually fine-tuned set of instrumentation. Instead, the investigator has—and probably will continue to have to—use equipment designed under strict constraints not always totally consistent with his or her continuously evolving needs. Additionally, because of the tremendous cost of space flight instrumentation, the option to build generic facilities that are designed to serve a variety of different investigators is often chosen. This questionnaire specifically deals with the first step in the process of defining the general requirements for such generic facilities, based on the technology of sample positioning, manipulation, and measurement in low gravity.

This questionnaire has been designed to provide a useful data base for the planning of future facilities. For those who are not familiar with containerless space experimentation or not ready to answer all the questions, a preliminary indication of their technical interest can be obtained by the items marked with a double asterisk (**).

SURVEY OF EXPERIMENTAL REQUIREMENTS
CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY

NAME:

AFFILIATION:

ADDRESS:

TEL.:

FAX:

**1.0 GENERAL EXPERIMENT DESCRIPTION

**1.1 DISCIPLINE OF THE INVESTIGATION:

FLUID DYNAMICS _____

MATERIALS SCIENCE:
- GLASSES AND CERAMICS _____
- POLYMERS _____
- METALS AND ALLOYS _____
- BIOTECHNOLOGY _____
- ELECTRONIC MATERIALS _____
  - SUPERCONDUCTORS _____
  - COMPOSITES _____

SURFACE/INTERFACIAL PHENOMENA _____

CRITICAL PHENOMENA/
FUNDAMENTAL PHYSICS AND CHEMISTRY _____

ATMOSPHERIC / PLANETARY SCIENCE _____

HIGH TEMPERATURE PHYSICS AND CHEMISTRY _____

OTHER (Please specify) _____

1.2 ACCELERATION LEVEL REQUIREMENTS

**1.2.1 MAXIMUM ALLOWABLE CONTINUOUS ACCELERATION LEVEL:

**1.2.2 MAXIMUM TOLERABLE TRANSIENT ACCELERATION LEVEL:
FREQUENCY RANGE:
DURATION:

**1.2.3 RELEVANCE OF LOW GRAVITATIONAL ACCELERATION TO PHENOMENA UNDER STUDY:

1.3 IMPORTANCE OF CONTAINERLESS CONDITIONS

**1.3.1 EFFECT OF CONTAINER (CONTAMINATION, SURFACE INTERFERENCE, HIGH TEMPERATURE REQUIREMENT,...)

**1.3.2 SENSITIVITY TO RESIDUAL POSITIONING FIELDS:

SHAPE DEFORMATION:	UNDESIRABLE____	UNACCEPTABLE____
SURFACE CHARGE:	UNDESIRABLE____	UNACCEPTABLE____
SURFACE FLOW:	UNDESIRABLE____	UNACCEPTABLE____
INTERNAL FLOW:	UNDESIRABLE____	UNACCEPTABLE____
TRANSLATIONAL OSCILLATION:	UNDESIRABLE____	UNACCEPTABLE____
ROTATIONAL MOTION:	UNDESIRABLE____	UNACCEPTABLE____
SHAPE OSCILLATION (MELT):	UNDESIRABLE____	UNACCEPTABLE____

2.0 SAMPLE REQUIREMENTS

**2.1 SIZE AND SHAPE

2.2 FORM (SOLID, LIQUID), GENERAL PHYSICAL PROPERTIES (MECHANICAL, THERMAL, ELECTRICAL, OPTICAL, COMPOSITION)

**2.3 NUMBER OF SIMULTANEOUSLY DEPLOYED SAMPLES

**2.4 TOTAL NUMBER OF SAMPLES NEEDED

2.5 STORAGE REQUIREMENTS (ENVIRONMENTAL CONDITIONS DURING STORAGE, TIME LIMIT,...)

2.6 SAMPLE DEPLOYMENT SPECIAL REQUIREMENTS

2.7 SAMPLE RETRIEVAL AND STORAGE, POST PROCESSING REQUIREMENTS

2.8 SENSITIVITY TO MECHANICAL HANDLING

**2.9 SAFETY REQUIREMENT DURING HANDLING AND PROCESSING
(CORROSIVENESS, TOXICITY)

2.10 PURITY RESTRICTION/MAXIMUM TOLERABLE IMPURITY LEVEL

3.0 PROCESSING ENVIRONMENT REQUIREMENTS

3.1 NATURE OF THE PROCESSING ENVIRONMENT:

** 3.1.1 VACUUM (MIN/MAX PRESSURE, PUMPING RATE):

** 3.1.2 GAS ENVIRONMENT:

. NATURE (INERT, OXIDIZING, REDUCING):

.PURITY REQUIREMENTS OR COMPOSITION FOR MIXTURES:

. ACCEPTABLE PARTICULATE CONCENTRATION LEVEL:

. MAJOR UNACCEPTABLE GASEOUS CONTAMINANTS:

. ACCEPTABLE GAS FLOW RATE DURING PROCESSING:

3.1.3 LIQUID ENVIRONMENT: YES:____ NO:____
LIQUID FOR SAMPLE QUENCHING: YES:____ NO:____

3.1.4 ENVIRONMENT TEMPERATURE:

** . SAMPLE ENVIRONMENT TEMPERATURE
TEMPERATURE RANGE:

UNIFORMITY (ACCEPTABLE SPATIAL THERMAL GRADIENT):

ALLOWABLE SHORT TERM FLUCTUATIONS AND REQUIRED LONG TERM STABILITY:

**ENVIRONMENTAL TEMPERATURE MEASUREMENT REQUIREMENTS:

. ACCURACY:

. SUGGESTED MEASUREMENT METHOD:

. SPECIAL REQUIREMENT FOR RADIATIVE BACKGROUND:

3.1.5 ENVIRONMENT HYDROSTATIC PRESSURE RANGE:
PRESSURE GRADIENT REQUIRED: YES____ NO____
PRESSURE GRADIENT RANGE:
RATE OF CHANGE:

**3.2 SAMPLE PROCESSING TEMPERATURE RANGE:
UNIFORMITY (MAX. TOLERABLE THERMAL GRADIENT):
REQUIRED THERMAL GRADIENT IN SAMPLE: YES____ NO____
RANGE OF ACCEPTABLE RATE OF CHANGE FOR HEATING:
FOR COOLING:
MAXIMUM DESIRED COOLING RATE:
RAPID QUENCHING DESIRED: YES____ NO____
COOLING RATE:

SAMPLE TEMPERATURE CONTROL:
. ADVOCATED METHOD OF SAMPLE HEATING:
. ADVOCATED METHOD OF SAMPLE THERMAL CONTROL:

. THERMAL DEADBAND FOR CONTROL:

3.3 EXPERIMENT CHAMBER IDEAL DIMENSIONS:

**3.4 EXPERIMENT CHAMBER PURGING, OUTGASSING REQUIREMENTS:

4.0 SAMPLE POSITIONING REQUIREMENTS

4.1 CRUCIAL POSITIONING PARAMETERS:
** 4.1.1 TRANSLATIONAL STABILITY (MAXIMUM FLUCTUATION ALLOWABLE):
** 4.1.2 ROTATIONAL STABILITY (MAXIMUM FLUCTUATION ALLOWABLE):

4.1.3 SHAPE STABILITY OF MELT (MAXIMUM PERCENT DEVIATION FROM IDEAL SPHERICITY):

4.1.4 CONTROLLED MELT SHAPE DISTORTION CAPABILITY:
YES_____ NO_____
ACTIVE SHAPE MODULATION CAPABILITY:
YES_____ NO_____

** 4.1.5 CONTROLLED SAMPLE ROTATION CAPABILITY:
YES_____ NO_____
ROTATION RATE RANGE:
ACCURACY OF ROTATION CONTROL:

4.1.6 MULTIPLE SIMULTANEOUS SAMPLES POSITIONING:
YES_____ NO_____
APPROXIMATE NUMBER:

4.1.7 CONTAINERLESS SAMPLE TRANSPORT:
YES_____ NO_____
DISTANCE:

**4.2 TYPICAL EXPERIMENT DURATION:

**4.3 REQUIREMENT FOR ABSOLUTE ABSENCE OF MOTION OR FLOW:
YES_____ NO_____
DURATION:

4.4 SAMPLE FREE-DRIFT WITHIN EXPERIMENT CHAMBER:
YES_____ NO_____
FOR DURATION:

5.0 SAMPLE PROPERTIES AND ENVIRONMENT PARAMETERS MEASUREMENTS

** CRITICAL PROPERTIES TO BE MEASURED AND ACCURACY:

5.1 OPTICAL DIAGNOSTICS
(INDICATE WHETHER A PREFERENCE OR AN ABSOLUTE REQUIREMENT)

** 5.1.1 SAMPLE OBSERVATION:
.DIRECT OBSERVATION: YES_____ NO_____
.VIDEO IMAGES: YES_____ NO_____
RESOLUTION: STANDARD (NTSC) _____
HIGHER RESOLUTION (SPECIFY)_____

FRAME RATE:
COLOR OR B/W:
. PRIMARY FEATURES OF INTEREST:
CONTOUR (RESOLUTION):
INTERNAL STRUCTURE (RESOLUTION):
SURFACE STRUCTURE (RESOLUTION):
DYNAMIC BEHAVIOR (TIME CONSTANT):
MEASUREMENT DURATION:

. FIELD OF VIEW (IN TERMS OF SAMPLE SIZE):

5.1.2 IMAGE RECORDING:

.VIDEOTAPES: YES_____ NO_____
.CINEFILMS: YES_____ NO_____

5.1.3 REAL-TIME DOWNLINK OF IMAGING: YES_____ NO_____

5.1.4 FLOW FIELD VISUALIZATION: YES_____ NO_____
.ADVOCATED TECHNIQUE:

.VELOCITY RANGE OF FLOWS:
. 1D____ 2D_____ 3D_____

5.1.5 SAMPLE OPTICAL PROPERTIES MEASUREMENTS:
PARAMETERS OF INTEREST:

5.2 SAMPLE THERMAL MEASUREMENTS:
** 5.2.1 PARAMETERS TO BE MEASURED AND ACCURACY:
SURFACE TEMPERATURE (ACCURACY AND RESOLUTION):

INTERNAL TEMPERATURE (ACCURACY AND RESOLUTION):

SPATIAL THERMAL GRADIENT (RESOLUTION):

LIST OTHER PARAMETERS OF INTEREST:

** 5.2.2 MEASUREMENT RATE: MAXIMUM_____readings/sec
MINIMUM_____readings/sec

5.2.3 ADVOCATED MEASUREMENT TECHNIQUES:

5.2.4 DYNAMIC RADIANCE MEASUREMENT (RECALESCENCE):
YES_____ NO_____
RESPONSE TIME CONSTANT: _____ sec.

5.2.5 THERMAL IMAGING: YES_____ NO_____
RESPONSE TIME CONSTANT:_____ sec.

**5.3 OTHER SAMPLE PARAMETERS TO BE MEASURED AND ACCURACY:

**5.4 ENVIRONMENT PARAMETERS TO BE MEASURED AND ACCURACY:
TEMPERATURE:
PRESSURE / VACUUM RANGE:
HUMIDITY:
ACCELERATION MAGNITUDE (RESPONSE TIME AND FREQUENCY RANGES):

ATMOSPHERIC GAS COMPOSITION (PRINCIPAL COMPONENTS AND RESOLUTION):

POSITIONING DEVICE PARAMETERS (ACOUSTIC PRESSURE LEVEL, ELECTRIC FIELDS, RF POSITIONING POWER,...):

OTHERS:

6.0 DATA REQUIREMENTS

6.1 DATA ACQUISITION MEDIUM (OPTICAL, DIGITAL,...):

6.2 DIGITAL DATA STORAGE REQUIREMENTS:_____ Mbytes per expt.

6.3 DIGITAL DATA DOWNLINK REQUIREMENTS (RATE, VOLUME, DURATION):

6.4 VIDEO DATA STORAGE AND DOWNLINK REQUIREMENTS (REAL-TIME AND VIDEOTAPES):

7.0 EXPERIMENT PERFORMANCE

** 7.1 REAL-TIME INTERACTION WITH EXPERIMENT: YES_____ NO_____

7.2 EXPERT OPERATOR INTERFACE: YES_____ NO_____

** 7.3 REAL-TIME EXPERIMENT PARAMETERS CHANGE: YES_____ NO_____
LIST PARAMETERS TO BE CONTROLLED:

** 7.4 IN-FLIGHT DATA ANALYSIS WITH ON-BOARD COMPUTERS:
YES_____ NO_____

7.5 DATA UPLINK CAPABILITY: YES_____ NO_____

7.6 REMOTE EXPERIMENT CONTROL FROM GROUND: YES_____ NO_____

7.7 COMPLETE EXPERIMENT AUTOMATION POSSIBLE: YES_____ NO_____

7.8 REAL-TIME DISPLAYS OF PARAMETERS TO OPERATOR:
LIST PARAMETERS REQUIRED:

7.9 REQUIRED HUMAN INTERVENTION: YES_____ NO_____

LIST ACTIVITIES:

** 7.10 POWER REQUIREMENT ESTIMATES: AVERAGE_____ Watts
PEAK_____ Watts
AVERAGE ENERGY REQUIRED PER RUN:_____Watt-Hours
AVERAGE RUN DURATION:_____ Minutes
TYPICAL NUMBER OF RUNS:_____
MIMIMUM NUMBER OF RUNS:_____

8.0 TECHNOLOGY DEVELOPMENT REQUIRED

** 8.1 AREAS OF NEW TECHNOLOGY DEVELOPMENT FOR SPACE EXPERIMENT:

** 8.2 GROUND-BASED INVESTIGATIONS REQUIRED FOR SPACE EXPERIMENT DEVELOPMENT
. SOUNDING ROCKET EQUIPMENT (5 to 15 minutes low gravity)
. KC-135 TEST FACILITY (20 sec. low gravity)

. OTHERS:

** 9.0 ADDITIONAL COMMENTS

PLEASE RETURN FILLED QUESTIONNAIRE TO:

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FOR MORE INFORMATION ON QUESTIONNAIRE: (818) 354 7125

Appendix B

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Appendix C

Containerless Experimentation in Microgravity
Workshop Program

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY
PASADENA HILTON, JANUARY 17-19, 1990
HOSTED BY THE JET PROPULSION LABORATORY
CALIFORNIA INSTITUTE OF TECHNOLOGY

PROGRAM

WEDNESDAY JANUARY 17, 1990
PLENARY SESSION I
9:00 am TO 12:00 pm

9:00 am Introductory Remarks and Orientation
(W. Johnson, California Institute of Technology)

9:15 am Charge of the Workshop
(M.C. Lee, Program Scientist, Microgravity Science and Applications Division)

9:35 am NASA Flight Program Review
(L. Spencer, Program Manager Microgravity Science and Applications Division)

9:55 am Ground-based and Microgravity Containerless Positioning Technology and Facilities
(M. Barmatz, Jet Propulsion Laboratory)

10:25 am Break

10:40 am The US Containerless Experiments
(W. Hofmeister, Vanderbilt University)

11:10 am The European Containerless Experiments
(P. Behrman, European Space Agency)

11:40 am The Japanese Containerless Experiments
(H. Azuma, National Aerospace Laboratory)

12:15 pm Lunch Break

PLENARY SESSION II
1:15 pm TO 5:00 pm

1:15 pm High Temperature Thermophysical Properties
(J. Margrave, Rice University)

1:45 pm Containerless Processing of Undercooled Melts
(J. Perepezko, University of Wisconsin)

2:15 pm Discussion

2:45 pm Break

3:00 pm Containerless Synthesis of Interesting Glasses
(M. Weinberg, University of Arizona)

3:30 pm Protein Crystallization in Microgravity
(A. McPherson, University of California at Riverside)

4:00 pm Fluid Physics under Microgravity
(E.H. Trinh, Jet Propulsion Laboratory)

4:30 pm Discussion

5:00 pm Splinter Sessions Organization Meeting

RECEPTION AND BUFFET DINNER AT THE JET PROPULSION LABORATORY 6:00 pm to 9:00 pm
VON KARMAN AUDITORIUM

BUS SERVICE FROM THE HILTON TO JPL WILL START AT 5:30 pm

THURSDAY 18 JANUARY, 1990

SPLINTER SESSIONS I
9:00 am to 12:00 pm

Five splinter session papers in each discipline to be announced
Rooms to be announced

1. THERMOPHYSICAL PROPERTIES AND VERY HIGH TEMPERATURE CHEMISTRY
   Session Chairman: P. Nordine, Containerless Processing Inc.
   Session Co-Chairman: A. Miller, NIST
   Session Recorder: A. Morrison, Jet Propulsion Laboratory

2. MATERIALS PROCESSING
   Session Chairman: R. Bayuzick, Vanderbilt University
   Session Co-Chairman: A. Fripp, Langley Research Center
   Session Recorder: M. Barmatz, Jet Propulsion Laboratory

3. FLUID DYNAMICS AND INTERFACIAL PHENOMENA
   Session Chairman: E. Trinh, Jet Propulsion Laboratory
   Session Co-Chairman: P. Marston, Washington State University
   Session Recorder: J. Watkins, Jet Propulsion Laboratory

SPLINTER SESSIONS II
1:15 pm to 4:15 pm

Five splinter session papers in each discipline to be announced
Rooms to be announced

1. THERMOPHYSICAL PROPERTIES AND VERY HIGH TEMPERATURE CHEMISTRY
   Session Chairman: W. Johnson, California Institute of Technology
   Session Co-Chairman: R. Hauge, Rice University
   Session Recorder: A. Croonquist, Jet Propulsion Laboratory

2. MATERIALS PROCESSING
   Session Chairman: R. Hopkins, Westinghouse
   Session Co-Chairman: E. Ethridge, NASA MSFC
   Session Recorder: G. Gutt, Jet Propulsion Laboratory

3. FLUID DYNAMICS AND INTERFACIAL PHENOMENA
Session Chairman: A.T. Chai, Lewis Research Center
Session Co-Chairman: M. Parang, U. of Tennessee
Session Recorder: A. Thomas, Jet Propulsion Laboratory

SESSION CHAIRMEN WORKING MEETING
4:30 pm to 6:00 pm

FRIDAY 19 JANUARY, 1990
PLENARY SESSION III
8:30 am to 12:30 pm
SPLINTER SESSIONS REPORT AND PANEL DISCUSSIONS

8:30 am Summary Report on Thermophysical Properties and High Temperature Chemistry Sessions

9:00 am Discussion

9:30 am Summary Report on Materials Processing Sessions

10:00 am Discussion

10:30 am Break

10:45 am Summary Report on Fluid Dynamics and Interfacial Phenomena Sessions

11:15 am Discussion

11:45 am Panel Members Closing Comments

12:30 pm Workshop Adjournment

PANEL MEMBERSHIP:

R. Bayuzick E. Ethridge P. Nordine R. Hauge W. Johnson
W. Hofmeister M. Lee A. Cezairlayan M. Weinberg E. Trinh
J. Perepezko T. Wang
NASDA, ESA, CNES, DLR Representatives (TBA)

CONTAINERLESS EXPERIMENTATION IN MICROGRAVITY
PASADENA HILTON, JANUARY 17-19, 1990
SPLINTER SESSIONS PROGRAM

SPLINTER SESSION I
Thursday, January 18, 1990
9:00 am to 12:00 pm

1. THERMOPHYSICAL PROPERTIES AND VERY HIGH TEMPERATURE CHEMISTRY

Session Chairmen: P. Nordine and A. Cezairlayan
Session Recorder: A. Morrison

9:00-9:20
Y.W. Kim (Lehigh University): "Real-Time Measurement of Materials Properties at High Temperatures by Laser-Produced Plasmas"

9:20-9:40
H.J. Fecht (Caltech): "Thermodynamic Properties and Crystallization Kinetics at High Liquid Undercooling"

9:40-10:00
R. Hauge (rice University): "Thermophysical Properties Measurements using Electromagnetic Levitation"

10:00-10:20
S. Krishnan, J.K.R. Weber, P.C. Nordine, and R.A. Schiffman (Intersonics Inc): "Non-Contact Temperature Measurement of Liquids at High Temperatures using Polarization Techniques"

10:20-10:40
Y. Bayazitoglu (Rice University): "Measurement of Thermal Conductivity and Thermal Diffusivity"

10:40-11:00
A.P. Miller and A. Cezairlayan (NIST): " A Dynamic Technique for Measuring Surface Tension at High Temperatures in a Microgravity Environment"

11:00-12:00
Discussion

2. MATERIALS PROCESSING

9:00-9:20
R. Willnecker and I. Egry (German Aerospace Research Establishment and Institute for Space Simulation, DLR):" Experiments for Electromagnetic Levitation in Microgravity"

9:20-9:40
H. Lenski and R. Willnecker (Dornier GmbH and DLR): " Electromagnetic Processing Onboard Spacelab"

9:40-10:00
P. Neuhaus, G. Lohofer, I. Egry, and R. Willnecker (German Aerospace Research Establishment and Institute for Space Simulation, DLR):"TEMPUS-First Results"

10:00-10:20
E.C. Ethridge, R.B. Johnson, and C. Feng (Marshall Space Flight Center and University of Alabama at Huntsville): " Reluctant Glass Formers and their Applications in Optical Lens Design"

10:20-10:40
W.K. Rhim (Jet Propulsion Laboratory): "Containerless Protein Crystal Growth Technology"

10:40-11:00
H.C. Foley (University of Delaware):"A systematic Investigation of the Preparation and Properties of Composite Carbon Molecular Sieves Containing Inorganic Oxides"

11:00-12:00
Discussion

3. FLUID DYNAMICS AND INTERFACIAL PHENOMENA

9:00-9:20
J. Hartfield, E. Curtis, and P. Farrell (University of Wisconsin): "Supercritical Microgravity Droplet Vaporization"

9:20-9:40
S. Putterman, G. Williams, and M Barmatz (UCLA and Jet Propulsion Laboratory): " Using a Microgravity environment to Probe Wave Turbulence"

9:40-10:00
E.G. Lierke and A.P. Croonquist (Battelle Frankfurt and Jet Propulsion Laboratory): " Drop Evaporation in a Single-Axis Acoustic Levitator"

10:00-10:20
R.E. Apfel and R.G. Holt (Yale University): "Rheological Properties, Shape Oscillations and Coalescence of Liquid Drops with Surfactants"

10:20-10:40
P. Marston (Washington State University): " Optical Scattering Methods Applicable to Drops and Bubbles"

10:40-11:00
K. Ohsaka (Jet Propulsion Laboratory): "Undercooling of Acoustically Levitated Molten Drops"

11:00-12:00
Discussion

SPLINTER SESSION II
Thursday January 18, 1990
1:15 pm to 4:15 pm

1. THERMOPHYSICAL PROPERTIES AND VERY HIGH TEMPERATURE CHEMISTRY

1:15-1:35
B. Dunn and S. Crouch-Baker (UCLA): " Containerless Synthesis of Ceramic Materials using Microwave Heating"

1:35-1:55
A. Cezairlayan, R.F. Chang, and G.M. Foley (NIST): " A High-Speed Spatial (Linear) Scanning Pyrometer: A Tool for Diagnostics, Temperature Mapping, and Property Determinations at High Temperatures"

1:55-2:15
J. Wallace (Casting Analysis Corp.): " Multiple Sensor, Multi-Frequency Eddy Current Monitor for Solidification and Growth"

2:15-2:35
C.A. Hahs (Oak Ridge National Laboratory): " High-Temperature Metal Purification using a Compact, Portable RF Heating and Levitation System on a Wake Shield"

2:35-2:55
R.J. Fox (Oak Ridge National Laboratory): "Compact RF Heating and Levitation System for the NASA Modular Electromagnetic Levitator"

2:55-3:15
S. Krishnan, J.K.R. Weber, R.A. Schiffman, and P.C. Nordine (Intersonics Inc.): " Optical Property Measurements as a Diagnostic Tool for the Control of Materials Processing"

3:15-4:30
Discussion

2. MATERIALS PROCESSING

1:15-1:35
C. Rey (Intersonics Inc.): " Some Considerations for Various Containerless Positioning Systems and their Science Capabilities"

1:35-1:55
M. Barmatz (Jet Propulsion Laboratory): " High Temperature Acoustic and Hybrid Microwave/Acoustic Levitators for Materials Processing: Progress Report"

1:55-2:15
E.G. Lierke, H. Loeb, and D. Gross (Battelle Institute, Frankfurt): "A New, Simple Electrostatic-Acoustic Hybrid Levitator"

2:15-2:35
W.K. Rhim (Jet Propulsion Laboratory): " Feedback-Controlled Electrostatic and Electromagnetic Sample Positioners"

2:35-2:55
G. Gutt (Jet Propulsion Laboratory): " A Computer Model of the Electrostatic Positioning System"

2:55-3:15
J.K.R. Weber, S. Krishnan, R.A. Schiffman, and P.C. Nordine (Intersonics Inc.): "Containerless Processing of Amorphous Ceramics"

3:15-4:30
Discussion

1:15-1:35
Y. Bayazitoglu (Rice University): " Surface Tension and Viscosity from Oscillations of Viscous Droplets"

1:35-1:55
A.L. Fripp, W.J. Debnam, R.T. Simchick*, and P.G. Barber** (Langley Research Center, *Lockheed Engineering and Sciences Co., and **Longwood College): "Radiographic Instrumentation for DPM Experiments"

1:55-2:15
A. Hmelo, J. Allen, and K.L. D'Amico (Vanderbilt University and Exxon Research): "The Metrology of Spherical Shells using Synchrotron X-Ray Micro Tomography"

2:15-2:35
A.V. Anilkumar and T.G. Wang (Vanderbilt University): "Drop Coalescence Studies"

2:35-2:55
J. Rudnick and M. Barmatz (UCLA and Jet Propulsion Laboratory): "Oscillational Instabilities in Single Mode Acoustic Levitators"

2:55-4:30
Discussion

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15. Supplementary Notes

Mother 326587

16. Abstract

The First Containerless Experimentation in Microgravity Workshop was held January 17–19 in Pasadena, California. The workshop organizers' principal goals were first to provide scientists from academia and from industrial and government research institutions an opportunity to acquaint themselves with the past, current, and future scientific investigations carried out in the Containerless Science programs of the Microgravity Science and Applications Division of the National Aeronautics and Space Administration as well as the European and the Japanese Space Agencies. The second goal was to assess the ongoing technological development program for low gravity containerless experimentation instruments. The third goal was to obtain recommendations concerning rigorous but feasible new scientific and technological initiatives for space experiments using noncontact sample positioning and diagnostics techniques. The specific output of the workshop is an initial set of recommendations for the development of the technical capabilities of future space-based instrumentation. The concensus among the workshop attendees is that there seems to be a strong support for experimental investigations in microgravity and that this support would be strengthened by closer interaction between the user community and the flight equipment developers. Such a cooperation would help ensure that priorities are assigned to scientific returns.

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