Electrostatic Levitation Technology for Thermophysical Properties of Molten Materials

by

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Abstract

Measurements of thermophysical properties of undercooled liquids often require some kind of levitator which isolates samples from container walls. We introduce in this presentation a high temperature/high vacuum electrostatic levitator (HTHVESL) which promises some unique capabilities for the studies of thermophysical properties of molten materials. Although substantial progress has been make in the past several months, this technology is still in the development stage, therefore, in this presentation we only focus to the present state of the HTHVESL(1) and point out other capabilities which might be realized in the near future.

The schematic diagram of the present HTHVESL is shown in Fig. 1, and a photograph of a molten aluminum sample being levitated between a pair of electrodes is show in Fig. 2. The electrode assembly is positioned at the center of the chamber and all the necessary equipment for levitation, heating, and diagnostics are located around the chamber. The chamber is evacuated to a high vacuum for reasons described below. The electrode assembly used in the present levitator has two pairs of side electrodes surrounding the bottom electrode. Damping voltages applied on these side electrodes prevent sample oscillation in the lateral direction.

The HTHVESL constructed at JPL has the following characteristics which may be relevant for the measurements of thermophysical properties:

(i) <u>The ESL requires a certain amount of sample charge and applied electric field</u> in order to exert a positioning force on the sample. For the sample levitation in 1-g laboratory, the gravitational force should be balanced out by the electrostatic force which is given by the product of the sample charge and the applied electric filed. As long as this requirement is met, the ESL can levitate any objects which either a solid or liquid, conductor or insulator at various temperatures and pressures. ESL encounters limitations when the processing environment breaks down before a required electric filed is applied, or if the sample fails to maintain sufficient amount of charge. A high vacuum environment was chosen in our system to avoid dielectric breakdown on one hand and also to maintain a clean processing environment on the other hand. Sufficient sample charge is ensured by the UV photoelectric emission while the sample is in the lower temperature range and by the thermionic emission in the temperature range about 1400K. Instead of using a separate UV source, we are utilizing the UV component of the xenon lamp.

So far we have successfully repeated melting/solidification cycle in a number of sample materials (melting temperatures are shown in parenthesis): In (157 C), Sn (232 C), Bi (271.44 C), Pb (327 C), In 0.69 w% Sb (492.5 C), Al (670 C), Ge (938 C), Cu (1083 C), Ni (1455 C), Zr (1855 C), and some silica based glass-ceramics. In case of zirconium we have completed more than 400 quantitative undercooling experiments. Less understood at this point is the sample-charge loss which seems to occur in the volatile materials as the sample started outgassing with rising temperature. We could not melt zinc due to charge loss since it has high vapor pressure near the melting temperature. The UV intensity we used did not seem to be sufficient to overcome the charge loss.

(ii) Stability of a levitated sample was achieved by a three-dimensional feedback control [2]. The sample position is detected by a pair of position detectors, compared with the preset position, and both position correction and damping signals are generated by a microprocessor. This signal is amplified by high voltage amplifiers before distributed to appropriate electrodes. In a thoroughly outgassed sample the instability is within 0.05 mm in all directions and even better stability is expected if the system is isolated from the floor vibration. Sometimes we have observed jitters in a vertical direction (oscillating center of mass approximately 0.2 mm in amplitude) as the sample outgassed on its way toward the thermionic temperature. However, as soon as the sample temperature reaches thermionic temperature, the jitter subsides and the sample recovers its stability in the higher temperature region.

Sample rotation has been observed time to time. This happened particularly when a nonspherical sample was coupled to the jittery motion during the outgassing period. However, as soon as the sample started to melt, the rotation subsided and the remaining rotation along the vertical axis was usually a fraction of a Hz. Such quick reduction of rotation rate upon melting is not fully understood at this point. If the sample material is well outgassed and is spherical it is less likely to induce rotation. In a separate experiment we have established a method of systematically inducing rotation in a levitated conducting sample. This rotation capability may be useful for the thermal diffusivity measurements, surface tension measurements, and perhaps for the shaping.

Oscillation (with a fixed center of mass) of the levitated melts do not usually come into play unless the volume of the melt is large or the melt has relatively low surface tension. We have observed induced oscillation in a large molten lead (Pb). Apparently the position control frequency might have been in resonance with the characteristic frequency of the lead drop. In a smaller lead drop such oscillations did not appear. This phenomenon may be exploited to design a method by which one can systematically induce specific mode of resonant oscillations and may be able to measure surface tension and viscosity in undercooled melts. Studying dynamic nucleation due to oscillation will be an interesting possibility.

Very little is known at the present time as far as internal flow within a melt is concerned. more studies may be needed in this subject. In case of a quietly levitated water drop holding a constant surface charge we have observed very little flow (probably about 1 mm/sec).

(iii) Sample heating is provided by a 1-kilowatt UV-rich high-pressure xenon arc lamp (ILC, model LX 1000CUV). The radiation produced by the bulb is roughly collimated into a 5 cm beam by a parabolic reflector at the back of the bulb housing. The beam is then focused by a fused quartz lens into a small spot in order to maximize the light flux on the sample. Since the focused spot size could not be reduced to less than 5 mm, a spherical mirror was placed opposite the lens in order to collect most of the xenon light beam that misses the sample. The temperature of a 2.5 mm diameter zirconium sphere could be varied from room temperature to 2270 K by adjusting the iris in front of the xenon lamp. Without the mirror the maximum temperature did not exceed 1750 K. We are in the process of installing a 2-kilowatt xenon lamp in order to process higher melting materials such as niobium. Use of a high power laser will have a clear advantage if one wants to keep the pyrometer free from interference by a broad band heating source. Fig. 3 shows a typical temperature profile of a cooling zirconium melt.

In summary, a high-temperature/high-vacuum electrostatic levitator was constructed for containerless materials processing. It can levitate various molten materials in vacuum with good stability and quiescence. Levitation of 2 to 3 mm samples having density up to 19.3 has been demonstrated. Wide open structure provides easy access to the sample from various diagnostic instruments. The superheating-undercooling-recalescence cycles can be repeated while maintaining good positioning stability. We have melted a number of metals, alloys, semiconductors, and even glass and ceramics. Using a 1 kW xenon lamp, we can extend the present temperature range to include the niobium melting temperature. Since sample heating is decoupled from the levitation, one can also study various low melting materials. Systematic sample rotation around vertical axis has been established and a way to induce systematic sample oscillation looks promising.

The levitator described in this presentation is primarily focused on the Earth-based applications. It can, however, be readily converted for operation in a reduced-g environment. A lesser control force required in space may permit the levitator operating in gaseous environments.

This work was carried out at the Jet Propulsion Laboratory and the California Institute of Technology under contract with the National Aeronautics and Space Administration.

References

1) Won-Kyu Rhim, Sang K. Chung, Daniel Barber, Kin F. Man, Gary Gutt, Aaron Rulison, and R. Erik Spjut, "An Electrostatic Levitator for High Temperature Containerless Materials Processing in 1-g," Rev. Sci. Instr. (submitted).

2) W.K. Rhim, M. Collender, M.T. Hyson, W.T. Simms, and D.D. Elleman, Rev. Sci. Instrum. **56**, 307 (1985).

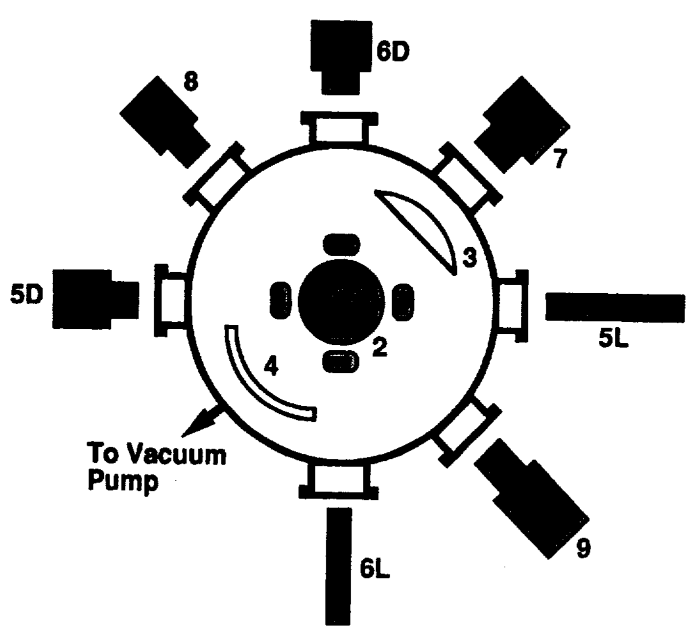


Fig. 1 Schematic diagram of the high temperature-high vacuum electrostatic levitator designed for ground-based applications. 1 is the sample, 2 is the electrode assembly, 3 is the focusing lens, 4 is the spherical reflector, 5D and 6D are the position detectors, 5L and 6L are the He-Ne lasers, 7 is the 1 kW xenon lamp, 8 is the video-camera with a telephoto lens, and 9 is the pyrometer.

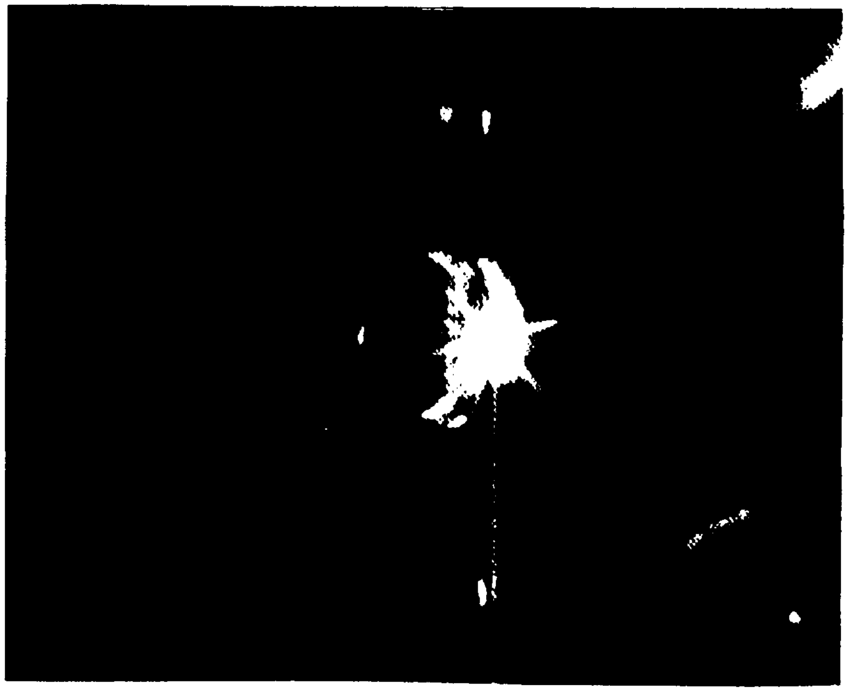


Fig. 2 A 3 mm size molten aluminum being levitated between top and bottom electrodes. The sample was heated by a 1 kW ILC xenon lamp before the spherical mirror was installed.

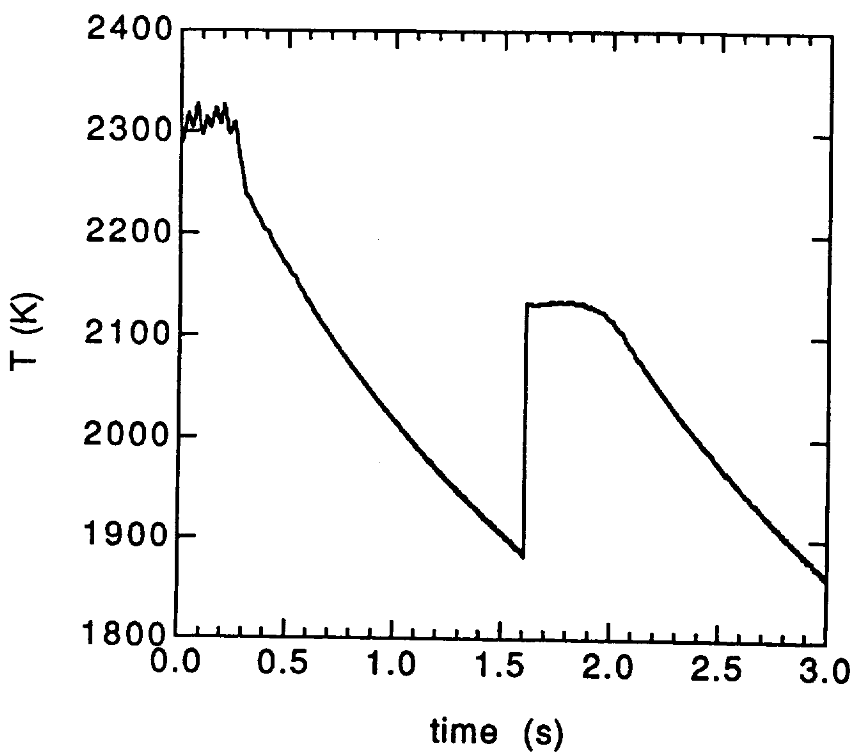


Fig. 3 Temperature versus time as a superheated zirconium drop undergoes radiative cooling.

WHY ELECTROSTATIC SAMPLE POSITIONING ?

- o Open structure - clear sample viewing
- o Quiescent positioning
  - no internal flow, no vibration, no oscillation, or rotation
- o Decoupled sample positioning and heating
- o 1-g as well as reduced-g application
- o Both conducting and nonconducting samples
- o Operable both in vacuum and controlled gas environments

Comparison with Electromagnetic positioner (TEMPUS)

	Electromagnetic	Electrostatic
Sample materials conductors semiconductors insulators	 yes ? no	 yes yes yes
Position stability	passive	active control
Internal flow	can be serious	no internal flow
Heating &Positioning	coupled	decoupled
Openness around the sample	more closed	more opened
Achievable temp.	2500 C (TEMPUS)	2500 C using 2 kw lamp

ACHIEVEMENTS IN FY'92

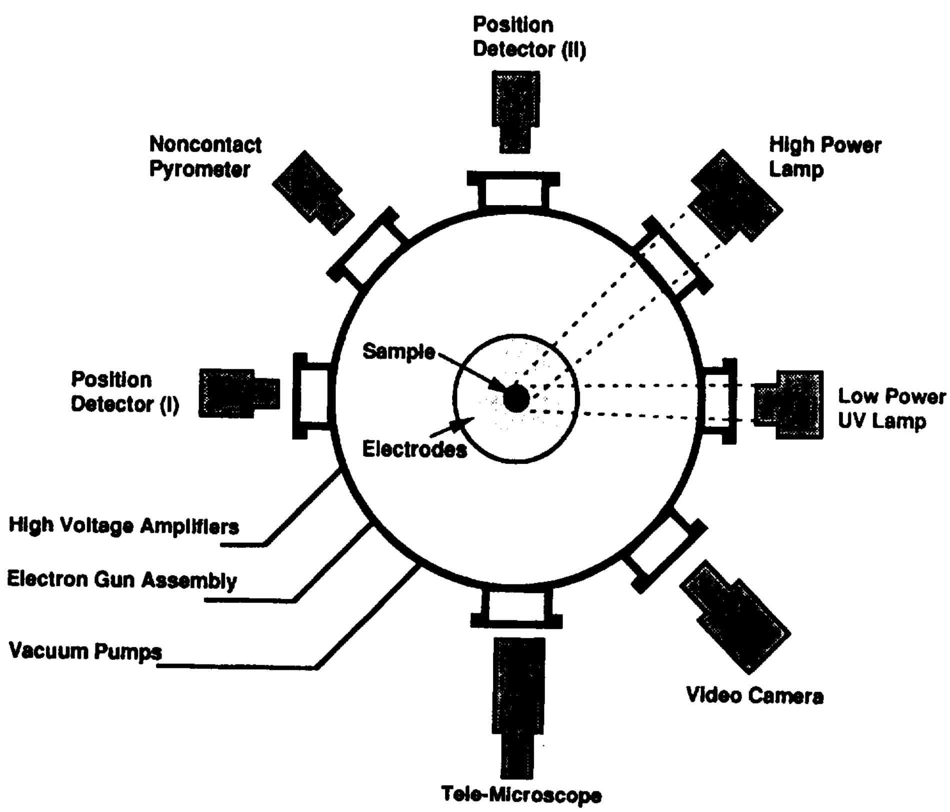
Levitation of high density materials against gravity in vacuum
(~3mm dia. size spheres of In, Pb, Al, Sn, Bi, $In_{0.69wt\%}Sb$, Ge, Cu, Fe, Ni, Ti, and Zr spheres have been levitated in vacuum) $V \alpha \sqrt{r\rho}$

Melting and solidification of high density materials
Melting, undercooling, recalescence and solidification of In(157 C), Sn(232 C), Bi(271.44 C), Pb(327 C), $In_{0.69wt\%}Sb$(492.5 C), Al(670 C), Ge(938C), Cu(1083C), Ni(1455 C), and Zr(1855 C)

Achieved ~2000 C with 1 KW xenon lamp

Developed a sample handler/multi-sample storage system

Sample preparation by e-beam heating(produced outgassed spherical samples)



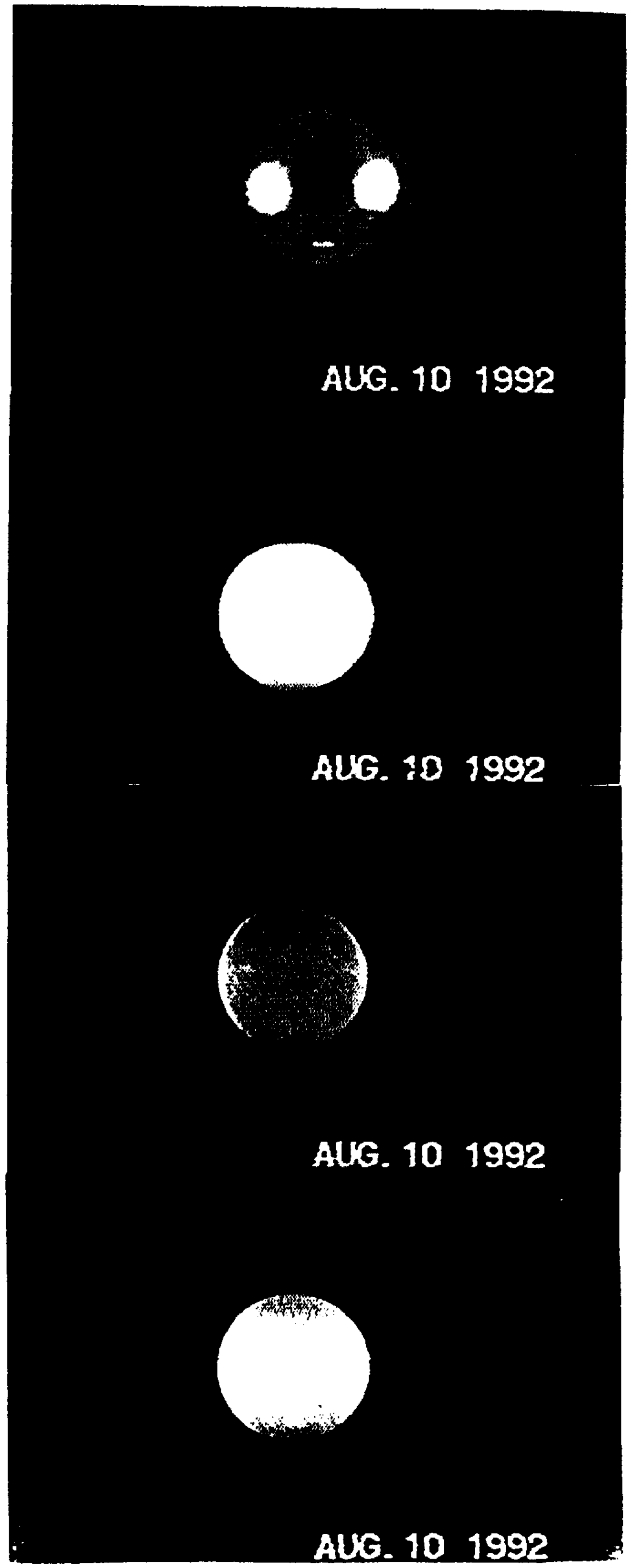
Zirconium
(Tm = 1885 C,
diameter= 2.5 mm)

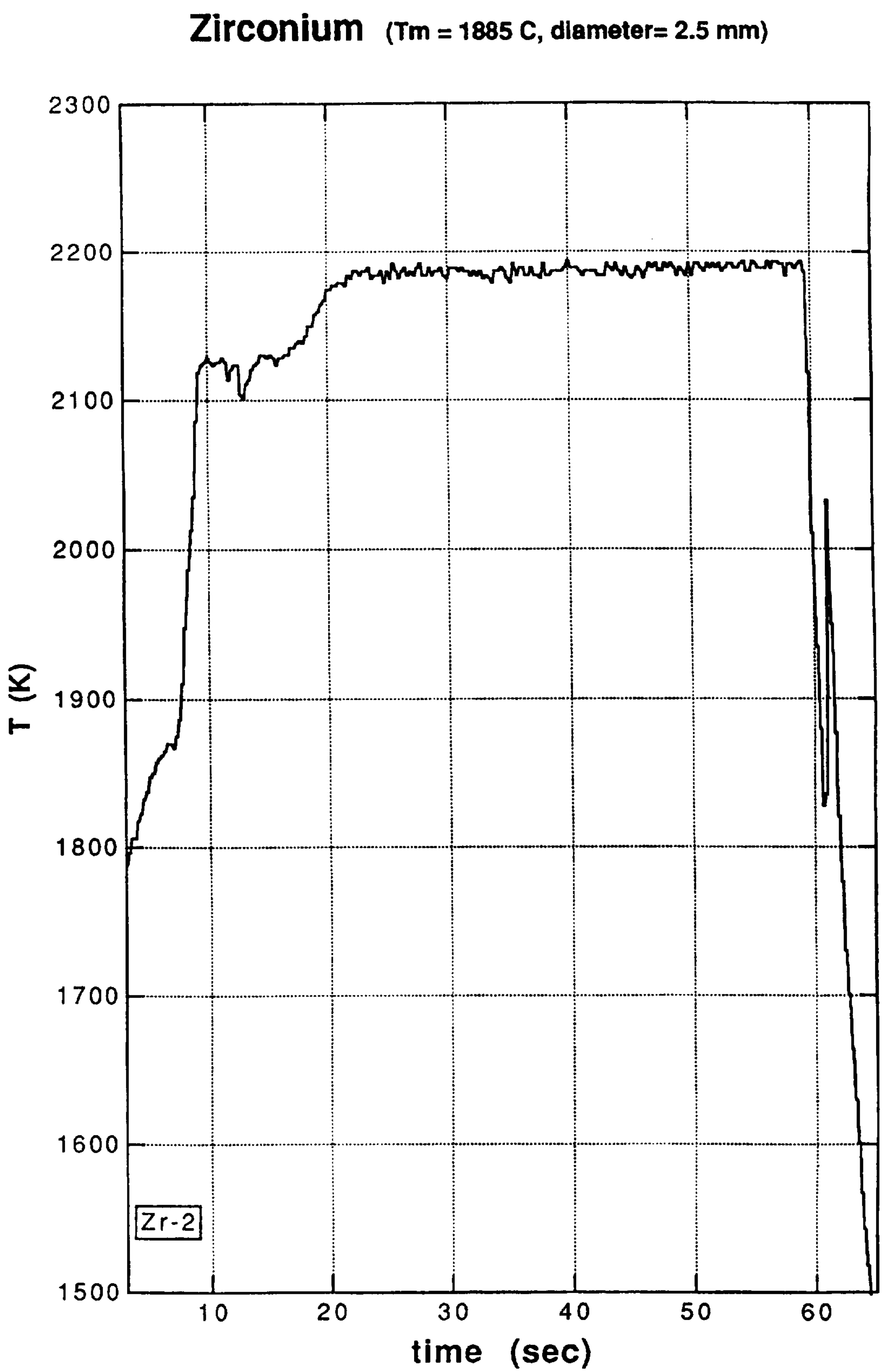
(a) superheated state

(b) Heating lamp off

(c) Undercooled state

(d) Recalescence



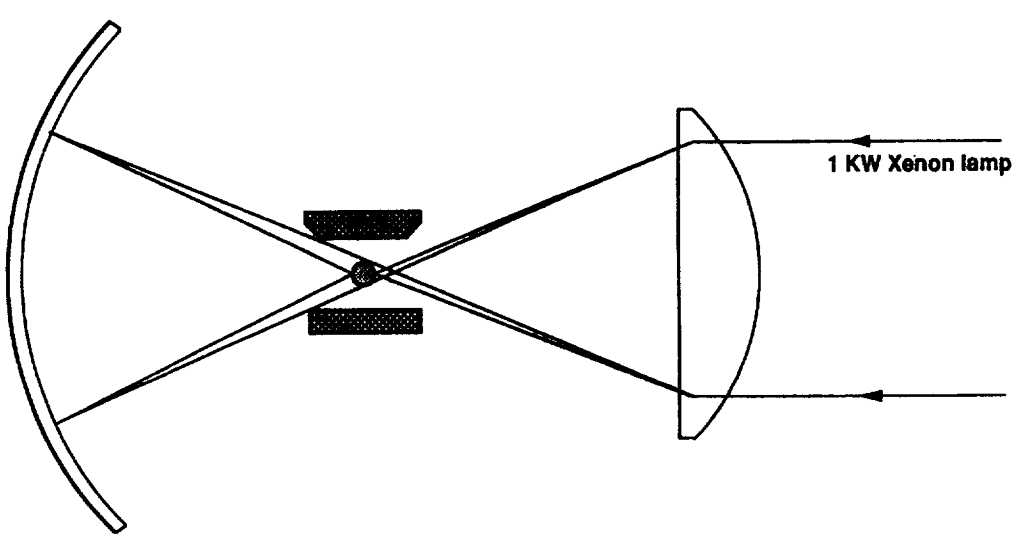


Zirconium (Tm = 1885 C, diameter= 2.5 mm)
2300
2200
2100
2000
1900
1800
1700
1600
1500
T (K)
Zr-2
10
20
30
40
50
60
time (sec)

USER FRIENDLY 1-G LEVITATOR
(capabilities)

	New System	Present system
Pressure:	10(-10) mmHg	10(-7) mmHg
Temp.:	up to 2600 C (2 KW)	up to 2000 C (1 KW)
Pos. Stability:	3-d control (~100 micron)	yes
Preheating /outgassing:	yes	yes
Sample size:	1~3.5 mm dia.	2.5 ~ 3.5 mm dia.
Sample density:	up to 20	up to 12
Sample rotation & vibration:	yes	no
Multi-sample storage and handling :	yes	no
Pyrometer:	DAPP(?)	single color(4Hz)
High speed imaging:	yes (>1000 fr/sec)	30 fr/sec
Noncontact sputtering:	?	no

PRESENT HEATING SYSTEM
(2000 C with 1 KW Xenon lamp)



Electrostatic Containerless Processing Technology

Scientific Objective

LONG RANGE OBJECTIVE is to select appropriate sample materials from metals, alloys, semiconductors, and ceramics and to process in the quiescent environment of electrostatic positioner in one-g and in the KC-135, and to investigate thermophysical properties, kinetics, and resulting microstructures of processed materials.

SHORT TERM OBJECTIVE:
a) Undercoiling and nucleation of metals, alloys, and semiconductors in controlled environment (Active Nucleant in bulk and on surface, and Mechanical Disturbances)

b) Specific heat, Viscosity, Surface tension, and Solidification velocity

c) Comparison with other results
(emulsion method, EM levitation, or drop tube method)

d) Collaboration with universities and other NASA centers.

PANEL DISCUSSION SUMMARY

Friday, October 23, 1992

The Thermophysical Properties of Molten Materials included a wide ranging open discussion and caucus moderated by Mike Robinson, John Berry, Sulekh Jain, Ared Cezairliyan, and Ray Taylor. The salient features of that discussion have been summarized by T.K. Glasgow and inescapably reflect his personal experience and bias.

- The casting industries in the United States are definitely important, representing at least $100 billion annually for metals; direct employment may be estimated at 600,000 persons. Cast products which include shaped castings and mill products, are critical to aerospace, automotive, heavy equipment, and virtually all other manufacturing enterprises. Castings of semiconductors, not included in the above estimates, are equally critical to the electronics industry. All these areas are subject to intense international competition; continuous improvements in quality and efficiency are imperative to maintain market share and our national trade balance.

- The casting industry could be well served by the availability of thermophysical properties of both liquids and solids. Numerical modelling of casting processes has reached a state of maturity such that it is a seriously considered tool. Process simulation is no longer an academic curiosity. For example at least one automobile manufacturer requires vendors of casting equipment to supply a computer model of the casting process with their bids. Computers of moderate cost and sufficient number crunching power are now available to handle the complex problems associated with fluid flow, radiative heat transfer, shrinkage, phase change, stress development, and microstructural evolution. All modelling suffers however from the paucity of reliable thermophysical data. Thus the tool of numerical modelling, so successful in aerodynamics, has not approached its potential in metal casting.

- The problem with data availability and reliability is bad enough for solids, including mold materials, but is much worse for liquids. Only a few universities have the capability of making measurements above 1000° C. Nor have theoretical developments been adequate to provide much help. If well developed theory were available then some properties could be determined from "reverse engineering", i.e. from the well characterized behavior of specific castings. And theory could allow extension of a few data points to a complete curve. Reliable theoretical work thus has extremely practical consequences. The challenge is immense, much greater than we can expect any single company or small consortium to handle. Thermophysical properties data can be a national resource, part of the industrial infrastructure, just like reliable energy, transportation and communication systems.

- Data requirements include liquidus and solidus temperatures, thermal conductivity, thermal diffusivity, specific heat, density, heat of fusion, viscosity, surface tension, species diffusivity, electrical resistivity, and emissivities, all as functions of composition and temperature as appropriate. These data are needed in detail, not for pure elements but for industrially significant alloys, first for a few representative materials. Each industry, e.g, steel, aluminum, nickel, lead, copper, etc., has a very large number of alloys in its total repertoire, but significant understanding could be gained by thorough examination of the most important alloys in each group. The broad attendance at this workshop indicates the importance of thermophysical properties in all the major metal, glass, and semiconductor manufacturing industries.

- A system to disseminate data is already in place, the Center for Information and Numerical Data Analysis and Synthesis (CINDAS), at Purdue University. Facilities exist for measurement of properties at Purdue, the NIST, at Rice University, and at Intersonics Corporation, among others. New techniques for property measurement made possible by fast electronics and inexpensive computers are being created for example at NIST and NASA Lewis. But funding for generation of new data has been very limited. And existing data needs critical examination both against theory and experimentally in casting practice.

- Given the breadth of need and the limited resources likely to be available it is obvious that a focused program plan is required. It is recommended that complete data sets be developed for a few simple alloy systems of importance to each major industrial sector based on the recommendations from that sector. Prudence dictates that measurements be made in more than one laboratory and probably using more than one technique. The measurements must be compared against theory and against well characterized casting practice. The information developed should be shared throughout the appropriate industrial sector

- Because thermophysical property data is a national resource plans must be made for the early domestic dissemination of all information. It may be noted that in the past most such data has been shared freely internationally. But if a competitive advantage is to be gained by generation of reliable process simulation input data then methods for protection of that data should be considered. The data bases maintained at Purdue already include at least one available only on a "need to know" basis for the SDIO program.

- Again because of the breadth of the need for thermophysical property data and the different types of aid required it appears that various responsibilities fall naturally to different organizations. Clearly it is the purview of the National Science Foundation to sponsor research at universities to improve theory and develop new instrumentation for examination of thermophysical material behavior. Given adequate funding it would appear the responsibility of the Department of Commerce to support the country's major industries by sponsoring measurements of commonly needed properties and dissemination of such data. ARPA, especially in its new roles, should also fund property measurement and critical comparison with casting practice for alloys important to advance technologies. The National Laboratories could offer their experience with materials science to perform critical evaluations of data. The DOD and NASA should include property measurement, process simulation, and fully characterized production of all new components, releasing this experience for use by US industry in a planned manner. Industry in general, the professional societies and their members have the obligation to examine the gains possible through use of reliable data in process simulation, to identify opportunities for investment, to alert funding agencies to this need, and to monitor overall program balance and progress.

Agenda

Thermophysical Properties of Molten Materials

Airport Marriott
Cleveland, Ohio
October 22-23, 1992

Wednesday, October 21

6:00 P.M. Registration (Twain Foyer)

Thursday, October 22

7:30 A.M. Registration; Continental Breakfast (Twain Foyer)
8:30 Welcome/Logistics: Tony Overfelt, Auburn University (Riverboat Ballroom)
8:40 Welcome: Thomas Glasgow, NASA Lewis Research Center
8:45 Welcome: Mike Knasel, Ohio Aerospace Institute

9:15-12:00 **Property Needs and Databases**
Chairperson: Sulekh C. Jain, GEAE

9:15 Data Needs for Aerospace Investment Castings
Sulekh C. Jain, General Electric Aircraft Engines
9:45 The Importance of Properties in Modeling
A.F. Giamei, United Technologies Research Center
10:15 Thermophysical Property Issues: Now and for the Future
J.W. Zindel, Ford Motor Company
10:45 BREAK
11:00 Thermophysical Property Sensitivity Effects in Steel Solidification
Tony Overfelt, Auburn University
11:30 Establishment of Computerized Databases on Thermophysical Properties
C.Y. Ho, CINDAS, Purdue University

12:00 Lunch (Becky Thatcher Room)

1:30-5:00 **Experimental Techniques**
Chairperson: Tony Overfelt, Auburn University

1:30 Dyanamic Measurements of Thermophysical Properties of Metals and Alloys at High Temperatures
Ared Cezairliyan, NIST
2:00 Containerless Measurements on Liquids at High Temperatures
Richard Weber, Intersonics, Inc.
2:30 Liquidus Temperature and Optical Property Measurement by Containerless Techniques
Collin Anderson, Intersonics Inc.
3:00 BREAK
3:15 Detection of Velocity in High Temperature Liquid Metals
A.C. Argyropoulos, University of Toronto

3:45	An Overview of the Measurements of Thermophysical Properties and Some Results on Molten Superalloy and Semiconductor Materials R. E. Taylor, TPRL, Purdue University
4:15	Optical Properties and Emissivities of Liquid Metals and Alloys ... Shankar Krishnan, Intersonics Inc.
4:45	Presentations of Opportunity
6:00	Day 1 Closing Remarks, Tony Overfelt, Auburn University
7:00-10:00	**Joint AFS Heat Transfer Committee/Workshop Meeting** (Riverboat Ballroom)

Friday, October 23

7:30 A.M.	Continental Breakfast (Twain Foyer)
8:00-9:30	**Theoretical Predictions** (Riverboat Ballroom) **Chairperson: Ared Cezairliyan, NIST**
8:00	Thermophysical Properties of Simple Liquid Metals: A Brief Review of Theory David G. Stroud, Ohio State University
8:30	A Thermodynamic Approach to Obtain Materials Properties for Engineering Applications Y. Austin Chang, University of Wisconsin - Madison
9:00	An Extended Laser Flash Technique for Thermal Diffusivity Measurement of High Temperature Materials J.M. Khodadadi, Auburn University
9:30	BREAK
9:45	**Panel Discussion** (Riverboat Ballroom) Mike Robinson, PCC Airfoils Inc., Moderator John Berry, University of Alabama - *THEORY* Ared Cezairliyan, NIST Sulekh Jain, GEAE - *NEEDS* Ray Taylor, Purdue TPRL - *EXPERIMENTAL*
12:00	Lunch (Huck Finn Room)
1:30	**Caucus** (Riverboat Ballroom) Discussion Leader: Tony Overfelt, Auburn University
3:00	Adjourn

Thermophysical Properties of Molten Materials Workshop
October 22-23, 1992
Attendees List

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Jacob Zindel	Ford Scientific Labs	Dearborn, MI 48121-2053	313/845-8559

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Dr. Sulekh C. Jain	General Electric Aircraft Engines	Cincinnati, OH
Dr. Jake Zindel	Ford Motor Company	Dearborn, MI
Tim Williams	General Motors	Warren, MI
Dr. Tony Giamei	United Technologies Research	E. Hartford, CT
Dr. John Tu	Howmet Corporation	Whitehall, MI
Mike Robinson	PCC Airfoils, Inc.	Beachwood, OH
Dr. Richard Weber	Intersonics, Inc.	Northbrook, IL
Mike Tims	CTC, Inc.	Johnstown, PA

Government Laboratories

Mr. Tom Glasgow	NASA Lewis Research Center	Cleveland, OH
Dr. Ared Cezairliyan	NIST	Gaithersburg, MD
	MSFC, JPL	
	SANDIA, ORNL, LLNL	

Universities

Dr. Tony Overfelt	Auburn Space Power Institute	Auburn, AL
Prof. John Berry	University of Alabama	Tuscaloosa, AL
Prof. Ray Taylor	TPRL, Purdue University	W. Lafayette, IN
Prof. Austin Chang	University of Wisconsin`	Madison, WI
Prof. Jim Baird	University of Alabama	Huntsville, AL
Dr. C.Y. Ho	CINDAS, Purdue University	W. Lafayette, IN
Prof. Tony Hmelo	Vanderbilt University	Nashville, TN

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13. ABSTRACT ***(Maximum 200 words)***

The role of accurate thermophysical property data in the process design and modeling of solidification processes was the subject of a 2-day workshop held in Cleveland, Ohio on October 22 and 23, 1992. The workshop was sponsored by NASA Lewis Research Center and cosponsored by the National Institute of Standards and Technology (NIST), the Ohio Aerospace Institute, Auburn University, and the Heat Transfer Committee of the American Foundrymen's Society (AFS). Organized by co-chairs Tony Overfelt of Auburn University and Sulekh Jain of General Electric Aircraft Engines, the workshop was attended by 58 engineers and scientists from industry, national laboratories, and universities. The workshop was divided into three sequential sessions dealing with (1) industrial needs and priorities for thermophysical data, (2) experimental capabilities for measuring the necessary data, and (3) theoretical capabilities for predicting the necessary data. In addition, a 2-hour panel discussion of the salient issues was featured as well as a 2-hour caucus that assessed priorities and identified action plans.

14. SUBJECT TERMS
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