MIDWEST RESEARCH INSTITUTE

REPORT

CONTAINERLESS HIGH TEMPERATURE PROPERTY MEASUREMENTS
BY ATOMIC FLUORESCENCE

FINAL TECHNICAL REPORT
March 18, 1987

NASA Grant No. NAG8-465
MRI Project No. 8084-S

Grant Period: March 15, 1984 - December 31, 1986

by

Paul C. Nordine and Robert A. Shiffman

Prepared for

Microgravity Sciences and Applications Program
Contract Monitor: L.B. Gardner/JA64
NASA-George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

CONTAINERLESS HIGH TEMPERATURE PROPERTY MEASUREMENTS
BY ATOMIC FLUORESCENCE

FINAL TECHNICAL REPORT
March 18, 1987

NASA Grant No. NAG8-465
MRI Project No. 8084-S

Grant Period: March 15, 1984 - December 31, 1986

by

Paul C. Nordine and Robert A. Shiffman

PREFACE

This is the final technical report for NASA Grant No. NAG8-465 for research carried out at Midwest Research Institute during the period March 15, 1984 - December 31, 1986. This work was directed by Dr. Paul C. Nordine, Principal Investigator, with the assistance of Dr. Robert A. Schiffman and Dr. Gordon Fujimoto. Drs. Nordine and Shiffman are appropriate contacts concerning technical aspects of the work. Administrative questions should be directed to Dr. W. D. Glauz, Engineering and Materials Sciences Department.

Approved for

MIDWEST RESEARCH INSTITUTE

W. D. Glauz

Dr. W. D. Glauz, Director
Engineering and Materials
    Sciences

March 18, 1987

ii

TABLE OF CONTENTS

I. INTRODUCTION AND SUMMARY

I. <u>INTRODUCTION AND SUMMARY</u>

Containerless processing in space provides an opportunity to greatly increase the temperature at which routine, well controlled processing of materials is possible. The thermodynamic inevitability of container interactions may thus be defeated. Even the best containers would contaminate processed materials to about 1 ppm at 1,500K, and more at higher temperatures. For example, silicon crystals grown from the melt in silica containers contain a few ppm oxygen. On the other hand, less than 0.05 ppm oxygen remains after these crystals are zone refined in a process for which the liquid silicon contacts no foreign material.

This project was aimed to advance the art of containerless high temperature processing and material property measurements. We have developed methods for non-contact suspension, heating, and property measurement for materials at temperatures up to 3,680K, the melting point of tungsten. New, scientifically interesting results have been obtained in Earth-based research. These results and the demonstration of new methods and techniques form a basis for further advances under the low gravity environment of space where containerless conditions are more easily achieved.

Containerless high temperature material property investigations that have been completed in this and our earlier projects include measurements of fluorine/LaB_6 reaction kinetics[1] at 1,000 - 1,500K; optical property measurements on sapphire[2] (Al_2O_3) at temperatures up to the melting point (2,327K); and vapor pressure measurements for LaB_6 at 2,000 - 2,500K[3], for molybdenum up to 2,890K[4] and for tungsten up to 3,680K[4].

Gas jet levitation[5-8], which is applicable to any solid material, and electromagnetic levitation[4] of electrical conductors were used to suspend the materials of interest. Non-contact heating and property measurements were achieved by optical techniques, i.e., laser heating, laser induced fluorescence measurements of vapor concentrations, and optical pyrometry for specimen temperatures.

Accurate temperature measurement requires spectral emittance data. We have analyzed actual emittance data for metals to conclude that this need cannot be met by the methods of multi-color pyrometry.[9] Instead, an instrument is being developed that determines emittance from the measured degree of polarization of light emitted by the specimen.[10] Unlike multi-color pyrometry, this is an exact method which is applicable to liquids which have smooth and specular reflecting surfaces.

Other methods of non-contact temperature measurement have been investigated.[11] Relative concentration measurements for different electronic states of atomic tungsten were measured,[4,11] from which specimen temperature could be calculated. A similar approach is possible using LIF measurement of vapor velocities.[8,11] A third method adapted ideal gas thermometry to very high temperatures by making LIF measurements[11] of Hg-atom concentrations in argon adjacent to hot specimens and in the ambient gas at locations where the gas temperature was known. These techniques are accurate

1

but less precise than optical pyrometry. The materials, temperatures, and ambient gas conditions which may be used are also restricted.

Our more recent work has emphasized research on liquids, for which Earth-based operation is more difficult and Space-based operations will provide research opportunities that cannot be realized on Earth. This work employs electromagnetic (EM) levitation of electrically conducting liquids in combination with laser heating to temperatures above that achieved by just-levitated specimens. This approach avoids the increase of EM forces on the specimen that occurs when EM heating is used to vary temperature and promises more stable operation over a wide temperature range. Laser in-duced fluorescence is used to monitor vapor species vs specimen tempera-ture. These methods have been tested in vaporization studies on solid molybdenum.

The focus of our future high temperature property measurements will include: (1) spectral emittance measurements on pure liquid metals for which a variety of properties (enthalpy, apparent heat capacity, density, etc.) vs apparent temperature are reported in the literature,[12][16] (2) thermo-dynamic properties calculated from component activity measurements on liquid alloys, and (3) thermodynamic properties of solid phases calculated from component activity measurements on liquids in equilibrium with the solid.

II. RESEARCH RESULTS

Much of the research performed under this grant has been reported in the literature.[2][4,8,9,11] Abstracts for papers that have been submitted for publication,[2,4] and for papers in preparation[8,11] are given in Section V.

The published work mentioned above involves containerless high temperature research on solid materials, for which much progress in Earth-based experimentation has been achieved.

In the remainder of this section we describe first the apparatus developed for research in liquids, then the method and theory for specimen emittance measurement. Methods by which these techniques may be used for thermodynamic property measurements on liquids are described in Section III.

Figure 1 presents schematic diagrams of the apparatus for which the identified components are described in the figure caption. The vacuum chamber is pumped by a 1200 l/s cryopump that typically achieves a pressure less than 10^{-9} torr during experiments on hot levitated materials. Vapor concentrations are measured by laser induced fluorescence or mass spectrom-etry, apparent specimen temperature measurements by optical pyrometry, and specimen emittances by a technique to be described later. The EM generator supplies approximately 5 Kw of 450 KHz or 3-8 MHz power for levitation of metals and other materials that are relatively good electrical conductors. The specimen temperature may be adjusted by changing the EM power and by separate laser heating. A feed back power controller adjusts the CO_2 laser power up to 300 watts to achieve constant specimen temperature. A laser
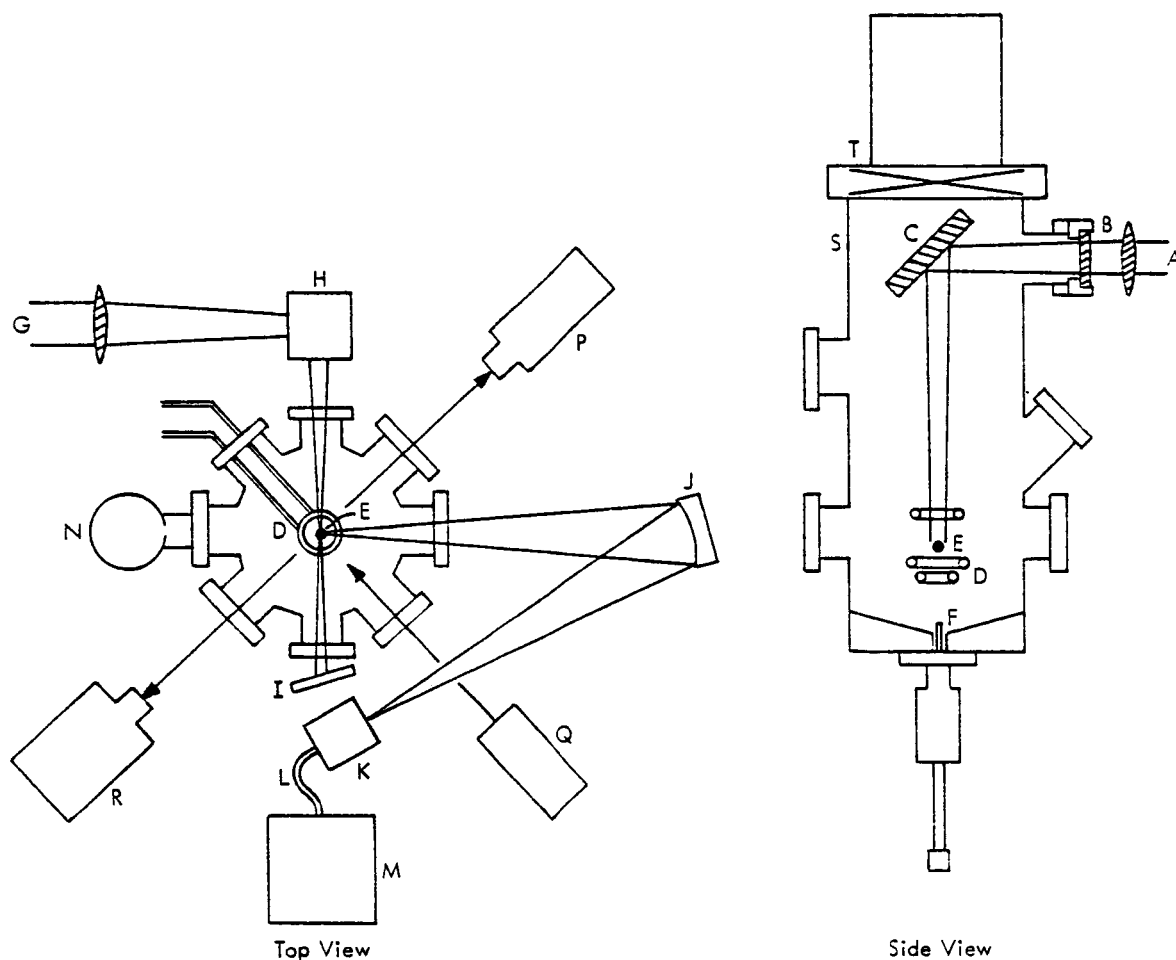
Top View   Side View

Figure 1 - Laser Heated Electromagnetic (EM) Levitation Apparatus for Containerless Experiments. A - CW CO_2 laser beam, up to 300 watts; B - ZnSe lens and window; C - internal laser mirror; D - EM levitation coil and leads to 5 KW, 450 KHz or 3-9 MHz induction generator; E - levitated specimen; F - retractable sample support; G - pulsed dye laser beam; H - laser beam steering device; I - laser beam stop; J - reflective lens for collecting laser induced fluorescence (LIF); K - aperture for spatially resolved LIF intensity measurements; L - optical fiber; M - monochromator, photomultiplier, boxcar averager, and chart recorder; N - quadrupole mass spectrometer; P - automatic optical pyrometer and laser power controller; Q - alignment laser; R - emittance measuring instrument; S - ultra-high vacuum enclosure; T - gate valve and 1200 l/s cryopump.

3

beam reflector is used inside the vacuum system to avoid a line of sight between the laser beam window and the hot specimen so that vapor will not deposit on the laser window. Metal vapors do deposit on the molybdenum reflector without impairing its function.

Solid tungsten was easily levitated and laser heated. LIF experiments on levitated and laser heated solid molybdenum were carried out to demonstrate operation of the apparatus. Liquid aluminum was levitated and heated with mass spectrometric detection of Al-vapor. However, the minimum temperature of aluminum in vacuum experiments was too high to avoid rapid specimen evaporation and aluminum vapor deposition on the optical windows. It is possible that lower temperature experiments can be done on aluminum and aluminum alloys with LIF detection under an inert gas pressure sufficient to cool the sample by thermal conduction. Experiments are planned on liquid zirconium, uranium, silicon, silicon alloys, and possibly liquid tungsten.

Figure 2 is a schematic diagram of the emittance measuring device. A He-Ne laser beam illuminates the levitated specimen at an angle of 90° to the direction at which the emittance measuring instrument collects light. This identifies the point on the specimen surface at which collected light is relected or emitted at an angle of 45° to the surface by a small spot of reflected He-Ne light observed in the emittance instrument. The instrument is adjusted so this spot falls on the instrument aperture. A filter removes the He-Ne light at $\lambda = 632.8$ nm and passes the spectral band at $\lambda = 665$ nm for which emittance is to be measured. A polarizing prism transmits, to separate detectors, the light polarized in a direction normal to (n) and in the plane (or parallel to the plane, p) of incidence for the He-Ne beam which equals the plane of light emitted and collected by the instrument.

Tingwaldt and Magdeburg[10] used this method to obtain emittance measurements on polished tungsten to better than ±0.9%. The method uses a special relation between reflectivities for the polarized components of light reflected at an angle of 45° to any opaque specular surface.

Reflectivities, r, for the two polarization states of emitted light are given by:

$$r_n = [\sin(\theta - \phi)/\sin(\theta + \phi)]^2$$

$$r_p = [\tan(\theta - \phi)/\tan(\theta + \phi)]^2$$

where θ is the angle of incidence and ϕ is the angle of refraction given by:

$$N \sin(\phi) = \sin(\theta)$$

N is the complex index of refraction for the reflecting material. The following simple relation between r_p and r_n is easily shown in the special case that $\theta = 45°$.

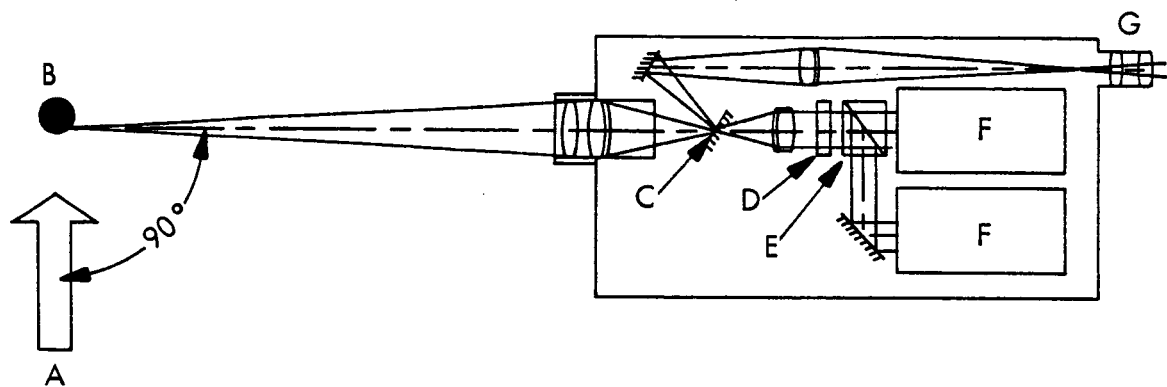$$r_p = (r_n)^2 \quad \text{if } \theta = 45°$$

4

Figure 2 - Emittance Measuring Instrument. A - Alignment laser beam, e.g., 632.8 nm He-Ne laser; B - levitated liquid specimen; C - instrument aperture; D - interference filter to pass 665 nm radiation; E - polarizing prism; F - photomultipliers; G - telescope for focusing and setting of the reflected spot formed by the alignment laser on the instrument aperture. Neutral density filters for measurements at very high temperatures and interference filters for measurements at several wavelengths are not illustrated.

For an opaque material, the emittance, e, and reflectance are related by:

$$r + e = 1,$$

which leads directly to:

$$e_n = 2 - e_p/e_n = 2 - I_p/I_n.$$

The ratio of emitted light intensities, which is measured by the instrument, is substituted for the ratio of emittances in the last part of the equation. Thus, e_n, is easily calculated from the measured intensity ratio.

The instrument is designed to obtain precise values of the emittance from simultaneous intensity measurements for the two polarized components of emitted light. This eliminates the effects on I_p/I_n of small variations in specimen temperature during the intensity measurements. Intensity ratios for normal and 45° emission are separately measured to obtain the normal spectral emittance usually needed for interpretation of optical pyrometry. Calibration of the two detectors is also obtained by measurements normal to the surface where $e_p = e_n$.

Consider now the errors in temperature that would result from ±1% errors in emittance measurement. The true temperature, T, is calculated from the apparent temperature, T_a (which is measured by a pyrometer) and the emittance by use of the following equation:

$$1/T - 1/T_a = (\lambda/C_2) \ln(e)$$

where $\lambda = 0.665$ µm (typically) and $C_2 = 14388$ µm·K. Suppose an alloy and its pure component liquid have equal measured temperatures and a ±2% error exists in the measured emittance ratio for the two liquids. The two temperatures, T_1 and T_2, will then differ due to the emittance error by an amount calculated from:

$$1/T_1 - 1/T_2 = (\lambda/C_2) \ln(0.98)$$

At, say, 2,000K, this equation gives $T_2 - T_1 = ±3.7K$. This accuracy in temperature allows accurate calculation of component activities and thermodynamic properties for the condensed phases.

III. FUTURE WORK

The Si-Ti binary phase diagram is given in Figure 3 to illustrate methods for thermodynamic property measurements. The shaded region of the diagram is the approximate range of temperature and composition in which (1) only a liquid phase occurs and (2) the vapor pressure is less than about 10^5 atm, which is a practical upper limit for vacuum experiments.

At all compositions and temperatures of the liquid, the Ti and/or Si vapor pressures exceed the minimum detection limit by LIF for our apparatus of about 10^9 atm. It is therefore possible to measure temperature and the mass spectrometer or LIF intensities for Ti and/or Si throughout the liquid region that is shaded in the figure.

The activities of the components in the alloy solution are calculated as the alloy to pure component intensity ratios. If, as illustrated in the figure, the alloy measurements extend to the liquidus temperatures, the component activities for the intermediate solid phases are obtained and the thermodynamic properties of the solid phases can be calculated. Experiments may be carried out at a variety of liquid compositions to obtain a complete thermodynamic description of the system.

The Si-Ti phase diagram illustrated in Figure 3 provides large departures from ideal solution behavior represented by the deep eutectic and sufficiently high melting intermediate phases so that the liquidus curves are within the experimentally accessible temperature range. Silicon is a very reactive material to which containerless experimentation is applicable, has a relatively low density which makes EM levitation easier, and becomes a good electrical conductor upon melting. For work on silicon and some silicides, it will be necessary to laser heat the solid to form the electrically conducting liquid before EM levitation can occur.
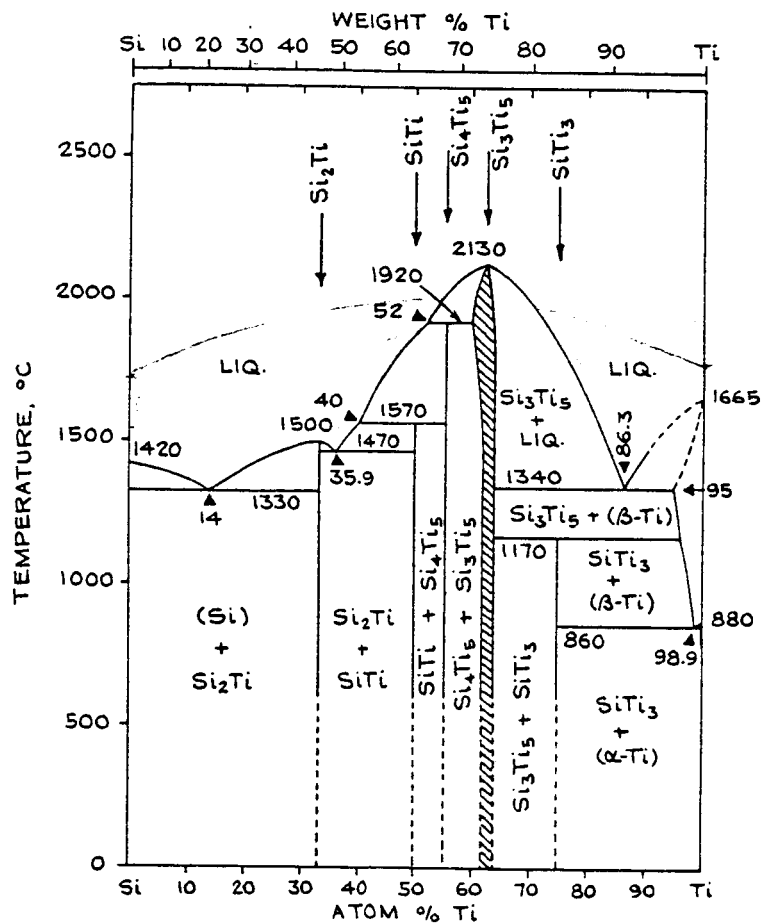
Figure 3 - Si-Ti Phase Diagram.[17] The shaded part labels liquid temperatures and compositions for which the vapor pressures are sufficient for LIF measurements but less than approximately 10^{-5} atm, which is a practical upper limit for experiments in a vacuum.

8

IV. REFERENCES

1. Nordine, P.C., and D. E. Rosner, <u>Fluorine Reactions with Refractory Ceramics</u>, AFWAL-TR-4188 (1982).

2. Nordine, P. C., and R. A. Schiffman, "Containerless Laser Induced Fluorescence Study of Sapphire and Polycrystalline Alumina Evaporation," <u>Advanced Ceramic Materials</u> (to be published).

3. Nordine, P. C., and R. A. Schiffman, "Containerless Laser Induced Fluorescence Study of LaB_6 Evaporation," <u>High Temp. Sci.</u>, 20, 1 (1985).

4. Schiffman, R. A., and P. C. Nordine, "Containerless Study of Metal Evaporation by Laser Induced Fluorescence," R. H. Doremus and P.C. Nordine, eds., <u>Material Processing in the Reduced Gravity Environment of Space</u> (to be be published), Mat. Res. Soc. Symp. Proc., Vol. 87 (1987).

5. Winborne, D. A., P. C. Nordine, D. E. Rosner, and N. F. Marley, "Aerodynamic Levitation Technique for Containerless High Temperature Studies on Isolated Liquid and Solid Samples," <u>Met. Trans.</u>, 7B, 711 (1976).

6. Rosner, D. E., and P. C. Nordine, "Mass Transport Requirements and a New Technique for Studying the Intrinsic Kinetics of High Temperature Gas/Liquid Reactions," <u>Physical Chemistry in Metallurgy, Proceedings of the Darken Conference</u>, R. M. Fisher, R. A. Oriani, and E. T. Turkdogan, eds., U.S. Steel Corp., Monroeville, Pennsylvania (1976), pp. 496-499.

7. Nordine, P. C., and R. M. Atkins, "Aerodynamic Levitation of Laser-Heated Solids in Gas Jets," <u>Rev. Sci. Instrum.</u>, 53, 1456 (1982).

8. Nordine, P. C., and R. A. Schiffman, "Imaging of Supersonic Levitation Jets by Laser Induced Fluorescence from Atomic Mercury." Presented at AIAA 25th Aerospace Sciences Meeting, Reno, Nevada, January 12-15, 1987. Paper in preparation for AIAA Journal.

9. Nordine, P. C., "The Accuracy of Multi-Color Optical Pyrometry," <u>High Temp. Sci.</u>, 21, 97 (1986).

10. Tingwaldt, C. P., and H. Magdeburg, "A New Optical Method for the Determination of Thermodynamic Temperatures of Glowing Metals," <u>Temperature, Its Measurement and Control in Science and Industry</u>, Vol. 3, Pt. 1, Reinhold Publishing Company, New York (1962), pp. 483-486.

11. Schiffman, R. A., and P. C. Nordine, "Temperature Measurements in Containerless Experiments." In preparation. To be presented at the 5th International Conference on High Temperature and Energy-Related Materials, Rome, May 25-29, 1987.

12. Margrave, J. L., "Heat Capacities of Liquid Metals Above 1500K," G. E. Rindone, ed., Materials Processing in the Reduced Gravity Environment of Space, Mat. Res. Soc. Symp. Proc., Vol. 9, pp. 39-42 (1982).

13. Bonnell, D. W., "Property Measurements at High Temperatures - Levitation Calorimetry Studies of Liquid Metals," Ph.D. Thesis, Rice University, Houston, Texas, May 1972.

14. Gathers, G. R., "Dynamic Methods for Investigating Thermophysical Properties of Matter at Very High Temperatures and Pressures," Lawrence Livermore National Laboratory Report UCRL-92054, Livermore, California, 1985; Rep. Prog. Phys. (submitted for publication).

15. Foley, G. M., M. S. Morse, and A. Cezairliyan, "Two-Color Microsecond Pyrometer for 2000 to 6000K," Temperature, Its Measurement and Control in Science and Industry, Vol. 5, Pt. 1, American Institute of Physics, New York, 1982, pp. 447-452.

16. Margrave, J. L., "Estimating Thermodynamic Properties of Solids and Liquids Over Wide Ranges of Temperatures," Proceedings of the Eighth Symposium on Thermophysical Properties, Vol. II: Thermophysical Properties of Solids and of Selected Fluids for Energy Technology, J. V. Sengers, ed., American Society of Mechanical Engineers, New York, 1982, pp. 31-44.

17. Svechnikov, V. N., Yu. A. Kocherzhinskii, L. M. Yupko, O. G. Kulik, and E. A. Shiskin, "Constitution Diagram of the System Titanium-Silicon," DOKL. AKAD. NAUK SSSR, 193, 2 (1970), pp. 393-396.

V. PUBLICATIONS

Papers published, submitted for publication, and in preparation are listed below. Abstracts of the papers submitted and in preparation are given in the appendix.

1. Nordine, P. C., and R. A. Schiffman, "Containerless Laser-Induced Fluorescence Study of LaB_6 Evaporation," High Temp. Sci., 20, 1 (1985).

2. Nordine, P. C., "The Accuracy of Multicolor Optical Pyrometry," High Temp. Sci., 21, 97 (1986).

3. Nordine, P. C., and R. A. Schiffman, "Containerless Laser Induced Fluorescence Study of Vaporization and Optical Properties for Sapphire and Alumina," Advanced Ceramic Materials (accepted for publication).

4. Schiffman, R. A., and P. C. Nordine, "Containerless Study of Metal Evaporation by Laser Induced Fluorescence," Mat. Res. Soc. Symp. Proc. 87, (accepted for publication).

5. Doremus, R. H., and P. C. Nordine, eds. <u>Materials Processing in the Reduced Gravity Environment of Space</u>, Mat. Res. Soc. Symp. Proc. <u>87</u> (to be published).

6. Nordine, P. C., and R. A. Schiffman, "Imaging of Supersonic Levitation Jets by Laser Induced Fluorescence from Atomic Mercury" (paper in preparation).

7. Schiffman, R. A., and P. C. Nordine, "Temperature Measurements in Containerless Experiments" (paper in preparation).

VI. <u>PRESENTATIONS</u>

1. Nordine, P. C., and R. A. Schiffman, "Containerless Laser-Induced Fluorescence Study of the Thermodynamics of Lanthanum Hexaboride," 4th International IUPAC Conference on High Temperature and Energy-Related Materials, Santa Fe, New Mexico, April 1984.

2. Nordine, P. C., and R. A. Schiffman, "Enthalpy of Boron Sublimation," Gordon Research Conference on High Temperature Chemistry, Wolfeboro, New Hampshire, July 1984.

3. Nordine, P. C., "Materials Processing in Space," Midwest Research Institute Annual Meeting, Kansas City, Missouri, May 1985.

4 Borthwick, D., P. C. Nordine, and R. A. Schiffman, "Radiative Life-times and Collisional Conversion Rates for the $^7D^o_1$ and $^7F^o_2$ States of Atomic Tungsten," Midwest High Temperature Chemistry Conference, Ames, Iowa, June 1985.

5. Nordine, P.C., "Containerless Processing," Space Commercialization Consortium Meeting, Washington, D.C., August 1985.

6. Nordine, P.C., "Containerless Processing," Chemical Engineering Seminar, Washington University of St. Louis, St. Louis, Missouri, October 1985.

7. Nordine, P. C., "Containerless Laser Induced Fluorescence Studies of Ceramic Materials," Materials Research Center Seminar, University of Missouri-Rolla, Rolla, Missouri, October 1985.

8. Nordine, P. C., "Strange Things that Happen to Materials in Space," Science Pioneers Symposium, Kansas City, Missouri, November 1985.

9. Nordine, P. C., and R. A. Schiffman, "Containerless Study of High Temperature Reactions by Laser Induced Fluorescence," 115th TMS-AIME Annual Meeting, New Orleans, Louisiana, March 1986.

10. Nordine, P. C., and R. A. Schiffman, "Containerless Study of High Temperature Reactions of Ceramic Materials by Laser-Induced Fluorescence," American Ceramic Society Symposium on Materials Processing in Space, Chicago, Illinois, May 1986.

11

11. Nordine, P. C., R. A. Schiffman, G. Fujimoto, and D. Borthwick, "Laser Induced Fluorescence Study of Tungsten Vapor," Gordon Research Conference on High Temperature Chemistry, Wolfeboro, New Hampshire, July 1986.

12. Nordine, P. C., "Containerless High Temperature Research with Laser Techniques," Chemistry Department Seminar, University of Nebraska, Lincoln, Nebraska, October 1986.

13. Schiffman, R. A., "Laser Fluorescent Property Studies of Evaporating Materials in Containerless Environments," Jet Propulsion Laboratory, Pasadena, California and Lawrence Livermore National Laboratory, Livermore, California, October 1986.

14. Schiffman, R. A., and P. C. Nordine, "Containerless Study of High Temperature Liquids by Laser-Induced Fluorescence," Symposium on Materials Processing in the Reduced Gravity Environment of Space, Materials Research Society Conference, Boston, Massachusetts, December 1986.

15. Nordine, P. C., and R. A. Schiffman, "Imaging of Supersonic Levitation Jets by Laser-Induced Flyorescence from Atomic Mercury", AIAA 25th Aerospace Sciences Meeting, Reno, Nevada, January 1987.

16. Schiffman, R. A., and P. C. Nordine, "Temperature Measurement in Containerless Experiments," to be presented at the 5th International Conference on High Temperature and Energy-Related Materials, Rome, May 1987.

VII. OTHER ACTIVITIES

1. Dr. Schiffman was chairman of the Symposium on Experimental Methods for Microgravity Materials Science Research and the Symposium on Experimental Techniques in High Temperature Science, 115th TMS-AIME Annual Meeting (March 1986).

2. Dr. Schiffman is chairman of the Thermodynamic Data Committee, American Society for Metals, Materials Science Division.

3. Dr. Schiffman is a member of the Alloy Phase Diagram Committee, American Society for Metals, Materials Science Division.

4. Dr. Schiffman is chairman of the 2nd International Symposium on Micro gravity Materials Science Research to be held at the 117th TMS-AIME Annual Meeting, Phoenix, Arizona, March 1988.

5. Dr. Nordine was vice chairman of the Gordon Research Conference on High Temperature Chemistry, Wolfeboro, New Hampshire, July 1986.

6. Dr. Nordine was co-chairman (with R. H. Doremus) of the Symposium on Materials Processing in the Reduced Gravity Environment of Space, Materials Research Society Conference, Boston, Massachusetts, December 1986.

7. Dr. Nordine is chairman of the Gordon Research Conference on High Temperature Chemistry, to be held in 1988.

APPENDIX

ABSTRACTS OF PAPERS SUBMITTED FOR PUBLICATION AND IN PREPARATION

CONTAINERLESS LASER INDUCED FLUORESCENCE STUDY OF VAPORIZATION AND OPTICAL PROPERTIES FOR SAPPHIRE AND ALUMINA

by

Paul C. Nordine and Robert A. Schiffman
Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri 64110
and
Department of Chemical Engineering
Yale University
New Haven, Connecticut

ABSTRACT

Evaporation of aluminum oxide was studied at temperatures between 1800K - 2327K, by laser induced fluorescence (LIF) detection of Al-atom vapor over sapphire and alumina spheres that were levitated in an argon gas jet and heated with a CW CO_2 laser. Apparent specimen temperatures were measured with an optical pyrometer. True temperatures were calculated from the LIF intensity vs temperature measurements and the known temperature dependence of the Al atom vapor concentration in equilibrium with Al_2O_3. Optical properties at the pyrometer wavelength, 0.66 μm, were calculated from the true and apparent temperatures. Similar results were obtained in separate experiments on self-supported 0.025 cm diameter Tyco sapphire filaments.

For Verneuil sapphire, the derived spectral absorption coefficient at λ = 0.66 μm and T = 2327K was k_λ = 0.013 cm^{-1}, in good agreement with the smallest previously measured value. The value of k_λ decreased with temperature in proportion to $\exp(8100/T)$. After heating to temperatures above 2200K, k_λ was unchanged in the temperature range 2000 - 2200K but decreased at lower temperatures such that an increase of k with temperature was observed between 1800 - 2000K. Addition of oxygen to the argon gas flow had no influence on the optical properties of sapphire but increased the spectral emittance of polycrystalline alumina.

Spectral absorption coefficients obtained for Tyco sapphire filaments were approximately 20 times larger than values for Verneuil sapphire. For Tyco sapphire, k_λ decreases with temperature under low pressure, vacuum evaporation conditions and increases with temperature for specimens evaporated under higher ambient pressures where vapor-solid equilibrium is approached.

The effects of impurities and dissolved oxygen on the high temperature optical properties of aluminum oxide are discussed.

CONTAINERLESS STUDY OF METAL EVAPORATION BY LASER INDUCED FLUORESCENCE

ROBERT A. SCHIFFMAN AND PAUL C. NORDINE
Midwest Research Institute, 425 Volker Boulevard, Kansas City, MO 64110

ABSTRACT

Laser induced fluorescence (LIF) detection of atomic vapors was used to study evaporation from electromagnetically levitated and CW CO_2 laser heated molydenum spheres and resistively heated tungsten filaments. Electromagnetic (EM) levitation in combination with laser heating of tungsten, zirconium, and aluminum specimens was also investigated. LIF intensity vs temperature data were obtained for molybdenum ($^7S_3$) atoms and six electronic states of atomic tungsten, at temperatures up to the melting point of each metal. The detected fraction of the emitted radiation was reduced by self-absorption effects at the higher experimental temperatures. Vaporization enthalpies derived from data for which less than half the LIF intensity was self-absorbed were $H_0^o = -636 \pm 24$ kJ/g-mol for Mo and 831 ± 32 kJ/g-mol for W. Space-based applications of EM levitation in combination with radiative heating are discussed.

IMAGING OF SUPERSONIC LEVITATION JETS
BY LASER INDUCED FLUORESCENCE FROM ATOMIC MERCURY

by

Paul C. Nordine and Robert A. Schiffman
Midwest Research Institute
425 Volker Boulevard
Kansas City, MO 64110

ABSTRACT

Gas jet levitation, in combination with radiant heating, allows containerless high temperature experiments on Earth with any solid material that does not sublime at the temperature of interest. When levitation occurs at low ambient pressures, supersonic jet velocities are required to supply a jet momentum flow rate adequate to support the specimen. Supersonic levitation jets were investigated in this work by laser induced fluorescence (LIF) from mercury vapor added to the argon gas flow. LIF via the Hg $^1S_0$ - $^3P_1$ transition was used to obtain photographic images of the jet structure and spatially resolved measurements of the radial, axial, and ambient pressure dependence of gas density in the jets. Jet velocities were also measured by focusing the laser beam to a fixed point in the jet and observing the dependence of image center location on the delay time between pulsed laser excitation and fluorescence detection. Levitation in supersonic free jets occurs at a distance from the nozzle at which the shocks are weak. Levitation stability and height are therefore insensitive to the shock structure because stagnation pressure losses in the shocks are small. Large jet stagnation pressure losses were observed for nozzle pressure ratios less than a critical value that increased with the nozzle Reynold's number. Then stable levitation was not achieved. For conditions where these stagnation pressure losses did not occur, the jet momentum flow rate could be accurately calculated from the velocity and density measurements.

TEMPERATURE MEASUREMENTS IN CONTAINERLESS EXPERIMENTS

Robert A. Schiffman and Paul C. Nordine
Midwest Research Institute
425 Volker Boulevard
Kansas City, Missouri 64110 USA

Research on non-contact temperature measurement in containerless experiments is reported in this paper. The methods are based on Boltzman's, Planck's, and the ideal gas laws. Temperature is derived from intensity measurements for laser induced fluorescence (LIF) from vapor or ambient gaseous species or light emitted by the specimen of interest.

Ideal gas thermometry was adapted to very high temperatures by making spatially resolved LIF measurements of Hg-atom concentrations in argon adjacent to hot alumina and sapphire spheres. The specimens were levitated in a gas jet and heated up to 2327K, the melting point of Al_2O_3, with a CW CO_2 laser. The product of concentration and temperature is nominally constant. Temperatures were calculated with a precision of $\pm$ 2% from the measured high temperature:ambient temperature Hg-atom concentration ratio. Corrections due to thermal (Soret) diffusion effects were shown to be small.

Temperature measurement based on Boltzman's law was investigated by LIF measurments of relative concentrations for different electronic states of tungsten vapor. Temperatures may also be derived from LIF measurements of vapor velocity distributions. Very precise concentration ratio or velocity measurements are required to obtain good precision in the resulting temperatures.

One-color optical pyrometry provides the high precision needed for high temperature property measurements but is accurate only if measured values for the specimen emittance are available. Accurate in-situ emittance measurements can be obtained on liquid specimens by measuring the degree of polarization for light emitted at 45° to the surface. The principles, design, and operation of a device based on this idea and its applications in containerless experiments will be discussed.

DISTRIBUTION LIST

1. Lloyd B. Gardner/JA64 (3 copies)
   George C. Marshall Space Flight Center
   Marshall Space Flight Center, Alabama 35812

2. NASA Scientific and Technical Information Facilities (2 copies)
   P.O. Box 8757
   Baltimore/Washington International Airport, Maryland 21240

3. Dr. Roger Crouch/EN (1 copy)
   NASA Headquarters
   Washington, D.C. 20546