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CONTAINERLESS HIGH TEMPERATURE PROPERTY MEASUREMENTS

FINAL TECHNICAL REPORT
December 6, 1991

NASA Contract No. NAS8-37427

Contract Period: September 21, 1988 - October 31, 1991

by

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for

Microgravity Sciences and Applications Program
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(NASA-CR-184271) CONTAINERLESS HIGH TEMPERATURE PROPERTY MEASUREMENTS Final Report, 21 Sep. 1988 - 31 Oct. 1991 (Intersonics) 14 p CSCL 22A

N92-15196

Unclas
G3/29 0061943

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PREFACE

This is the final technical report for NASA Contract No. NAS8-37427 for research conducted at Intersonics, Incorporated during the period September 21, 1988 - October 31, 1991. It continued work directed by Dr. Paul Nordine in two previous NASA projects, Contract No. NAS8-34383 at Yale University and Grant No. NAG8-465 at Midwest Research Institute. Technical questions may be directed to Dr. Nordine. Administrative questions may be directed to Ms. Betty Brewer, Contracts Manager.

Approved for

INTERSONICS, INCORPORATED

Dr. Charles A. Rey
President

December 6, 1991

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1. INTRODUCTION AND SUMMARY

Containerless processing in the low-gravity environment of space provides the opportunity to increase the temperature at which well controlled processing of and property measurements on materials is possible. The thermodynamic inevitability of container interactions [1] may thus be defeated.

This project was directed towards advancing containerless processing and property measurement techniques for application to materials research at high temperatures in space. The work was carried out in the ground-based containerless processing facility at Intersonics, Incorporated. The experimental work served three major goals, to: (i) apply new techniques to containerless property measurements, (ii) perform property measurements on materials of interest to NASA-supported scientists and (iii) extend the limits of ground-based containerless techniques.

Containerless conditions were achieved by electromagnetic levitation of liquid and solid metals and aerodynamic levitation of solid non-metals. These techniques were used in combination with cw CO_2 laser beam heating. Optical techniques used for non-contact property measurements were optical pyrometry for temperature, laser induced fluorescence for gaseous species concentrations, and laser polarimetry for optical properties and spectral emissivities.

Containerless high temperature material property investigations which have been completed in this work include measurements of the vapor pressure, melting temperature, optical properties, and spectral emissivities of solid boron [2,3]. The reaction of boron with nitrogen was also investigated by laser polarimetric measurement of boron nitride film growth [3]. The optical properties and spectral emissivities were measured [4-7] for solid and liquid silicon, niobium, and zirconium; liquid aluminum and titanium; and liquid Ti-Al alloys of 5-60 atomic percent titanium. Alternative means for non-contact temperature measurement in the absence of material emissivity data were evaluated [8]. In addition, the application of laser induced fluorescence for component activity measurements in electromagnetically levitated liquids was investigated, and the feasibility of a hybrid aerodynamic-electromagnetic levitation technique was demonstrated.

Thermodynamic criteria for cleaning a number of materials were evaluated [5-7,9]. Clean metal surfaces were achieved for Al, Si, Ti, Nb, Zr, and Ti-Al alloys. The purification of Zr [7] was particularly difficult due to the high temperature required to decompose the nitride. The purification of the ambient gas by reaction with metal vapor was also demonstrated and confirmed by laser induced fluorescence (LIF) measurements. Titanium vapor was detected by LIF only if its vaporization rate calculated from vapor pressure data exceeded the measured flow rate of gaseous impurities in the inert gas and from outgassing or leaks.

The research conducted in this project had important implications for several other investigations. The Ti-Al alloy emissivity data allowed Anderson et al. [10-12] to measure the true degree of undercooling that could be achieved in these alloys [11] and to obtain accurate liquidus temperature measurements [12] in this system. Substantial errors in the previous liquidus data were found in their work. Also, in work that was supported by the Air Force and by Los Alamos National Laboratory, it was possible to extend the optical property investigations to measurements on liquid aluminum oxide [13] and levitated liquid uranium metal [14].

This report concludes a sequence of three NASA-sponsored projects in Containerless Property Measurements. The two earlier projects were conducted at Yale University and at Midwest Research Institute. The present work benefited greatly from developments in polarimetric measurement of optical properties that originated at Rice University [15,16], and also included collaborative work with scientists from Vanderbilt University on the Ti-Al system [6,10-12]. NASA-supported work in the area of containerless processing and property measurements is continuing at Intersonics in two new projects funded under the recent NRA on Containerless Microgravity Experimentation.

2. RESEARCH RESULTS

Much of the research performed under this contract has been reported in the literature [2-9,17]. Abstracts for papers in preparation [6,7] are given in the appendix. In addition two sets of feasibility experiments were conducted which are described below.

2.1 Alloy Component Activity Measurements

Feasibility experiments were performed to evaluate the measurement of component activities and vapor pressures by laser induced fluorescence over electromagnetically levitated liquid alloys. Since emissivities of liquids can be measured with high accuracy via polarimetry, their absolute temperatures can be determined. Then activity values can be measured as the LIF intensity ratio over the alloy and its pure components at the same temperature.

Spatially resolved LIF intensity measurements are required in these experiments because the atom concentrations in the gas phase decrease with distance from the specimen surface. True equilibrium vapor pressures and activities are obtained only by extrapolating LIF intensity measurements to the specimen surface. The spatially resolved measurements proved to be impossible in the tightly wound coils necessary to levitate liquid titanium. They could be made in more open EM coil configurations, which might be possible if increased electromagnetic power was available. A 5 kW EM generator was used in this research.

The feasibility of an alternative technique for using more open EM coils was investigated, i.e., the combination of aerodynamic and electromagnetic levitation. This is described in the next section.

2.2 Hybrid Aerodynamic-Electromagnetic Levitation

The simultaneous use of aerodynamic and electromagnetic levitation offers the ability to extend the operating conditions available with either levitation technique. Aerodynamic forces reduce the EM field strength requirements and (i) allow reduced temperatures to be obtained and (ii) more open coil configurations consistent with the requirements of property measurement experiments. At the same time, electromagnetic centering and shaping forces stabilize the aerodynamic levitation of liquids. This has already been demonstrated in early work [18] to investigate electromagnetic heated and aerodynamically levitated liquid aluminum and uranium. The early work used a conically-shaped levitator which limited optical access to the specimen even though the EM coil did not. In the present feasibility study, the use of free jet levitation in combination with a more open EM coil architecture was investigated.

The apparatus is illustrated in Figure 1. Experiments were conducted with 3 mm diameter aluminum spheres. The spheres were levitated in the gas jet and conditions adjusted to center the specimen in the EM coil. Then the EM power was turned on and the specimens were heated.

Results from the feasibility experiments were as follows:

- With a 5 mm i.d. cusp coil, the specimen could not be positioned in the coil gap which was about 3 mm. The specimen levitated either above or below but not within the coil as the levitation gas flow rate was changed. It should be possible to levitate in the gap if the gap distance were increased.

- The specimen could be inserted into a coil of 10 mm i.d. by adjusting the levitation gas flow rate.

- Stable levitation continued as the specimen was EM heated to the melting point. The specimen tended to move down in the coil as its temperature increased and could be raised by decreasing the gas flow rate.

- Rapid oscillations of the solid specimens occurred during heating unless the gas jet and EM coil were carefully centered on the same axis.

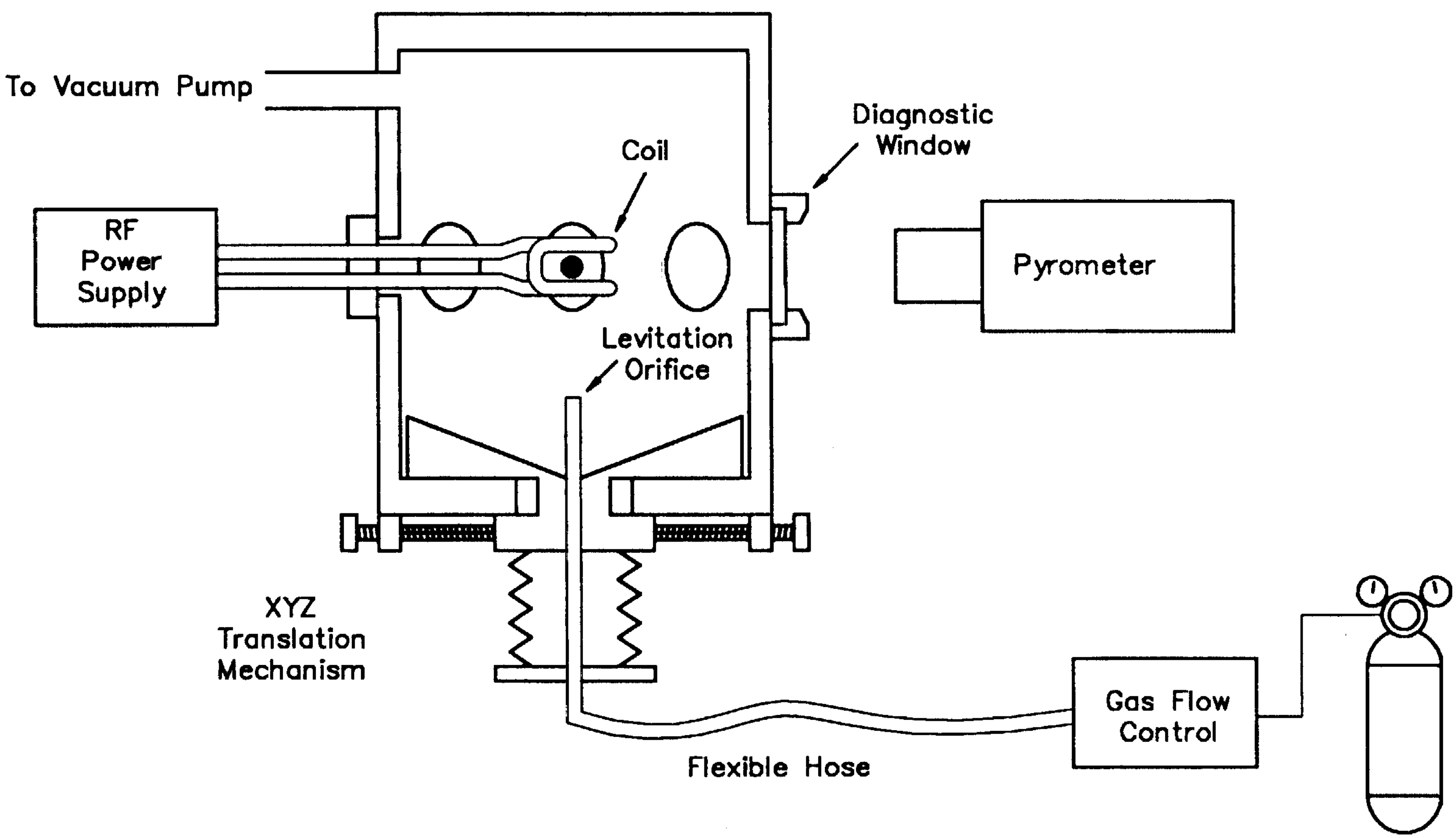

Figure 1 – Hybrid Aerodynamic–Electromagnetic Levitation Apparatus

- Upon melting, specimen stability was maintained for several seconds, it then became unstable. Small changes in the coil separation, centering, and other adjustments influenced the rate at which instabilities developed.

- Most stable levitation and melting behavior was achieved with the most open coils with inside diameters up to 10 mm and coil gaps up to 6 mm.

Hybrid aerodynamic-electromagnetic levitation of liquid metals was demonstrated to provide the basis for new experiments. Two qualities of the technique which extend existing capabilities are: (i) levitation with an open coil architecture, and (ii) extension to lower temperatures of the temperature range over which liquids can be levitated.

Levitation in an open coil will enable spatially resolved LIF measurements to be made up to the specimen surface. When the operating protocols for stable levitation of liquid metals are established, alloy activity and thermodynamic property measurements can be conducted on liquid alloys.

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APPENDIX

ABSTRACTS OF PAPERS IN PREPARATION

Spectral Emissivities and Optical Properties in the Titanium - Aluminum System

S. Krishnan, C.D. Anderson, J.K.R. Weber, P.C. Nordine, W.H. Hofmeister, and R.J. Bayuzick

The spectral emissivities, refractive indices, and extinction coefficients of solid, liquid, and supercooled liquid titanium-aluminum alloys were measured vs temperature by He-Ne laser polarimetry at 632.8 nm. The experiments were carried out under containerless conditions using electromagnetic levitation and heating supplemented by CO_2 laser beam heating. Small temperature dependencies to the normal spectral emissivities were observed for all the alloy compositions that were studied. The undercooled melts exhibited virtually constant spectral emissivities but emissivities varied slightly with temperature for the stable liquids. The optical properties and spectral emissivities of the liquid phase varied smoothly with composition. The spectral emissivities decreased by a factor of six and the optical constants differed two-fold between pure liquid titanium and aluminum. The spectral emissivity vs composition at 1800K showed a maximum near the equiatomic Ti-Al composition and a minimum near pure liquid aluminum. Up to 300K undercooling of the liquid was observed for near-equiatomic compositions.

Spectral Emissivities and Optical Properties of Solid and Liquid Zirconium

S. Krishnan, C.D. Anderson, J.K.R. Weber, P.C. Nordine

The spectral emissivities, refractive indices, and extinction coefficients of solid, liquid, and supercooled liquid zirconium were measured vs temperature by He-Ne laser polarimetry at 632.8 nm. The experiments were carried out under containerless conditions using electromagnetic levitation and heating supplemented by CO_2 laser beam heating. No significant differences in the normal spectral emissivities and optical properties were observed for these materials over the 2000 - 2128K temperature range for the solid and 2000 - 2650K for the liquid. The values e = 0.35, n = 2.85, and k = 4.20 were determined for the emissivity, index of refraction, and extinction coefficient, respectively. The emissivity value agrees with selected results from the recent scientific literature.