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For the Period 1 January 1991 - 31 December 1991

## MICROGRAVITY NUCLEATION AND PARTICLE COAGULATION EXPERIMENTS SUPPORT

Submitted to:

National Aeronautics and Space Administration Goddard Space Flight Center Greenbelt, Maryland 20771

Attention:

Dr. Joseph A. Nuth, III Astrochemistry Branch, Code 691 Laboratory for Extraterrestrial Physics Space Sciences Directorate

Submitted by:

L. U. Lilleleht Associate Professor

F. T. Ferguson Graduate Student Researcher

> J. R. Stephens Matrix, Inc.

SEAS Report No. UVA/528260/CHE92/107 March 1992

SCHOOL OF

ENGINEERING & APPLIED SCIENCE

University of Virginia Thornton Hall Charlottesville, VA 22903

**DEPARTMENT OF** CHEMICAL ENGINEERING

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#### UNIVERSITY OF VIRGINIA School of Engineering and Applied Science

The University of Virginia's School of Engineering and Applied Science has an undergraduate enrollment of approximately 1,500 students with a graduate enrollment of approximately 600. There are 160 faculty members, a majority of whom conduct research in addition to teaching.

Research is a vital part of the educational program and interests parallel academic specialties. These range from the classical engineering disciplines of Chemical, Civil, Electrical, and Mechanical and Aerospace to newer, more specialized fields of Applied Mechanics, Biomedical Engineering, Systems Engineering, Materials Science, Nuclear Engineering and Engineering Physics, Applied Mathematics and Computer Science. Within these disciplines there are well equipped laboratories for conducting highly specialized research. All departments offer the doctorate; Biomedical and Materials Science grant only graduate degrees. In addition, courses in the humanities are offered within the School.

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#### Status Report VII

Grant No. NAG-5-865

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#### I – SUMMARY

Modifications to the nucleation apparatus suggested by our first microgravity flight campaign have been completed. These included a complete "repackaging" of the equipment into three racks along with an improved vapor spout shutter mechanism and additional thermocouples for gas temperature measurements. The "repackaged" apparatus was used in two KC-135 campaigns: one during the week of June 3, 1991 consisting of two flights with Mg and two with Zn, and another series consisting of three flights with Zn during the week of September 23, 1991.

Our effort was focused then on the analysis of these data, including further development of the mathematical models to generate the values of temperature and supersaturation at the observed points of nucleation. The efforts to apply Hale's Scaled Nucleation Theory to our experimental data have met with only limited success, most likely due to still inadequate temperature field determination. We will therefore continue refining the mathematical models of temperature and concentration fields. In addition, we will also be developing new programs capable of accounting for any effects of residual convective currents and make use of the additional gas temperature information supplied by thermocouples within the chamber volume.

Our collaboration with the MARS Center in Naples, Italy has continued with their participation in the June 1991 KC-135 flight campaign and the development of a preliminary particle collector system design with Matrix, Inc. The collector is designed to capture particles from the region of nucleation and condensation, as well as from other parts of the chamber. We hope to have this collector ready to be attached to our microgravity apparatus in time for the next flight campaign.

Joint presentations of our progress on this project and that of our Italian colleagues have been scheduled for two forthcoming international conferences in Europe during April 1992.

### II – INTRODUCTION

The formation and interaction of fine-grained refractory particulates is of interest to diverse fields of science, including astrophysics. Although the homogeneous, vapor phase nucleation of some refractory materials has been investigated in terrestrial laboratories, accurate studies of any interaction and growth of the resulting fine-grained particulates have been greatly hampered by gravity, i.e. by particle settling and thermally driven convection currents. These difficulties, however, can be avoided in a microgravity environment, thus making it possible to produce uniform, "quiescent" suspensions of mono-disperse particulates in low-pressure gas for particle interaction and coagulation studies.

This project is a part of an on-going program initiated by researchers at NASA Goddard Space Flight Center to study the formation and growth of cosmic dust grain analogues under terrestrial as well as microgravity conditions. Its primary scientific objective is to study the homogeneous nucleation of refractory metal vapors and a variety of their oxides among others, while the engineering, and perhaps a more immediate, objective is to develop a system capable of producing mono-disperse, homogeneous suspensions of well-characterized refractory particles for various particle interaction experiments aboard both the Shuttle and the Space Station. Both of these objectives are to be met by a judicious combination of laboratory experiments on the ground and aboard NASA's KC-135 experimental research aircraft.

The University of Virginia Department of Chemical Engineering has provided support to NASA-GSFC researchers in all phases of this Microgravity Nucleation and Particle Coagulation Program since January 1, 1987 under Grant NAG-5-865. The contributions of the University of Virginia and its Matrix, Inc. subcontractor during this reporting period are summarized here.

### III – STATUS OF PROJECT

## A: University of Virginia Effort

Experimental Apparatus and Data from KC-135 Flights. During this reporting period we completed the modifications to the nucleation apparatus mentioned in Status Report VI which were deemed necessary from our first experiences with microgravity test runs in February 1990. These modifications included a complete "repackaging" of the equipment into three racks, installing an improved mechanism for operating the vapor spout shutter, a series of thermocouples stretched diagonally across the interior of the chamber for gas temperature measurements, a timer, and LED's to indicate on the video record when data were actively being acquired.

The "repackaged" apparatus was used to study the condensation of Mg and Zn vapors, each on two flights of our second KC-135 campaign during the week of June 3, 1991. The next or the third flight campaign had been scheduled for the week of September 9, 1991, but was canceled due to a plane malfunction and then rescheduled for the week of September 23, 1991. During that campaign we were able to collect data on three flights, all with Zn. Since then we have devoted our primary efforts on the video data analysis by determining the condensation front location and then applying the mathematical models for temperature and supersaturation ratio determination there.

<u>Modeling of the Nucleation Chamber.</u> One of the goals of this work has been to determine the temperature and the degree of supersaturation at the point of nucleation; first for the various relatively high volatility metals such as Mg, Zn, Pb, and Sn, then for intermediate volatility materials, and eventually for "super-refractory" materials of interest to planetary scientists and astrophysicists. As the temperature and concentration fields cannot be measured directly at every point within the chamber, these fields are derived from a combination of mathematical modeling and actual temperature measurements at locations unlikely to interfere with the nucleation process,

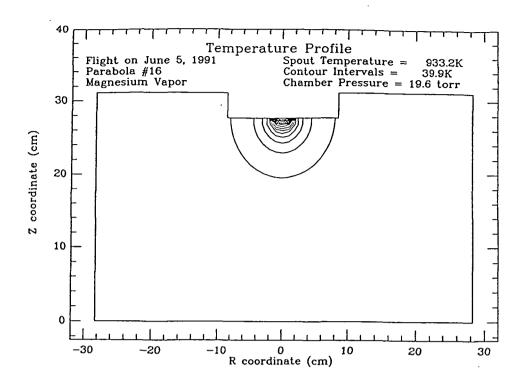


Figure 1: Temperature Profile for Flight on June 5, 1991

usually at chamber walls.

Numerical models for the temperature and concentration fields were developed in August 1990.<sup>1</sup> One of the programs calculates the temperature field within the viewing region of the nucleation chamber from the experimentally measured wall temperatures as boundary conditions. These temperature calculations assume only a two-dimensional heat conduction mechanism and a steady state. The second program starts with the temperature field produced by the first program and computes the transient diffusion of metal vapor into the viewing region. The degree of supersaturation at the observed point of condensation is then calculated from the predicted temperature and concentration at that point.

Figure 1 is a typical plot of the temperature distribution within the viewing region of the chamber. It is based on the experimental surface temperature data collected

<sup>&</sup>lt;sup>1</sup> "Modeling of Transport Processes in a Nucleation Chamber," F.T. Ferguson, Master of Science Thesis, Department of Chemical Engineering, University of Virginia, August 1990.

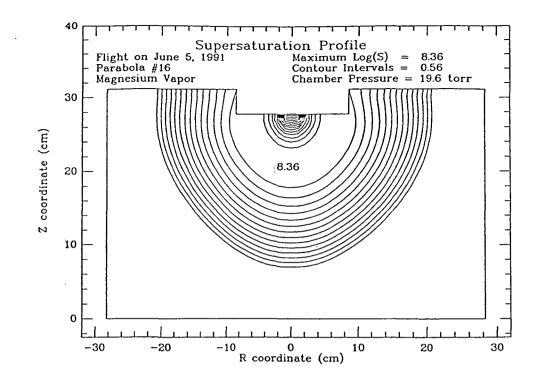


Figure 2: Supersaturation Profile for Flight on June 5, 1991

during a flight, and represents the temperature profile for one parabola only. Figure 2 is the corresponding supersaturation profile for this same parabola. Because of the large values of supersaturation involved with these calculations, the logarithm of the supersaturation ratio S is plotted in this Figure. In order to generate the values of temperature and supersaturation at the point of nucleation, the smoke cloud locations are first determined from the video tape of the experiment. The values of the temperature, T, and supersaturation ratio, S, are then interpolated from data files similar to the ones used to generate Figures 1 and 2.

We have been very interested in applying Hale's Scaled Nucleation Theory to our experimental data since this theory appeared to fit our earlier 1-g experimental data for Ag and SiO rather well.<sup>2</sup> Hale's theory predicts an essentially linear relationship between  $(\ln S)^{2/3}$  and (1/T). Another important feature of this theory is that the ratio

<sup>&</sup>lt;sup>2</sup> "Analysis of Experimental Nucleation Data for Silver and SiO Using Scaled Nucleation Theory," Barbara N. Hale, Paul Kemper, Joseph A. Nuth, J. Chem. Phys. 91 4314 (1989).

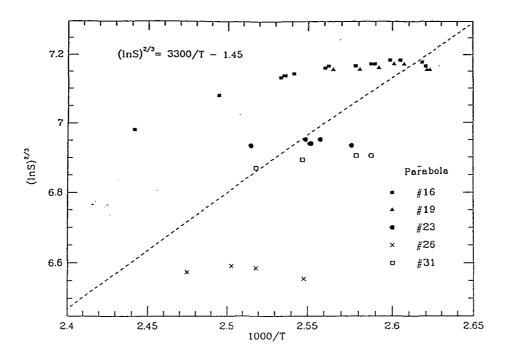


Figure 3:  $(\ln S)^{2/3}$  vs. (1000/T) for Mg

of the slope to the intercept from such a plot should yield the critical temperature of the material.

Figure 3 is a plot of  $(\ln S)^{2/3}$  vs. (1000/T) for 5 parabolas taken from a flight on June 5, 1991 with Mg. Although the best fit through all of the points yields a reasonable value for the critical temperature, approximately 2300K, there is considerable scatter in the data from individual parabolas, as well as from different parabolas. Note, however, that the data cover only a rather narrow range of temperatures.

Figure 4 is a similar plot of  $(\ln S)^{2/3}$  vs. (1000/T) for 3 parabolas with Zn from the September 25, 1991 flight. This plot has also a considerable amount of scatter. The critical temperature derived from these data is unrealistically high.

Even though the best fit lines through Figures 3 and 4 are in general similar, the data from the individual parabolas of the two flights have quite different characteristics. For example, consider the data for parabolas #16 and 19 from Figure 3

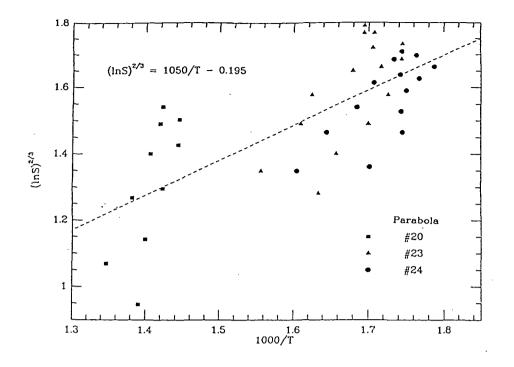


Figure 4:  $(\ln S)^{2/3}$  vs. (1000/T) for Zn

with Mg. The best fit line through these parabolas alone has a shallow slope and a positive intercept. The Zn data from Figure 4 for just parabola #20 give a best fit line with a much steeper slope and a large negative intercept. The most probable cause for such differences between these two cases lies in the calculated temperature profiles. The temperature fields used to develop Figure 3 are similar to the one shown in Figure 1. This temperature profile exhibits a very steep drop in temperature away from the spout area. The data for the zinc plot were based on a more extensive set of thermocouple temperature measurements which suggest that the gradients near the spout are not as steep. The actual temperature field is probably somewhere between these two extremes, and we are currently working to obtain a better representation of this profile.

The apparatus in June and September 1991 flights included the addition of a set of 5 small thermocouples which were suspended across the viewing region of

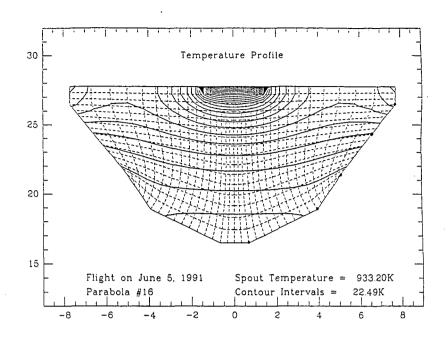


Figure 5: Temperature Profile Using Thermocouples Suspended in Gas

the nucleation chamber to measure gas temperatures. Because of the geometry of the computational gridwork of the current mathematical models, these measured temperatures could not be utilized directly. However, they are useful for comparing the computed temperature field with actual measurements at these five locations. We do intend to modify the temperature model to permit a more direct use of these data in our calculations along with suitable corrections for radiative heat transfer effects, if necessary.

We have already developed a computer program with a different gridwork to calculate the gas temperature in the region enclosed by the suspended thermocouples and chamber walls. Figure 5 is a typical temperature profile calculated using this new conduction program. Unfortunately, the smaller gridwork cannot be used in the diffusion program as yet since the transient concentration field requires a solution of the equations over the entire viewing region of the nucleation chamber.

### B: Matrix, Inc. Effort

During the contract period, John R. Stephens traveled to Johnson Spaceflight Center in Houston, Texas and participated in flight experiments aboard NASA's Reduced Gravity Aircraft in conjunction with other workers involved in the project. In addition to the researchers from the United States, Carlo Mirra and Pasquale Dell'Aversana from the Microgravity Advanced Research and Support (MARS) Center in Naples, Italy took part in these experiments. The trip covered the period from June 2 – 8, 1991. The apparatus to fly on the NASA aircraft was prepared for the flight on June 3, 1991, and we carried out flight experiments on June 4–7. John Stephens returned to Santa Fe on June 8. The flight series went very well with data being obtained on the condensation of Mg vapor to complement the data obtained last year on this metal. We also obtained data on the condensation of Zn in the last two flights of the series.

In addition to the substantial changes and improvements that were incorporated into the experimental system for this flight series, several future improvements were discussed including adding data on temperature and pressure in the chamber to the video data using a video data system. John Stephens is now looking into video annotation hardware that would be suited for this application. In addition to the video annotation, a major improvement to the data collection would be the capability of obtaining quantitative video data on light scattering from particles in the chamber in several colors simultaneously. To do this will require a monochrome video camera and a rotating filter wheel with various color filters or a color video camera that can be run with the automatic gain control (AGC) of the camera turned off. Such cameras are available, for example from Cohu, Inc. There are also VCR's available that allow turning off the AGC. John Stephens is now studying the trade-offs for these two systems.

During this reporting period John Stephens spent several months in Naples, Italy

working with our Italian co-workers to help them develop their microgravity program in conjunction with the current NASA program. In the spring of 1991, Professor Bussoletti, and also Drs. Mirra and Dell'Aversana from the MARS Center in Naples received funding for their program of microgravity condensation studies, which will be coordinated with our work in the United States.

While in Italy we developed the preliminary design for a particle collector system that will be added to the Goddard microgravity apparatus to collect particles from the chamber during and between parabolas. The collector is designed to capture particles from the region of particle nucleation and condensation as well as from other parts of the chamber. The particles will be collected on transmission and scanning electron microscope substrates and also on spectroscopic substrates to allow ultraviolet, visible and infrared analysis of the particles formed in the chamber. The particle collector will be mounted onto the Goddard microgravity system and we hope to fly this on the next KC-135 campaign. The collected particles will be analyzed by the Italian groups.

We also initiated a longer-term effort with the Italian workers to design a second generation apparatus to study particle nucleation and condensation with emphasis on reaching higher temperatures. Higher temperatures would allow us to study the nucleation of carbon and silicate materials that are of astrophysical interest. Two preliminary designs were considered: a high temperature diffusion cloud chamber, and an innovative furnace based on a hemispherical mirror furnace that has been used in sounding rocket flights. The MARS Center will continue to refine these concepts including performing hydrodynamic modeling of the vapor flow in these proposed systems with the aim of having final designs by the end of 1992.

# **IV – FUTURE WORK**

As no additional KC-135 flight campaigns are scheduled for the near future, the emphasis for the remainder of the contract period will be on analyzing the existing data and improving the equipment, particularly the data acquisition system.

We will continue refining the existing mathematical models for the temperature and concentration fields. In addition, we will also be developing new programs capable of accounting for any effects of residual convective currents and make use of the additional gas temperature information supplied by thermocouples in the chamber volume.

We are also planning SEM analyses of dust samples collected from various locations inside the chamber after selected flights in June and September, 1991. Since we do not as yet have the means of sampling during a parabola, these samples are expected to contain particles over a range of ages and therefore will have experienced exposure to a variable number of 0 - 1.8 g cycles. Consequently, it is unlikely that these pictures will yield much information on the size and growth mechanisms in zero gravity. They should, however, give us a good some idea of the shape and size range of particles the sample collector and our next generation video data subsystems which is now being designed should be able to handle.

Joint presentations of our progress on this project and that of our Italian colleagues have been scheduled for two forthcoming international conferences in Europe:

- European International Space Year Conference in Munich, Germany, March 30
  April 4, 1992, and
- VIIIth European Symposium on Materials and Fluid Sciences in Microgravity to be held in Brussels, Belgium, April 12-17, 1992.

### DISTRIBUTION LIST

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