

IJEMS

Iowa Joint Experiment in Microgravity Solidification

John R. Bendle and Steven J. Mashl, Ph.D.
Iowa State University, Ames, Iowa

Richard A. Hardin, Ph.D.
University of Iowa, Iowa City, Iowa

ABSTRACT

The Iowa Joint Experiment in Microgravity Solidification (IJEMS) is a cooperative effort between Iowa State University and the University of Iowa to study the formation of metal-matrix composites in a microgravity environment. Of particular interest is the interaction between the solid/liquid interface and the particles in suspension. The experiment is scheduled to fly on STS-69, Space Shuttle Endeavor on August 3, 1995. This project is unique in its heavy student participation and cooperation between the universities involved.

INTRODUCTION

The Iowa Satellite Project was formed at Iowa State University in 1991 with the single objective of designing and building a micro satellite, ISAT-1, to benefit the state of Iowa. ISAT-1 was designed and an engineering mock-up was built. The project was then put on hold until sufficient funding could be found to begin the construction phase. Then in October of 1994, the opportunity to fly an experiment presented itself. The experiment would be flown in a Smart Can with the Wake Shield Facility II under the Space Vacuum Epitaxy Center (SVEC) at the University of Houston. The necessary funds were available and the decision was made to modify the solidification experiment designed for ISAT-1 for flight in a Smart Can.

The final definition of the science objectives came in December of 1994. Because solidification is such an important part of the processing of composite metal parts it was selected as the focus of the experiment. The previous studies in this specific area are very limited and a great deal can be gained from the study of the solidification phase of metal matrix formation.

IJEMS is scheduled to run on Space Shuttle Endeavor, STS-69 on August 3, 1994. The experiment, consisting of four individual ingots of tin cadmium alloy with nickel particulate, will run over a 850 minute time span. The data and the ingots will be analyzed after the flight. Ground tests will also be performed after the flight to duplicate the conditions during flight. The two will then be compared to reveal what effect the absence of gravity has on the solidification process.

MANAGEMENT APPROACH

From the beginning, one of the goals of the IJEMS project was to promote student involvement. This provided the students with the hands on experience that the classroom lacks while keeping project costs low with the increased participation. The IJEMS team consisted of 2 postdoctorates, 3 graduate students and 15 undergraduate students. The academic backgrounds included aerospace engineering, computer engineering, electrical engineering, mechanical engineering and metallurgical engineering.

One of the most difficult and most beneficial aspects of the project was the multiple university involvement. The majority of the of the systems work was done at Iowa State University (ISU) while the science was done at both the University of Iowa and ISU. Constant communication and frequent meetings were essential to the successful completion of the project. Ultimately, the most encouraging aspect of the cooperative effort was the ability to utilize the strongest attributes of the two universities thereby increasing the teams overall abilities.

IJEMS SCIENTIFIC GOALS

Solidification is an important part of the processing scheme for many materials, and for cast parts, the solidification process can represent the final stage in which the structure and therefore the mechanical properties of a part are determined. As most materials contain particulates, either unintentionally in the form of inclusions, or intentionally as in the case of particle reinforced composites, the interaction between the moving solid-liquid interface and particulates suspended within the liquid is of particular significance. In the processing of metal-matrix composite (MMC) materials a uniform distribution of the reinforcing particles properly incorporated within the metal is critical to the mechanical soundness of the final product. Understanding the interaction of the particles with the solidification front is critical to the production of high quality MMC materials. An interested reader will find that the review by Lloyd (ref. 1) gives an overview of the state of MMC process technology using Al and Mg (aluminum and magnesium) alloys.

The scientific objective of the Iowa Joint Experiment in Microgravity Solidification (IJEMS) is to provide experimental data on the interactions between the solid/liquid (S/L) interface and suspended particulates during the directional solidification (DS) of Sn-Cd alloys under microgravity conditions. These microgravity (or space-based) experiments will then be compared with ground-based experiments. It is envisioned that these experiments will provide data for particle S/L interface interaction models (both for comparison with existing models, and perhaps development of new ones), insight into the effect of convection and buoyancy forces on the particle interface interaction, and should provide data for verification for numerical models of the process currently being developed at the University of Iowa.

A primary goal of this study will be to quantitatively evaluate the phenomena of particle pushing and entrapment. Depending on the conditions of the solidification process, a particle may be pushed or engulfed by the advancing S/L interface. When engulfment occurs, the particle is incorporated directly into the solid. If particles are pushed (as is often the case in multidirectional solidification), they will segregate into the last liquid to freeze. If this occurs, the locally high concentration of small particles behaves as a single large defect which can serve as a site for crack nucleation, potentially leading to failure.

Multiple, dissenting theories exist on the causes for pushing/entrapment behavior. A review of eight of these theoretical models is given by Asthana and Tewari (ref. 2), and compilations of papers on the field can be found in references 3 and 4. It is hoped that the data gathered through this research will provide insight into the pushing/entrapment phenomena, and clarify the accuracy of these existing theories.

Basics of the Theory for Particle S/L Interface Interaction

Central to the theoretical models of particle S/L interface models is the concept of a critical velocity V_c which is the velocity at which the transition from pushing to entrapment occurs. Of particular interest in this work, will be the variation in V_c which may occur as the experimental conditions are varied. Additionally, any preferential location of the particles within the individual phases present will be worthy of note. Also, the experimental evidence suggests several modes of particle-front interaction (ref. 2) such as no entrapment at all, entrapment only after a period of pushing, and instant engulfment. Past experimental evidence shows that higher solidification front velocities are necessary to capture finer particles. Also, evidence to date suggests that V_c is dependent not only on the physical properties of the liquid melt, but also on the geometric and physical properties of the particle. The interested reader is directed to ref. 2 for the details. A summarized list of specific factors which have been proposed and shown to effect pushing/entrapment behavior is given below:

- solid-liquid, solid-particle and particle-liquid interfacial surface energies
- the solid/liquid interface velocity, the value of V_c
- buoyancy forces acting upon the particle
- thermal conductivity of the particle and the liquid
- convective flow within the liquid
- particle size
- temperature gradient at the interface

· interface morphology

Two of these factors, buoyancy effects and convective flow will be eliminated or at least greatly reduced in the microgravity environment aboard the space shuttle. The minimization of these factors should allow a more accurate determination of the role of the remaining parameters in particle S/L interface interactions. Ultimately, the results of the space based experiments will be compared to identical ground based experiments in order to quantify the effects of microgravity. Generally, a clear transition between pushing and engulfment is blurred somewhat by these many influences on the process, making this a research area where additional work can make technological improvements possible if these many effects can be properly accounted for and predicted.

In the IJEMS experiments, four ingots will be melted and directionally solidified. There will be two rates of solidification used by varying thermal boundary conditions, and the ingots will be (two each) of a eutectic and off-eutectic composition. Two samples of each composition will undergo a given rate of solidification to give the four experimental runs to be made by the IJEMS payload. By varying both alloy composition and thermal boundary conditions, the particle interaction with different interface morphologies can be explored. Another possible variation not explored in the current IJEMS experiment is to examine the effect of interfacial energy through varying the type of particles used. Only Ni particles were used in the IJEMS tests. In the IJEMS a combination of a range of particle sizes, along with the variation in interface velocity which will occur as the fraction of metal solidified increases, and should provide data on pushing versus entrapment over a range of conditions.

Experimental Apparatus and Procedures

The space-based portion of this work will be performed within a canister aboard the space shuttle in which four directional solidification modules will be mounted. Each module holds a cylindrical crucible containing an ingot of Tin-Cadmium (Sn-Cd) alloy + particles as shown in Figure 1. Foil heaters (MINCO model HK5450) act to both melt the ingot and control the thermal conditions during solidification. As noted from Figure 1, there is a heater in the base used to both melt and control solidification. Also note the large "Melt Heater" wrapped about the bulk of the crucible; it is employed only in the melt cycle of the testing. Finally there is a narrow foil heater wrapped around the crucible opposite the base heater which is used to control the rate of solidification along the length of the sample and also to melt it.

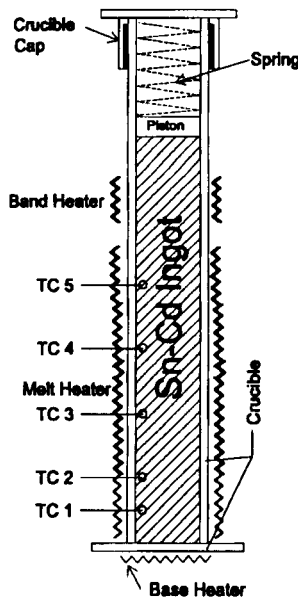


Figure 1 Schematic of the IJEMS experiment crucible

Thermocouples are placed on the "Base" and "Band" heaters to directly monitor and control them using on-off control, and the large "Melt Heater" is controlled using a thermocouple (TC 4) in the ingot. Five sheathed thermocouples (TC 1 through 5) are inserted through the crucible to approximately 1 to 2 mm into the sample, and sealed and fastened there using Omega high temperature epoxy. TC 1 through 5 serve to monitor conditions during solidification, and to track the position of the S/L interface over time. These thermocouples make up the primary experimental data recorded by the on-board computer, and can be used (following some corrections for heat loss) to determine the speed of the S/L interface. Following solidification on one of the samples, the heaters are turned off but temperature data is taken as the crucible cools. This "cool down" data will provide additional data for the heat loss corrections. During solidification, the thermal conditions in all modules are maintained so that solidification occurs directionally along the axis of the crucible as shown in Figure 2.

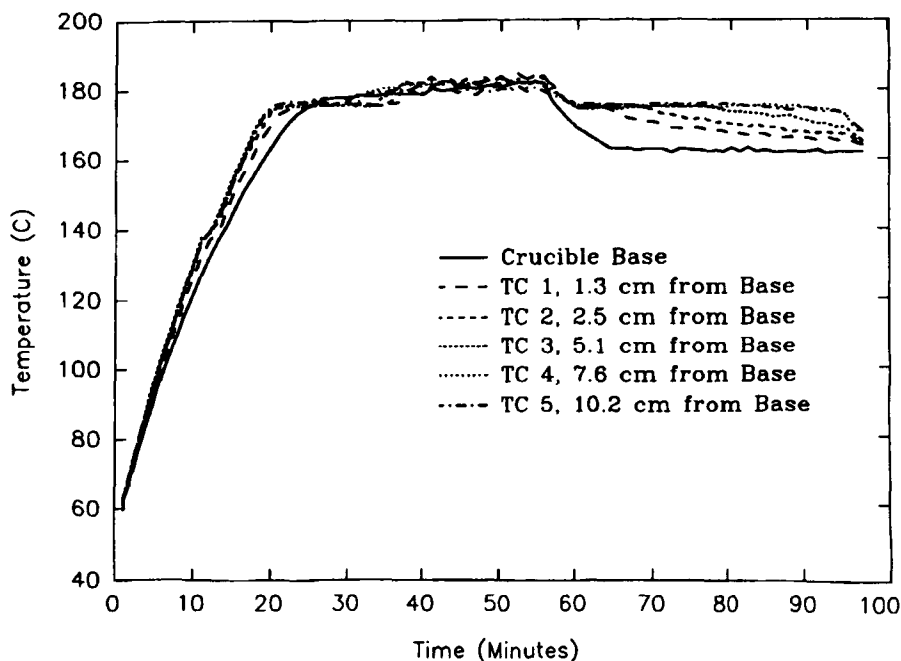


Figure 2 Temperature versus time for a mock-up test of the IJEMS experiment (refer to Figure 1 for positions of thermocouples, TCs)

Alloys of Sn and Cd were chosen for these tests because of their relatively low melting temperature (eutectic liquidus temperature T_L of 176 C, and the off-eutectic T_L used was 179 C). One reason for this is that the heaters used have a maximum temperature rating of 200 C, and other considerations were limitations on power, number of tests desired, and time constraints for the total duration of the experiment during the mission. Two of the ingots will be of the eutectic composition (67 wt.% Sn - 33 wt.% Cd) containing Nickel particles (from 1 μ m to 100 μ m in size). The thermal conditions are controlled in order to cause the solidification front to be planar in nature, characterized by alternating Cd and β -phase lamellae oriented normal to the growth front. The two remaining modules hold ingots having a slightly tin-rich, off-eutectic composition (70 wt.% Sn- 30 wt.% Cd) which also contain Nickel particles. Solidification of these ingots will be characterized by the growth of proeutectic β dendrites the remaining interdendritic liquid freezing to form eutectic.

Referring to Figure 1, the crucibles (made of 304 stainless steel tubes) were assembled under a vacuum with an undersized ingot being slid into the crucible with care taken not to damage the protruding thermocouples. The spring loaded piston was then assembled and the screw cap fastened down. Assembly under a vacuum eliminated air from being trapped inside the crucible (this may have led to Marangoni convection), and the spring loaded piston was carefully designed to accommodate the change in shape of the sample during melting or the metal floating about.

Given in Figure 2 is representative data for the IJEMS experiment for a laboratory mock-up with a eutectic sample. The two stages (melt and solidify) of the experiment are shown in Figure 2. First the melt stage (from 0 to 55 minutes) consists of a heat up period (0 to 20 minutes), the melting period (20 to about 35 minutes), and the hold period (shown here from 35 to 55 minutes to allow the sample to reach a superheated temperature ≈ 183 C that is adequately uniform prior to solidification). The second major stage is the solidification stage which is shown in Figure 2 from 55 to 95 minutes. Note that from 55 to 65 minutes the base heater is off as its set point is switched to 161 C, and the base heater kicks in again as its temperature hits 161 C. An improved control system would have eliminated oscillations in the base heater temperature. Fortunately, the observable effects of this were damped out to below the random variations and accuracy of the measurement system used. By setting the on-off band of the control system to a very small number some of this can be alleviated through rapid on-off cycling of the heaters.

Note that in the test case presented in Figure 2, TC 1, TC 2, and TC 3 break away from the eutectic temperature (indicating the passing of the S/L front) in a clearly directional fashion. Also, in this case, note that TC 4 and 5 drop below the eutectic temperature together which means the far end of the ingot is solidifying in an omnidirectional manner. This difficulty was overcome by using the "Band Heater" shown in Figure 1, and picking its set point carefully. In doing so, the rate of solidification and its directionality can be controlled better. As mentioned previously, there will be two rates of solidification used in the IJEMS; a relatively fast rate and a fairly slow one. The set points (temperatures) and duration times for the stages for the IJEMS experiments are given below:

Stage and Duration	Melt Heater	Base Heater	Band Heater
Melt (all tests), 70 min	183 C	183 C	183 C
Solidify (fast), 65 min	Off	163 C	177 C
Solidify (slow), 200 min	Off	167 C	192 C

These stages will then be duplicated in the matching ground-based experiments to acquire comparative data.

Smart Can INTERFACE

The Smart Can that IJEMS is flown in is modeled after the Get Away Special Can but has a few additional features. The aluminum can has an inside diameter of 20.0 inches and an inside height of 31.25 inches resulting in a 5.68 cubic foot internal volume. It will hold a maximum payload mass of 200 pounds. The can has a power feed capable of supplying a constant 20 amps at 28 volts to the experiment contained within. The can is rated to supply nominal power of 400 watts with a 1,400 watt maximum. It is also capable of supplying command and real-time telemetry via the Shuttle downlink. The can is fixed to the cross bay carrier that supports the Wake Shield Facility II.

IJEMS takes advantage of the power feed, making an internal power source unnecessary. The real-time data line is not used but the associated connector in the can lid is used for ground testing. The IJEMS structure is bolted to the top of the can with 24 bolts while the other end is supported in the can by 3 rubber bumpers. The environment in the can is a half an atmosphere of dry nitrogen.

IJEMS SYSTEMS

The systems portion of the project can be broken up into three major parts; structure, power and command and data handling. The systems had to be designed around the Smart Can and its environment while still making the science objectives possible.

Structure

The structure went through numerous iterations before the final design was reached. The factors under consideration were packing configuration, weight, and strength. The experiment had to fit in a manner that kept

the ingot heating elements away from the more sensitive electronics such as the computer. This also meant minimizing the conductive paths between the heaters and the electronics. The weight of the structure was not minimized but it was a contributing design factor to ensure that the maximum payload mass was not exceeded. The structure also had to be designed to withstand a 17g load factor, a 3 axis, 1 minute per axis, 9g rms random vibration test and a 2000 Hz swept sine vibration test. Once the design was finalized, its behavior was modeled using the ANSYS finite element program. The model showed a more than adequate ability to withstand the 17g load factor and the vibration test.

Next, the hardware phase began. All of the major components of the structure are made of 6061 aluminum, chosen for its light weight and high strength. The structure is made of two end plates. The top plate is 1/4 inch thick and has the 24 mounting holes milled in it. The bottom plate is 1/8 inch thick and has 3 rubber bumpers fixed to it. The top and bottom plate are connected by a plus shaped divider made of two intersecting plates, also 1/8 inch thick (figure 3). One of the dividers has a hole in it for the electronics box to pass through. All of these parts are connected to one another by 1 inch angle aluminum. Originally the angles were to be fastened using rivets but a miscalculation occurred and the holes were milled 1/16 of an inch larger than they should have been. There were no rivets the right size to fit the new holes. This led to the use of bolts with locking nuts at the fasteners. This increased the already conservative factor of safety.

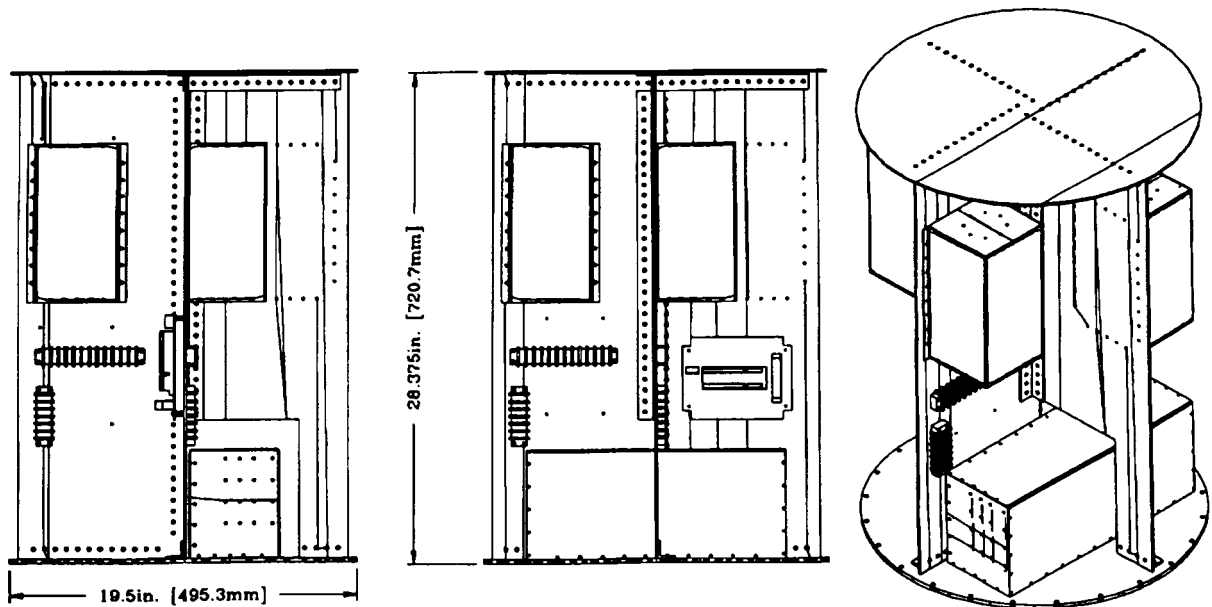


Figure 3 AutoCad Generated Assembly of IJEMS

Once completed, the structure underwent numerous tests to prove its integrity. The bare structure was first fitted with accelerometers and then spot tested with an impact hammer. The results yielded resonant frequencies almost exactly the same as those predicted in the finite element model. Next the fully loaded structure was tested on a shake table in all 3 axes for 1 minute per axis at 9g rms. The structure survived the test with only a minor complication. A few of the bolts that hold the experiment to the structure shook loose because no lock-tite had been applied. The structure fared well in the swept sine vibration test. Finally, once integrated into the Smart Can another set of vibration tests was run to determine if the characteristics of the combined modes would be problematic. A 3 axis, 30 second per axis, 7g rms vibration test was run along with a swept sine vibration test. Again the only problem was that 10 screws that fasten the experiment to the structure shook free during the vibration test because no lock tite was applied. Lock tite was applied after the test so the problem should not reoccur.

Power

The power supplied to the can from the cross bay carrier is in the form of four separately fused lines, each

at 5 amps and 28 volts. A custom power distribution was designed and built. Two of the lines pass through emi filters before the power distribution board and the other two and go directly to the board. On the board each line has triple redundant 120 C thermal fuses. The filtered lines then combine go into a DC to DC converter which outputs +5 and +/-12 volts to supply power to the electronics. The unfiltered lines combine and lead into a solid state relay backplane which supplies power to the heaters at 28 volts. that melt the ingots.

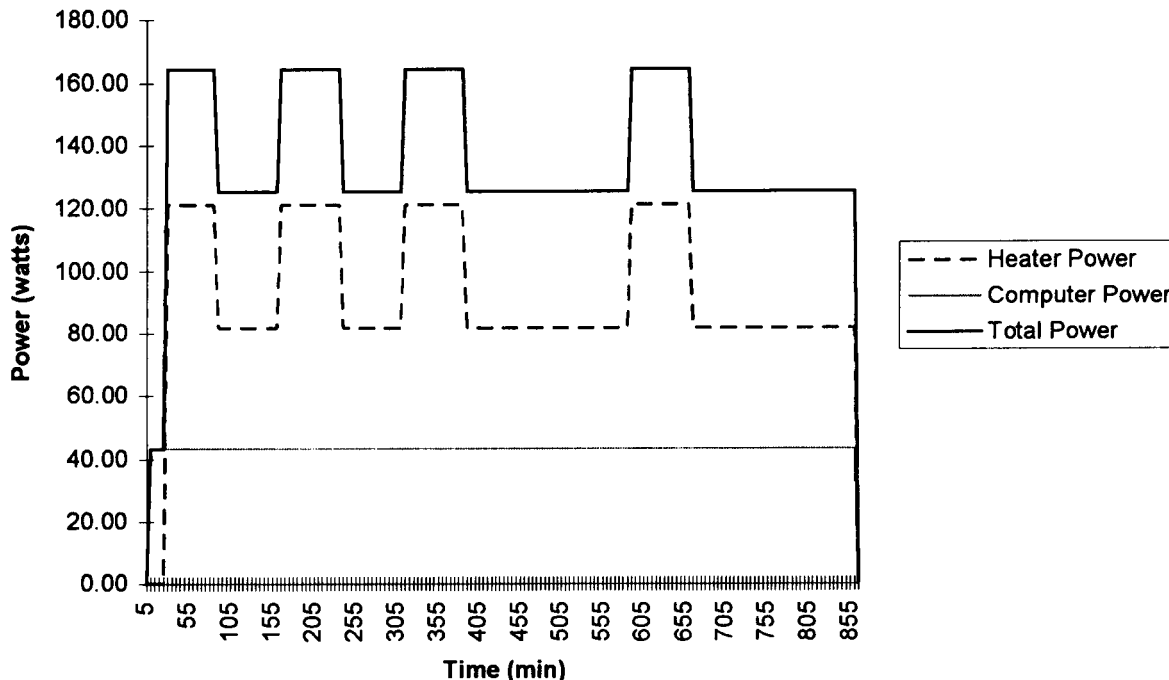


Figure 4 IJEMS Power Budget

Because IJEMS uses an external power source it was necessary to carefully budget the amount of power it consumed throughout the experiment. The power draw not only affects the experiment but also the Wake Shield Facility II because the power is drawn from the Wake Shield buss. It was therefore necessary to carefully schedule the operation of the experiment components and come up with a time dependent power budget (figure 4). The most critical concern was the operation of the heaters because they draw far more power than the electronics. The electronics draw 44 watts of power while the heaters draw as much as 122 watts of power. With a maximum combined power draw of 166 watts, the experiment draws far less power than the cross bay carrier is capable of supplying. The total power drawn over the 850 minutes is 1,941 watts.

Command and Data Handling

The command and data handling (CDH) system was the most complex of all the systems. The CDH system is comprised of a computer, a digital I/O board, two A/D boards, a four slot backplane, a solid state relay backplane and two screw terminals. The computer is a 486SLC single board with 2 MB of ram and 3 MB of onboard flash RAM. The digital I/O board is a 24 channel TTL compatible board. The A/D boards are 16 channel, 15 bit boards with built in cold junction compensators. The solid state relay backplane is a buffered 24 position backplane. The screw terminals are standard 16 position terminals.

The system was designed to be completely autonomous. When the power is turned on the computer boots up. The first action the computer does is a system test. The computer checks for open thermocouples and makes a record of any openings. Then the computer performs a controlled melt of each cell as described earlier, storing the critical thermocouple data in the flash drive. Once the four cells have melted and solidified the computer shuts itself down. In the event of a power interrupt or shut down at some point during the experiment the computer resets to its initial state. If power is reapplied, the system will run as before, thus remelting any ingots that have already

been melted. There for it becomes necessary to determine whether it is beneficial to reapply power or not. This is a time based decision so it is required that someone be present to make that decision during the entire time that the experiment is running.

The CDH system encountered a problem. All of the data from the experiment is stored in the 3Mb flash memory drive. The 3Mb was a sufficient amount of storage for the for experiment data so capacity was not thought to be a problem. Unfortunately, the single board computer had a bios chip that could not effectively use the 3Mb. Instead, the computer could only store 0.9Mb in the flash drive. If any more than 0.9Mb was stored in the flash drive it locks up and the information cannot be retrieved. The computer manufacturer was able to provide a new bios chip but not until 1 week after the experiment was delivered to Houston for integration. Therefore, thermocouple sampling rates had to be reduced to compensate for the reduced storage capacity.

PREFLIGHT HEALTH TEST

It is essential to have the ability to check the health of the system after it is installed into the Smart Can so that any problems can be revealed and remedied. A test apparatus was made so that anyone can check the health of the experiment, even if they know nothing about the experiment. All that is needed is a voltmeter and the single page of instructions. The test box interfaces with the experiment via the Bendix connector in the top of the can that was intended to be used for the real-time data link. The box has 6 electrical contacts and 3 LEDs.

The test box is important for three reasons. First, it allows for the testing of the power system. The 6 electrical contacts are +5 volts, -12 volts, +12 volts, +28 volts and two grounds. As long as each contact measures the voltage it is marked, the power system is healthy. Secondly, the LEDs indicate any open thermocouples. The number of times that the lights flash corresponds to a specific thermocouple that is not functioning. Finally, the LEDs also indicate when the test period is nearing the end. The experiment will begin to run once it has been on for 20 minutes. If the experiment began to run will still on Earth it could ruin the results. Therefore, after the payload has been on for fifteen minutes, the LEDs flash indicating that shutdown must occur within the next five minutes. The experiment can be turned on and off without concern as long as the 20 minute time limit is not exceeded.

CONCLUSION

The Iowa Joint Experiment in Microgravity solidification has thus far been a successful venture. The few minor problems that occurred seem insignificant when compared to the rest of the project. The entire endeavor took less than 6 months from its October 1994 inception to the delivery of hardware on March 25, 1995. The final configuration weight at delivery was 83 pound. The photographs below (figure 5) show the experiment in its final configuration.

Still in store for project is a successful flight and subsequent data retrieval. The ground base tests also have to be performed after the data is retrieved. Only after the data has been analyzed and the ingots have been examined can the project be deemed a complete success.

ACKNOWLEDGMENTS

It is with gratitude that the authors acknowledge the direction and guidance of Professor Christoph Beckermann of the University of Iowa and Professor Rohit Trivedi of Iowa State University. Without their leadership this project could not have been so expeditiously executed. The authors would like to acknowledge the Iowa Space Grant Consortium for their strong financial support. Also, this project would not have been possible without the financial support of the Institute for Physical Research and Technology at Iowa State University. Additional thanks goes to the Engineering Research Institute at Iowa State University for their expert help in milling and other metal work. Thank you to Toronto MicroElectronics for their donation of the computers and the accompanying technical support. A final and most sincere thank you goes to Space Industries, Inc. without whose help this project would not have been possible.

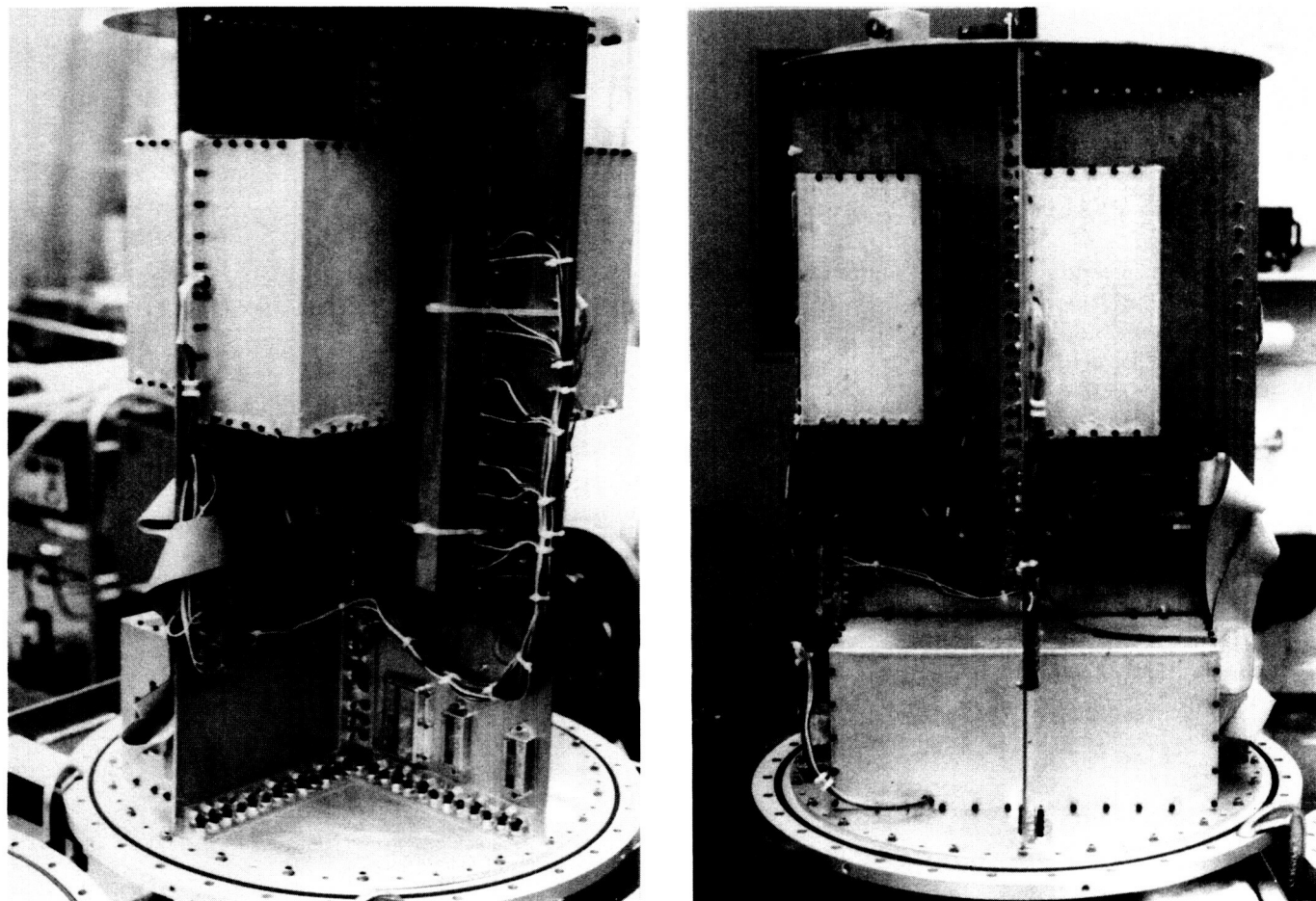


Figure 5 Photographs of IJEMS in Final Configuration

REFERENCES

1. Lloyd, D. J., "Particle reinforced aluminum and magnesium matrix composites," *International Materials Reviews*, Vol. 39, No.1, 1994, pp. 1-23.
2. Asthana, R., and Tewari, S. N., "Second phase particle-solidification front interactions: an evaluation of theoretical models," *Processing of Advance Materials*, Vol. 3, 1993, pp. 163-180.
3. *Solidification of Metal Matrix Composites*, edited by Pradeep Rohatgi, TMS, 1990.
4. *Processing of Semi-Solid Alloys and Composites*, Proceedings of the Second International Conference, edited by S.B. Brown and M.C. Flemings, TMS, 1993.