

# **Survey Analysis of Materials Processing Experiments Aboard STS-47: Spacelab J**

*R.J. Sharpe*

*The University of Alabama, Tuscaloosa, Alabama*

*M.D. Wright*

*Marshall Space Flight Center, Marshall Space Flight Center, Alabama*

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*R.J. Sharpe*

*The University of Alabama, Tuscaloosa, Alabama*

*M.D. Wright*

*Marshall Space Flight Center, Marshall Space Flight Center, Alabama*

National Aeronautics and  
Space Administration

Marshall Space Flight Center • MSFC, Alabama 35812

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## LIST OF ACRONYMS AND SYMBOLS

CHF	continuous heating furnace
GHF	gradient heating furnace
IF	image furnace
NASDA	National Space Development Agency
Pb	lead
PICA	phenolic-impregnated carbon ablator
Sn	tin
Te	tellurium
TPS	thermal protection system



## TECHNICAL MEMORANDUM

### **SURVEY ANALYSIS OF MATERIALS PROCESSING EXPERIMENTS ABOARD STS-47: SPACELAB J**

#### **1. INTRODUCTION**

The Shuttle program of the late 20th century ushered a new era of space science. Early Gemini and Apollo missions had touched on human-tended space science, although these experiments were viewed only as secondary objectives. Skylab, the first American Space Station, laid the foundation for experiments in space, based on a long-term human presence.

The Shuttle brought a new perception in the way space science was viewed. Engineers and scientists developed a reusable laboratory module designed to fit the orbiter's payload area. This reusable science laboratory allowed for scientists on Earth to design original experiments and submit them to be tested by Shuttle astronauts. The laboratory itself would be known as the Spacelab.

Shuttle astronauts conducted an estimated total of  $\approx 3,500$  experiments over a 20-yr period aboard Spacelab.<sup>1</sup> These experiments ranged from solar physics to biotechnology to life sciences, etc. The strongest majority of Spacelab's experiments, however, focused on materials science (almost 1,000 out of the 3,500).

Significant materials science experiments conducted on Spacelab J, which featured 20 life science experiments and 24 materials science experiments, are discussed here. The mission was a joint Japanese-American science venture that flew aboard Shuttle Endeavor on STS-47. Experiments focused on four categories—electronic and crystal materials science, metal alloy studies, glasses and ceramics studies, and fluid dynamics. The mission took place over a 9-day period, and NASA published a full report of science results in February 1995.<sup>2</sup> All experiments aboard Spacelab J were carried out under the direction of the Payload Operations and Control Center at Marshall Space Flight Center, Huntsville, Alabama.

## 2. ELECTRONIC/CRYSTAL GROWTH MATERIALS EXPERIMENTS

Electronic and crystalline materials experiments aboard Spacelab J focused on determining the most effective route for growing various forms of semiconductor crystals as well as experimentation in the specific shape in which the crystal would grow.<sup>3</sup> Primary instrumentation in crystal growth experiments consisted of three furnaces—the gradient heating furnace (GHF), used for investigating crystal formation; the image furnace (IF), used for crystal growing using the floating zone method; and the continuous heating furnace (CHF), used for generating high temperatures and fast cooling of crystal samples.<sup>4</sup> Significant experiments are discussed in detail in section 2.1.

### 2.1 Growth of Silicone Spherical Crystals and Surface Oxidation

Silicone, a metalloid element, is recognized as one of the most important semiconductor materials in the world. It is used in the circuitry of household electronics as well as in complex applications. When these silicone materials are produced, however, a specific amount of impurity must be introduced to the crystal to create either an abundance of free electrons or a lack of shared electrons (depending on the need).<sup>5</sup> The uniformity of this process—known as doping—is critical to the effectiveness of the crystal, albeit difficult. Dr. Tatau Nishinaga, scientist at the University of Tokyo, proposed growing silicone crystal spheres under microgravity aboard Spacelab J. In his experiment description, he listed the following reasons of significance for the experiment:

- Microgravity might eliminate the buoyancy-driven convection in the melt.
- Growth of crystals without a crucible is possible.
- The microgravity environment allows for possible discovery of a way to more effectively create a uniformity of impurities used in the crystal.

Although Nishinaga suggested that several experiments would be necessary to make the most concrete conclusions, time was a limiting factor. As a result, he proposed two short experiments, the first of which consisted of a premade silicone sphere crystal that would be inserted into the Spacelab Crystal Growth Facility and heated past its melting point. The crystal would begin to melt, but before the sample had entirely melted, the system would then be cooled. This allowed the remaining solid silicone in the center to serve as the seed crystal for growth in the second step. During the workup of the experiment, an infrared sensor would monitor the sample's temperature. A diagram of the instrumentation is shown in figure 1.

The second experiment involved insertion of a silicone rod into the heating unit, melting the end of the rod, and using the nonmelted portion of it as the source of the seed crystal. This crystal would avoid contact with the sides of the furnace, eliminating potential problems with contamination. The process was modeled after the Czochralski method of crystal growing in which seed crystals are placed on a rod and inserted into melted silicone. Mark C. Lee, payload commander aboard Spacelab J, is shown conducting the experiment in figure 2.

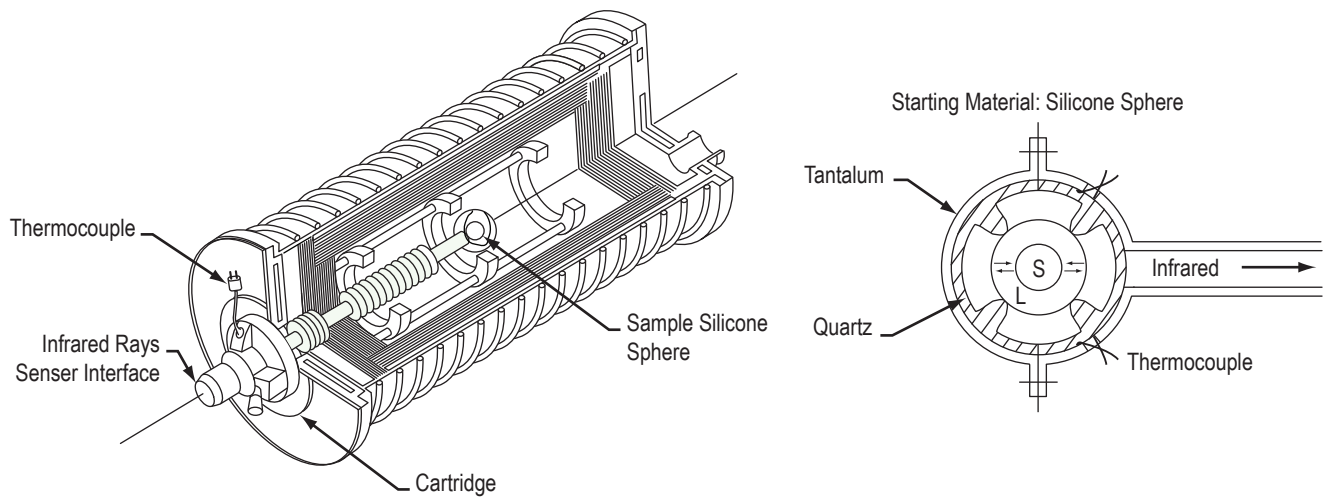


Figure 1. Diagrams of the first method of silicone spherical crystal growth.

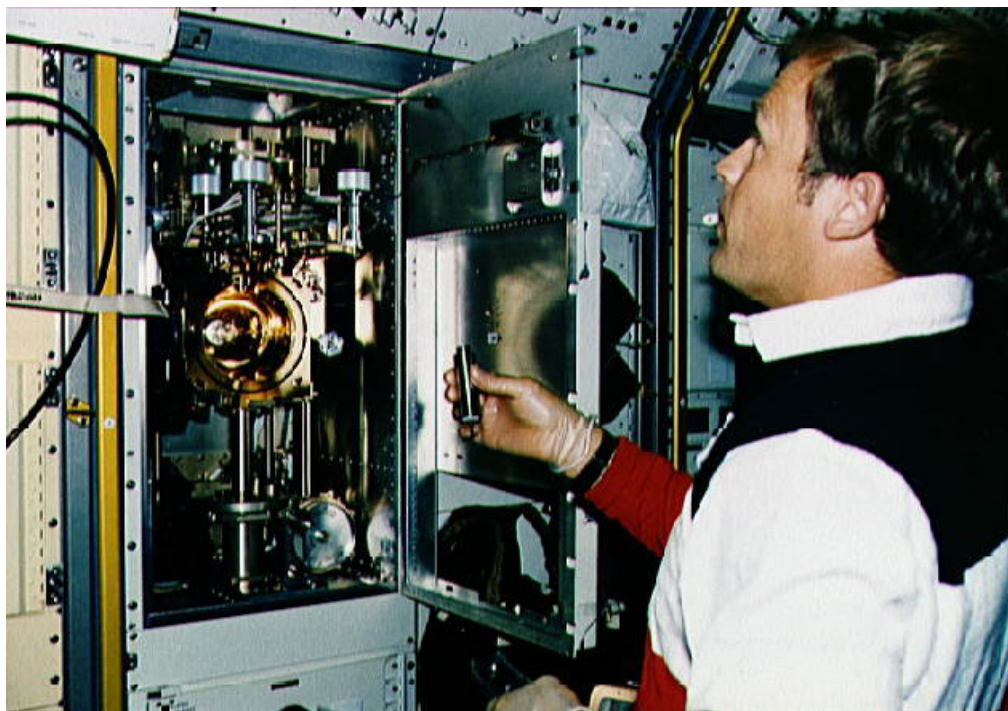


Figure 2. STS-47 Payload Commander Mark Lee conducts the Silicone Spherical Crystal Experiment aboard Spacelab J.

Upon completion of both experiments, observations were recorded, and the samples were brought back to Earth for analysis. The first part of the experiment showed promising results. A crystal began to grow within the time period; however, a crack in the tantalum shell covering the sample caused a significant loss of silicone material.<sup>6</sup> The second part of the experiment showed similar crystal growth. However, a portion of the crystal shattered when it came into contact with the furnace's surface. Scientists had to first reassemble the crystal before making any observations.

Once the crystals were polished, Nishinaga made his conclusions. He found that the crystals grown aboard the Spacelab, though slightly damaged in the experimentation, contained significantly less amounts of ridges (indicative of nonuniformity) than the same crystals grown on Earth. He reasoned that by preventing inconsistencies in the crystal growth chamber, he could grow silicone crystals with a more uniform impurity distribution. His conclusion was based on a prior experiment that took place on Spacelab 1 which showed that inconsistent heating temperatures caused striations due to a phenomenon known as Marangoni convection.

This experiment demonstrated an improvement on the way silicone-based semiconductors could be produced. Virtually all forms of electronics today must feature circuit boards. Nishinaga's experiment improved the foundation upon which many of those circuits could be developed.

## **2.2 Growth Experiment of Narrow Band-Gap Semiconductor Lead-Tin-Tellurium Crystals in Space**

Another experiment in improving semiconductor materials focused on the metal alloy lead-tin-tellurium (Pb-Sn-Te).<sup>4</sup> Pb-Sn-Te, like silicone-based compounds, bears strong potential for applications in circuits due to its properties as a semiconductor. In addition, this metal alloy is capable of detecting a wide range of frequencies of light in the infrared, making it a useful material for applications in heat sensor technology; i.e., home security systems, space imaging, etc. However, the synthesis of high-quality Pb-Sn-Te crystals on Earth without major defects has proven difficult due to the varying densities of fluids on Earth under different temperatures.<sup>7</sup>

Tomoaki Yamada, a Japanese scientist at the NTT Basic Research Laboratory, proposed growing Pb-Sn-Te crystals in a microgravity environment. He hoped that his approach would aide not only in the discovery of the mechanism through which the crystals were formed, but would also contribute to the production of higher quality crystals. He explained in his description of the experiment that a key factor in the electrical qualities of the crystal was the ratio of Pb:Sn in the mixture as well as the uniformity of the impurities present in the alloy.

Using the GHF, a seed crystal of the Pb-Sn-Te was inserted into a melt of the same material at  $\approx 1,000^\circ\text{C}$  ( $1,832^\circ\text{F}$ ) inside a crucible made of boron nitride. This crucible was protected by quartz and then covered in a tantalum shell. The GHF was then cooled to allow crystallization to occur in one direction starting from the seed crystal. The experiment's workup is shown in figure 3.

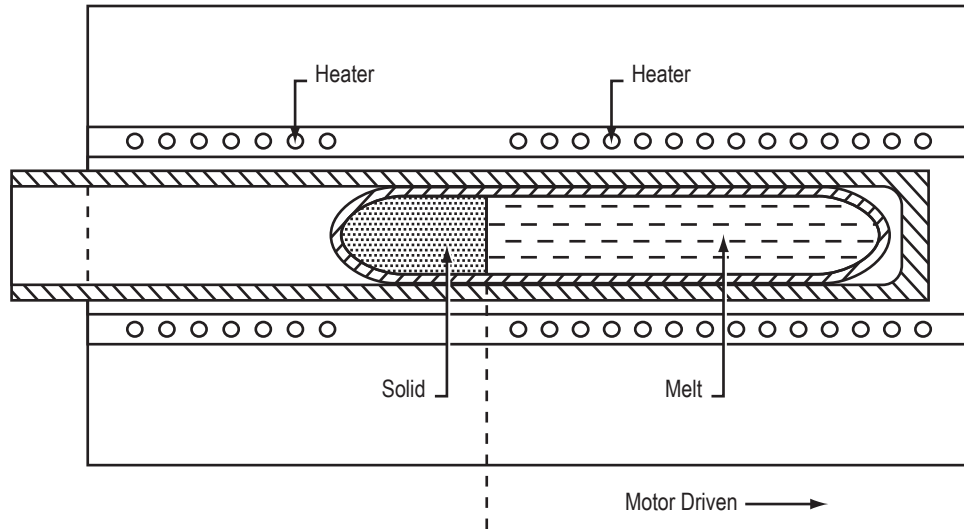


Figure 3. Diagram of the growth workup of Pb-Sn-Te crystals.

Results showed that the crystallized melt did grow cylindrically in a direction away from the seed crystal, but a strong concentration of subgrains (crystals that grow in a different orientation than the rest of its neighbors) was present near the seed-melt interface.<sup>8</sup> Yamada found that small amounts of the melt sample escaped via an opening in the boron-nitride crucible. These escaped pieces continued cooling and eventually formed spherical Pb-Sn-Te crystals. Yamada concluded that this took place due to a graphite spring (being used to push the melt material against the seed crystal) that had encouraged crystallization.

Upon examination, researchers found that the crystals grown in space showed a marked increase in mixture uniformity as well as a constant ratio of Pb:Sn. Furthermore, the space-grown crystal possessed electrical properties superior to those of the same crystals grown on Earth. The results confirmed Yamada's hypothesis that the Pb-Sn-Te crystals grown in microgravity would have more useful properties than those grown on Earth. Using these conclusions, scientists learned how to more effectively develop these metal alloy crystals. As a result, a greater standard was achieved for the production of infrared-sensitive instruments.



### 3. METALS AND ALLOYS EXPERIMENTS

When NASA launched Spacelab J in 1992, a great deal of research had taken place in the study of metals and alloys and their applications. Scientists hoped to discover metals that could be mixed to attain desired properties as well as what processes would be needed to most effectively create those metals. On Spacelab J's experiments, three questions would be addressed—how the structure of the metal changes as it solidifies, what takes place at the interface of two metals when they are mixed, and what thermal and physical properties did these materials possess.<sup>3</sup>

#### 3.1 Study on Solidification of Immiscible Alloys

A challenge facing scientists in metals studies was whether a process could be established to develop a metal alloy out of materials that had previously been immiscible in uniform distribution. (Until this point, only highly segregated alloys of these metals had been developed.<sup>4</sup>) In earlier experiments in microgravity, scientists had attempted to create an alloy of aluminum, lead, and bismuth among others, but upon observation, the alloy synthesized did not show a uniform distribution of both metals across the entire material. Figure 4 is an image showing the nonuniformity that was achieved in a sample of aluminum and lead.

Scientists at the Tokyo Institute of Technology, led by Dr. Akihiko Kamio, proposed a similar experiment to be carried out on Spacelab J.<sup>9</sup> His experiment and results are discussed below.

Dr. Kamio and his team proposed using four different samples for experimentation in the mixing of immiscible alloys. Three samples consisted of mixtures of indium and aluminum (although the amount of aluminum would vary) with the fourth sample consisting of copper and lead. Each of these four samples were inserted into a container made of graphite within a tantalum cartridge. The cartridge, however, would have an ultrasonic vibrator attached to it that would be used later in the experiment. The apparatus was then placed into the GHF and initially set to a temperature of 955 °C (1,751 °F). The mixture remained at this temperature for 56 min during which time the vibrator was turned on for 10 min to better mix the two metals. The heat was then reduced to 639 °C (1,182 °F) for 30 min so that the metals would cool but still remain melted. When completely cooled, the solid alloy was stored for further examination on Earth.

When the samples were brought back to Earth and studied, Kamio and his group found that the copper-lead sample performed exceptionally well.<sup>10</sup> The copper-lead mixture showed a uniform distribution of each metal across the alloy. Furthermore, Kamio found the structure of the alloy to consist of a prominent copper matrix with a series of nonuniform lead rods. The aluminum-indium samples were damaged, however, due to a leak in the sample containers. This resulted in a large loss of data from the experiment.



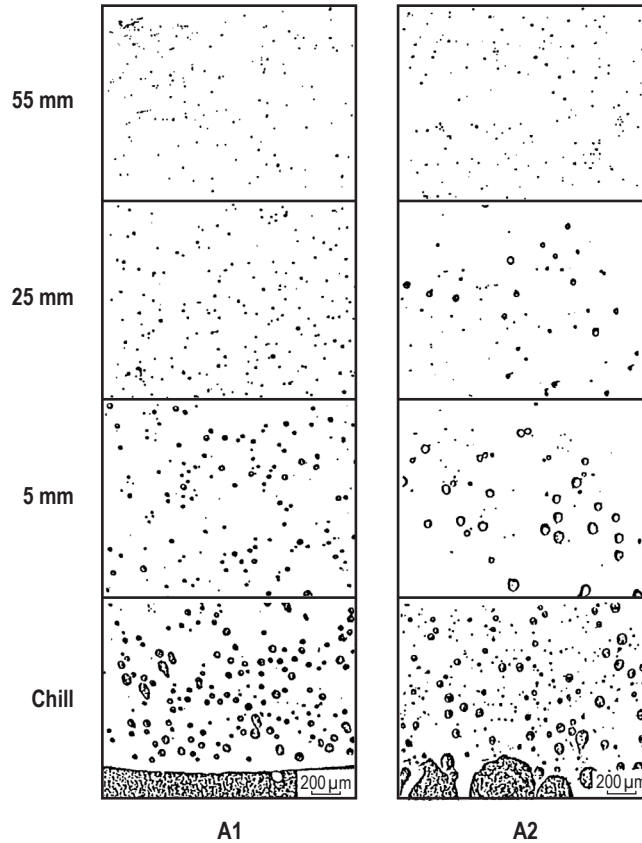


Figure 4. Alloy of aluminum-10% lead developed before Spacelab J experiment. The lead (the dot shapes), rather than distributing in uniformity, shows a preference to one direction.

Nevertheless, the research demonstrated that two metals found to be immiscible in uniformity on Earth could be made into alloy given the correct conditions. This result allowed scientists to continue experimentation to discover a way to produce these alloys on Earth. In practice today, the study of using immiscible metal alloys has found applications in the development of superconductors.<sup>11</sup>

### 3.2 Fabrication of Very-Low-Density, High-Stiffness Carbon Fiber/Aluminum Hybridized Composites

Carbon fiber composites represent a choice substance for strong, yet lightweight materials applications. For example, various areas of the Space Shuttle's thermal protection system (TPS) use carbon-carbon composites as the primary material. Recently developed at Ames Research Center, phenolic-impregnated carbon ablators (PICAs) are being tested for use as the heat shield material of the Orion spacecraft. On Spacelab J, an experiment was conducted to determine if a process could be developed to synthesize a carbon composite material that is both very strong and very low in density.<sup>4</sup> The project was led by Dr. Tomoo Suzuki, scientist at the Tokyo Institute of Technology, who developed an experiment to test whether carbon fiber composites held together

by aluminum alloys could be effectively developed in microgravity. Figure 5 illustrates the structural difference between composites made on Earth and in microgravity.

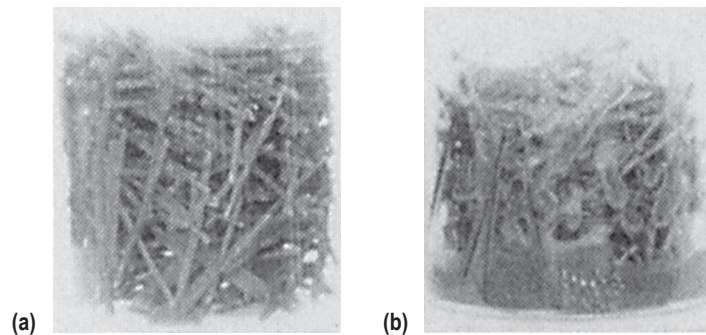


Figure 5. Example of how gravity affects the development of composite materials. Shown is a nylon thread composite produced in (a) microgravity and (b) on Earth.<sup>4</sup>

Dr. Tomoo chose for his mixture a carbon fiber that would be coated with an aluminum alloy. The alloy works as a bonding material as it melts with heating and then begins to cool, giving the material its desired low density and strength.<sup>12</sup> The decision to use aluminum was based on a similar experiment that was conducted on a National Space Development Agency (NASDA) of Japan's TT-500A rocket which was designed to simulate microgravity for up to 6 min. (This experiment was in large part a failure to sample overheating.) In a short workup, six samples of carbon fibers would be coated with aluminum using a process known as vacuum evaporation. The fibers would then be divided into lengths of 1 mm and placed in a capsule for heating at 700 °C (1,292 °F). This heating process would take  $\approx 10$  min.

Once completed, the capsule would be transferred to the CHF where it would undergo constant heating for the remainder of the flight. The materials would then be stored for examination once the Shuttle returned to Earth. When the samples were examined, it was found that the heating process (initial heating followed by continuous milder heating) on all six samples was a success.<sup>13</sup> The bonding of the aluminum to the carbon fibers was effective in each sample. An image of the CHF in operation is shown in figure 6.

Furthermore, examination of the materials' physical properties showed that they were rigid with a density less than a tenth of aluminum. Elastic and compression strength tests were also conducted, and the composite performed with less promise than Tomoo and his team had expected. Tomoo hypothesized that this was due to an unexpected coagulation of some of the aluminum alloy, which acted as a defect to the structure of the composite.



Figure 6. STS-47 Mission Specialist Jan Davis works at the CHF on Spacelab J.

Overall, the experiment was a major success as the desired material was fabricated without any major problems. However, the unexpected aluminum coagulation suggested that more research was needed to eliminate the defect. This experiment was one of many that sought to achieve the common goal of developing a strong, yet lightweight material resistant to both extremes in temperature as well as force and pressure. In addition, it laid the foundation for a new family of extreme condition materials development. Carbons and their composite fibers lie at the forefront of materials development today with research in ablative materials (PICA, TPS mentioned earlier) as well as all-purpose structural materials such as fullerene and nanotube carbon materials.

## 4. GLASS AND CERAMIC EXPERIMENTS

The Glass and Ceramics Branch of Spacelab J focused on examining and manipulating the properties of glass.<sup>3</sup> Payload specialists aboard Spacelab J used both the IF and the acoustic levitation furnace to conduct their experiments, the ultimate goal being to better understand the physical properties of different types of glass and assist in the developing of new techniques for the preparation of higher quality glasses. One such experiment is outlined in section 4.1.

### 4.1 High Temperature Behavior of Glass

In an experiment designed to compare the physical properties of glass on Earth to those in space, Kyoto University Scientist Naohiro Soga developed a Spacelab experiment to examine samples of glass under extreme temperature.<sup>14</sup> The experiment had two main goals—to determine the viscosity (or a sample's ability to flow) of a glass sphere in microgravity and to use that data to confirm that the presence of gravity is not a major factor in these physical properties.

Using the IF (used in this experiment so that the sample could be seen while heating), a small sample of glass is melted. This glass sample would also be filled with gold particles so that flow of the melted glass can be readily observed. A video camera is set up in front of the IF for recordings to be viewed on Earth. A diagram of the experiment is shown in figure 7.

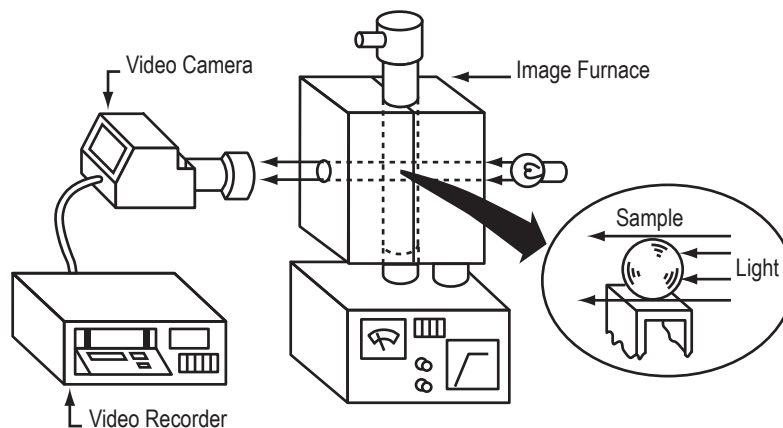


Figure 7. Workup of the High-Temperature Glass Experiment.

Soga specifically hoped to find that the volume-temperature relationship of glass in microgravity was comparable to that of glass on Earth. In addition, he hoped to obtain information from the gold pieces about the nature of how a melted glass sphere flows. He found that as the glass sphere was heated, there was a spike in volume increase near the 400–500 °C (752–932 °F) tempera-

ture range. In addition, at about 600 °C (1,112 °F), a large amount of bubbles began to appear within the sphere. Both of these results had not been experienced when the same experiment was conducted on Earth. The gold particles, although heated at one point past their melting point, retained their original shape.

Through the results of this experiment, Dr. Soga confirmed that the glass had slightly different properties in microgravity than it possessed on Earth. In practice today, the observations made here offered scientists a stronger awareness of the behavior of glass in high temperature for various aircraft.<sup>16</sup>

## 5. FLUIDS EXPERIMENTS

Experimentation in fluid dynamics aboard the Spacelab J represented another major goal in that the issue of gravity-induced buoyancy would be removed.<sup>4</sup> In microgravity, scientists could reconcile the effects on fluids caused by gravity from those that were induced by other factors. The discoveries in fluids from this mission, amongst others, enabled scientists to effectively develop and process new materials in order to understand what physical factors would be affected by a gravity-absent environment. One such discovery is shown in section 5.1.

### 5.1 Study of Bubble Behavior

Understanding the flow of bubbles within a fluid on Earth is a relatively simple concept (they will inevitably travel to the surface of the fluid), but once gravity is removed, the effect of buoyancy no longer applies and bubbles are left largely unaffected. However, other forces still act on bubbles within a fluid which are often masked on Earth due to the dominating buoyant force and can be readily observed in microgravity. Dr. Hisao Azuma, scientist at the National Aerospace Laboratory, designed an experiment aimed at understanding these effects.

The test was designed to analyze the effect of surface tension of bubbles within a silicone oil fluid.<sup>17</sup> The effect of surface tension on the sides of bubbles had been noted previously but was unobservable on Earth due to the much larger buoyant force. Azuma also knew that a bubble in microgravity will migrate towards the direction of higher temperature because of a difference in surface tension. He sought to find how fast different bubbles would migrate due to the liquid's Marangoni number (a unitless value showing the ratio of surface tension to viscosity). By using a temperature gradient of a hot surface on one side of the fluid and a cool surface on the other side, the speed could be measured.

When the experiment began, an unexpected event took place in that the injected bubble traveled to the center of the fluid before heating began.<sup>18</sup> Azuma later proposed that this took place because electrical charges were induced when the bubbles were injected through a syringe. As the experiment continued, the speeds of different bubbles were observed. As the data came in, the speeds of the bubbles were found to be lower overall than were originally predicted. It was decided that this was the result of the number of moving bubbles affecting the speed of all bubbles within the fluid. Also, it was noted that bubbles near the hot wall began to travel faster than predicted towards the hot surface. This was explained in that the bubbles were found to be attracted to other bubbles on the hot surface.

These results gave scientists a better comprehension of how fluids behaved in a microgravity environment and, as a result, a stronger understanding of factors to consider in aerospace materials development. Today, these factors are important considerations when developing a tool for oxygenating water in both gravity and microgravity.<sup>19</sup>

## **6. CONCLUSIONS**

The findings of Spacelab J were a remarkable accomplishment in studying the physical properties of materials for the applications of aerospace technology. Each experiment worked within a greater field of interest to better understand how materials designed on Earth would behave in outer space. In addition, Spacelab J was an international landmark in cooperation between NASA and the NASDA. Scientists and engineers from both nations developed the instrumentation of the laboratory, selected the experiments, and assigned the astronauts that would ensure the success of the mission.



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14. ABSTRACT  This Technical Memorandum (TM) is a survey outline of materials processing experiments aboard Space Shuttle Mission STS-47: Spacelab J, a joint venture between NASA and the National Space Development Agency of Japan. The mission explored materials processing experiments including electronics and crystal growth materials, metals and alloys, glasses and ceramics, and fluids. Experiments covered include Growth of Silicone Spherical Crystals and Surface Oxidation, Growth Experiment of Narrow Band-Gap Semiconductor Lead-Tin-Tellurium Crystals in Space, Study on Solidification of Immiscible Alloys, Fabrication of Very-Low-Density, High-Stiffness Carbon Fiber/Aluminum Hybridized Composites, High Temperature Behavior of Glass, and Study of Bubble Behavior. The TM underscores the historical significance of these experiments in the context of materials processing in space.					
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Marshall Space Flight Center, Alabama  
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