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# The Advanced Automated Directional Solidification Furnace

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## ABSTRACT

The Advanced Automated Directional Solidification Furnace (AADSF) is a five zone tubular furnace designed for Bridgman-Stockbarger, other techniques of crystal growth involving multiple temperature zones such as vapor transport experiments and other materials science experiments. The five zones are primarily designed to produce uniform hot and cold temperature regions separated by an adiabatic region constructed of a heat extraction plate and an insert to reduce radiation from the hot to the cold zone. The hot and cold zone temperatures are designed to reach 1600°C and 1100°C respectively. AADSF operates on a Multi-Purpose Experiment Support Structure (MPESS) within the cargo bay of the Space Shuttle on the United States Microgravity Payload (USMP) missions. Two successful flights, both employing the directional solidification or Bridgman Stockbarger technique for crystal growth have been made, and crystals of HgCdTe and PbSnTe grown in microgravity have been produced on USMP-2 and USMP-3 respectively. The addition of a Sample Exchange Mechanism (SEM) will enable three different samples to be processed on future flights including the USMP-4 mission.

## 2. INTRODUCTION

One of the major applications of microgravity is in materials, in particular the processing of electronic materials, and metals and alloys by directional solidification, and also by chemical vapor transport. There are many reasons for such studies in low earth orbit; these include the need to reduce the fluid flow caused by gravity driven convection, the possibility of reducing the solutal Rayleigh numbers by several orders of magnitude, and the ability to study the effects of small accelerations on fluid flow and solidification. In addition, microgravity will reduce a material's tendency to deform under its own weight immediately after solidification when it is weakest. The possibility of containerless processing, including float zone techniques will also aid in the production of more pure material, with less defects induced by contact with the container walls. NASA has built and flown on the shuttle two versatile, complex furnace systems specifically designed for materials processing. These are the Crystal Growth Furnace (CGF)[1], and the AADSF[2]. Of these the CGF is designed to fly within the Spacelab, and in a man tended environment; the samples are loaded by the crew members on board the orbiter, but the furnace is designed to run autonomously once the samples have been inserted. In contrast, the AADSF operates on the MPESS structure within the cargo bay of the orbiter; the samples are loaded prior to launch and are inaccessible once the mission is underway. As such the AADSF is subject to the harsh environment of outer space with wide fluctuations of temperature, and has undergone rigorous ground testing to simulate these conditions. Both CGF and AADSF were built by Teledyne Brown Engineering in Huntsville, AL.

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## 3. HARDWARE

AADSF consists of three major constituent sections of hardware, namely the Experimental Apparatus Container (EAC), the Data Acquisition System (DAS) and the Signal Conditioning and Control System (SCCS). The functions of these three elements are summarized in Table 1.

The EAC is an environmental container designed to isolate the heating element and the translation mechanisms of the furnace system within a controlled environment. As such it is vacuum tight, with feedthroughs for required power, coolant loops and monitoring devices. During use, the EAC normally operates with a positive pressure of argon. The outer jacket of the furnace is 20.3 cm in diameter and incorporates the coolant loops on the outside. The coolant used during flight is freon, which is provided through the MPESS carrier as part of the USMP mission. Ground based testing uses water for cooling.

A major component of the EAC is the five zone furnace module itself. The zones consist of beryllium oxide cores wrapped with platinum-rhodium (60/40) furnace wire. The inner diameter of the furnace is 2.54 cm, but with the high temperature metallic insert the working diameter is restricted to 1.91 cm. The gradient region is shown schematically in Figure 1. The hot zone is 25.4 cm long and the cold zone 12.7 cm long. The hot zone is equipped with a hot guard heater to prevent "rolloff" of the temperature, while the gradient region can be adjusted by means of a booster heater just above the adiabatic zone. Similarly, the cold zone is maintained more isothermal by means of a short guard heater. Temperature control of the zones is by means of thermocouples positioned close to the elements themselves. There are two thermocouples per heater element, thus ensuring continuity of operation in the case of a failure of one of the thermocouples. Two schematics of the EAC and its components are shown in Figure 2.

The adiabatic zone consists of a re-configurable assembly with a cold plate or adiabatic ring, a hot plate, both constructed of Zircar AL-30AA, a heat extraction plate of Inconel 718, and a high temperature insert composed of Haynes alloy 214. The nominal thicknesses of these components are 4.32mm, 1.60 mm and 7.62 mm for the hot plate, heat extraction plate and cold plate respectively. The configuration can be modified to include 1X, 2X or 3X of each of these components in any combination. The USMP-2 flight used the 1X configuration to obtain a high gradient. The USMP-3 flight used the 2X configuration.

The translation mechanism consists of a d.c. motor driving a lead screw. Translation rates in the range of 0.5 to 50 mm per hour are available. This wide range of speeds allows for extremely slow growth for directional solidification, and rapid insertion or withdrawal for other scientific reasons such as the quenching in of an interface. Experimenters have regularly used rapid withdrawal for examining the shape and position of the solidifying interface.

The Sample Exchange Mechanism will be added for the USMP-4 Mission. This facility does not affect the thermal characteristics, and in fact will be retro-fitted to some of the existing furnace assembles. Three samples will be accommodated, and will be processed in sequence. The cartridge design will not change, and the samples will be securely held during launch and landing. Due to the need to withdraw all of the samples above the opening to the furnace, the new model will be taller than the original AADSF. This means that it can no longer be accommodated at one side of the orbiter on the MPESS, but has to be positioned on the center line. This modification, in fact, may help the science, as will be described below.

The other two main components of the AADSF system, as shown in Table 1, are the Data Acquisition System and the Signal Control and Conditioning System. These are housed outside the EAC, being bolted to the MPESS as shown in Figure 3.

Other related components of the AADSF include the cabling to provide the system with 28 volt d.c. power from the main bus of the orbiter, and the fact that in the event of a computer malfunction, the AADSF can be controlled by means of the orbiter on-board computer which can re-boot the system be means of a program called the Bootstrap and Table Load Software (BTLS). This would be done by the orbiter crew during the mission.

## 4. SCIENCE

The first two flights of the AADSF examined the solidification of two solid solution semiconductor alloys, namely mercury cadmium telluride and lead tin telluride. As shown in Figure 4, these alloys exhibit different fluid flow characteristics as driven by gravity on earth. In the case of lead tin telluride, the component rejected at the interface is less dense than the bulk liquid, and so complete mixing ensues. In the case of mercury cadmium telluride, the rejected component is denser and fluid flow is restricted to a small region close to the interface. The two experiments thus complement each other very well. In the flight experiments, the intentions of both investigators was to examine the amount of fluid flow present under different conditions of small residual accelerations. The experiments are shown in Table 2.

The results from the mercury cadmium telluride experiment showed that low gravity levels alone would not result in diffusion controlled growth. Even with static residual acceleration levels lower than $10^{-6}g_0$, there was considerable fluid flow. Figure 5a shows the effect in the first half of the mission when the vector had a component which was destabilizing with respect to the solute. There is three dimensional fluid flow in the direction of the vector manifested by the pushing of the higher density mercury rich material in the direction of the residual transverse vector. The degree of inhomogeneity is in fact greater than on earth. On the other hand, as shown in Figure 5b, when the vector is towards the solid, the flow is reduce, even with a large (1.55 $\mu$g) transverse residual component. This flight gave clear demonstration of the nature of three dimensional fluid flow in microgravity.

## 5. MISSION

As mentioned above, the AADSF is attached to the MPESS carrier within the payload bay of the orbiter. The arrangement, and how the AADSF fits with other payloads is shown in Figure 3. This is the configuration as used for USMP-2 and USMP-3. For USMP-4, the AADSF is taller, due to the addition of the sample exchange mechanism; this results in the necessity of moving the instrument to the center line of the orbiter. This latter factor has significant implications on the science. During shuttle missions, there can be restrictions on the attitude in which the orbiter can fly. These restrictions can lead to undesirable residual acceleration vectors on the experiment itself. In the case of USMP-2, the two different attitudes shown in Figure 4 produced widely differing results. There are two principle origins of the residual acceleration vectors. The first of these is the drag on the orbiter caused by residual ionized particles. This varies with the night and day cycles of the orbiter. The second contributor is the gravity gradient factor which is caused by the fact that he experiment may be located away from the center of gravity of the orbiter. Thus, the experiment may lie within a slightly different orbit with respect to the center of gravity, and there is an effective acceleration component introduced. The situation and the calculations can be quite complex, and many safety and operational needs of the orbiter influence the possible attitudes. The re-positioning of the AADSF to the centerline of the orbiter makes these calculations easier, and means that it will be easier to obtain desired residual accelerations.

## 6. REFERENCES

1. R. Srinivas and D. A. Schaefer, "Crystal Growth Furnace: An Overview of the System Configuration and Planned Experiments on the First United States Microgravity Laboratory Mission," Paper 92-0786, AIAA, 30th Aerospace Sciences Meeting and Exhibit, Reno, NV, January 1992.
2. J. LeCroy and D. Popok, "Design of a High Thermal Gradient Bridgman Furnace", Paper 94-0336, AIAA, 32nd Aerospace Sciences Meeting and Exhibit, Reno, NV, January 1994.

**Experimental Apparatus Container (EAC)**

Houses the five zone Furnace Module with associated freon cooling loop
Translation System, and associated motors and relays
Muffle Tube or Cartridge containing the experimental Ampoule and its instrumentation
Sample Exchange Mechanism (SEM), and its motors and relays

**Data Acquisition System (DAS)**

16-bit microprocessor which controls the command and data interfaces
Provides discrete inputs, analog inputs, serial/digital input/outport ports, and relay drivers
Receives uplinked commands, processes them and transmits them to the SCCS

**Signal Conditioning and Control System (SCCS)**

Controls the furnace parameters for the five zones
Can operate autonomously with pre-programmed furnace temperatures and translations
or can receive uplinked commands from the DAS to modify experimental parameters during the mission
Transmits data to the DAS for downlink

Table 1. The components of the AADSF System

**USMP-2**

GROWTH OF SOLID SOLUTION SINGLE CRYSTALS
Dr. Sandor L. Lehoczky, Marshall Space Flight Center, Huntsville, AL
Principal Investigator
**Successfully flown - March 1994 on STS-62**

**USMP-3**

COMPOUND SEMICONDUCTOR GROWTH IN A MICROGRAVITY ENVIRONMENT
Dr. Archibald L. Fripp, Langley Research Center, Hampton, VA
Principal Investigator
**Successfully flown - March 1996 on STS-75**

**USMP-4**

FIRST FLIGHT of AADSF with SAMPLE EXCHANGE MECHANISM
THREE EXPERIMENTS - up to 3 different teams
Candidates not chosen at time of going to press
**Scheduled for October 1997 on STS-87**

Table 2. AADSF Experiments

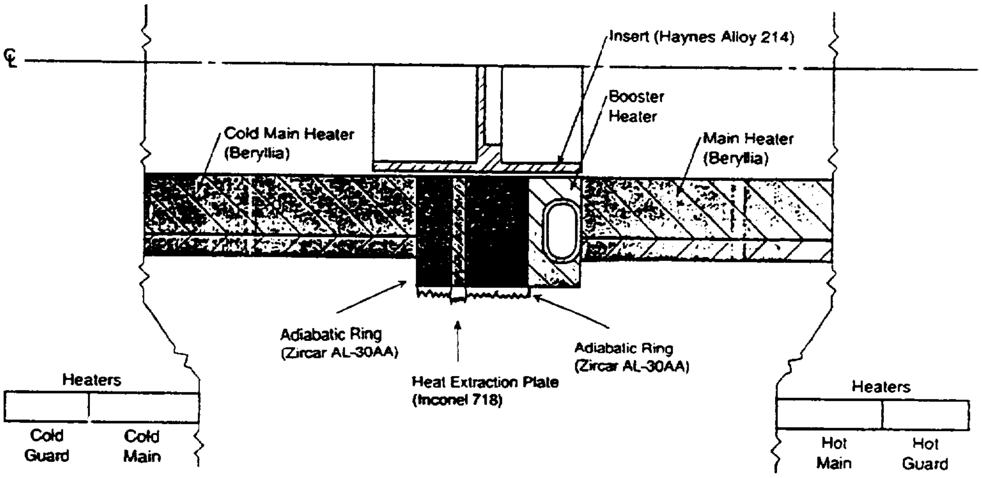


Figure 1. Schematic of AADSF High Gradient Region

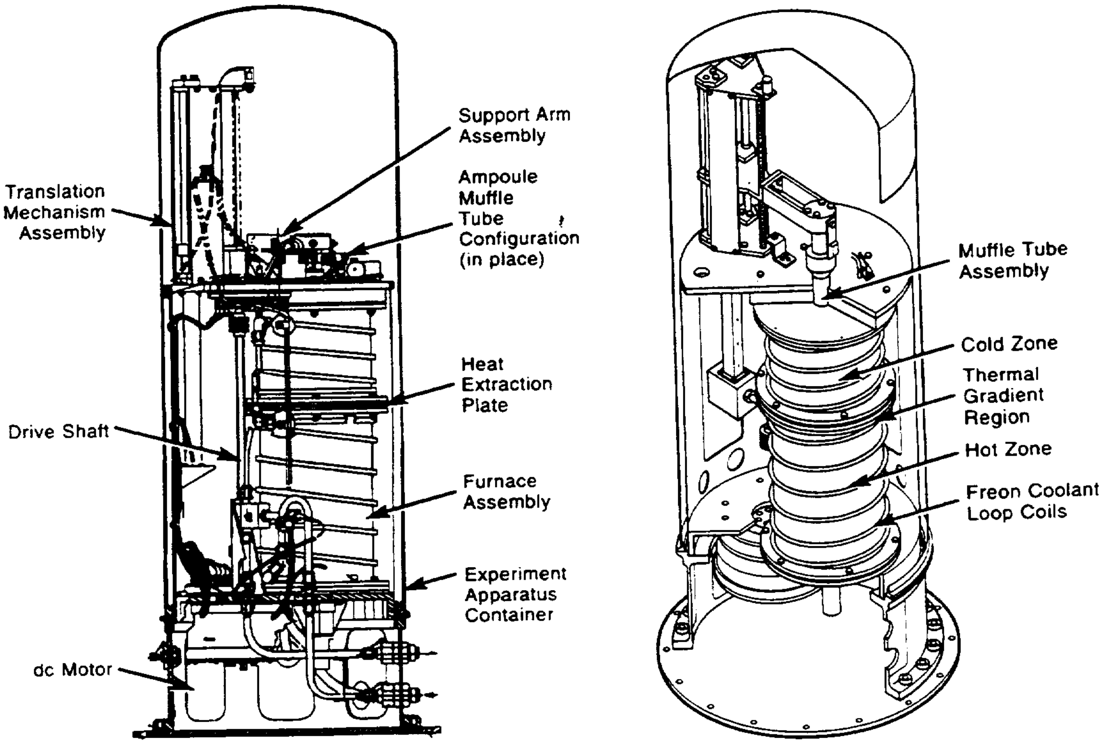


Figure 2. Two views of the Experiment Apparatus Container

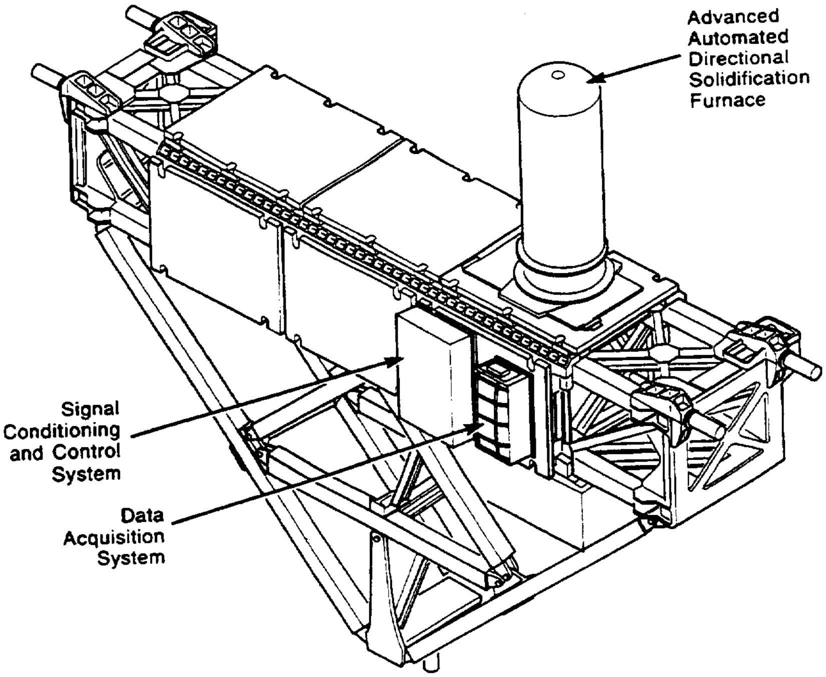


Figure 3. Advanced Automated Directional Solidification Furnace on Shuttle Carrier

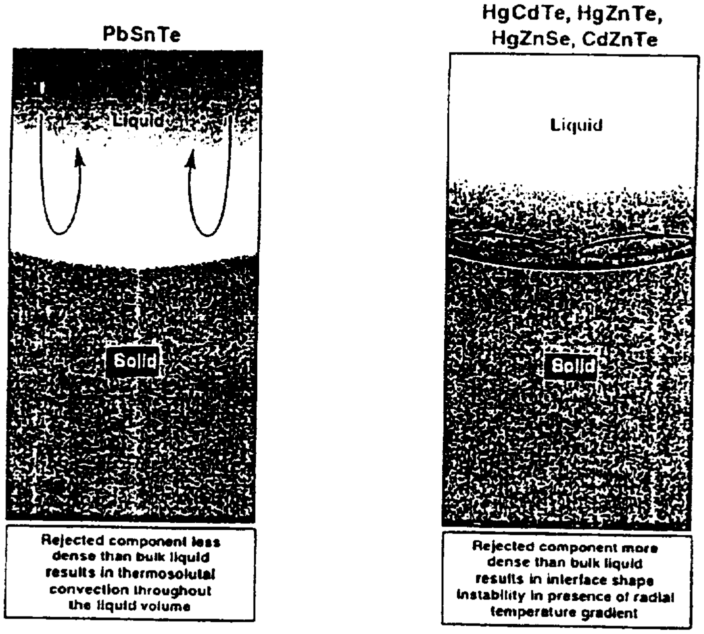
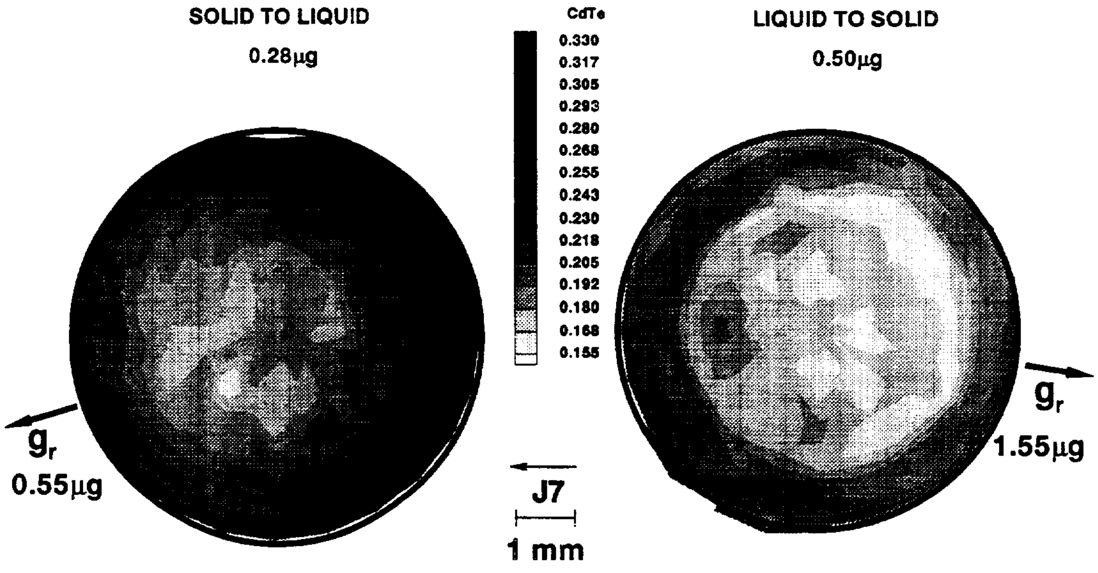


Figure 4. Gravity Effects in the Solidification of Pseudobinary Solid Solution Systems



(a) +YVV/-ZLV Attitude (b) -XLV/-ZVV Attitude

Figure 5. USMP-2/AADSF Flight Sample of $Hg_{1-x}Cd_xTe$