

# **PROCESSING EUTECTICS IN SPACE**

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### SUMMARY

Six areas of experimental investigation were dealt with under NASA contract NAS 8-29669 with the objective of investigating possible improvements in eutectic structures by low-gravity processing.

The areas dealt with were

- 1. Improvements in structural perfection;
- 2. Study of growth conditions for bending eutectic structures;
- 3. Study of processes for the formation of thin sheets of eutectics;
- 4. Evaluation of floating-zone techniques;
- 5. Processing of off-eutectic compositions.

The aspects of eutectic processing which hold the greatest potential for improvements from low-gravity processing conditions are those in which the density of the components has the greatest difference, and also those where the weight of the molten material poses a problem in terms of support.

In the areas studied, the formation of thin sheets, and by extrapolation any thin section, and the floating zone technique of processing will benefit from low-gravity processing. Within these areas, it is important to continue the study of microstructural perfection and thermal control, since the value of a given solidified piece depends on the degree to which the microstructures provides the desired properties. Off-eutectic compositions add a degree of freedom to the formation of <u>in situ</u> composites by allowing adjustment of the composition to provide properties not attainable with the eutectic composition.

The results of this study indicate that without a substantial improvement in thermal control and solidification techniques, the goal of a flat solid-liquid interface with an independently variable thermal gradient in the liquid at the solid-liquid interface is not obtainable.

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Such a goal, however, is a necessary condition for processing offeutectic compositions, where the greatest potential for engineering of materials with the unique eutectic-type microstructure lies. Preliminary results on modifications of processing equipment suggest that equipment can be designed to suppress radial heat flow in the traditional methods where an ingot is contained in a crucible which is passed through a furnace and quench in series. Such suppression should serve to flatten the interface independent of the thermal gradients.

In addition to further studies on improvements in heat flow which could be obtained in the traditional techniques by design changes, a more advanced concept should be pursued consisting of a crucible-less solidification technique related to zone melting. This concept, in addition, would be advantageously pursued in a low-g environment.

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### INTRODUCTION

NASA programs have been directed toward exploring the nearly weightless environment provided by orbiting spacecraft to conduct experiments which will lead to manufacturing products in space for use on earth. Further studies are required to determine which materials would derive most benefit from production in low-g. One experiment which should be conducted involves exploring this low-g environment for the unidirectional solidification of eutectics and off-eutectics to determine if more perfect structures can be attained. The attainment of defect-free microstructures is particularly important in developing these eutectics for nonstructural applications.

Studies which have been done in an earth-based laboratory environment have generally not yielded specimens with the degree of perfection required of the eutectic microstructure to provide test data to evaluate their nonstructural applications. It has been recognized that the low-g environment of an orbiting space laboratory provides a unique environment to re-examine the process of solidification with the goal of producing better microstructures.

The objective of this program is to evaluate the feasibility of using the space environment for producing eutectics with microstructures which can be of value on earth. In carrying out this objective, evaluative investigations were carried out on the technology of solidification in a 1-g environment to provide sound baseline data for planning space laboratory experiments.

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### EXPERIMENTAL

### Improvement in Structural Perfection

<u>Background</u>. Under contract NAS8-28724, a plate-like eutectic Al-CuAl<sub>2</sub> was investigated on the ground and in space. In these studies some improvement in the microstructure was observed in the space processed samples. Because rod-like eutectics are as important and are probably more important for nonstructural applications, it was felt that the possibility of improving the microstructure of rod forming eutectics should be evaluated as well.

In order to run meaningful experiments in space, it was felt that a detailed study of solidification and the microstructure of a rod forming eutectic should be conducted to obtain base line information. The system selected was a model eutectic,  $Al-Al_3Ni$ , which, when unidirectionally solidified, forms rods of  $Al_3Ni$  in an Al matrix. This system has been the subject of many studies which have defined its crystallography and mechanical properties. These are treated in Refs. 1-11. The crystallographic orientations between the  $Al_3Ni$  rods and the aluminum matrix have been found to be as follows: the orthornombic  $Al_3Ni$  grows in the<010> direction in a face-centered cubic aluminum matrix growing in the<110> direction. The  $Al_3Ni$  rods are faceted, with the facets being parallel to the (111), (110), and (112) planes of the aluminum matrix.

It is known that processing variables are directly related to the degree of perfection which is obtained in the microstructure. The types of defects which occur on this scale are caused by a curved solid-liquid growth interface, which leads to colony structures, and less catastrophically to creation or annihilation of  $Al_3Ni$  rods, and changes in the solidification rate, which results in changes in the number of rods per unit area. In addition, there appears to be a minimum value of the thermal gradient to solidification rate ratio, G/R, required by this system to suppress colony growth. It is also known that impurities in the melt lead to break-up of the microstructure because of rejection by the growing solid until a concentration is reached where incorporation into the solid is forced to occur.

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<u>Experimental Studies</u>. In these experiments attempts were denoted to develop methods of evaluating eutectic microstructure and be evaluated at methods of solidification in an attempt to develop techniques which would give good microstructures.

Initially, ingots were directionally solidified by extraction from a furnace into a cooling zone using several different standard furnacequench systems. It was generally found, by examination of cross sections of the ingots, that the liquid-solid interface was not flat, and further the rate of solidification was not constant. The several systems varied markedly, however, in the deviation from the ideal of flat interface and constant solidification rate.

System I consisted of a graphite resistance element furnace with a lowering mechani n which held the ingot in its crucible on a watercooled pedestal. This is shown in Fig. 1. The lowering speed was adjustable, but remained constant at a specific setting. Because the water-cooled pedestal followed the lowering mechanism the effective quench position also moved relative to the hot zone, causing the solidliquid interface to move at a changing rate. The results of a solidification run using the aluminum-nickel eutectic is shown in Fig. 2.

System II consists of an induction heated graphite susceptor through which the crucible is drawn into a fixed position flowing water quench, shown in Fig. 3. This system has the advantage that it can produce very high thermal gradients, and the quench position is fixed with respect to the heat source. While the rate of sol'dification is grossly constant, the system as employed was designed for high power requirements, and thus is difficult to control to the required degree for low-melting point systems such as the aluminum-nickel or the aluminum-copper eutectic. The results of a solidification run using the aluminum-nickel eutectic are shown in Fig. 4.

System III combined features of Systems I and II in that it was a resistance heated furnace system for thermal stability but with a fixed position quench to stabilize the solidification rate. This is shown in Fig. 5. A thermal gradient at the solid-liquid interface of 110°C/cm could be obtained, but studies of longitudinal sections showed the interface was not flat, as indicated in Fig. 6.

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For System IV, a modification to the solidification equipment was made by adding an isothermalizing furnace liner to System III. This liner was purchased from Dynatherm Corporation, and consists of an Inconel material tube with sodium as a working fluid. It is isothermal above  $450^{\circ}$ C with a maximum working temperature of  $1100^{\circ}$ C. The quench system was also modified so that any length of tube could be drawn through the furnace and quench system. This configuration is shown in Fig. 7.

Using System IV, a number of runs were made using the aluminumnickel eutectic to examine the effects of various growth rates and separations of quench and furnace. In each case, cross-sections transverse to the ingot axis were used to examine the structure of the ingots.

The final addition made was to add a shield system around the ingot below the furnace but above the quench for the purpose of eliminating radial heat flow. This radial heat flow suppressor had a varying cross section in order to match the thermal gradients in the ingot. It is shown in Fig. 8.

<u>Microstructural Evaluation</u>. As found in previous studies, the Al<sub>3</sub>Ni rods in the aluminum-nickel eutectic appear to grow with equal factor in two directions: one is parallel to the ingot axis, while the is in the neighborhood of  $15^{\circ}$  to the solidification direction. This is rise to effects in the transverse sections which appear to be variations in fiber density.

If the aluminum-nickel ingots had perfect microstructure, the Al<sub>3</sub>Ni rods would be equally spaced and would be in an hexagonal array. In evaluating the structure, the average nearest neighbor distance is determined, and the distribution of distances is obtained. The dispersion of the distribution can be used as a measure of the deviation from the perfect hexagonal array.

Bends, kinks, and branches in the structure all constitute defects, as does the existence of cellular structure.

Two Al-Al<sub>3</sub>Ni specimens were subjected to an analysis by Dr. Theo Kattamis, Institute of Materials Science, University of Connecticut. These specimens were solidified in Systems I and II (resistance heating, moving quench, and induction heating, fixed quench, respectively). The remaining specimens were examined in transverse section to indicate progress in achieving the goal of improving the microstructure.

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Results of the Microstructural Study. Illustrations of the typical faults observed in teh aluminum-nickel system are shown in Figs. 9, 10 and 11, which illustrate fiber dual orientation "density waves" in the transverse section caused by the dual orientation as seen in a longitudinal section, and colony structure. A high-quality structure in transverse section, showing only the deviation from hexagonal perfection, is shown in Fig. 12.

Table I and II indicate the ingots grown in Systems I and II, and examined at the University of Connecticut. Figures 13 and 14 show the variation in solidification rate for these furnace systems, obtained from measured characteristic nearest neighbor distances using copperaluminum eutectic ingots.

The distribution of the characteristic dimension, i.e. the nearest neighbor distance, for the Al-Al<sub>3</sub>Ni ingots studied, is given for the ingot from System I in Fig. 15, and from System II in Fig. 16.

Examination was made in a similar fashion but less extensively, of ingots solidified in System III and also in System IV. Data on characteristic distance variation and growth rate are given in Table III and Table IV for these systems. These results are presented graphically in Figs. 17 and 18, showing the data on nearest neighbor distance variation.

The result of this study shows that at a condition of a high thermal gradient in the liquid or a suppressed radial heat flow, the solid-liquid interface in the aluminum-nickel eutectic tends to be flat, with a reduction in the tendency to form colonies. The high thermal gradient reduces the distance along the ingot axis over which the temperature, as a function of radial distance from the center of the ingot can be the eutectic temperature, thus flattening the interface. This becomes less effective as the diameter of the ingot becomes larger. Suppressing the radial heat flow improves the flatness of the interface without the penalty of dependence on the ingot size. It appears that the rate of solidification is also made more nearly constant with the use of the radial heat suppressor device. Further experience with this technique will be required to optimize its use.

### Bending of Eutectic Structures

<u>Background</u>. The application of eutectics with their unidirectional microstructure will require the fabrication of parts with varying cross sections and non-cylindrically symmetric cross-sections. Under such circumstances, the attempt to directionally solidify a eutectic alloy in the desired shape will cause the structure to deteriorate if the part is not designed to accommodate the degree to which a directionally solidifying eutectic can follow changing contours. Therefore, this task was directed toward examining the degree to which the structure of a directionally solidified ingot can follow changes in contour. It was felt that once this was evaluated, it could then be compared to how a eutectic structure follows ontours in space.

<u>Experimental Studies</u>. During the directional solidification of both the aluminum-copper and the aluminum nickel systems, gas bubbles could be entrapped both in the body of the melt and attached to the crucible walls. Examinations of the microstructure were made at such defect points to elucidate the effect on the microstructure of the required changes in growth direction. Examination was also made of a high temperature alloy cent and directionally solidified on a doublepedestal-shaped mold.

<u>Microstructural Evaluation</u>. Entrapped gas pockets were observed in ingots of both Al-Al<sub>2</sub>Cu and Al-Al<sub>3</sub>Ni. The resulting growth patterns are shown in Figs. 19 and 20. In addition to these relatively large pockets, small voids were observed in the body of several ingots. Sections through such voids in the Al-Cu and Al-Ni systems are shown in Fig. 21. For the lead-tin eutectic system solidified in thin-film form, growth around a hole is shown by Fig. 22. The response exhibited by a Ni-Nb-Al eutectic alloy to solidification in a double pedestal mold is shown in Fig. 23.

Since the copper-aluminum system produced parallel plates in its microstructure, the response of microstructure to an obstacle in its normal growth direction should depend on the orientation of the plates with respect to the surfaces of the obstacle. It has been observed that terminations of either rods or plates in eutectic systems upon encountering obstacles does not significantly disrupt the structure; the renucleation of the microstructure produces the greatest problem. Given the studies performed so far, whether in this work or in other investigations, it is difficult to separate the temperature effects from the

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shape effects when studying the areas where the eutectic microstructure is non-regular due to an obstacle in the solidification path.

Results of the Microstructural Examination. It has been found by other workers, and confirmed by this examination that  $90^{\circ}$  bends or horizontal surfaces are to be avoided when designing products to be made by unidirectionally solidifying eutectic materials. Contours diverging at angles up to  $45^{\circ}$  from the growth direction still permit relatively regular growth of the microstructure, but beyond that value, a broken up structure results. In the case of fiber-type eutectic materials, regions may be denuded of fibers when growth from a new surface is required due to an obstacle or contour change.

Formation of Thin Sheets of Eutectics

<u>Background</u>. Many of the uses for eutectics require relatively thin sections. Therefore, it was of interest to determine the conditions under which the regular eutectic microstructure could be obtained in thin sheet form directly. The next step would be to compare these results with those obtained in a space experiment to determine if more perfect thin microstructures could be attained.

Experimental Studies. Three experimental operations were performed to elucidate the nature of eutectic growth in thin sheet form. Vacuum arc melting of small buttons resting on a water-cooled copper hearth was investigated as a way to obtain rapid cooling and high gradients where the button was in contact with the hearth. A second experiment was run where a piece of lead-tin eutectic was placed between two copper rods, each cooled, and melted with a zone heater. In this case, the eutectic wet the copper rods, and growth was initiated by traversing the molten eutectic region out of the hot zone. This operation is schematically shown in Fig. 24. The third experiment consisted of melting traveling zones in thin sheets of the lead-tin eutectic. These sheets were formed by rolling previously cast eutectic compositions to thicknesses of the order of 25 microns (0.001 inch). They were supported on sand-blasted pyrex microscope slides and positioned under a hot wire which travelled over them, melting a zone as it progressed. This set-up is shown schematically in Fig. 25.

<u>Microstructural Examination</u>. The buttons which were vacuum-arc melted and frozen on the cold hearth were sections and polished for microstructural evaluation. The structures observed are shown in Fig. 26. It can be seen that the characteristic sizes obtained in the micro-

structure are about the same as those obtained in the more usual solidification techniques. It appears that the heat transfer from the melted buttons to the cold hearth was not particularly rapid.

In the case of the lead-tin eutectic melted between two copper rods, it can be clearly seen in Fig. 27 that a certain distance is required for nucleation and rearrangement of the microstructure into the regime in which it can grow. This initiation phenomenon will always occur unless the solidification is initiated from a previously solidified specimen.

The attempt to grow thin sheets of the lead-tin eutectic appears to be promising. Experimental difficulties aside, Fig. 28 shows some of the structure obtained in the zoned sheets. Viewed perpendicular to the growth plane, the structure consists of lamellae growing in the direction of the moving zone. The lamellae thus have their characteristic size in the plane of view, and up to the film thickness perpendicular to the plane of view. Transverse sections of the thin film lead-tin eutectic samples are shown in Fig. 29.

As reported in Ref. 12, small areas of perfect lamellar lead-tin eutectic structure were grown by the above method. It appears that for sheets of thickness less than some critical dimension, the shape of the solidifying specimen exerts an influence on the structure which is not found in larger specimens. Pursuit of this technique could lead to interesting results in terms of eutectic microstructure.'

### Floating Zone Techniques

<u>Background</u>. Restricting the size of the molten section of an ingot being directionally solidified provides a means of obtaining very high thermal gradients. In addition, it can, if well controlled, be restricted to a sufficiently narrow zone so that the molten material is supported by the surface tension of the material itself, with no requirement for a crucible. It should also be capable of providing a constant rate of solidification, a requirement for obtaining a good microstructure.

A ring source of radiant energy is commonly provided to form the molten zone when the specimen is in the form of a cylindrical ingot. If the melting temperature is sufficiently high, radiative cooling will provide a sufficient source of heat removal to maintain size control. As the melting point of the material to be zoned decreases, additional sources of cooling must be provided, since radiation will be inadequate.

<u>Experimental Studies</u>. In the present study, the shape of the material in the molten zone was examined for some high melting point materials. The effect of the weight of the molten material in causing the melted material to run out of the zone was clearly observed as shown in Fig. 30. In such a case, processing in zero-gravity conditions would clearly be advantageous.

The main examination of zone melting was done in conjunction with studying the solidification and microstructure of off-eutectic compounds. In particular, the zone melting apparatus used provided for a crucible to contain the material. The main material studied was the lead-tin system, in which the eutectic melts at 183°C. The apparatus is shown in Fig. 31. Using 4 mm diameter rod specimens, the interface had clearly discernable curvature. Reducing the dimension to 2 mm improved the flatness of the interface.

A modification to the above system was investigated which provided zero contact between the molten material and the crucible. This consisted of forcing gas at relatively low pressure and flow rate between the ingot and the crucible from below. When the temperature of the zone region was raised to cause melting, the molten material expanded and flowed down the lower part of the still-solid rod until its attempt to reach the crucible walls was stopped by the gas flow. An equilibrium diameter of the molten zone was reached when the restriction provided by the molten flow raised the pressure of the gas flowing up the tube to the value required to support the molten material. Additional experimentation with this technique will be required to determine if it will provide a simulation of containerless solidification using zone melting techniques.

### Off-Eutectic Solidification

<u>Background</u>. To use materials with the microstructure obtained by unidirectional solidification of eutectic, it is important to be able to control the number of rods in a matrix or to be able to produce a rod-like microstructure from phases which normally form plate-like microstructures, or vice-versa. All of these things are theoretically possible in off-eutectic solidification, where the ability to vary the composition adds a degree of freedom relative to the fixed composition eutectic.

In off-eutectic solidification, there is no constraint on composition, but it is more difficult to obtain a controlled structure. It is important in this type of solidification to minimize any effects which might disturb the boundary layer which forms in front of the solidifying interface. In Ref.12, for example, it is pointed out that convection currents can have deleterious effects on the microstructure. In vertical growth under gravity conditions, another problem exists if the density of the major part of the liquid above the solid is greater than the density of the boundary layer. In such a case, the heavier liquid tends to displace the boundary layer liquid. These effects can be minimized in a zero-g environment.

Experimental Studies. As pointed out in Ref.13, convection is less of a problem in the unidirectional solidification of off-eutectic compositions if a zone melting technique is used. In addition, very high gradients can be obtained. These experiments have been repeated using the lead-tin system, and have verified that in the zone melted and solidified region, the composition is in fact the gross off-eutectic composition. At the same time a eutectic-like microstructure was formed. For small diameter rods (of the order of 2 mm diameter) the solid-liquid interface in a zone melting system will be relatively flat. As the diameter increases, more curvature appears, which is an undesirable effect.

<u>Results</u>. The results of this experimentation were obtained with the zone furnace illustrated in Fig. 31. The microstructures obtained are illustrated in Figs. 32, 33 and 34 for compositions, 30w/o lead -70w/o tin, 38.1w/o lead - 61.9w/o tin, and 45w/o lead - 55w/o tin. To check the composition after zone melting, a portion of the 30w/o lead-70w/o tin sample was examined with the electron microprobe. A quantitative element analysis gave 30.2w/o lead, 69.8w/o tin.

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### DISCUSSION AND CONCLUSIONS

The technologies of solidification studied in this program show that a significant improvement in thermal control during the solidification process must be made before even the earth-based processes will exhibit their full potential in terms of controlled microstructure. The solidification process is a complex one from the thermal design point of view, and a complete analysis of the thermal character of a given method of unidirectional solidification is required in order to improve that particular process. Such thermal design analysis will be similarly critical for a space laboratory experiment.

This program has also shown that a desirable microstructure can be obtained by directional solidification under very high thermal gradients when the composition is off the eutectic one. This important result provides a degree of freedom in materials design not available using eutectic compositions.

- It is concluded from the studies completed in this program that
- further studies of thermal processing should be made using means of suppressing radial heat flow in the solid-liquid interface region during solidification by withdrawal from a furnace through a fixed position quench;
- 2. solidification employing basically zone techniques should be further studied;
- 3. the effects of the thermal gradient on the resulting microstructure should be evaluated;
- 4. an analytical approach should be coupled with the experimental studies to elucidate the temperature distribution within the solidifying specimen as a means of significantly improving the design of unidirectional solidification systems.

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### Table I

### Microstructural Analysis of Al-Al<sub>3</sub>Ni Eutectic Ingot Solidified in Furnace System I

Specimen A73-475; Rate = 3.5 cm/hr; Section 10 cm from head Data Supplied by the Materials Science Institute, University of Conn.

Fiber Diameter	1.10	micrometers
Fiber Spacing - nearest neighbor approach	3.30	micrometers
Fiber Spacing/Fiber Diameter	3.0	
Perfection <sup>#</sup> of hexagonal array - dispersion of		
fiber spacings	0.297	,
Volume fraction of Al <sub>3</sub> Ni fibers	9.7%	

\*Perfection = zero dispersion

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### Table II

### Microstructural Analysis of Al-Al<sub>3</sub>Ni Eutectic Ingot Solidified in Furnace System II

Specimen A73-482; Rate = 5 cm/hr; Section 10 cm from head Data supplied by the Materials Science Institute, University of Conn.

Fiber Diameter	1.28	micrometers
Fiber Spacing - nearest neighbor approach	2.78	micrometers
Fiber Spacing/Fiber Diameter	2.17	
Perfection" of hexagonal array - dispersion of		
fiber spacings	0.606	
Volume fraction of Al <sub>3</sub> Ni fibers	10.5%	

**\***Perfection = zero dispersion

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### Table III

### Structural Perfection Analysis of Al-Al<sub>3</sub>Ni Eutectic Specimens

Furnace System III

Ingot <u>No.</u>	Section	Withdrawal	Average* Inter-Fiber Spacing	Dispersion** of Inter-Fiber Spacing Curve
UDS721-		cm/hr	micrometers	
10	l	4.0	1.58	0.067
11	4 <b>T</b>	12.1	1.57	0.027

\*Average fiber spacing determined from number of fibers per unit area \*\*Dispersion of curve obtained by measuring actual inter-fiber spacings

### Table IV

### Structural Perfection Analysis of Al-Al<sub>3</sub>Ni Eutectic Specimens

### Furnace System IV

Ingot <u>No.</u> UDS721-	Section No.	Withdrawal Rate	Average <sup>#</sup> Inter-Fiber Spacing	Dispersion** of Inter-Fiber <u>Spacing Curve</u>
27	8	4.0	1.65	0.031
	9	4.0	2.07	0.090
	10	4.0	2.73	0.199
	11	4.0	2.25	0.229
	12	4.0	2.88	0.378
	13	4.0	2.88	0.920
34	7	4.0	2.12	0.454

\*Average fiber spacing determined from number of fibers per unit area \*\*Dispersion of curve obtained by measuring actual inter-fiber spacings

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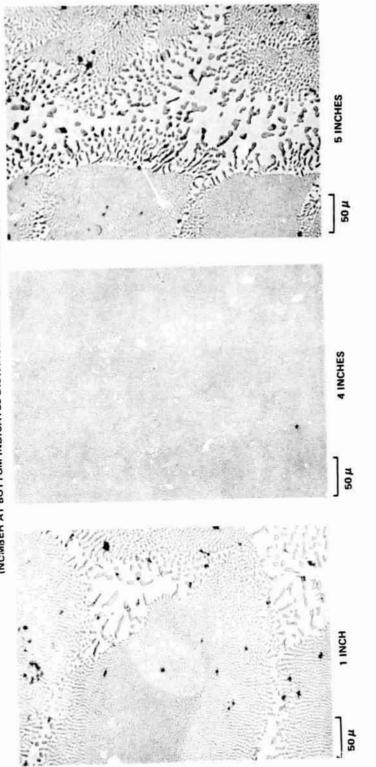
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N911721-12 VIEWING PORT UPPER WATER COOLED ELECTRODE ARGON IN-A GRAPHITE HEL!X HEATER TUBE ALUMINA ........BLS LOWER WATER SIGHT PORT FOR COOLED ELECTRODE OPTICAL PYROMETER ARGON IN WATER COOLED FURNACE SUPPORT -PEDESTAL TRAVERSE DIRECTION TRAVERSE ATTACHMENT WATER IN WATER OUT

# FIGURE 1. SYSTEM I - GRAPHITE DIRECTIONAL SOLIDIFICATION FURNACE

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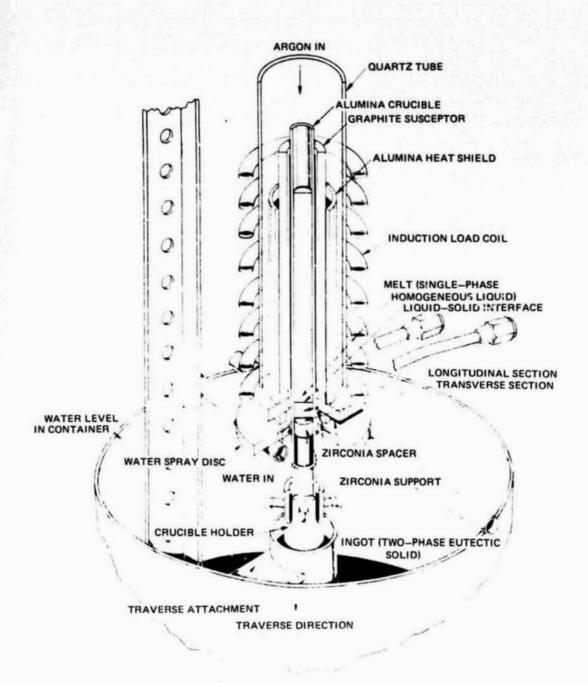
(INCOT A73-475 R = 3.5 cm/hr GRAPHITE RESISTANCE CURNACE) (NUMBER AT BOTTOM INDICATES DISTANCE FROM HEAD OF INGOT)

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FIGURE 2. TRANSVERSE SECTIONS OF CONTROLLED AI-AI3Ni EUTECTIC

SOLIDIFIED IN SYSTEM I FURNACE

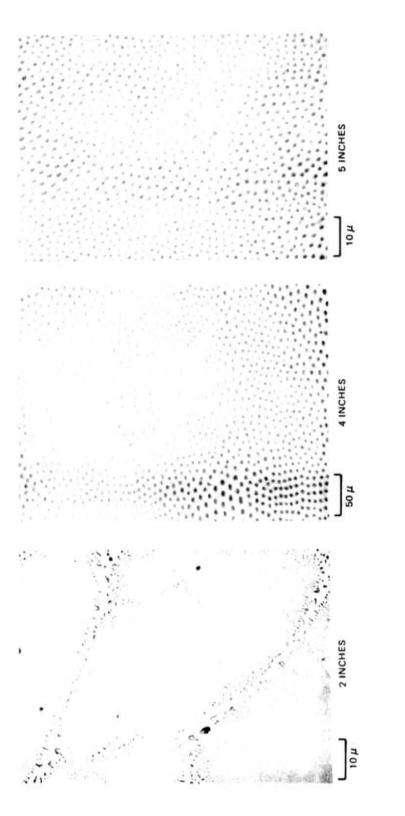
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### FIGURE 3. SYSTEM II INDUCTION HEATED HIGH THERMAL GRADIENT DIRECTIONAL SOLIDIFICATION APPARATUS

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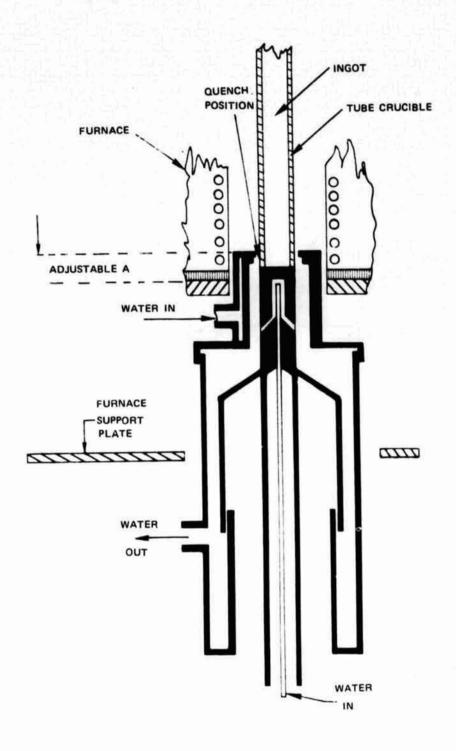


# FIGURE 4. TRANSVERSE SECTIONS OF CONTROLLED AI-AI3NI EUTECTIC INGOT DIRECTIONALLY SOLIDIFIED IN SYSTEM II FURNACE

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### FIGURE 5. SYSTEM III - RESISTANCE HEATED HIGH-GRADIENT FURNACE

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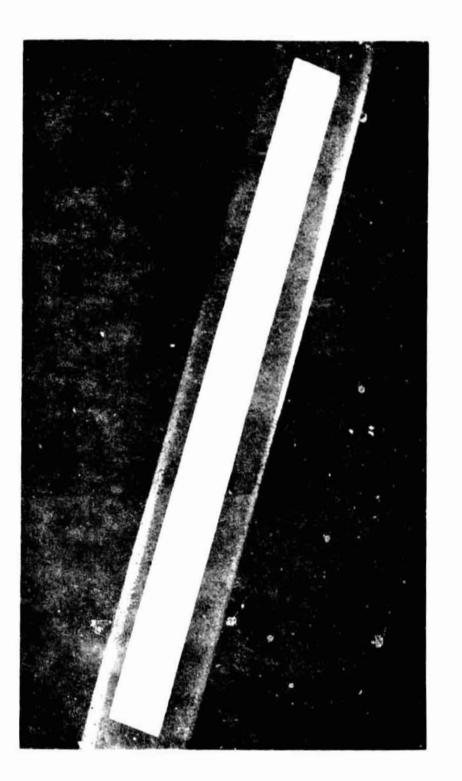


FIGURE 6. LONGITUDINAL SECTION OF AI-AI3NI INGOT 721-32 DIRECTIONALLY SOLIDIFIED IN SYSTEM III FURNACE SHOWING INTERFACE CURVAIURE

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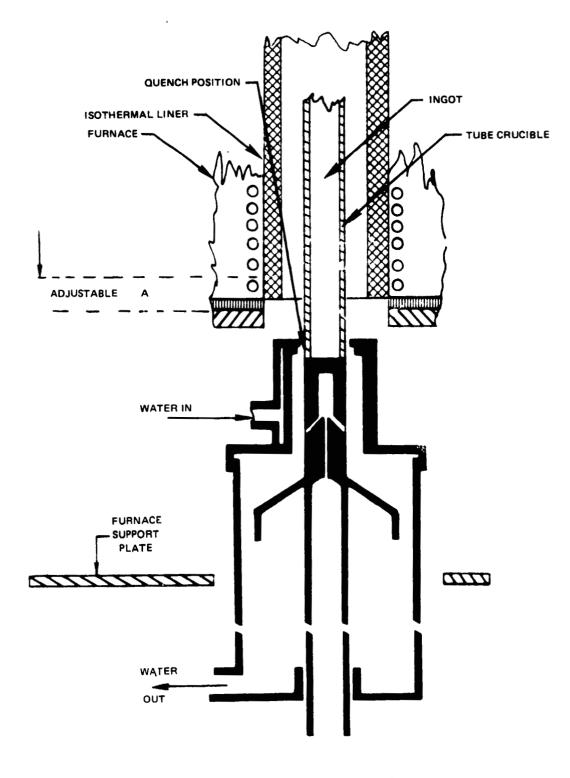


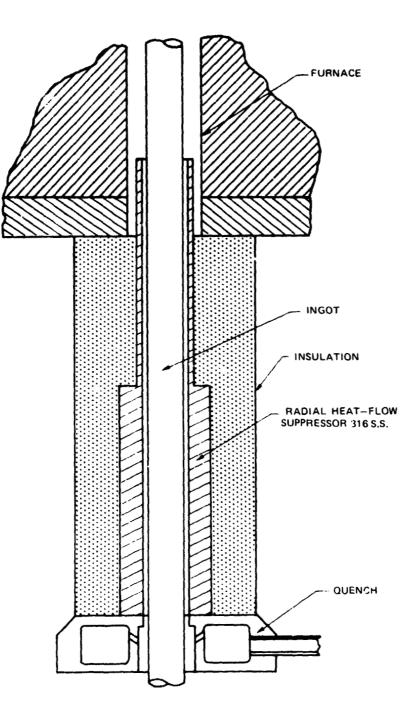
FIGURE 7. SYSTEM IV – MODIFIED RESISTANCE HEATED HIGH–GRADIENT FURNACE

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### FIGURE 8. RADIAL HEAT-FLOW SUPPRESSOR IN POSITION RELATIVE TO FURNACE, QUENCH, AND INGOT

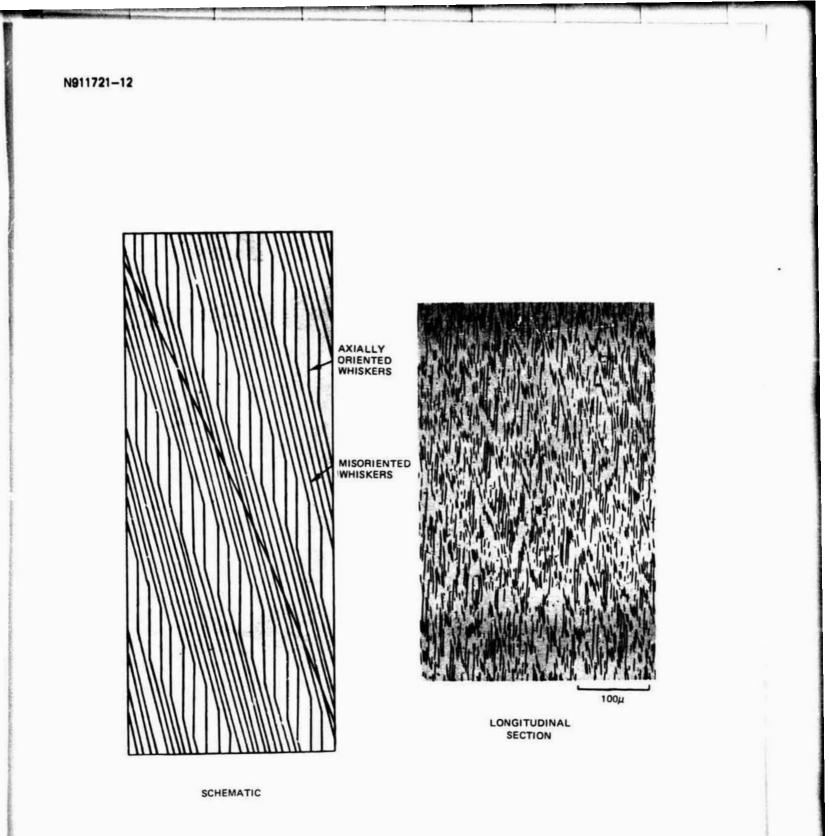
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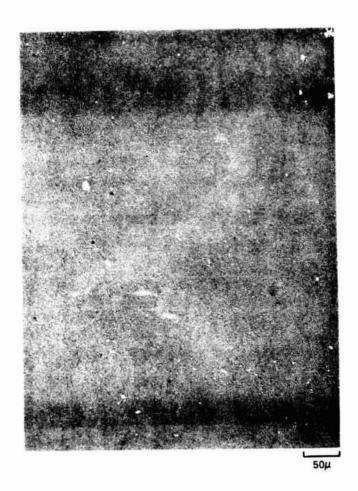


### FIGURE 9. SCHEMATIC DRAWING, AND MICROGRAPH OF LAYERS IN DUAL ORIENTATION AI-AI<sub>3</sub>Ni MICROSTRUCTURES

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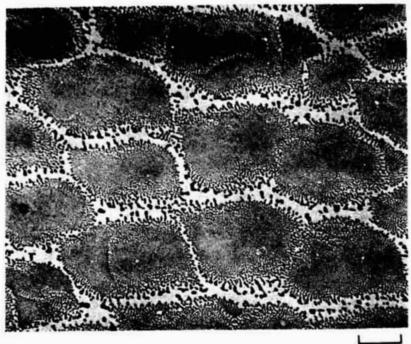
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### FIGURE 10. TRANSVERSE SECTION OF CONTROLLED AI-AI<sub>3</sub>Ni EUTECTIC "DENSITY WAVES" DUE TO DUAL ORIENTATION OF AI<sub>3</sub>Ni RODS)

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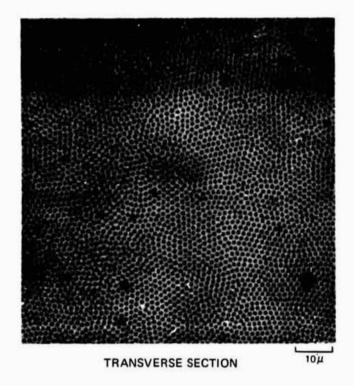


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FIGURE 11. TRANSVERSE SECTION OF AI-AI<sub>3</sub>Ni EUTECTIC SHOWING COLONY STRUCTURE

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## FIGURE 12. HIGH QUALITY STRUCTURE IN AI-AI3NI EUTECTIC



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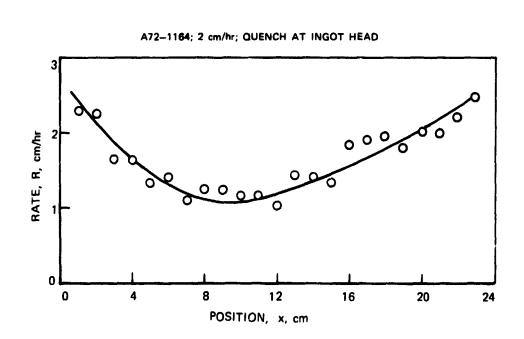


FIGURE 13. SYSTEM I - SOLIDIFICATION RATE VS POSITION IN A DIRECTIONALLY SOLIDIFIED AI-AI<sub>2</sub> Cu INGOT

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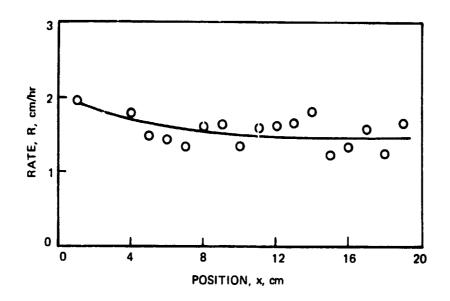
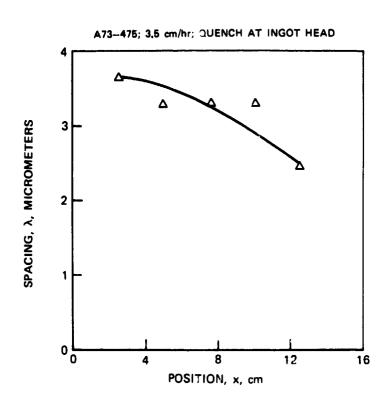


FIGURE 14. SYSTEM II - SOLIDIFIED RATE VS POSITION IN A DIRECTIONALLY SOLIDIFIED AI-AI<sub>2</sub> Cu EUTECTIC INGOT

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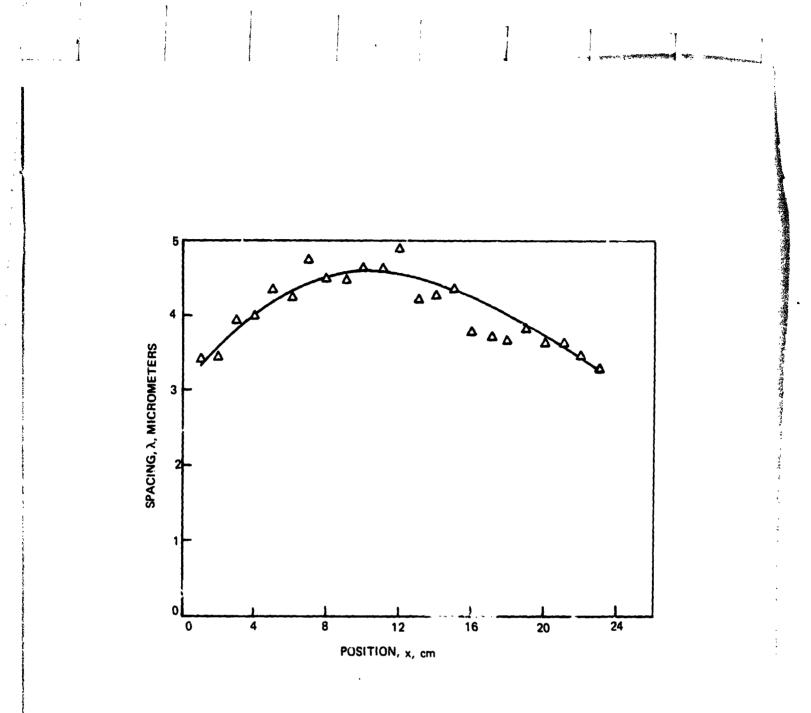
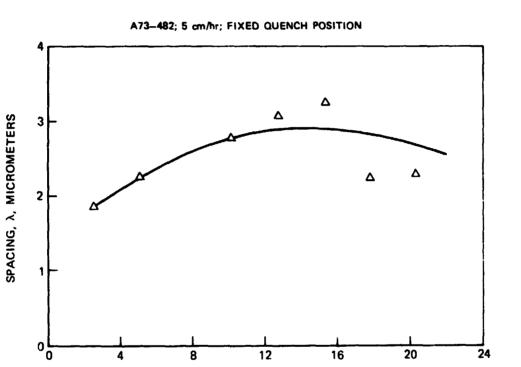


FIGURE 15. SYSTEM I - CHARACTERISTIC SPACING VS POSITION IN A DIRECTIONALLY SOLIDIFIED AI-AI<sub>2</sub> Cu iNGOT



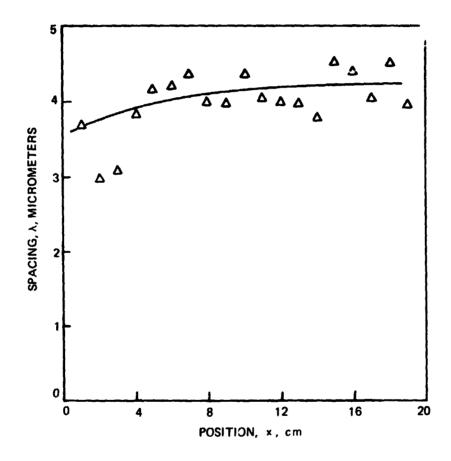
# FIGURE 16. SYSTEM II - CHARACTERISTIC SPACING VS POSITION IN A DIRECTIONALLY SOLIDIFIED AI-AI<sub>3</sub>Ni EUTECTIC

POSITION, x, cm

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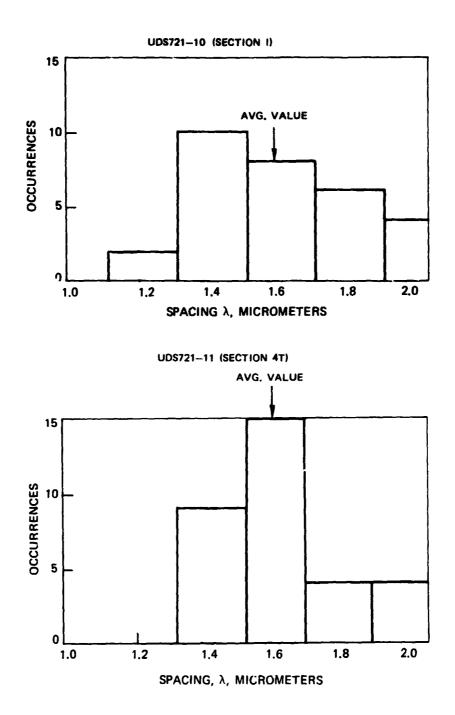
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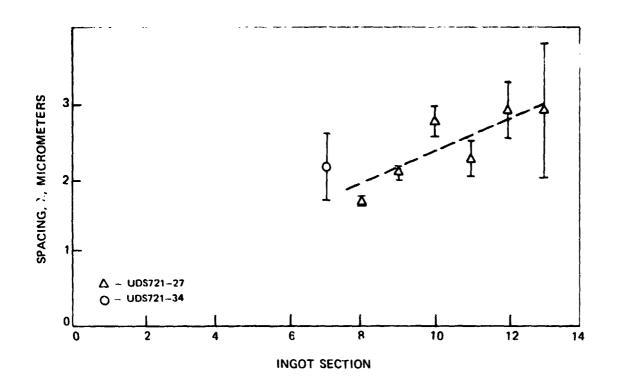
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FIGURE 18. SYSTEM IV - CHARACTERISTIC SPACING VS INGOT SECTION IN A DIRECTIONALLY SOLIDIFIED AI-AI<sub>3</sub>Ni EUTECTIC INGOT

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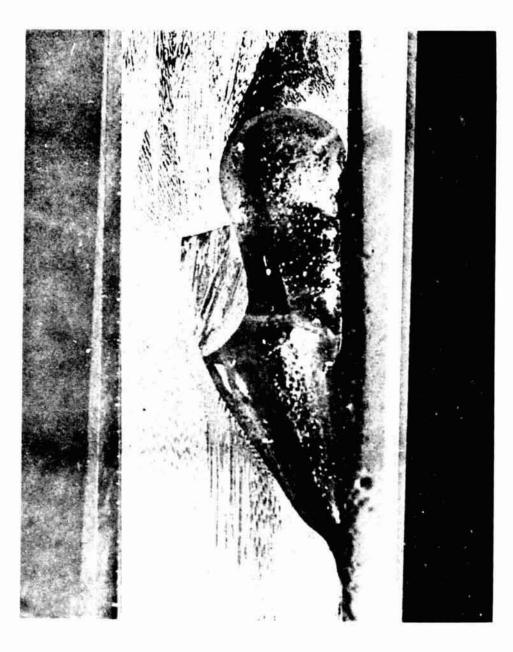
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### FIGURE 19. GROWTH PATTERN OF THE AI-AI<sub>2</sub>Cu LAMELLAR STRUCTURE AROUND AN ENTRAPPED GAS POCKET

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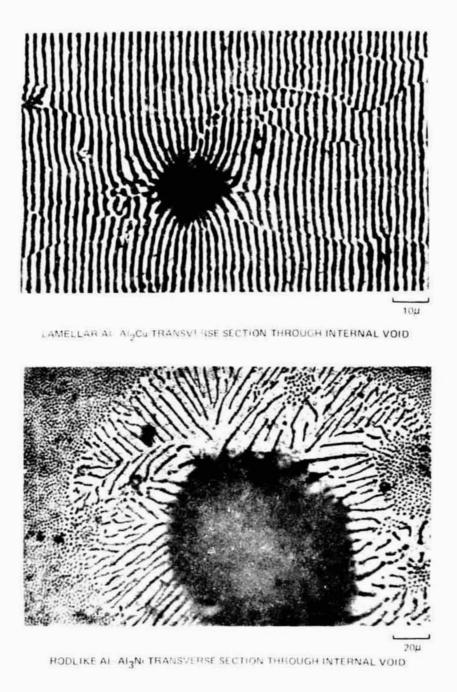


### FIGURE 20. GROWTH PATTERN OF THE AI-AI<sub>3</sub>Ni FIBROUS EUTECTIC AROUND AN ENTRAPPED GAS POCKET

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#### FIGURE 21. ACCOMMODATION OF EUTECTIC MICROSTRUCTURE TO REGIONS REQUIRING CHANGES IN GROWTH DIRECTION

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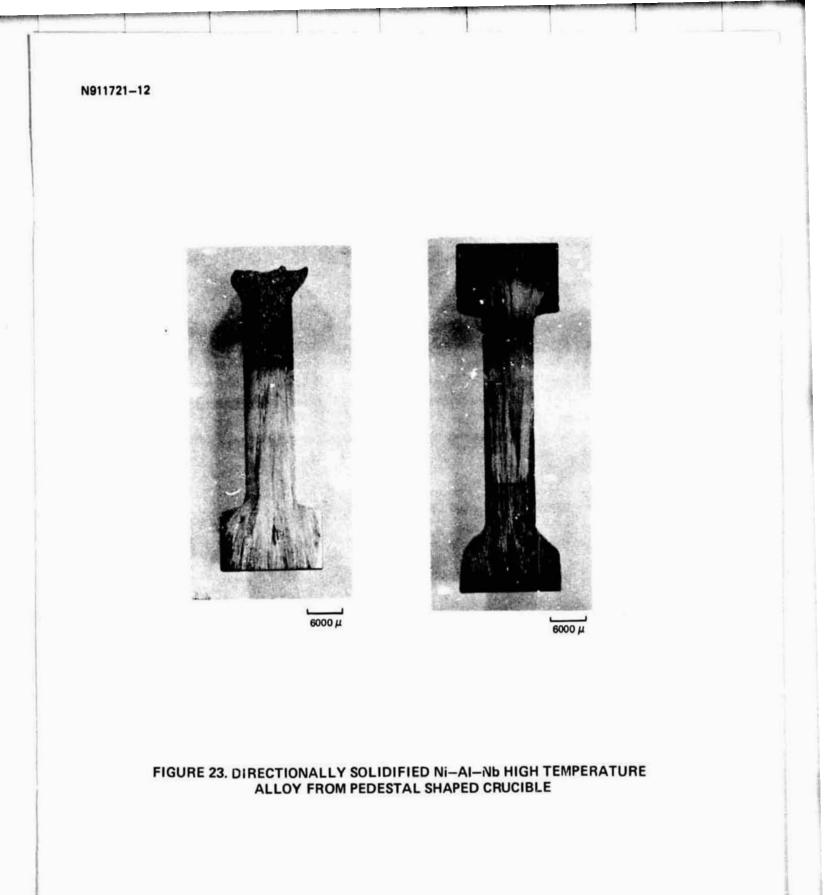


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## FIGURE 22. GROWTH OF THE LEAD-TIN LAMELLAR STRUCTURE AROUND A VOID DURING THIN FILM SOLIDIFICATION

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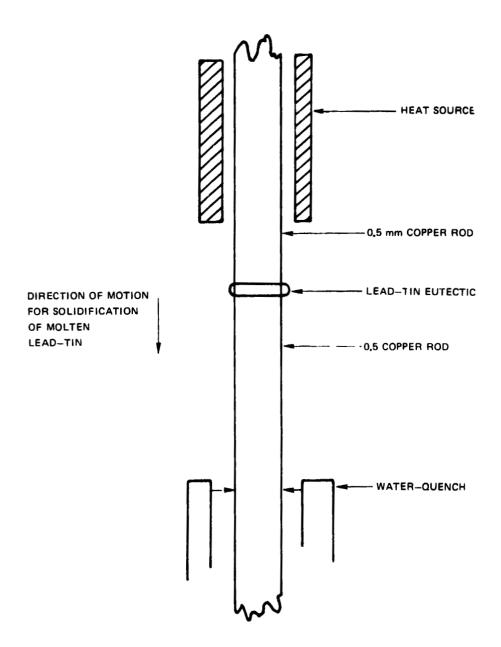
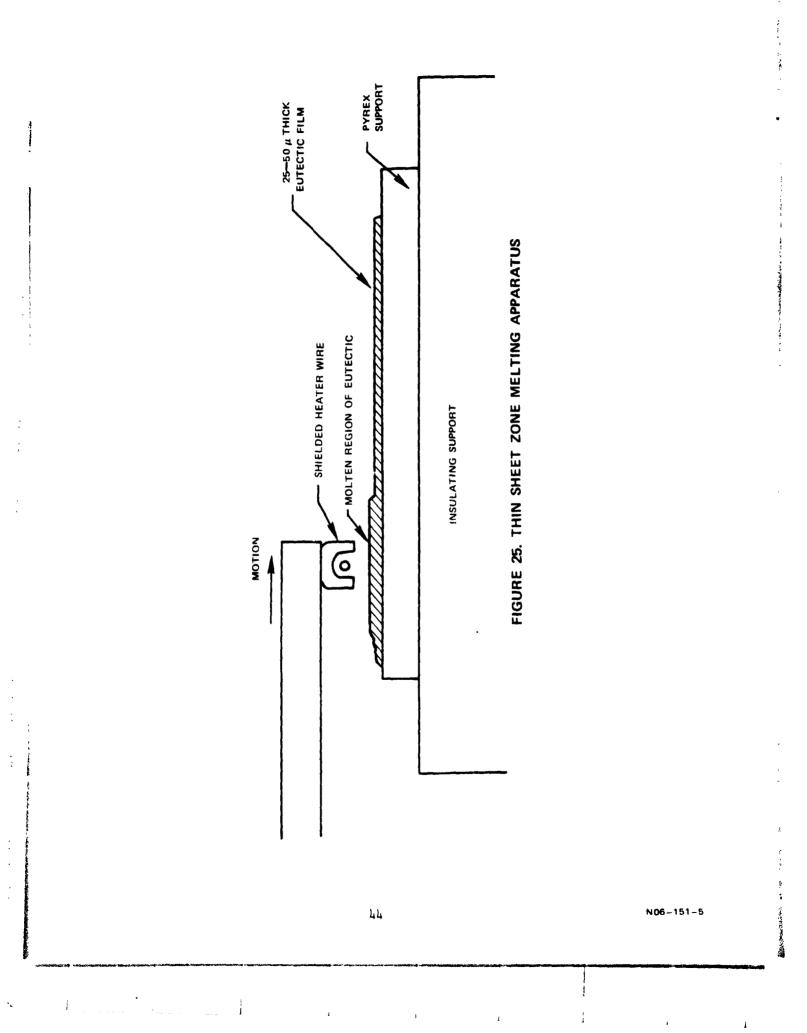


FIGURE 24. SCHEMATIC ILLUSTRATION OF THIN-SHEET LEAD-TIN SOLIDIFICATION

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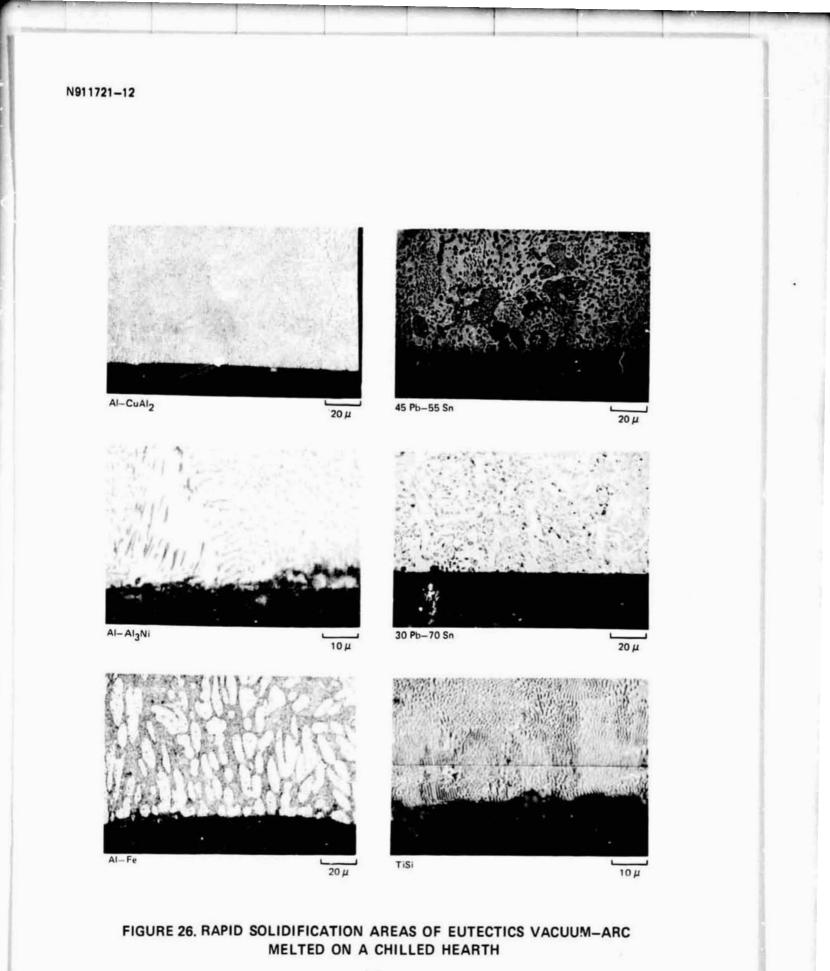
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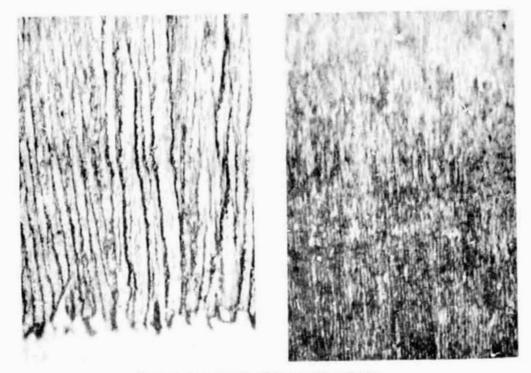


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UDS 721–31



DETAILS OF MICROSTRUCTURE WITHIN GRAINS

## FIGURE 27. DIRECTIONALLY SOLIDIFIED THIN SHEET OF LEAD-TIN EUTECTIC

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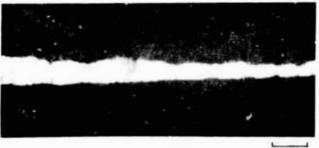
10µ

## FIGURE 28. MICROSTRUCTURE IN THE PLANE OF GROWTH OF 40 MICROMETER THICK Pb-Sn EUTECTIC FOILS

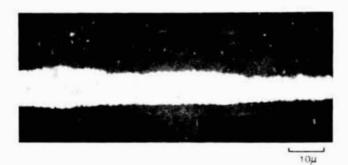
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10µ



20μ

FIGURE 29. MICROSTRUCTURE OF TRANSVERSE SECTIONS OF THIN FILM LEAD-TIN EUTECTIC

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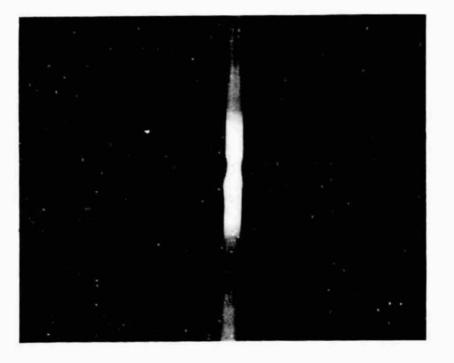
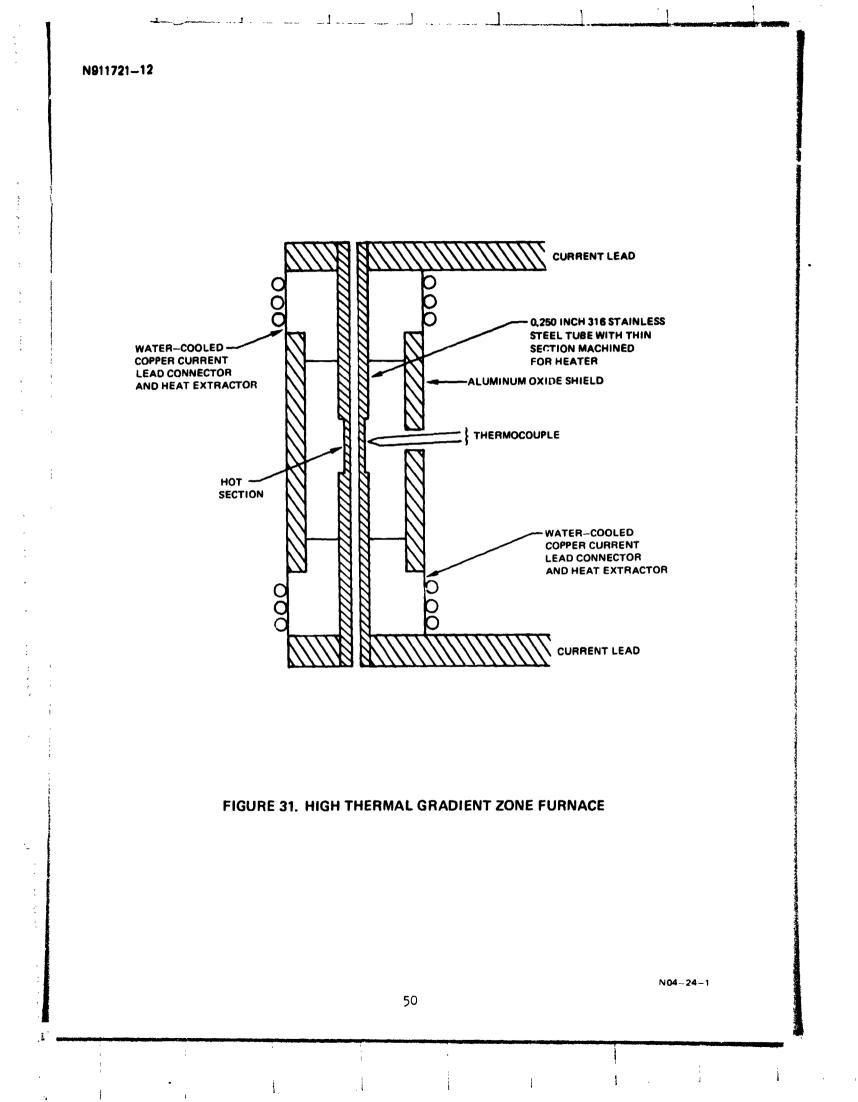
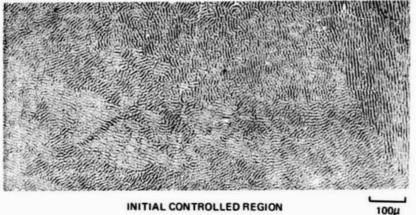


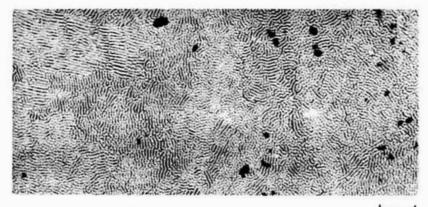
FIGURE 30. COLD CATHODE MELTED ZONE IN AI203

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UDS 721-41 330°C/cm @ 0.8 cm/hr (EUTECTIC: 38.1% LEAD)





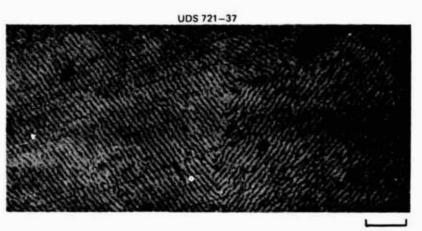
2 cm INTO CONTROLLED REGION

100µ

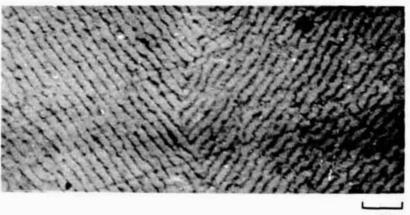
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#### FIGURE 32. MICROSTRUCTURE IN 30% LEAD-70% TIN ZONE DIRECTIONALLY SOLIDIFIED OFF-EUTECTIC ALLOY





20 µ

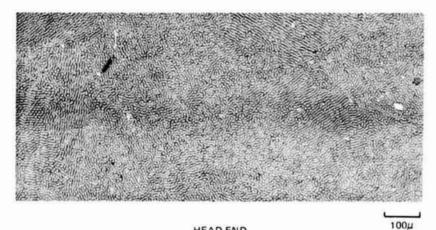


10 µ

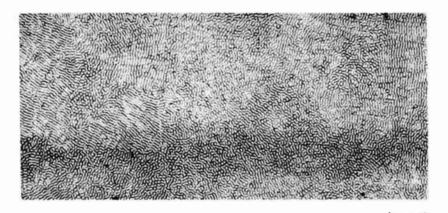
FIGURE 33. MICROSTRUCTURE OF LEAD-TIN EUTECTIC ZONE MELTED AT 3.4 cm/hr WITH A THERMAL GRADIENT OF 165° C/cm

N03-46-2

#### UDS 721-40 330°C/ cm @ 0.8 cm/hr (EUTECTIC: 38.1% LEAD)



HEAD END



6 cm PAST HEAD END

#### FIGURE 34. MICROSTRUCTURE IN 45% LEAD-55% TIN ZONE DIRECTIONALLY SOLIDIFIED OFF-EUTECTIC ALLOY

N04-155-1

100µ