P24P

N911360-24

RESEARCH STUDY ON MATERIALS PROCESSING IN SPACE, M566 EXPERIMENT

PHASE B AND C REPORT ON CONTRACT NAS8-28724

AUGUST 1, 1972 - JUNE 18, 1974

Prepared for

NASA-GEORGE C. MARSHALL SPACE FLIGHT CENTER
MARSHALL SPACE FLIGHT CENTER, ALABAMA 35812

PREPARED BY:

Frank C. Douglas
Francis S. Galasso

United Aircraft Research Laboratories

UNITED AIRCRAFT CORPORATION

EAST HARTFORD, CONNECTICUT 06108

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

SUMMARY

Specimens of the aluminum-33 wt% copper eutectic partially melted and resolidified in the low effective gravity of the orbiting Skylab were examined and characterized with respect to microstructural defects and thermal conductivity values. The results obtained were compared with similar evaluations of ground-based simulation melt-resolidification experiments and as-prepared unidirectionally solidified specimens. Assistance was supplied to Marshall Space Flight Center on a consulting basis during the operation of the program as required during ground-based testing and simulation of the Skylab furnace system.

Thermal conductivity data and electrical resistivity data at temperatures from 25°C to 400°C did not show significant differences between ground and space processed specimens.

Because the furnace and capsule configuration were fixed for the space-laboratory operations, no significant changes could be made in its design, In this furnace, solidification of the remelted portion of the specimens as performed in the ground-based and space-laboratory experiments could not proceed at a constant rate. It was observed, however, that single grain renucleation occurred in both cases using the single grained starting specimens under the operating conditions of a one-hour constant furnace temperature soak period prior to the cool-down cycle. In contrast, when a three-hour soak period was used in a ground-based test, six different grains were nucleated.

A methodology of evaluating the defects in the Al-Al_2Cu structure was implemented with Dr. Theo Kattamis, Materials Science Institute, University of Connecticut. A specimen from Skylab 3 showed signs of instability in growth and several grains were found in the ingot. The specimen from Skylab 4 did not show such marked instability in growth and was found to contain fewer defects than the ground-processed specimens. This agrees with data from Georgia Institute of Technology which showed that there were fewer defects in both their Skylab 3 and 4 specimens than in ground processed specimens.

INTRODUCTION

The objective of this program was to assist the Marshall Space Flight Center S&E-PE Laboratory in conducting and evaluating experiments on the unidirectional solidification of eutectic alloys in the nearly zero-g environment of an orbiting space laboratory.

Three major areas of investigation were pursued: (1) planning of experiments on the ground which contributed to optimization of the experiment to be conducted in space; (2) assistance to MSFC in conducting the planned experiments; and (3) evaluation of flight samples after they have returned from being processed in space.

In exploring the effects of processing materials under reduced g-forces, the unidirectional solidification of eutectics could provide important information on the gravitational effects operative in earth-bound solidification, and could lead to more perfect structures than are commonly obtained. The attainment of defect-free microstructures is particularly important in developing eutectics for nonstructural applications.

A number of research studies have been devoted to the production of aligned eutectic and off-eutectic microstructures in which the microstructure was thoroughly analyzed. Thus, several well characterized eutectic alloy systems exist which are suitable for use as a model. The aluminum-33 wt% copper system was selected by MSFC for use as such a model system.

LABORATORY TEST PROGRAMS

Specimen Fabrication

In order to provide specimens for the M566 Composite Casting Experiment which were to be used to determine the effect of a low g on the eutectic solidification phenomenon, it was concluded that each of the initial specimens should consist of a single grain in the region where nucleation of structure after a melt-back would occur. The M566 experiment was designed to use specimens 0.635 cm dia by 12.7 cm long, threaded on one end to facilitate anchoring to a cold plate and a radiation element to heat the other end with provisions for slowly lowering the temperature. Slightly greater than one-half the specimen length would be melted. Thus, an acceptable initial specimen would require a minimum of 6.35 cm of single grain eutectic material. The single grain end would be threaded and used as the cold end. The experimental procedures for producing the twenty-two specimens included the preparation of master-heats, the remelting and solidification of the eutectic alloy under controlled conditions, and the examination of the resulting ingots. The eutectic forms between an aluminum-rich solid solution (α, Al) and an intermetallic compound (θ, $CuAl_2$) at 67 percent by weight of aluminum. The phase diagram is shown in Fig. 1.

Preparation of Master Heats

Master heats of the Al-$CuAl_2$ eutectic alloy were melted and cast within a vacuum induction furnace shown in Fig. 2. The charge, composed of 99.999+% zone refined aluminum and spectrographic copper, was placed in a new high purity alumina crucible, the chamber evacuated to a pressure of 2×10^{-5} torr and then back-filled with argon gas, and the melt brought to a temperature of about 900°C. After holding the melt at 900°C for 60 minutes, the metal was allowed to cool to about 600°C at which temperature it was poured into a split copper mold and allowed to solidify under an argon atmosphere. Figure 3 shows this copper mold which yielded 4 cast bars 3/8 inch in diameter and 8 inches long.

Directional Solidification Apparatus and Procedures

The apparatus used to solidify the Al-$CuAl_2$ eutectic unidirectionally (shown in Figs. 4 and 5) consists basically of a graphite resistance furnace which heats tubular high purity alumina crucibles containing the 3/8 inch diameter cast bars of eutectic composition under a dynamic argon atmosphere. The alumina crucibles are held in a graphite holder which is mounted on a water-cooled pedestal. This assembly is lowered vertically at varying rates corresponding to nominal solidification rates using a variable-speed controller apparatus. The temperature of the liquid, controlled by the power input to the resistively heated graphite tube, was held at about 850°C. Either one or three eutectic ingots were directionally solidified during a test run. An assembly consisting of three alumina crucibles contained within a graphite holder is shown in Fig. 6.

Examination Procedures

After the completion of the directional solidification process, the ingots were separated easily from the alumina crucibles without any apparent sign of a reaction between the eutectic alloy and the crucible. However, because of the irregular internal surface of the crucible, this separation was accomplished by breaking away the alumina crucible. The ingots were then sectioned as shown in the insert of Fig. 6 approximately 1.2 cm from the tail end. This transverse section was then polished and etched to determine whether a single grain had been produced. Only ingots having a single grain at this section were examined further by cutting the ingot at a distance of 12.7 cm toward the head end from the original section. This transverse section was also polished and etched to determine the number of existing grains. The 12.7 cm long specimens were then centerless ground from an original diameter of 1.03 cm to 0.635 cm. The surface of these specimens was then macro-etched to determine the length of the single grain that was produced at the tail end. If the single grain length was 6.35 cm or greater the specimens were then finish machined to the ampoule design (shown in Fig. 7; the threaded end corresponding to the tail end of the ingot) as specified on drawing number 615B707 which was furnished by the Westinghouse Astronuclear Laboratory.

The transverse sections were polished by conventional means through Linde A and both the transverse sections and the specimen surfaces were etched with Keller's reagent, which consists of 95 ml H_2O, 1 ml HF, 1.5 ml HCl, and 2.5 mil HNO_3, for 20-30 seconds.

Directionally Solidified Specimens

Directionally solidified ingots of the Al-$CuAl_2$ eutectic alloy with a microstructure consisting of lamellae essentially parallel to one another were successfully produced as previously described in the experimental procedure. These ingots were 1.03 cm in diameter by approximately 25.4 cm long (see insert Fig. 6) and were solidified at a nominal rate of 2 cm/hr. In the growth of these ingots, a multigrained structure would be present at the head end, two to three grains at the center, and in general a single grain at the tail end.

Figure 8 is a transverse section of one of the few Al-$CuAl_2$ eutectic ingots which produced a multi-grained structure near the tail end. This figure shows both macroscopically and microscopically the difference between a grain boundary and a subgrain boundary. A grain boundary is defined here as a change in lamellar orientation where the boundary completely encloses a volume of material. A subgrain boundary is a lower angle change in lamellar orientation but is mostly distinguished by the fact that the boundary disappears within an encompassing grain.

Figure 9 shows a transverse section of one of the many Al-$CuAl_2$ eutectic ingots which produced a single grain near the tail end. This tail end section was typical of all the specimens that were used in fulfilling the objectives of this program.

The photographs in Fig. 10 represent two of the finished Al-$CuAl_2$ specimens that were produced. These specimens met both the single grain length requirement and the design specifications as stated in the contract. The only difference between the two specimens shown in Fig. 10 is that the threads in one were machined whereas the threads in the other specimen were ground. As can be seen the machined threads were chipped at some of the thread tips in two areas 180° apart from one another which was caused by the very nature of the single grain specimens. When the sharp tool bit makes a point contact with the material there is a tendency for the lamellae to chip off when the bit is parallel to the lamellae. The severity of this chipping varied from specimen to specimen. However, in the grinding process, there is a full and uniform contact made between the abrasive and the material which prevents this phenomenon from occurring. Although both type specimens were considered adequate for their intended application, once the practice of grinding the threads was incorporated it was considered as the best way of producing good quality specimens.

Twenty-two specimens, as listed in Table I, were produced to be used for the space-processing and ground-based processing of eutectics.

Materials Analysis

Chemical analyses were made on various aluminum-copper eutectic specimens in addition to the initial analyses of the starting materials and resulting directionally solidified eutectics. The results of the major component analyses are presented in Table II. An analysis by a combination of atomic absorption and emission spectroscopy was used to determine impurity levels. The results obtained, including those determined by the Westinghouse Astronuclear Laboratory, are presented in Table III.

Specimen Evaluation - Structure

Single-grained aluminum-copper eutectic specimens supplied to NASA-Marshall Space Flight Center were encapsulated for experiments in the Skylab M512 facility for solidification under low-g conditions, and for base-line experiments conducted on the ground at Marshall Space Flight Center.

The specimens received from Marshall Space Flight Center for examination at the United Aircraft Research Laboratories and the University of Connecticut were labelled M566-5 (Skylab 3, 10^{-4} g, max temp. 844°C), M566-7 (Skylab 4, 10^{-4} g, max temp. 867°C) M566-11 (ground-based experiment, max temp. 867°C) and M566-15 (ground-based experiment max temp. 844°C). In addition, an unprocessed original specimen A72-987A was used for comparison in the determination of defect density.

The evaluation of the eutectic specimens was based on observation of the microstructure resulting from the growth conditions. Defect types and evaluative procedures are specified in detail in Appendix I. The results of applying these procedures yielded a comparison between specimens or between sections of the same specimen in terms of the lamellar spacing, λ, which also yields the rate of solidification, the number density of discontinuities in the lamellar structure as observed in transverse sections, the average length of lines connecting such discontinuities in units of lamellar spacing (a characteristic length for the eutectic microstructure), and the average distance between such lines of discontinuity.

Specimens M566-15 (ground processed) and M566-5 (Skylab 3) were formed by melting back an unprocessed specimen to about 61 mm from the threaded (cool) end. The maximum temperature of the melt was 844°C. The microstructure and defect density data are presented in Table IV. Unfortunately the Skylab-3 sample M566-5 developed an instability which resulted in a pinching off of the melt starting at 75 mm from the threaded end. As a consequence of this instability two grains grew from the seed; they were clearly visible at 75 mm from the threaded end and by 100 mm a third grain was present. The defect density was found to be quite high in this sample.

A view of specimen M566-5, as received, is shown in Fig. 11. Note the indentation about 3/4 of the way up the sample which was described above. Figure 12 shows the interface and adjacent structure while Fig. 13 shows transverse sections along the specimen. Similar transverse sections for M566-15 (ground processed) are shown in Fig. 14.

Table V presents data for A72-987A (an unprocessed original specimen), M566-11 (ground processed) and M566-7 (Skylab 4). The latter two samples were formed by melting back unprocessed specimens to 57 mm from the threaded end. The maximum temperature of the melt was 867°C. M566-7 unlike the Skylab 3 specimen showed only a slight indentation. Cross sections made every inch revealed that the specimen was single grained along its length. Note from data in Table V that the perfection of the microstructure is better than that of the ground based sample. For example, the number of defects is 10 to 30% improved for sample M566-7, processed at 10^{-4} g.

The transverse sections of each of these specimens is shown in Figs. 15-17.

Data from Georgia Institute of Technology for M566-10 (ground processed), M566-6 (Skylab 3) and M566-9 (Skylab 4) are presented in Table VI. It can be seen that they measured improved microstructures in both space processed samples.

Freezing rate data can be obtained from the relation $R = (0.125/\lambda)^{2.222}$ with in micrometers and R in cm/hr. Graphical presentation of the variation of lamellar spacing made in Fig. 18 for specimens M566-15 (ground processed) and M566-5 (Skylab 3) which were exposed to a maximum furnace temperature of 844°C, Fig. 19 presents

the same type of data for specimens M566-11 (ground processed) and M566-7 (Skylab 4) processed at 867°C maximum furnace temperature.

The lamellar spacing data obtained on Specimens A72-962B, M566-6, M566-9, and M566-10 are plotted in Fig. 20 with the permission of the Georgia Institute of Technology.

Specimen Evaluation - Thermal and Electrical Conductivity

Samples were measured over the approximate range 50 to 400°C by Dynatech R/D Co. They were supplied in the form of rods 6.29 mm diameter and 12.66 mm long and having a density of 3370 Kg m^{-3}. The rod was fitted with specially prepared end pieces to facilitate assembly in the apparatus and small holes were drilled some 3 mm deep at regular intervals to facilitate attachment of thermocouples.

Prior to any thermal conductivity measurements the sample was laid on knife edges a fixed distance apart, a thermocouple was fixed to the central section and current leads clipped on each end. A steady d.c. current was applied and the potential difference across the knife edges together with that across a calibrated 0.001 ohm shunt in series with the sample were measured on a potentiometer. The current was reversed and the potential differences measured again in order to eliminate thermal voltages. The electrical resistivity was determined in terms of the ratio of the potential difference, the value of the shunt, and the distance between the knife edges and the area of cross section of the sample.

The sample was fitted into a matching tapered hole in a large copper heat sink and a heating unit cap with a 6 mm diameter self-contained central heater was attached to the other end of the sample containing the hole. A heavy nickel cylindrical guard tube with a heater cap was fitted around the sample and anchored securely to the heat sink. Thermocouple instrumentation was attached both to the top and bottom and along the length of the guard tube. The composite sample configuration was mounted on a fluid cooled base with a subsidiary heater and surrounded by a further cylindrical heater and an outer shroud. The interspaces and surrounds were filled with a low conductivity insulating powder.

A steady temperature distribution was maintained in the system and by adjustment of the heaters on the guard, the heat sink and the outer cylindrical guard, both the temperature of the top part of the sample and guard cap were made identical and the gradient in the guard tube was matched to that along the sample in order to insure that heat losses were kept to an absolute minimum. At equilibrium conditions the temperature gradient along the rod was obtained from readings of the thermocouples and the d.c. power dissipated on the central heater was measured using a precision resistor network.

The thermal conductivity was derived in terms of the power dissipation, the temperature gradient, and the dimensions of the rod.

Electrical resistivity measurements were carried out at the same time as the above measurements. Current leads had been fixed to the sample heater cap and the heat sink. After each thermal conductivity measurement electrical resistivity measurements were taken as described previously but in this case the potential difference across the sample was measured using the "like" arms of the thermocouples.

The overall temperature of the system was adjusted by means of the various heaters and the mean temperature was changed at regular increments up to approximately 400°C. A repeat electrical resistivity point was obtained on knife edges at room temperature to check for any changes due to heat treatment.

The collected results of the thermal conductivity measurements are presented in Fig. 21. These show that a measurement of thermal conductivity is insufficiently sensitive to distinguish effects due to space processing of the directionally solidified eutectic.

In addition to the thermal conductivity, measured at 25°C and near 400°C, the electrical conductivy was ascertained, as shown in Fig. 22. The ratio of these numbers, according to the law of Wiedemann and Franz, should be constant. This is found to be experimentally verified for simple metals; the measurement of this ratio thus allows the metal composite to be compared with simple metals.

The Wiedemann-Franz law states that:

$$L = \frac{K}{\sigma T} = \text{constant}$$

where L is the Lorentz number.

$$L = \mathcal{L} \times 0.7416\ (10)^{-8}\ \frac{\text{joule}^2}{\text{coulomb}^2\text{-deg K}^2}$$

If current and heat conduction are both carried by a free-electron gas, the factor $\mathcal{L} = 3$. For metals and highly degenerate semi-conductors, a first order theoretical calculation gives $\mathcal{L} = (\pi)^{2/3}$ The Lorentz number is thus $2.7\ (10)^{-8}$ $(\text{joules/coulomb - deg K})^2$. (Ref. 1) For simple metals, the experimental value is $2.44\ (10)^{-8}$, which agrees with the values obtained for the eutectic listed in Table VII. Thus, it must be concluded that the eutectic system should exhibit the properties of a simple metal at temperatures above room temperature.

Low Temperature Investigation

The variation of resistivity with temperature at low temperatures was determined by Dr. L. Lacy, Space Sciences Laboratory, MSFC, on specimens supplied by UARL to determine if significant variations might appear due to theseparation of phases in the microstructure. These data are given in Table VII, and only smooth variations in values were observed. Accoustical attenuation was also investigated by Dr. Lacy, but it appeared that no significant differences would be elucidated by such a study unless they were sufficiently gross as to be easily observed by other techniques.

A low temperature (77°K) X-ray scan was made of an eutectic specimen to determine if the details X-ray diffraction patterns of the Al-Al_2Cu system as a function of temperature would provide a significant means of evaluating differences between ground and low-g processing. From the data attained it was judged that a detailed study was not warranted. As a result of the investigation, however, some useful information was recorded on cell size change. The evaluation was carried out using the (112) reflections from the Al_2Cu phase. This phase is tetragonal with a space group designation D_{4H}^{18} (I4/mcm). The unit cell has dimensions a_o = 6.066Å, c_o = 4.874Å. The present study determined the d-spacing of the (112) planes, given as 2.12Å, to be 2.1242Å at room temperature and 2.1204Å at liquid nitrogen temperature. The change is thus 0.0038Å, or approximately 2%.

DISCUSSION AND CONCLUSIONS

It has been shown that eutectics can be successfully unidirectionally solidified in a relatively simple NASA apparatus consisting of a cold end and a heater at the other end which can be slowly cooled. The microstructure produced in this equipment was as good over a short length as that formed in an apparatus normally used to grow eutectics on the ground which involves withdrawing a molten sample from a heated region.

The sample given to UARL from Skylab 3 showed a large indentation in the resolidified portion of the ingot and contained more than one grain. Because of this instability in growth a meaningful comparison with specimens controlled on the ground could not be made.

The Skylab 4 sample did not show such a marked indentation and its microstructure was less faulted than a ground processed sample unidirectionally solidified in similar equipment. This agrees with results from Georgia Institute of Technology which show improved microstructure for Skylab 3 and Skylab 4 specimens sent to them.

From studies at UARL, University of Connecticut and Georgia Institute of Technology it appears that better microstructure was obtained in space processed samples. However, because of the small number of samples involved more studies should be conducted to determine the mechanism by which low-g affects the fault density.

REFERENCES

1. A. J. Dekker, "Solid State Physics", Prentice-Hall, (1959) p300.

APPENDIX

METHOD OF MEASURING MICROSTRUCTURAL PERFECTION

1. Lamellar Spacing (λ) micrometers

   a. Lines of length "a" are drawn perpendicular to lamellae in defect-free areas of transverse sections, using many photomicrographs of the same section.

   b. The average number of lamellae, X, crossing the "a" cm length of line is measured.

   c. $N_A = n_t \left(\frac{M^2}{ab}\right)(10^{-4}\ \lambda^2)$

      where: M is magnification

2. Fault Density (N_A) No. per $(100\lambda)^2$ area

[Average number of terminations and other defects such as ripples per $(100\lambda)^2$ area in tranverse sections].

   a. The number of terminations and defects, n_t, on each photomicrograph is measured.

   b. The average n_t per section and specimen is determined.

   c. $N_A = n_t \left(\frac{M^2}{ab}\right) \quad (10^{-4}\ \lambda^2)$

      where: a and b are dimensions of photomicrograph, cm
      λ is lamellar spacing, micrometers
      M is magnification

3. Average Defect Spacing Along Lamellae (d_L/λ) unitless

   a. Several lines parallel to the lamellae are drawn, spaced evenly across the photomicrograph.

   b. The average distance, d_L, between defects along a test line is measured and averaged for several photomicrographs and transverse sections.

   c. d_L is divided by λ.

4. Average Length of Mismatch Line (L_{A_o}) cm $(100)^2$ area

(The Average length of mismatch line normalized for a $(100\lambda)^2$ area in transverse sections)

a. Several randomly oriented lines on each photomicrograph of transverse section are drawn.

b. The total number of intersections, n, between test line and mismatch lines is measured per total length of test line, d, cm.

c. For each photomicrograph $P_L = \frac{\Sigma n}{\Sigma d}$ is calculated.

(P_L is number of intersections per cm)

d. P_L is averaged over many photomicrographs and transverse sections.

e. $L_{A_o} = P_L \frac{\pi}{2} M \ (10^{-4} \lambda^2)$

where: λ is in μ and M is magnification.

5. Angle Between Lamellae and Growth Direction (α)

This angle is measured in a longitudinal section

Figure A-1 illustrates the relationships between the transverse and longitudinal structure, showing the angular relationships of these for determining lamellar orientation.

Table I

Description of Al-$CuAl_2$ Eutectic Specimens
Prepared at UARL
for the NASA M566 Composite Casting Experiment

Specimen Number	Ingot Number	Heat Number	Finished Threads	Length of Single Grain from Tail End (inches)
1*	A72-868	A72-866	Machined	5
2*	-928	-915		> 2 1/2
3*	-937	-936		> 2 1/2
4	-941B	-936		5
5	-941C	-936		5
6	-946A	-944		2 3/4
7	-951B	-948		5
8	-951C	-948		4 7/16
9	-957C	-953		5
10	-962A	-958		4
	-962B+			
11	-964B	-963		4 5/8
	-964C++			
12	-977B	-967		5
13	-982A	-979		3 5/8
14	-982B	-979		3 1/4
	-987A+++			
15	-989A	-989	Machined	3 1/4
16	-1018B	-1017	Ground	4 7/8
17	-1021B	-1019		2 1/2
18	-1021C	-1019		3 3/8
19	-1026A	-1024		5
20	-1026C	-1024		3 1/16
21	-1033A	-1029		5
22	-1043C	-1036	Ground	2 11/16

*These specimens were produced individually in separate directional solidification runs. The remaining 19 specimens were produced as part of a triple run and designated either A, B or C. These specimens were all produced at a nominal solidification rate of 2 cm/hr and an average thermal gradient in the liquid of about 39°/cm.

+Specimen A72-962B was sent to the Georgia Institute of Technology for their evaluation program.

++Specimen A72-964C was used to obtain baseline data on the thermal conductivity of the directionally solidified specimen.

+++Specimen A72-987A was used to obtain defect density data on an unprocessed original.

Table II

M566 Al-$CuAl_2$ Eutectic Specimen Major Constituent Analyses
UARL - Target Composition 33.0% Cu by Weight

Specimen Designation UARL	NASA		Notes	Copper Wt.%	
Orig. D.S. Test Ingot				33.18±.05	
A72-941B	M566-4		Composition of orig. ingots	33.42±.06	
A72-941C	M566-5		by wet chemical methods -	33.14±.01	
A72-946A	M566-6 (Tail Piece)		UARL	33.72±.10	
A72-951B	M566-7			32.98±.06	
A72-951C	M566-8			33.32±.02	
A72-957C	M566-9			33.49±.05	
A72-964B	M566-11		Ground based - as prepared	33.3±.1	
			remelted portion-UARL	33.6±.2	
A72-964B	M566-11	sec 2	1 cm section of ground based	33.5	
		sec 10	melt-back expt.	33.9	
		sec 11	Electron Microprobe - UARL	33.6	
Final Solidification Direction		point 1	0.65 cm length including melt-	33.8	
		2	back zone: Electron Micro-	31.4	
		3	probe points ~30 μmeters	34.7	
		4	apart - UARL	32.6	33.0 avg
		5		34.3	
		6		30.8	
		7		33.5	
		P	Interface region	60.5	
		11		34.3	
		12		28.6	
		13		40.9	
		14		32.2	34.4 avg
		15		34.5	
		16		35.6	
		17		34.5	

Table II (Contd)

Specimen Designation UARL	Specimen Designation NASA	Notes	Copper Wt.%	Position *
Orig. D.S. Test Ingot				
A72-946A	M566-6 sec 4A	Sample examined by Georgia	32.65	4.32
Final Solidification Direction ↓	4	Institute of Technology X-ray	32.17	13.5
	3A	fluorescence	33.16	20.9
	3	Skylab processed at 10^{-4}g	32.82	28.1
	2		33.12	35.1
A72-957C	M566-9 sec 5	Sample examined by Georgia	32.39	7.1
Final Solidification Direction ↓	4	Institute of Technology X-ray	32.67	13.6
	3	fluorescence	32.42	20.8
	2	Skylab processed at 10^{-4}g	32.91	27.7
	1		33.13	34.8
A72-962A	M566-10 sec 5	Sample examined by Georgia	32.10	7.1
Final Solidification Direction ↓	4	Institute of Technology X-ray	32.99	13.2
	3	fluorescence	32.80	21.2
	2	Ground processed at MSFC	32.85	27.9
	1		33.29	35.0

*mm from interface with remelt section

Table III

Impurity Level Analyses on M566 Al-$CuAl_2$ Eutectic Material

UARL Impurity Analyses on Al, Cu, and Al-$CuAl_2$ Eutectic Material Used for M566 Specimens By Emission Spectroscopy and Atomic Absorption

Accuracy - ±50% of amount shown

Sample No.	Material	Fe	Na	Mg	Ni	Pb	Si	Nb	- ppm
A66-253	Cu	34	5	30	< 1.5	5	< 100	< 100	
A71-194	Al	77	45	3.5	48	53	< 50	< 100	
A72-1049	Cu-Al Eutectic	50	25	15	31	38	< 50	< 100	

UARL Impurity Analyses on M566 Al-$CuAl_2$ Specimens by Emission Spectroscopy and Atomic Absorption

Accuracy - ±50% of amount shown

NASA Specimen No. M566-	UARL Ingot No. A72	Fe	Na	Mg	Ni	Pb	Si	Nb	- ppm
4	941B	37.6	44.1	2.5	71.3	22	--	< 100	
5	941C	39.4	59.0	2.3	64.0	17	--	< 100	
6*	946A	41.7	95.7	2.6	122.7	< 25	--	< 100	
7	951B	32.0	33.9	1.4	66.6	30	--	< 100	
8	951C	29.9	22.4	1.8	62.3	< 13	--	< 100	
9	957C	63.8	36.3	4.2	74.8	< 15	--	< 100	

Data obtained by Westinghouse AstroNuclear Laboratories Method - Emission Spectrography

Accuracy - .3 to 3 x amount shown

NASA Specimen No. M566-	UARL Ingot No. A72-	Fe	Na	Mg	Ni	Pb	Si	Nb	- ppm
5	941C	< 10	200	10	10	30	50	100	
6	946A	< 10	200	10	10	30	50	100	

Table IV

Defect Analysis in Al-$CuAl_2$ Specimens

Data Supplied by Dr. Theo Kattamis
Materials Science Institute
University of Connecticut

Skylab Specimen M566-15 (UARL A72-989A) Processed at 1g - MSFC - Max Temp 844°C

Position*	25	50	Interface	75	100
λ	4.21	4.67		4.33	2.42
N_A	1585	1627		1526	823
L_{A_o}	0.572	0.571		0.555	0.172
d_L/λ	8.03	8.60		6.80	10.34
α	5.53	6.0		4.7	5.1

Skylab 3 Specimen M566-5 (UARL A72-941C) Processed at 10^{-4}g - Max Temp 844°C

λ	4.19	4.68	5.23	4.86	3.2
N_A	1392	1986	2423	2520	1521
L_{A_o}	0.514	0.574	1.007	1.239	0.631
d_L/λ	6.14	8.08	4.90	4.63	5.95
α	8.9	7.6	8.1	6.7	6.1

KEY

Width of lamellar pairs - micrometers λ

No. of defects per $(100\lambda)^2$ N_A

Average length of defect line per $(100\lambda)^2$ μ^2 area L_{A_o}

Distance between defect lines in units of lamellar spacing d_L/λ

Average angular position with respect to a ref., deg. measured in a longitudinal section α

*distance from threaded end in mm
**ingot showed evidence of instability during solidification

Table V

Defect Analysis in Al-$CuAl_2$ Specimens

Data Supplied by Dr. Theo Kattamis
Materials Science Institute
University of Connecticut

Unprocessed Eutectic (UARL A72-987A) Processed at 1g - MSFC - Max Temp 867°C

Position	25	50	Interface	75	100
λ	4.02	4.30		4.49	4.62
N_A	1223	1341		1471	1484
L_{A_o}	0.459	0.436		0.549	0.764
d_L/λ	7.50	5.88		7.80	7.15
α	6.9	5.8		5.3	4.6

Skylab Specimen M566-11 (UARL A72-964B) Processed at 1g - MSFC

Position	25	50	Interface	75	100
λ	4.14	4.21	4.58	4.06	3.72
N_A	999	772	1324	1126	1203
L_{A_o}	0.382	0.346	0.613	0.440	0.490
d_L/λ	10.59	13.42	7.64	8.86	7.95
α	7.6	7.15	9.45	7.1	6.35

Skylab Specimen M566-7 (UARL A72-951B) Processed at 10^{-4}g - Max Temp 844°C

Position	25	50	Interface	75	100
λ	3.44	3.07	7.17	3.97	3.42
N_A	994	959	1050	970	825
L_{A_o}	0.311	0.285	0.550	0.389	0.210
d_L/λ	9.56	13.41	4.84	8.88	14.09
α	5.6	7.9	5.0	5.9	5.6

Same Key as Table IV

Table VI

Defect Analysis in the Resolidified Part
of the M566 Al-$CuAl_2$ Specimens

Data Supplied by Dr. J. L. Hubbard
Georgia Institute of Technology

Skylab 4 Specimen M566-9 (UARL A72-957C) Processed at 10^{-4}g

Section No.	1	2	3	4	5	I**
X *	88.8	81.7	74.8	67.6	61.1	54
λ	3.40	3.56	3.70	3.79	4.10	
P_L	247	273	253	247	233	
L_{A_Q}	.449	.544	.543	.549	.615	
d_L/λ	8.02	7.39	7.24	7.07	7.03	

Skylab 3 Specimen M566-6 (UARL A72-946A) Processed at 10^{-4}g

Section No.	1.	2	3A	4	5	I**
X *	96.4	89.4	82.2	74.8	68.4	61.3
λ	3.40	3.62	3.84	3.92	4.32	
P_L	273	267	213	273	247	
L_{A_Q}	.496	.549	.493	.659	.724	
d_L/λ	7.35	6.79	7.81	6.49	6.09	

Skylab Specimen M566-10 (UARL A72-962A) Processed at 1g - MSFC

Section No.	1	2	3	4	5	I**
X *	103.3	96.2	89.5	81.5	75.4	68.3
λ	2.82	3.65	3.84	4.13	4.47	
P_L	427	280	273	280	267	
L_{A_Q}	.533	.586	.633	.751	.837	
d_L/λ	5.60	6.63	5.92	5.67	5.69	

*distance from threaded end in mm
**I = interface

Key for Table VI

Width of lamellar pairs, micrometers	λ
Average length of defect line per $(100\lambda)^2$ area (units $cm/(100\lambda)^2$ ^{2})	L_{A_o}
Distance between lamellar fault lines in units of lamellar spacing	d_L/λ
No. of intersections of a test line with lamellar faults	P_L

Table VII

Resistivity Data on Al-$CuAl_2$ Material

Supplied by Dr. L. Lacy, SSL, MSFC

	Resistivity at 25°C $\mu\Omega$ cm	Resistivity Ratio $\rho(300^{\circ}K)/\rho(4.2^{\circ}K)$	Density (gm/cm^3)
Eutectic, as cast	4.25	11	
Eutectic, unidirectionally solidified			
Approx ∥ to lamellae	4.26	11	3.5232±
⊥ to lamellae	5.1	--	.0005
$CuAl_2$	9.83	12	4.378-4.317
Al	2.66		2.69
Cu	1.67		8.93

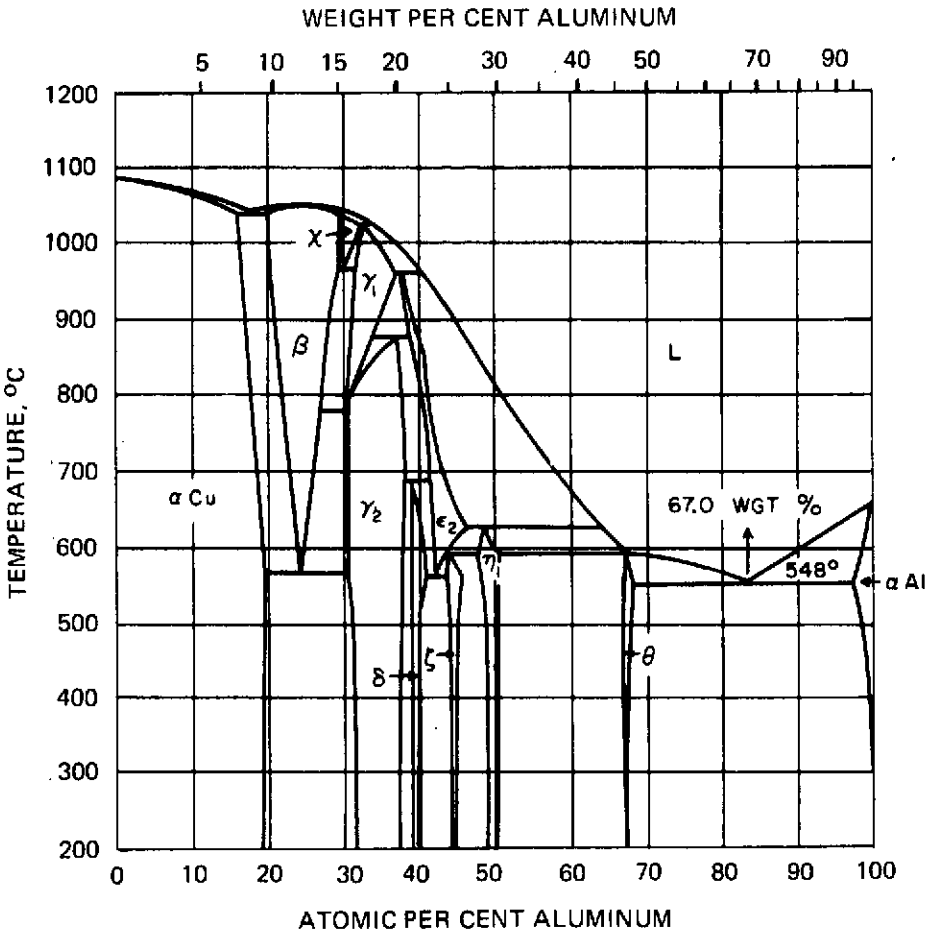


FIGURE 1. COPPER ALUMINUM PHASE DIAGRAM

N06–45–1

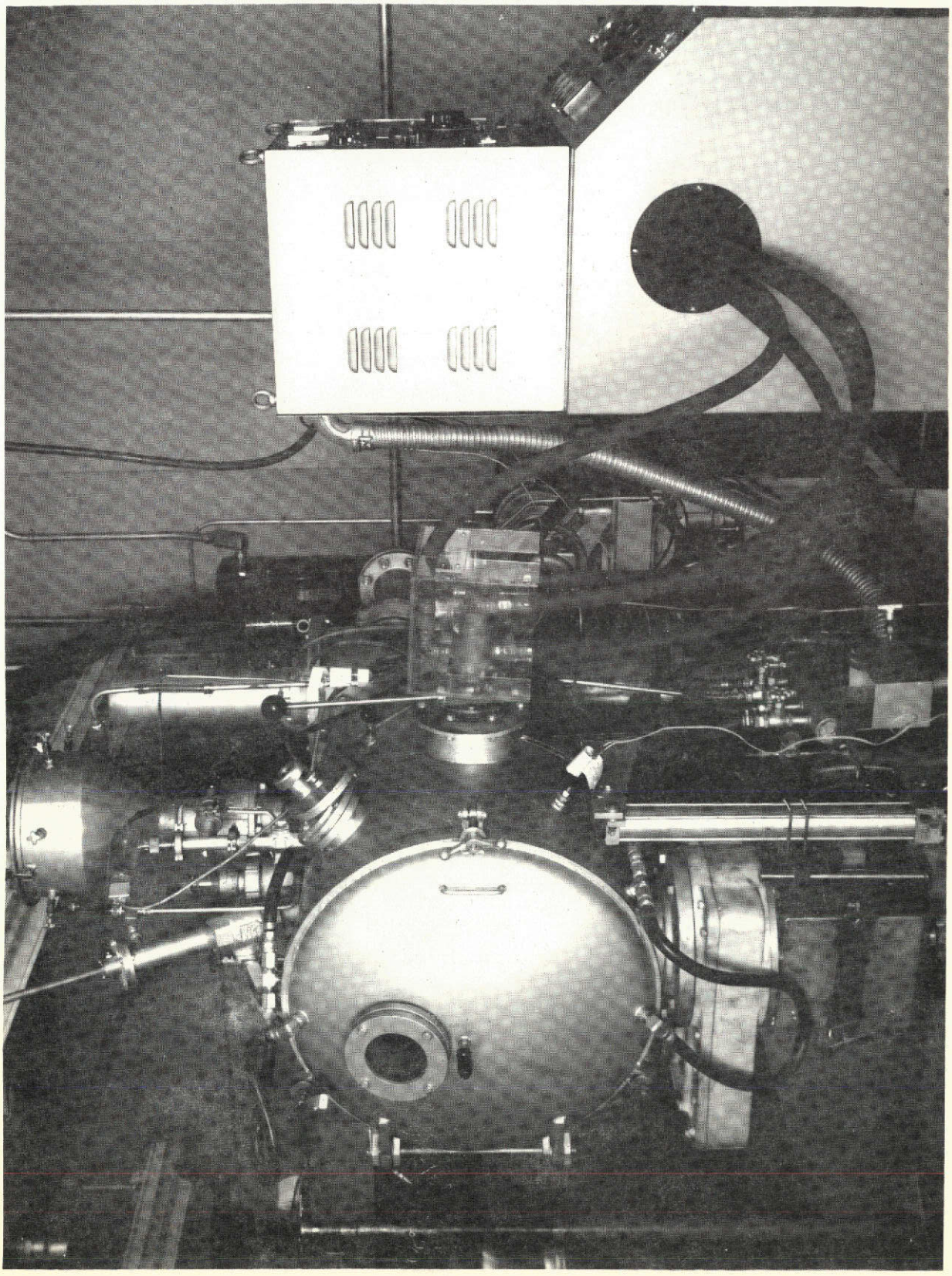


FIGURE 2. VACUUM INDUCTION MELTING AND CASTING FURNACE

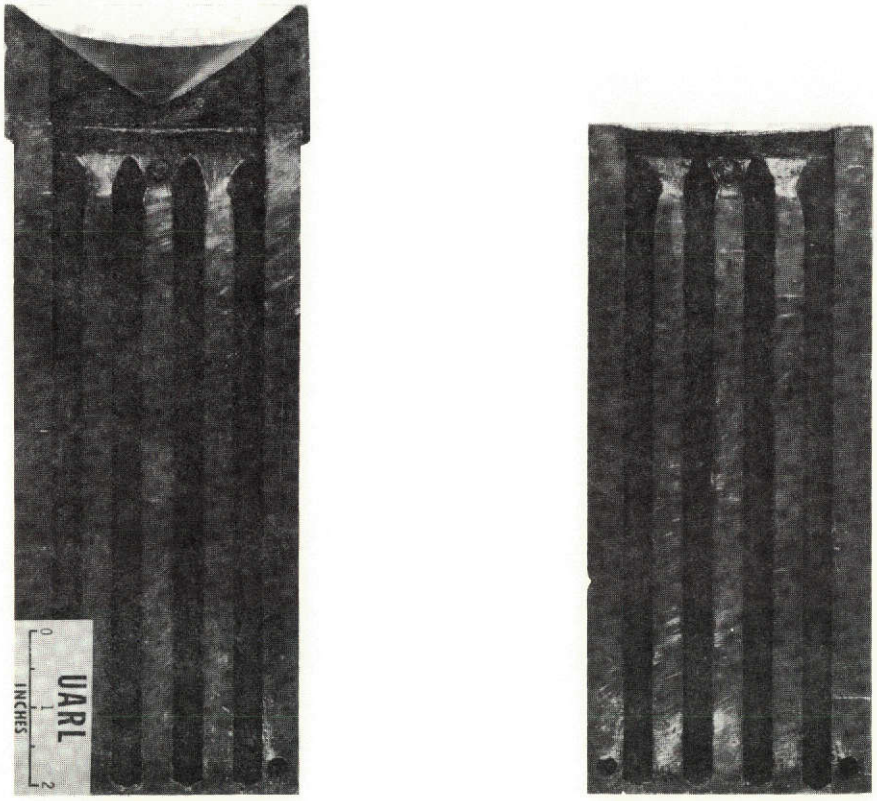


FIGURE 3. 3/8 INCH DIAMETER BAR, SPLIT COPPER MOLD

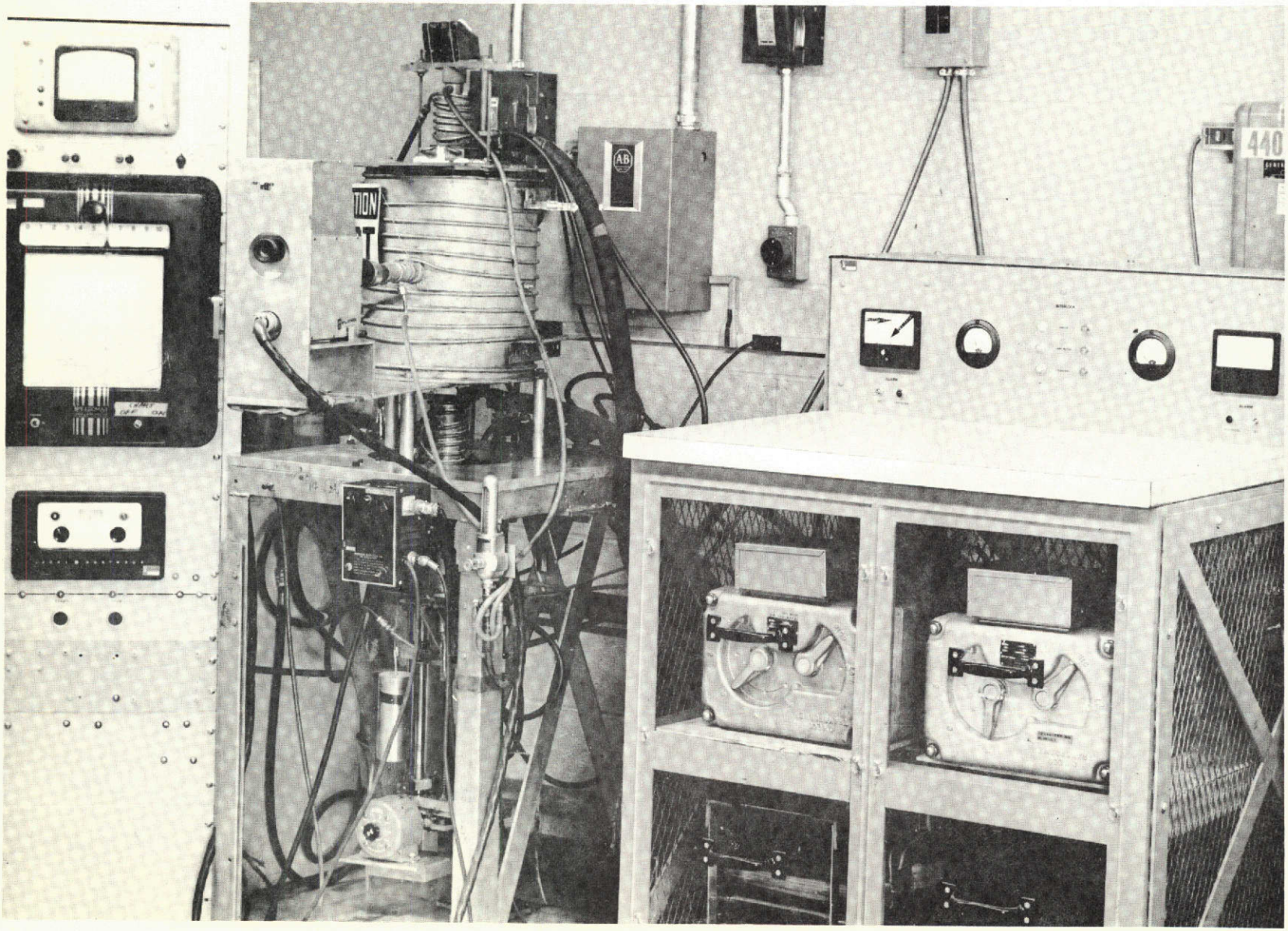


FIGURE 4. GRAPHITE DIRECTIONAL SOLIDIFICATION FURNACE

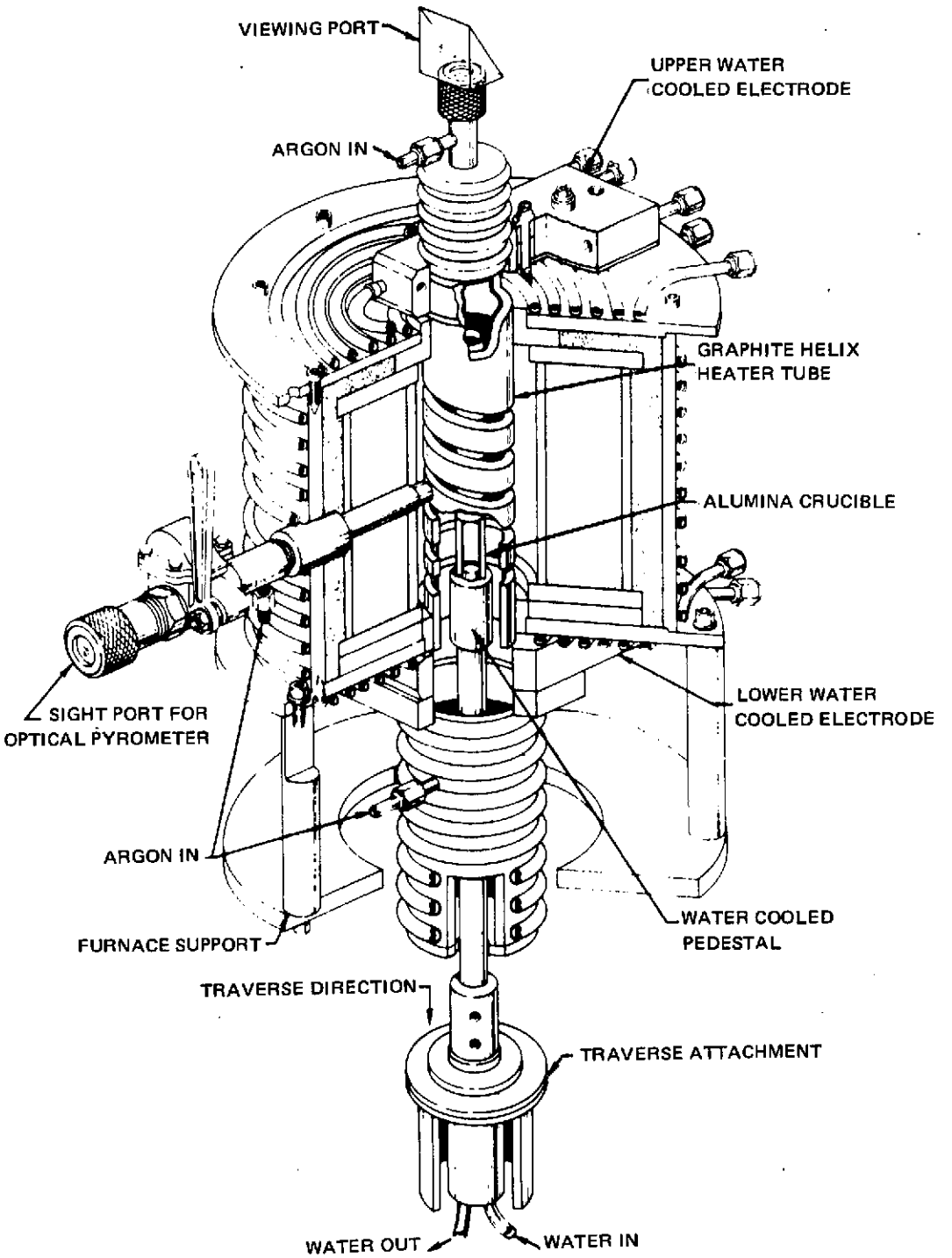


FIGURE 5. GRAPHITE DIRECTIONAL SOLIDIFICATION FURNACE

N06–77–2

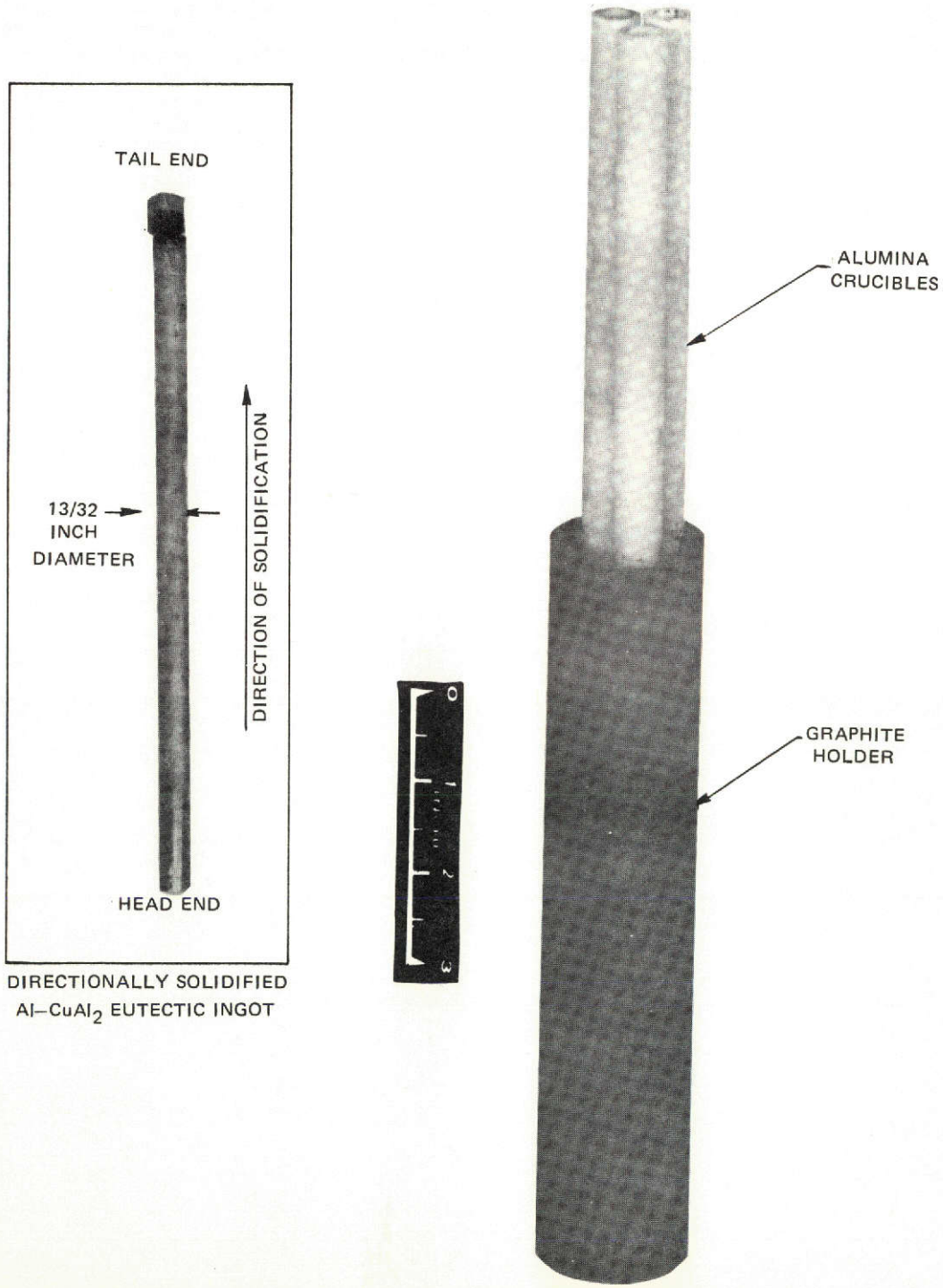


FIGURE 6. TRIPLE DIRECTIONAL SOLIDIFICATION ASSEMBLY

DRAWING NO. 615B707

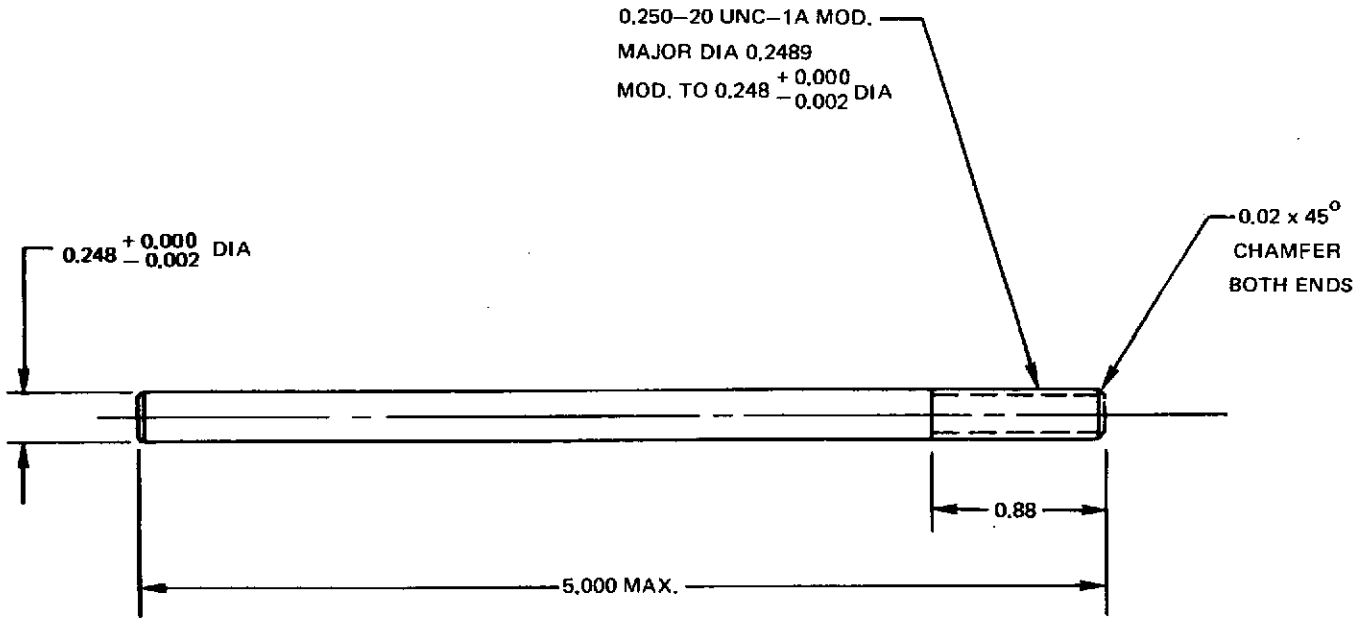


FIGURE 7. AMPOULE–EXP. M566 ZERO GRAVITY EXPERIMENT CARTRIDGES

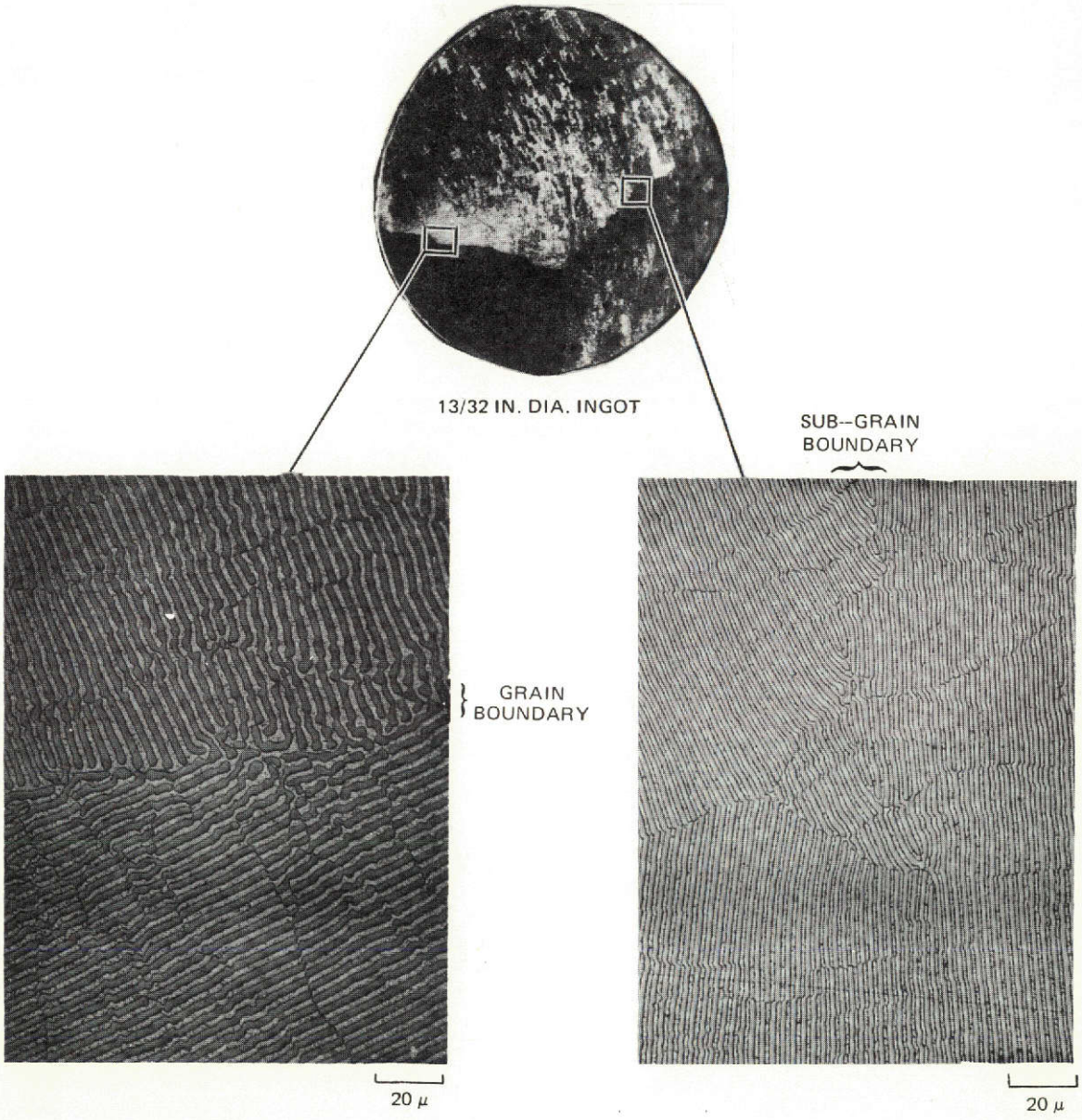
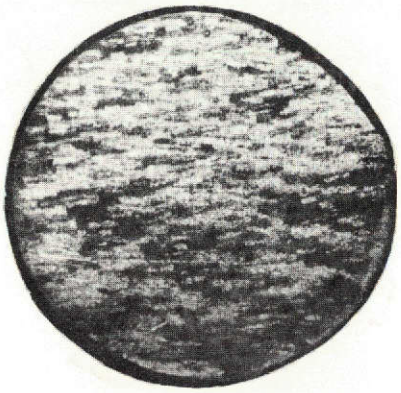


FIGURE 8. TRANSVERSE SECTION OF MULTI–GRAIN Al–$CuAl_2$ EUTECTIC INGOT



13/32 INCH DIAMETER INGOT

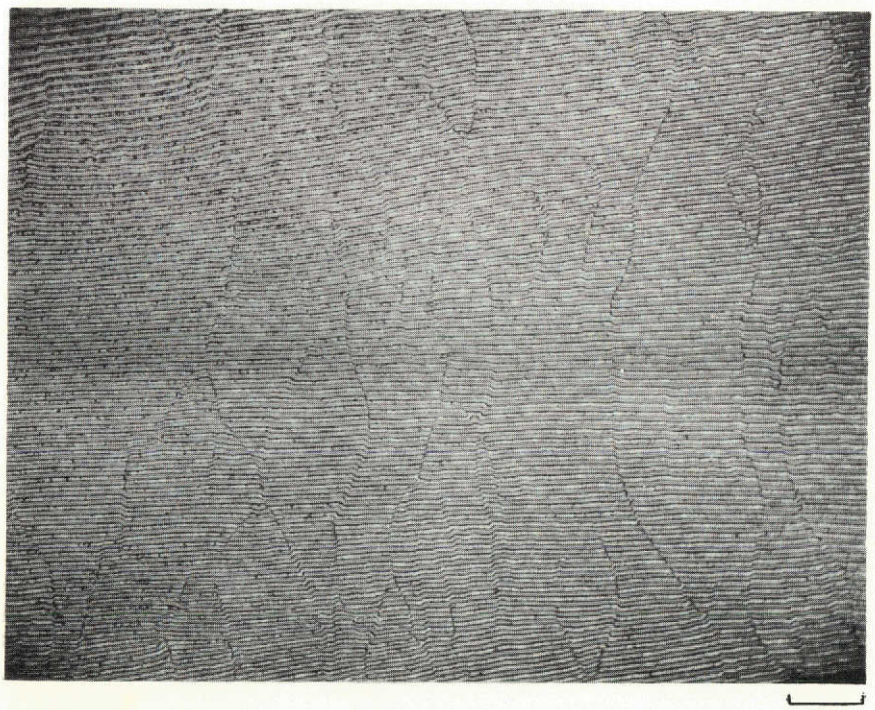
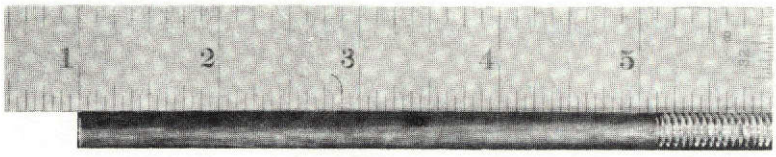
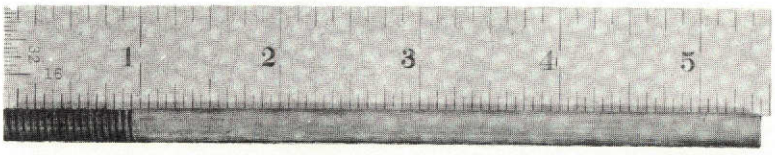


FIGURE 9. TRANSVERSE SECTION OF SINGLE GRAIN Al-$CuAl_2$ EUTECTIC INGOT



MACHINED THREADS



GROUND THREADS

FIGURE 10. FINISHED SINGLE GRAIN Al-$CuAl_2$ SPECIMENS

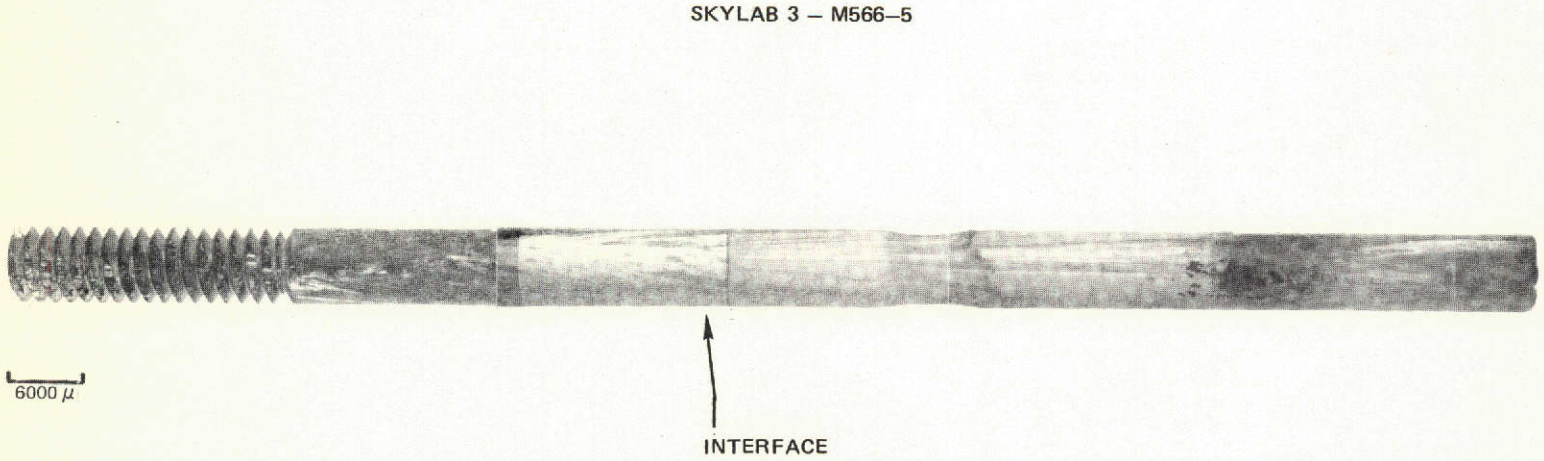


FIGURE 11. ALUMINUM–COPPER EUTECTIC SPECIMEN SOLIDIFIED IN SPACE

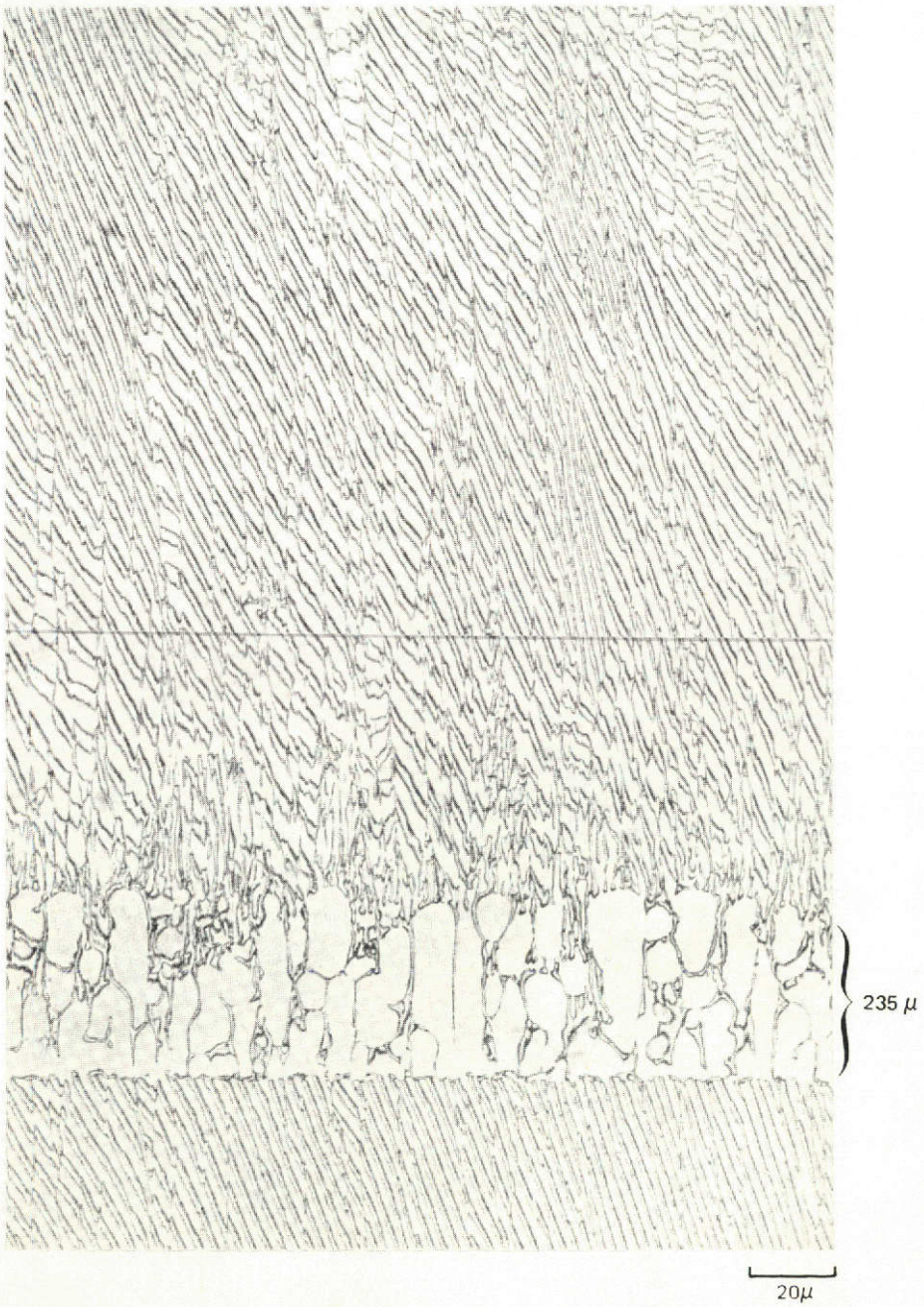
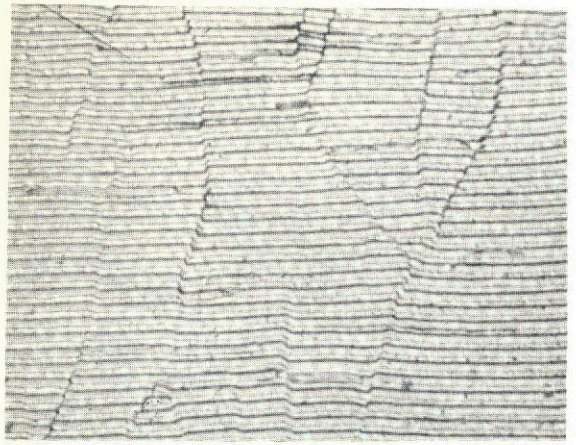
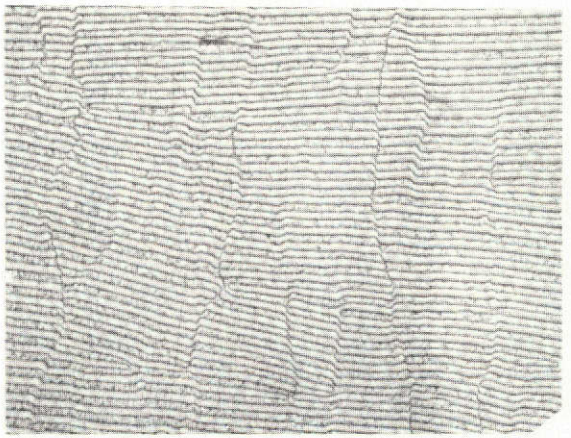


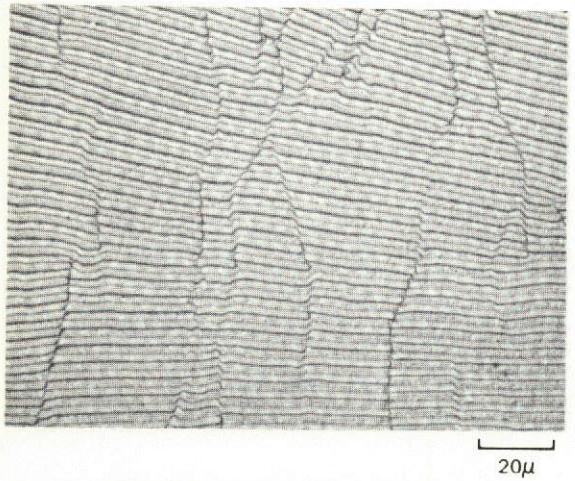
FIGURE 12. ALUMINUM–COPPER EUTECTIC M566–5 (SKYLAB 3)
MELT BACK INTERFACE AND ADJACENT REGION



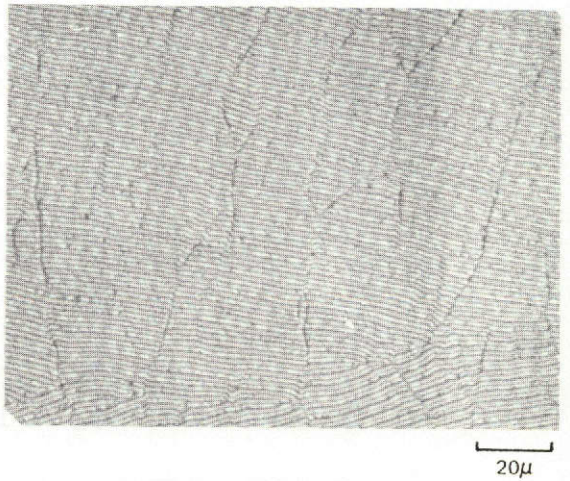
SECTION 1 (ORIGINAL)



SECTION 2 (ORIGINAL)

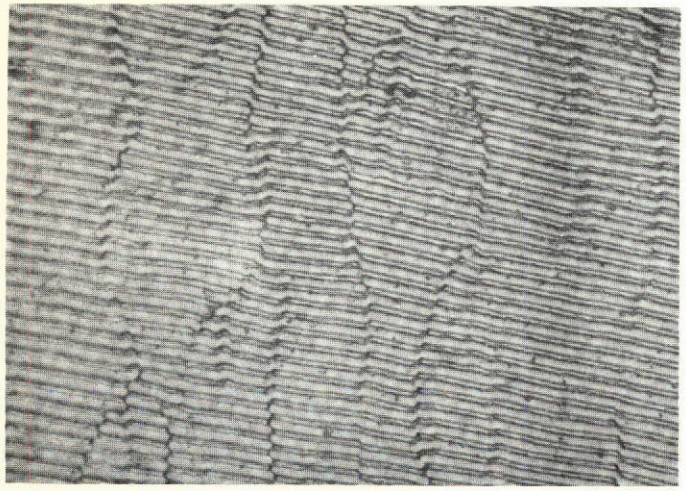


SECTION 3 (RE–SOLIDIFIED)



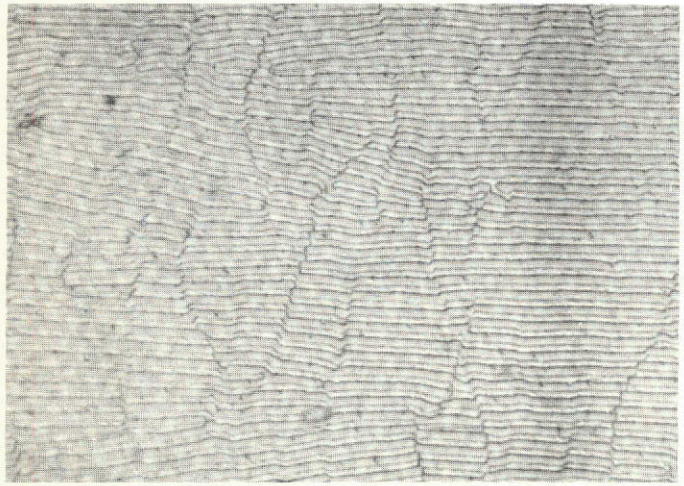
SECTION 4 (RE–SOLIDIFIED)

FIGURE 13. TRANSVERSE SECTIONS OF Al–Al_2Cu EUTECTIC SPECIMEN M566–5 REMELTED IN SKYLAB 3

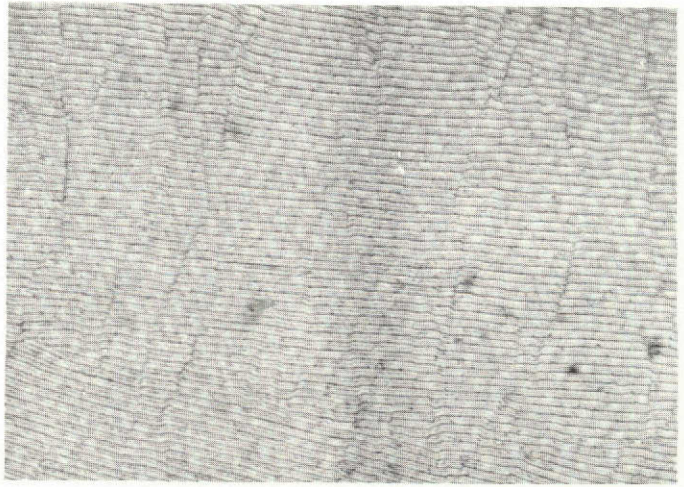


SECTION 1 20μ

SECTION 2 20μ

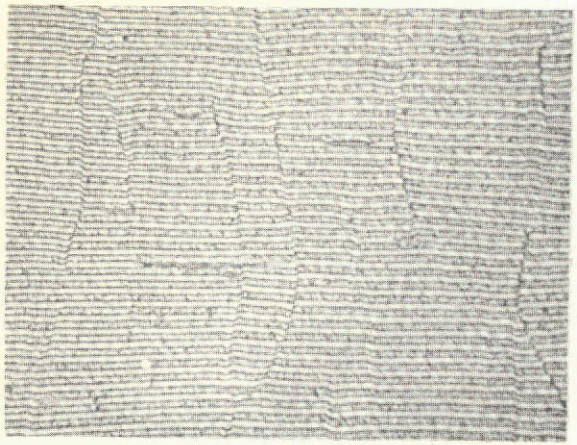


SECTION 3 20μ

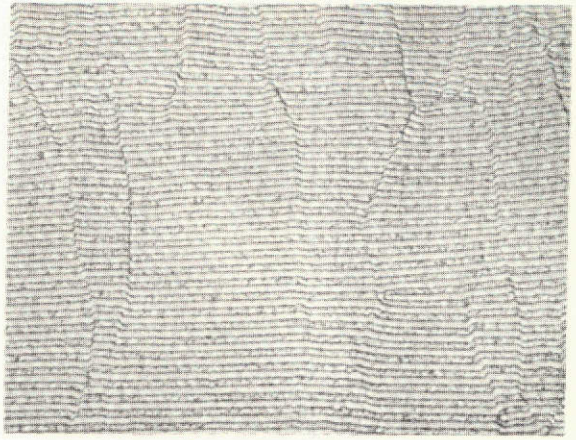


SECTION 4 20μ

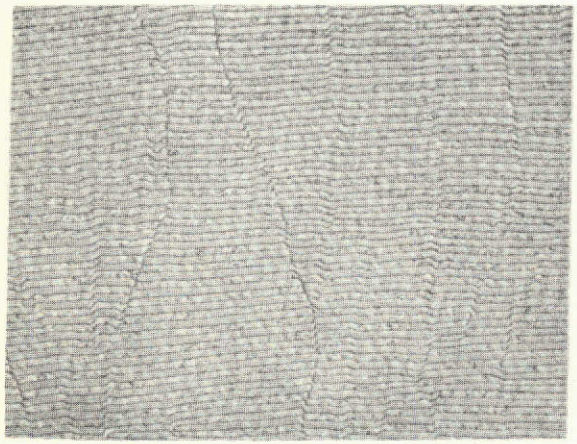
FIGURE 14. TRANSVERSE SECTIONS OF Al–Al_2Cu EUTECTIC SPECIMEN M566–15 (GROUND BASED TEST)



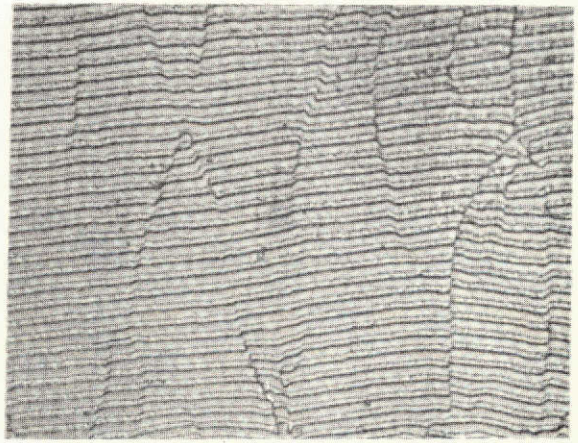
SECTION 1



SECTION 2



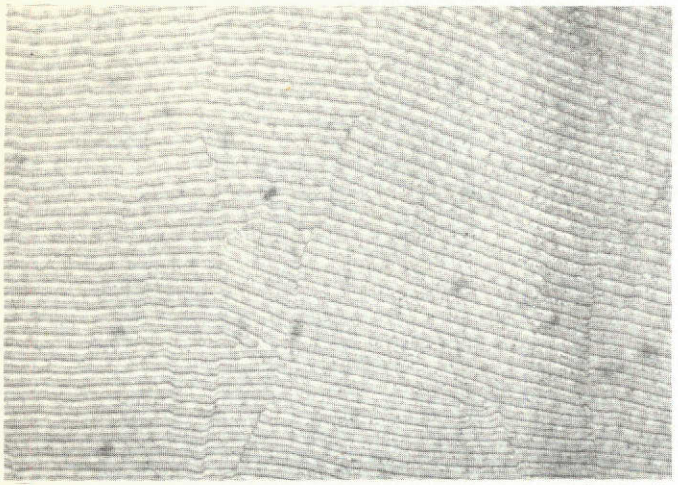
SECTION 3



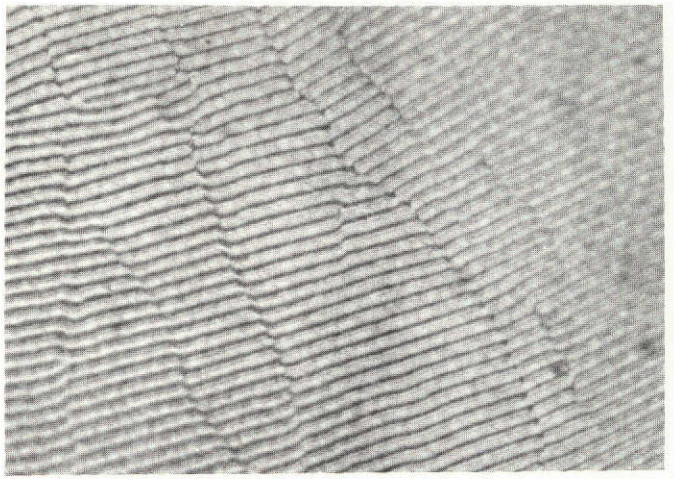
SECTION 4

FIGURE 15. TRANSVERSE SECTIONS OF Al–Al_2 Cu EUTECTIC SPECIMEN A72–987A

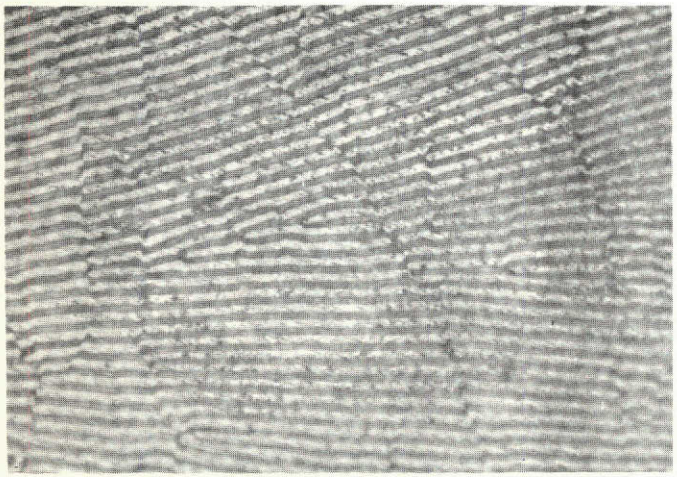
FIGURE 16. TRANSVERSE SECTIONS OF Al–Al_2Cu EUTECTIC SPECIMEN M566–11 (GROUND BASED TEST)

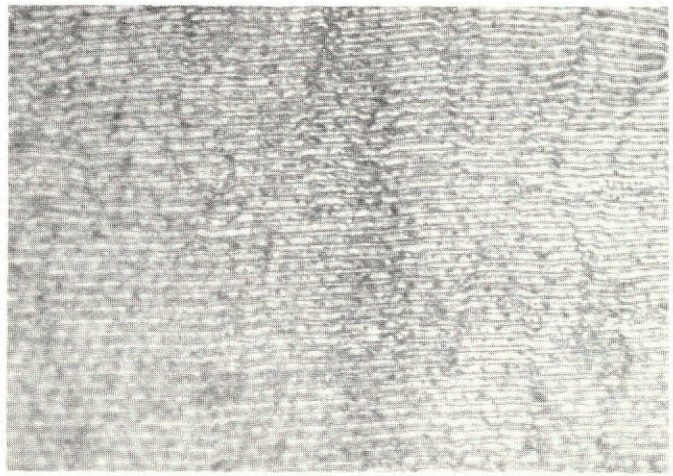


SECTION 1 20μ



SECTION 2 20μ





SECTION 4 20μ

FIGURE 17. TRANSVERSE SECTIONS OF Al–Al_2Cu EUTECTIC SPECIMEN M566–7 (SKYLAB 4 TEST)

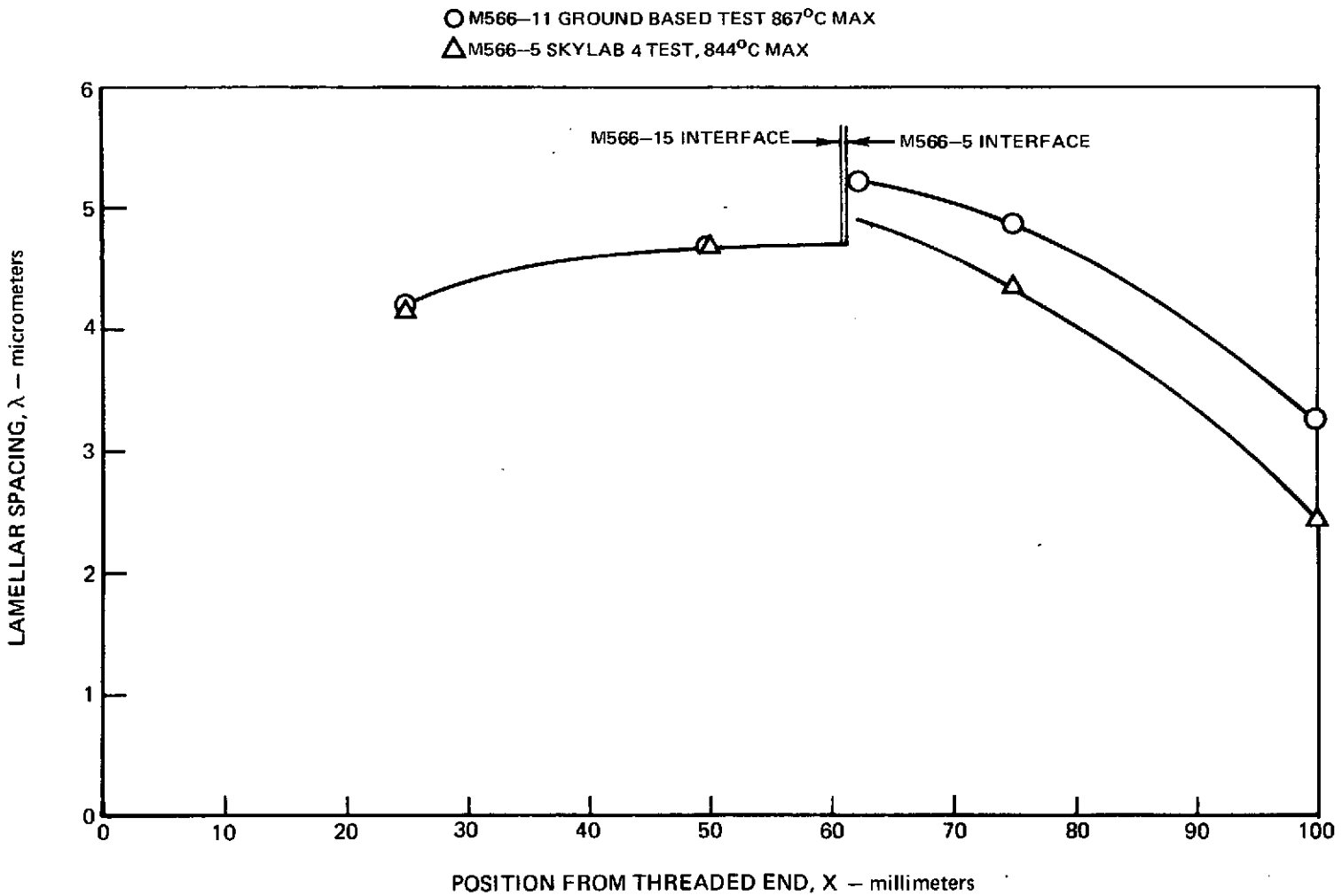


FIGURE 18. COMPARISON OF LAMELLAR SPACINGS IN Al–Al_2Cu EUTECTIC SPECIMENS

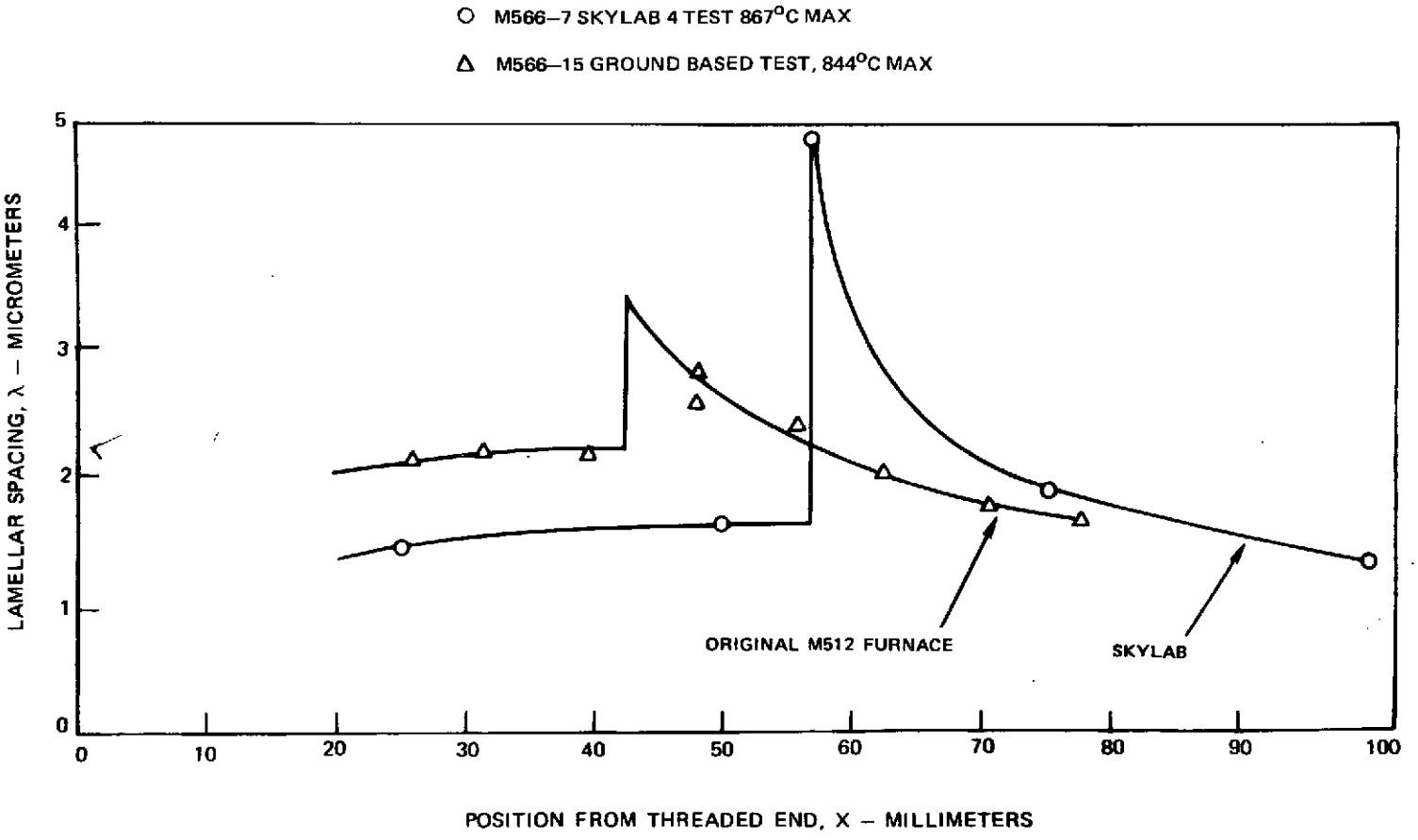


FIGURE 19. COMPARISON OF LAMELLAR SPACINGS IN Al–Al_2Cu EUTECTIC SPECIMENS

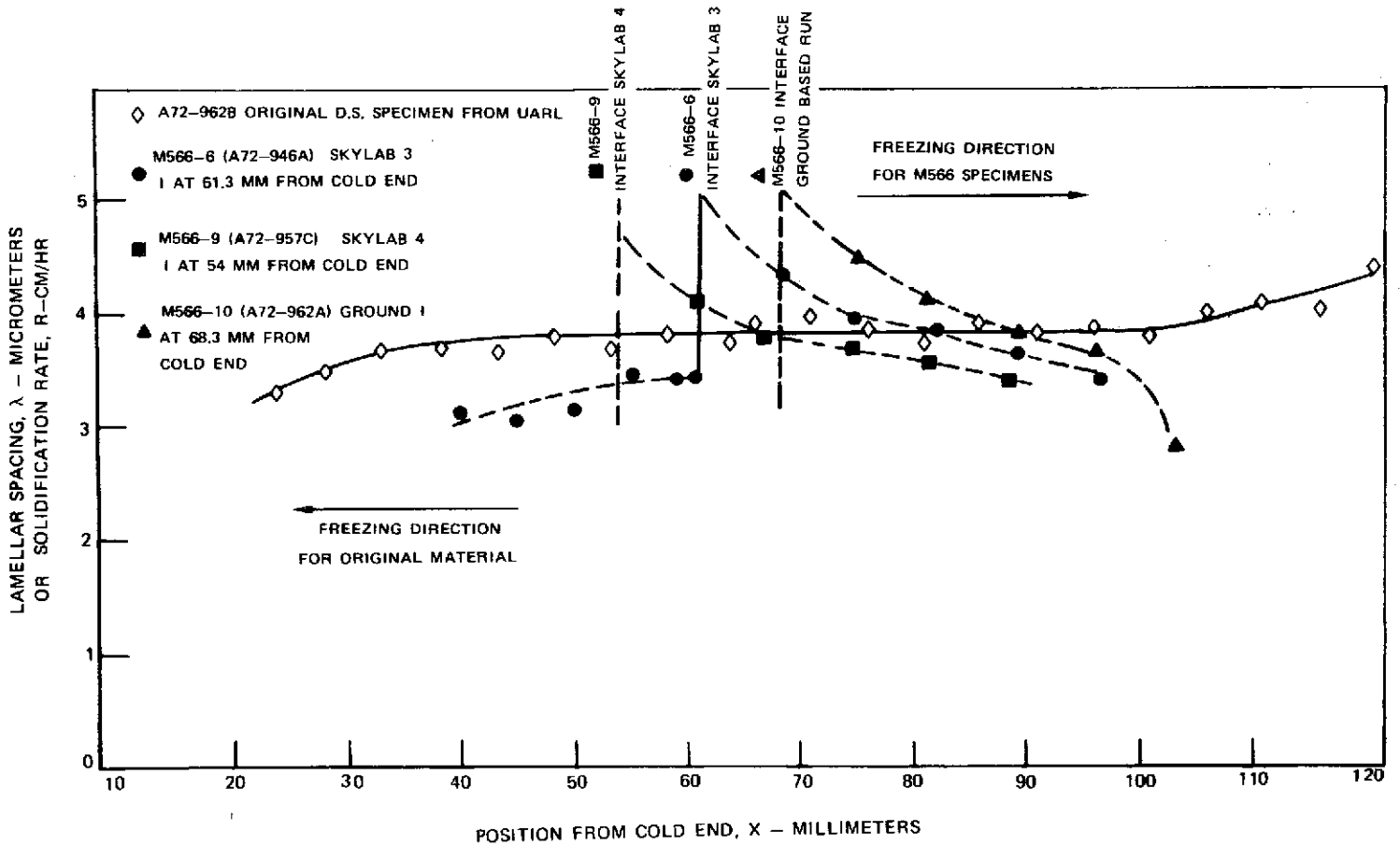


FIGURE 20. LAMELLAR SPACINGS OF M566 Al–Al_2Cu SPECIMENS EXAMINED BY THE GEORGIA INSTITUTE OF TECHNOLOGY

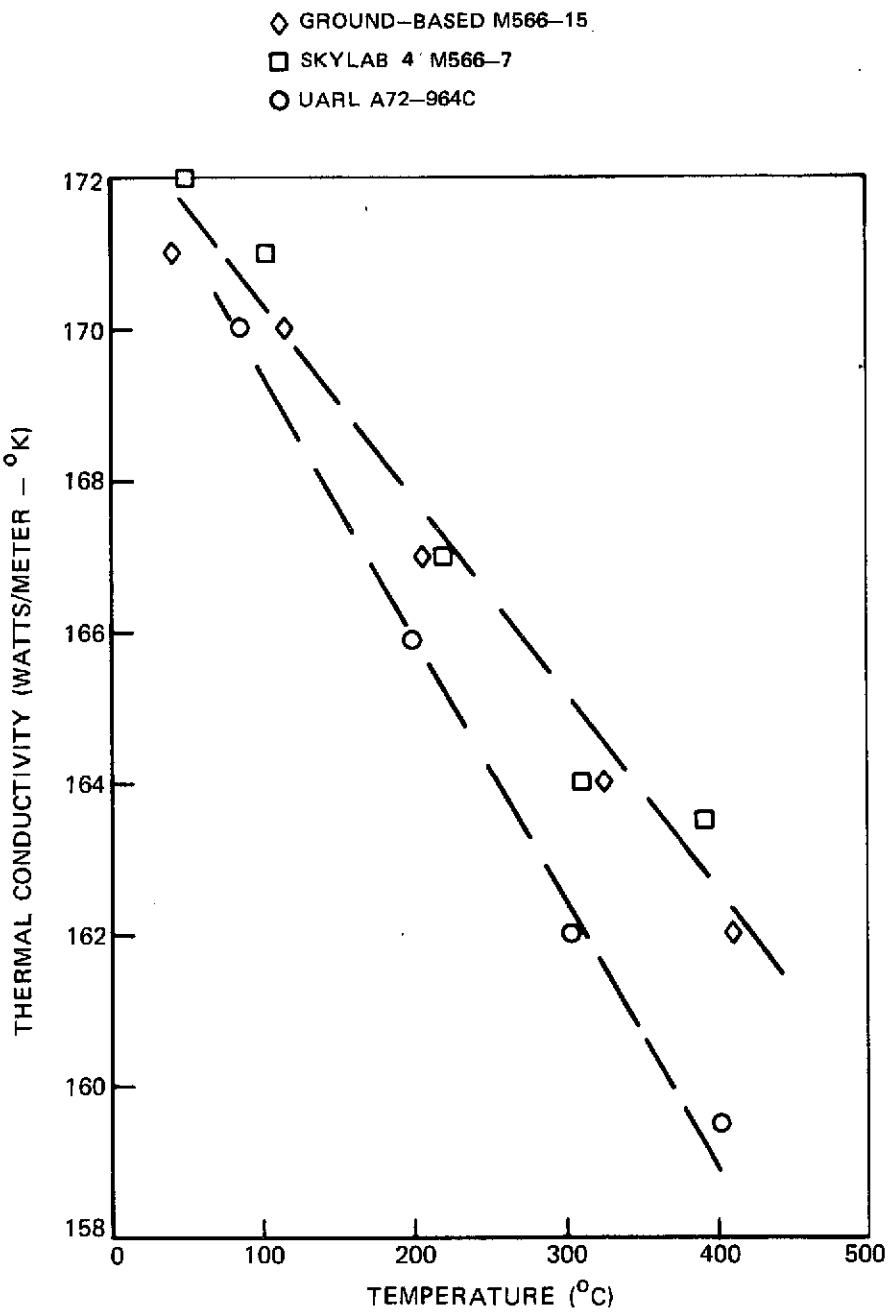


FIGURE 21. THERMAL CONDUCTIVITY MEASUREMENTS ON Al–Cu EUTECTIC ALLOYS COLLECTED RESULTS

N04–13–1

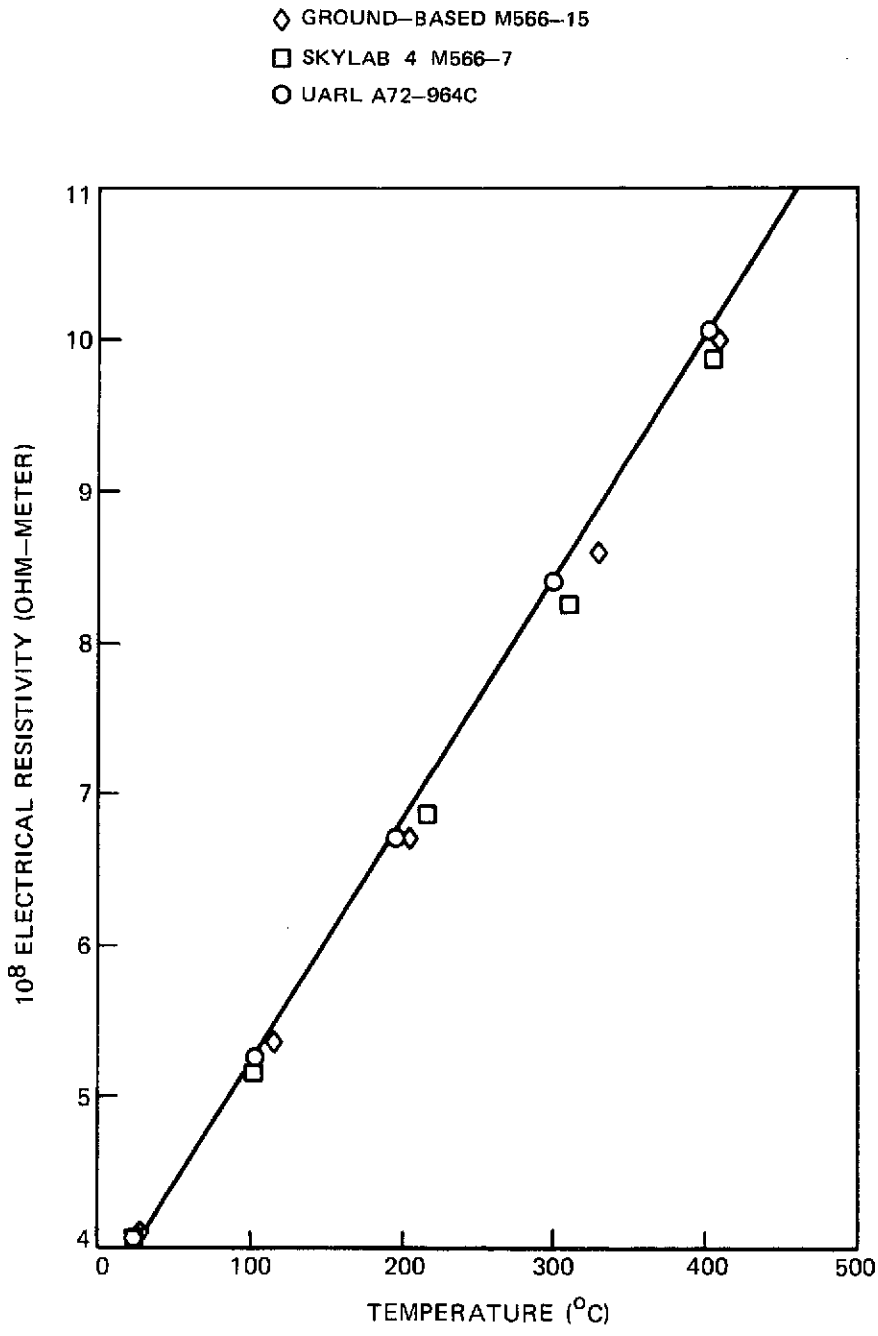


FIGURE 22. ELECTRICAL RESISTIVITY MEASUREMENTS ON Al–Cu EUTECTIC ALLOYS COLLECTED RESULTS

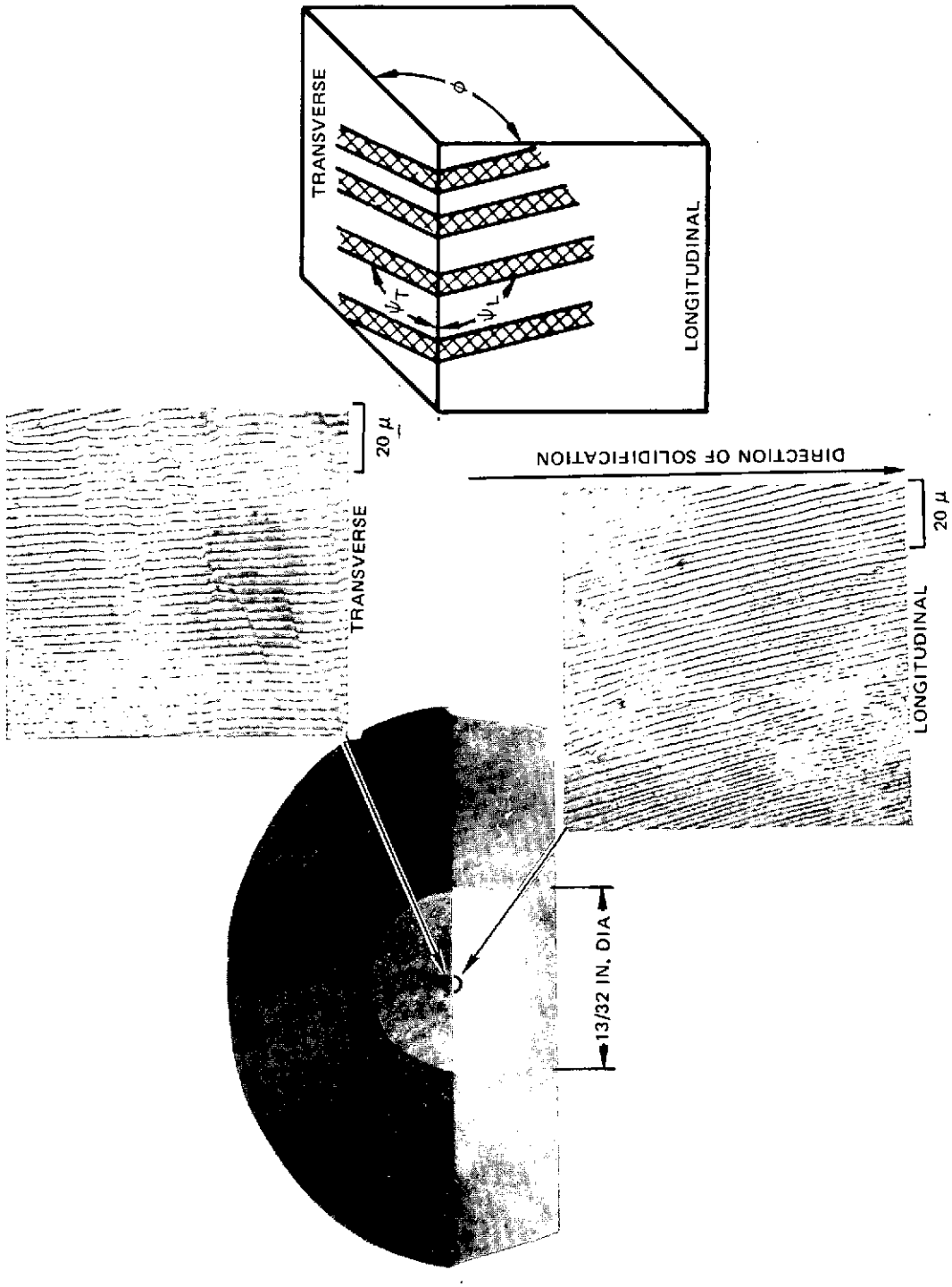


FIGURE A–1. MEASUREMENTS REQUIRED TO DETERMINE TRUE LAMELLAR ORIENTATION