N77-17681

SPACE PROCESSING ON SKYLAB AND ASTP

R. S. Snyder
Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

ABSTRACT

The Skylab and Apollo-Soyuz Test Project (ASTP) missions provided the opportunity to examine the influence of micro-gravity on the processing of various materials. The majority of the experiments dealt with the solidification of alloys, semiconductors, and composite materials or basic liquid-liquid and liquid-solid interactions necessary to understand complex processing. The space results have yielded basic data and increased knowledge of fundamental materials behavior. Potential advantages of space processing to several materials disciplines have been identified.

INTRODUCTION

In the following paragraphs the results of physical and engineering experiments in metallurgy, fluids handling and crystal growth on two space missions, Skylab and the Apollo-Soyuz Test Project (ASTP), will be discussed. The presentation is incorporated into a colloquium concerned with bioprocessing in space because there are similarities in process objectives in the two fields and the results may trigger some ideas in biological applications even though the metallurgists and solid state physicists were concerned with metals and semiconductors. Some of the properties explored in space should have utility in all scientific fields. For example, many disciplines are now expressing an interest in freely suspending their material and then performing manipulations or measurments with no contamination or uncontrolled disturbance. Homogeneous isotropic materials as well as ordered highly anisotropic material have been proposed for space investigation.

The unique advantage of the space environment for modifying or improving the properties of materials is the long term weightlessness. The extended weightlessness in space is created by the motion of the spacecraft under the influence of its high orbital velocity and the gravitational pull of earth. Objects located at the center of mass within the spacecraft are not accelerated by any additional outside force and they will "float". Objects displaced from this point will follow trajectories prescribed by well known equations of motion. These motions have been frequently photographed in space. Space is also characterized by a vast vacuum capacity and the possibility of attaining a vacuum significantly below the capability of any vacuum pump system on earth. The Skylab and ASTP experiments, however, were designed to utilize primarily the micro-gravity advantage of space.

These two missions encompassed a wide variety of experiments intended to explore the capabilities of the space environment and the details of each of them cannot be given in this brief overview paper. Several review articles of the experiments are already available[1] in addition to the individual reports of the principal investigators.

SKYLAB

A group of experiments that could be done in a common facility on Skylab, shown in Figure 1, were selected in 1969. An electron beam was incorporated to melt metals typically used in welding and to form freely floating metal spheres. The brazing of two tubes with an exothermic chemical reaction was proposed to investigate the capillary flow of the melt around the tube ends. The growth of a gallium arsenide single crystal in a solution of liquid gallium was designed to investigate an improved method to grow this important semiconductor material. All control functions, battery power and storage containers were included in this facility.

By 1971, interest in space processing had developed considerably. The Apollo 14 mission had flown three simple demonstrations of high interest to the program: electrophoresis; melting and solidification of composite materials; and fluid behavior under various thermal conditions. These experiments, done during the return of the Apollo from the moon, showed both the advantages and some of the difficulties of processing materials in space. In 1972, an additional set of eleven experiments were proposed to use the furnace concept of the gallium arsenide experiment but modified for multiple purpose use. (Figure 2) These are listed in Table 1 to show the variety of different experiments that were planned for this versatile facility. As is shown, several experiments were done on Skylab III and repeated with additional samples on Skylab IV. Since the furnace processed three sample cartridges each time, a significant amount of material was returned to Earth for analysis.

The "Vapor Growth of IV-VI Compounds" experiment investigated a technique for growing crystals of electronic materials by condensing vapor evolved from a heated source of the same material. Turbulence in the vapor due to the imposed temperature gradient limits the results on earth but swirling convection currents should not occur in weightlessness. The results of this experiment were unanticipated since up to ten times more crystalline material was produced in space. Dr. Wiedemeier proposes that this is primarily due to an inadequate theory used to determine growth on the ground as well as in space and the Skylab results will modify the fundamental understanding of this technology on Earth. Additionally, the structural perfection of the space grown crystals was clearly superior and the size of one crystal, shown in Figure 3, was six times larger than ever achieved.

Two experiments with different semiconductor materials, germanium and indium antimonide, were done to give a direct comparison of ground and space characteristics. A single crystal, contained in a cylindrical ampoule, was partially remelted and then solidified in space. The principal objective was to achieve improved homogeneity of a specific impurity by removing thermal convection at the solidification interface. The availability of precise electrical measurements systems and high resolution techniques to study segregation inhomogeneities on the order of fractions of a micrometer make semiconductor materials excellent candidates for space processing. Figure 4 shows a comparison of Earth and space-grown crystal of indium antimonide from Dr. Gato's experiment.

Dr. Hans Walter made crystals in a way that is impossible on Earth. A containerless melt was formed on the end of a seed crystal and then directionally solidified to form single crystals typified by Figure 5. The initial samples were cylinders of single crystal indium antimonide, one end fastened in a heat sink and the other in a cavity that was heated above the sample melting point. The cylinder was partially melted in space forming a spherical melt in contact with the solid seed material. The temperature of the cavity was then slowly lowered. The melt solidified first near the seed and finally at the tip in contact with the inner wall of the cavity. Although the melt was surely spherical, the final oblong crystal shape was determined by the forces intrinsic to the solidification process itself. These crystals were all characterized by flat surface facets and decreased internal imperfections.

The measurement of diffusion of one particle species through another in liquids is difficult on Earth because gravity will act on any small difference in particle density to produce convection currents. Dr. Ukanwa designed an experiment to detect and measure any disturbances to pure diffusion caused by any source. In this experiment, cylindrical rods of zinc containing radioactive zinc-65 atoms confined to a zone at the end of the cylinder were melted and solidified in space. By slicing the cylinder on Earth, the diffusion of radioactive zinc atoms was measured, the results show agreement with theory, again confirming the absence of convection currents driven by a temperature gradient.

APOLLO SOYUZ TEST PROJECT (ASTP)

The success of the Skylab experiments led to the proposal of over 30 experiments that could be accommodated in the Skylab furnace with minimum modification. The ASTP timeline allowed only seven experiments to be done by utilizing almost the entire mission in the heat-up, soak at high temperature and controlled cool-down required to fulfill the experiment requirements. The experiments selected for ASTP are listed in Table II. Three proposals were follow-on to Skylab experiments in which the same scientists wanted to obtain additional data. The USSR proposed an experiment to melt several different material combinations during the joint Russian American part of the mission but their analysis has not been completed.

The ASTP experiment on "Surface Tension Induced Convection" further investigated the elimination of convection currents in liquids metals. The Skylab experiment on diffusion in zinc indicated that gravity-induced thermal convection is effectively eliminated in space processing. However, as a result of this condition, it was hypothesized that convection effects caused by surface tension gradients may become significant in space processing. Since surface tension gradients occur due to thermal or concentration differences which are not gravity dependent factors, the objective of this experiment was to determine if similar surface tension induced convection effects would result from concentration gradients. The experiment concept consisted of melting three samples of bi-metallic materials (lead and lead-gold alloy) in wetting and non-wetting capsules in the multipurpose furnace, allowing inter-diffusion of the two components, and then resolidification. If no convective stirring effects due to the surface tension difference between the materials exist, then a normal concentration - distance profile for the gold would result. The preliminary examination of the flight samples indicates that a normal concentration-distance profile of gold in the sample was obtained. Autoradiographs compare favorably with distance predictions based on the zinc self-diffusion data obtained on Skylab indicating that the diffusion of gold in lead was in the typical liquid diffusion range.

"Monotectic and Syntectic Alloys", investigated the effects of weightlessness on the melting and solidification of two material systems, lead-zinc and aluminum antimonide. The objectives were to investigate phase segregation effects in low gravity for the immiscible binary lead-zinc and to determine the influences of low-gravity solidification on the microstructural homogeneity and Stoichiometry of semiconducting compound aluminum antimonide. The large density difference between the two metals makes it difficult to avoid severe gravity separation of the phases during solidification of earth-prepared systems. The comparison of the microstructure of the ground and space processed material, Figure 6, clearly shows an improvement of homogeneity obtained in space.

The experiment, "Interface Marking in Crystals", was developed to study quantitatively the basic solidification behavior of high temperature melts under near zero-gravity conditions and answer questions raised by the earlier experiment by Professor Gatos of MIT on Skylab. The experimental hardware on ASTP differed from the related Skylab experiment in that an electrical pulsing unit was utilized to provide interface demarcation during solidification from the melt. Evaluation of the demarcation lines which were clearly established throughout the samples show that the microscopic growth rate was subject to an initial transient which did not stabilize immediately. The observed rate behavior in space was comparable to the ground-based samples and at variance with the behavior predicted by theory. Segregation analysis again showed striking differences between one-gravity and zero-gravity conditions.

ASTP experiment "Processing of Magnets" was conducted to determine the potential advantage of space processing to modify critical magnetic properties by improving chemical homogeneity, morphological and crystalline perfection of the magnetic substructure. Preliminary results now indicate that a magnetic alloy of manganese and bismuth resolidified directionally in space has attained a significantly higher coercive strength.

The objectives of "Crystal Growth from the Vapor Phase" were to continue the Skylab investigation into the effects of micro-gravity on the structure of single crystals of mixed systems and to determine the mass transport rates of these systems using the chemical transport technique. The materials, germanium selenide and germanium telluride were chosen to be similar to Dr. Wiedemeier's Skylab experiment. The ASTP experiment gave close agreement of data which confirms predictions that the vapor transport technique is suitable for the growth of crystals of significantly better quality in a micro-gravity environment. Crystals of high structural uniformity were produced and the vapor transport rate in space has consistently been greater than predicted by earth models resulting in much larger single crystals than can be produced on earth by identical techniques.

Experiment MA-131, "Halide Eutectic Growth", was developed to investigate production in space of a eutectic mixture consisting of continuous fibers of LiF embedded in a NaCl matrix. When grown on earth, this material has not realized its full potential for optical transmission due to discontinuous fibers resulting from convection currents in the melt. Dr. Yue's related Skylab experiment demonstrated that continuous fibers of NaF embedded in a NaCl matrix could be grown in space by the directional solidification technique resulting in a material that exhibited improved optical properties in comparison to earth-grown samples. This technique was again successfully utilized in the ASTP experiment and improved optical transmission was obtained. Figure 7 shows a comparison of the optical transparency for the best Earth-grown material and the sample from space.

Demonstrations of fluid banding in space and two electrophoresis experiments done on ASTP are described elsewhere in this Colloquium. There are other bioprocessing experiments that could be done advantageously in space and they could be related to the solidification experiments just discussed. The ease with which liquids can be immobilized and manipulated should have relevance to bioprocessing. Ideas for new experiments should be developed and proposed to NASA for the Spacelab missions of the 1980's.

REFERENCES:

1. Proceedings of the Third Space Processing Symposium - Skylab Results, Marshall Space Flight Center, April 30 - May 1, 1974

   Apollo-Soyuz Test Project - Preliminary Science Report, NASA TMX-58173, February 1976

SKYLAB EXPERIMENTS	SKYLAB MISSION II	III	IV
MATERIALS PROCESSING FACILITY			
M551: METALS-MELTING EXPERIMENT, R. R. POORMAN, MSFC ASTRONAUTICS LAB	X		
M552: EXOTHERMIC BRAZING EXPERIMENT, J. R. WILLIAMS, MSFC PRODUCT ENG. LAB	X		
M553: SPHERE-FORMING EXPERIMENT, E. A. HASEMEYER, MSFC ENG. LAB	X		
M555: *GALLIUM ARSENIDE CRYSTAL-GROWTH EXPERIMENT, R. S. SEIDENSTICKER, WESTINGHOUSE RESEARCH LAB			
MUTTIPURPOSE FURNACE SYSTEM			
M556: VAPOR GROWTH OF IV-VI COMPOUNDS, H. WIEDEMEIER, RENSSELAER POLYTECHNIC INSTITUTE		X	X
M557: IMMISCIBLE ALLOY COMPOSITIONS, J. L. REGER, TRW SYSTEMS		X	X
M558: RADIOACTIVE-TRACER DIFFUSION, A. O. UKANWA, MSFC SPACE SCIENCES LAB		X	
M559: MICROSECRATION IN GERMANIUM, F. A. PADOVANI, TEXAS INSTRUMENTS		X	
M560: GROWTH OF SPHERICAL CRYSTALS, H. U. WALTER, UNIV. OF ALABAMA		X	X
M561: WHISKER-REINFORCED COMPOSITES, T. KAWADA, NATIONAL INSTITUTE FOR METALS RESEARCH		X	X
M562: INDIUM ANTIMONIDE CRYSTALS, H. C. GATOS, MIT		X	X
M563: MIXED III-V CRYSTAL GROWTH, W. R. WILCOX, USC		X	X
M564: ALKALI HALIDE EUTECTICS, A.S. YUE, UCLA		X	
M565: SILVER GRIDS MELTED IN SPACE, A. DERUYTHERRE, KATHOLIEKE UNIV., LEUVEN, BELGIUM		X	
M566: COPPER-ALUMINUM EUTECTIC, E. A. HASEMEYER, MSFC PRODUCT ENG. LAB		X	X
EXPERIMENTS PERFORMED ON EACH MISSION:	3	11	7

* NOT FLOWN BECAUSE STORAGE AREA PREEMPTED BY SKYLAB REPAIR KIT.

TABLE I

ASTP MULTIPURPOSE FURNACE EXPERIMENTS

MA-010:	MULTIPURPOSE FURNACE - J. McHUGH, WESTINGHOUSE
MA-041:	SURFACE TENSION INDUCED CONVECTION - R. E. REED, OAK RIDGE
MA-044:	MONOTECTIC AND SYNTECTIC ALLOYS - C. Y. ANG, USRA/MSFC
MA-060:	INTERFACE MARKING IN CRYSTALS - H. C. GATOS, MIT
MA-070:	PROCESSING OF MAGNETS - D. J. LARSON, GRUMMAN
MA-085:	CRYSTAL GROWTH FROM THE VAPOR PHASE - H. WIEDEMEIER, RPI
MA-131:	HALIDE EUTECTIC GROWTH - A. S. YUE, UCLA
MA-150:	MULTIPLE MATERIALS MELTING - I. IVANOV, USSR

TABLE II

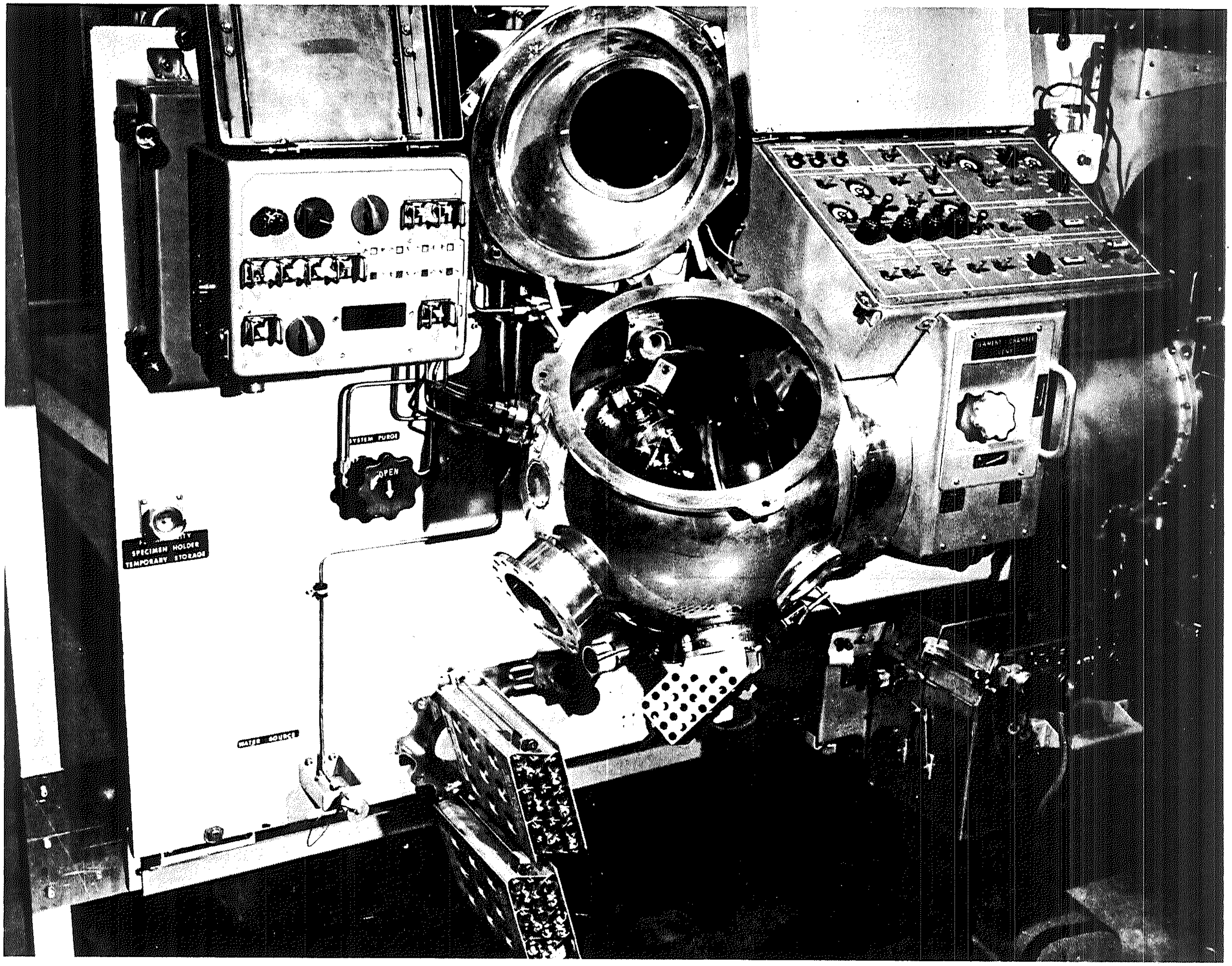


Figure 1.- M512 space processing experiment facility on Skylab.

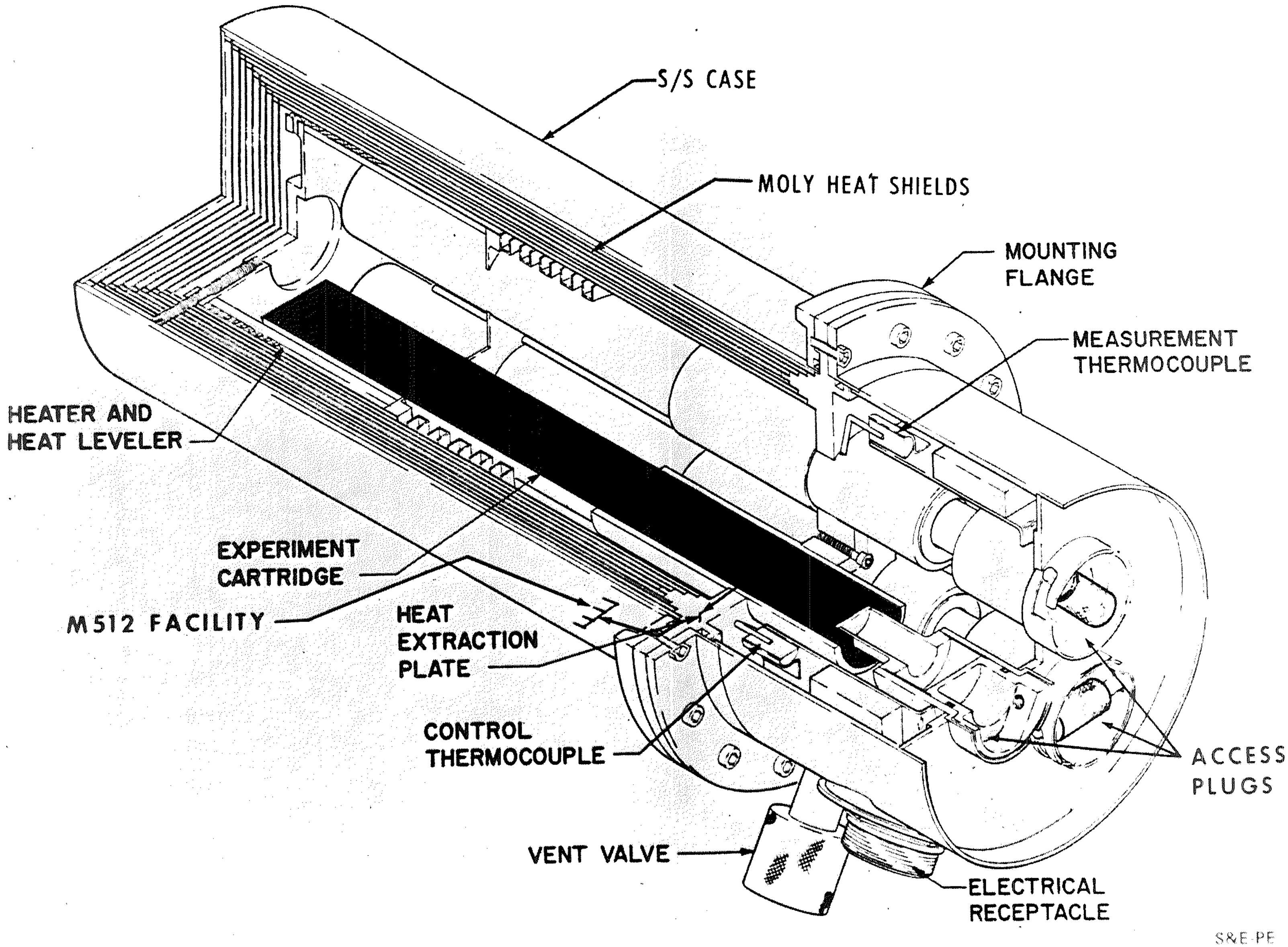


Figure 2.- M518 multipurpose electric furnace.

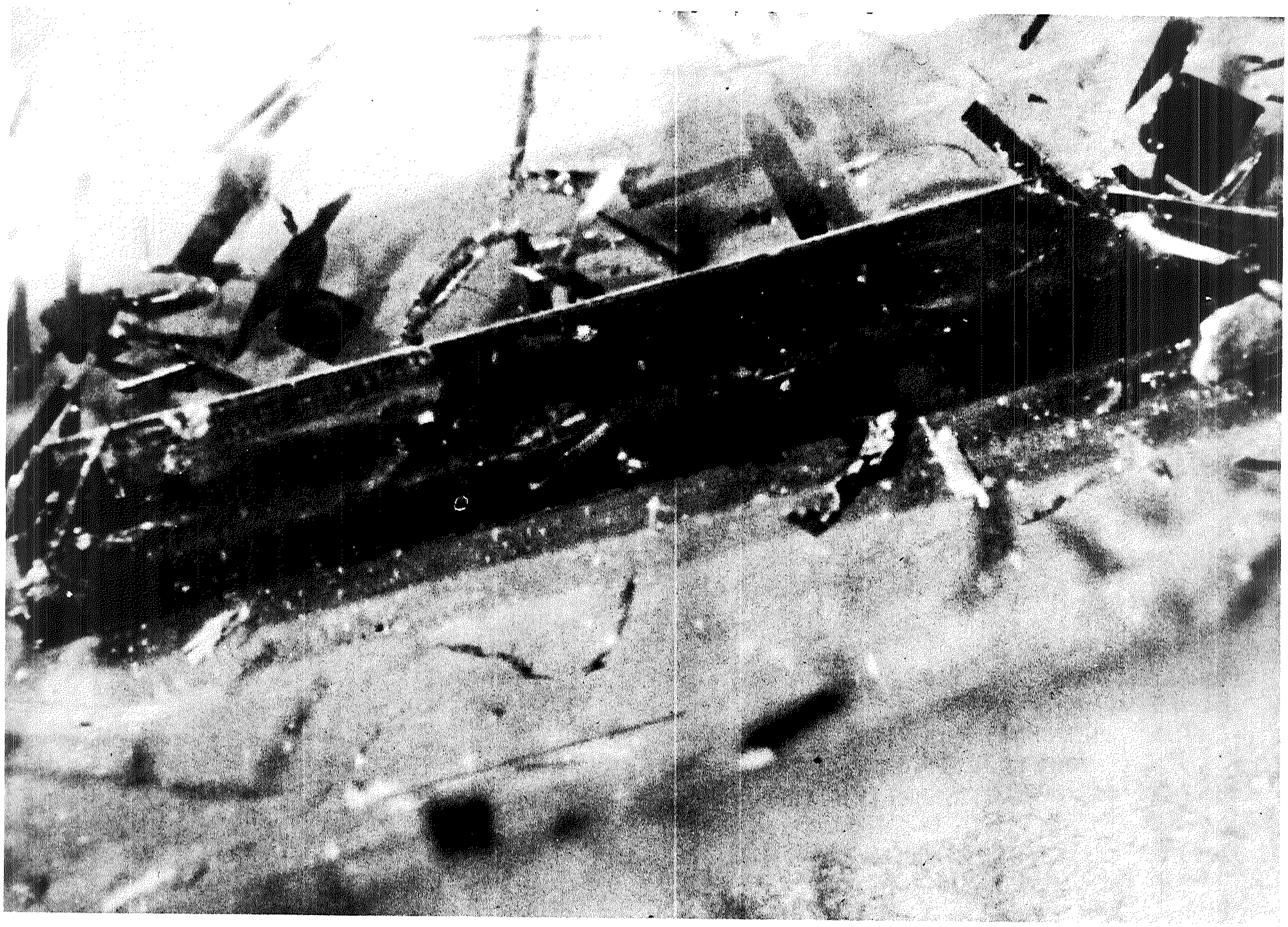


Figure 3.- Space-grown (zero g) semiconductor crystal (germanium selenide); Skylab experiment M556, single crystal, 20-mm length).

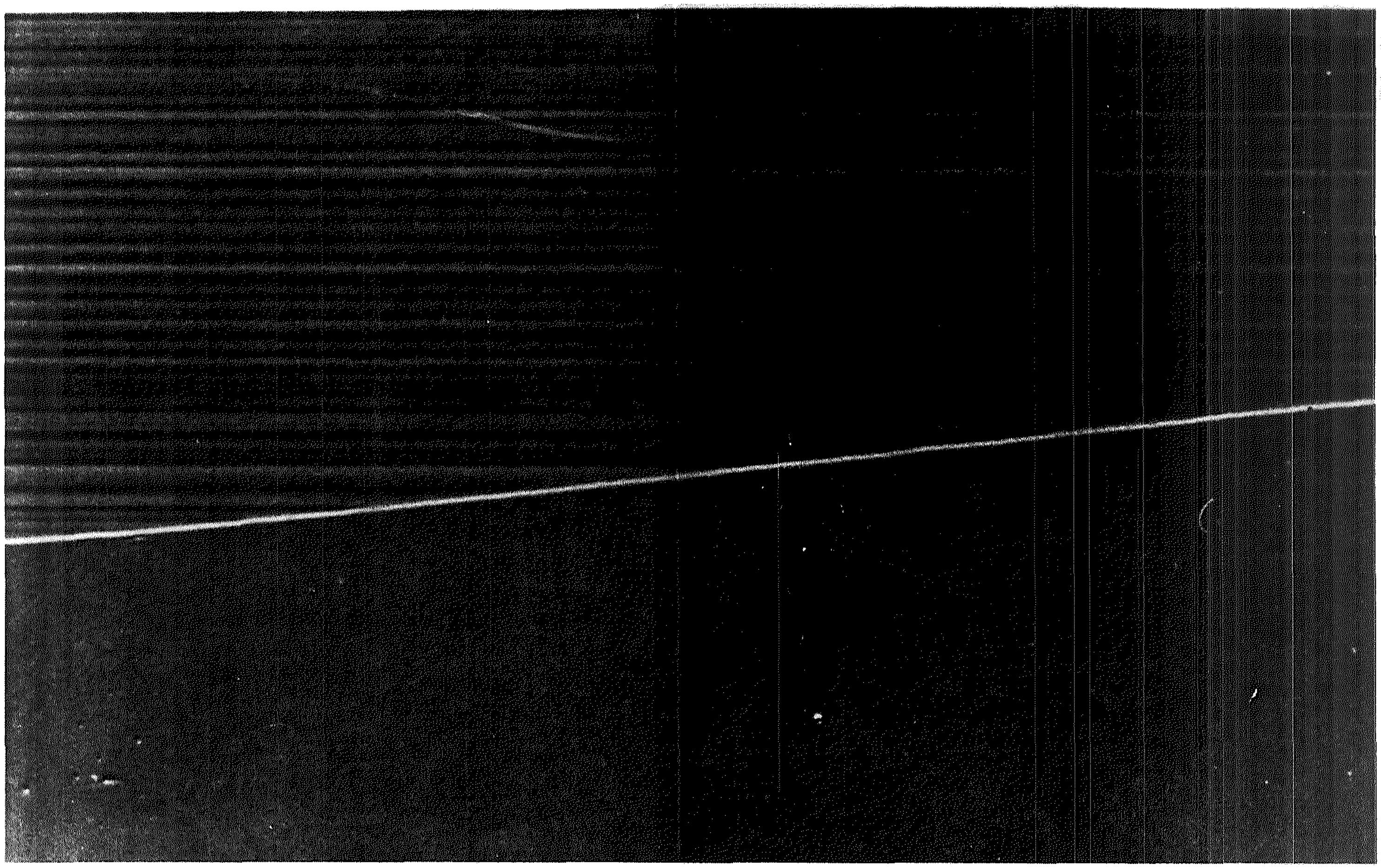


Figure 4.- Segment of the initial regrowth interface of tellurium-doped InSb crystal (B-1); dopant inhomogeneities are seen in the Earth-grown (upper) section; no dopant inhomogeneities are present in the space-grown (lower) section; 125X magnification.

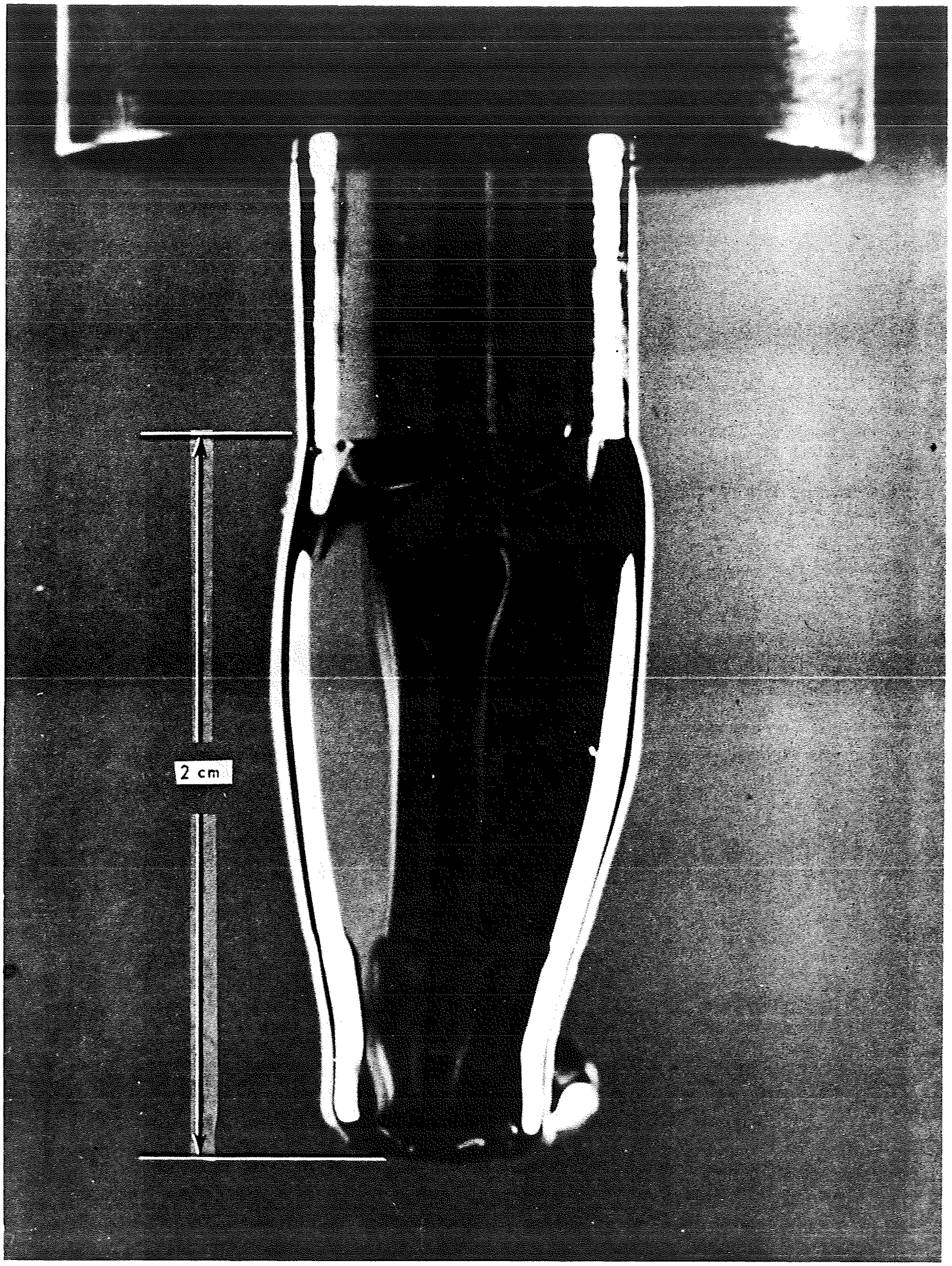
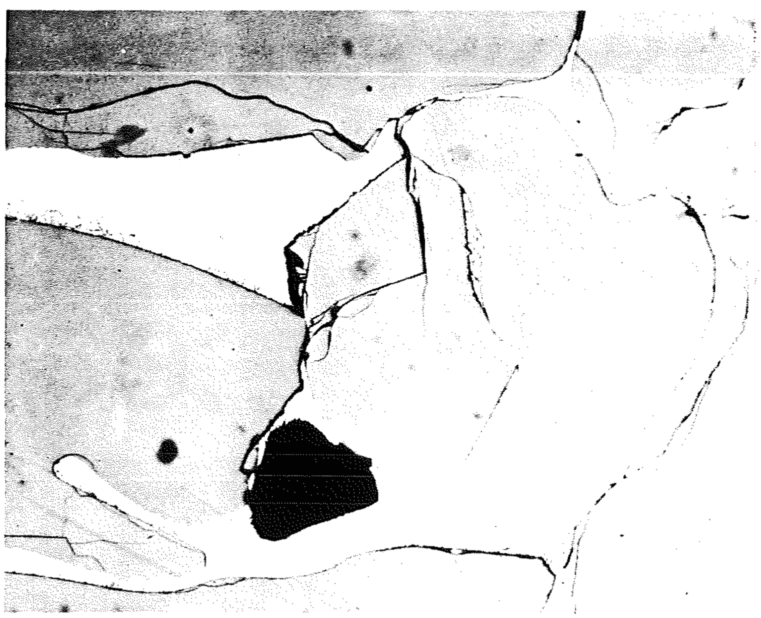


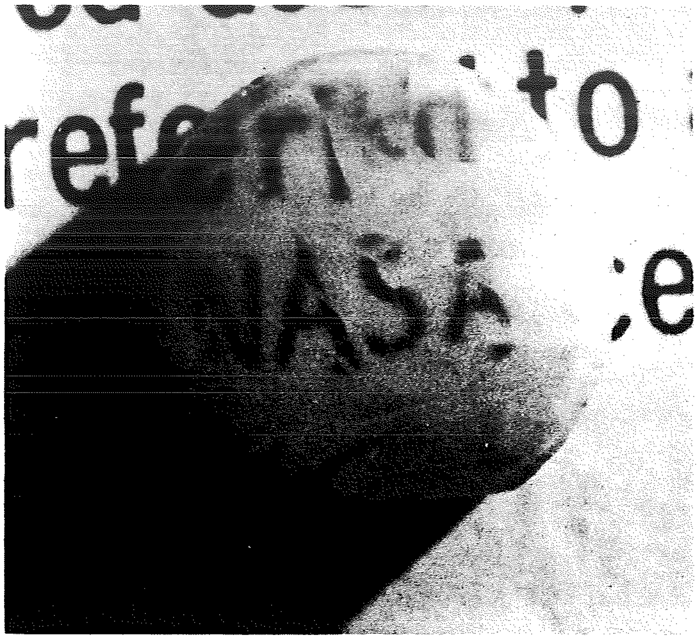
Figure 5.- Space-grown (containerless) single crystal of semiconductor indium antimonide; Skylab experiment M560 (special illumination).

(a) Low g (A185).

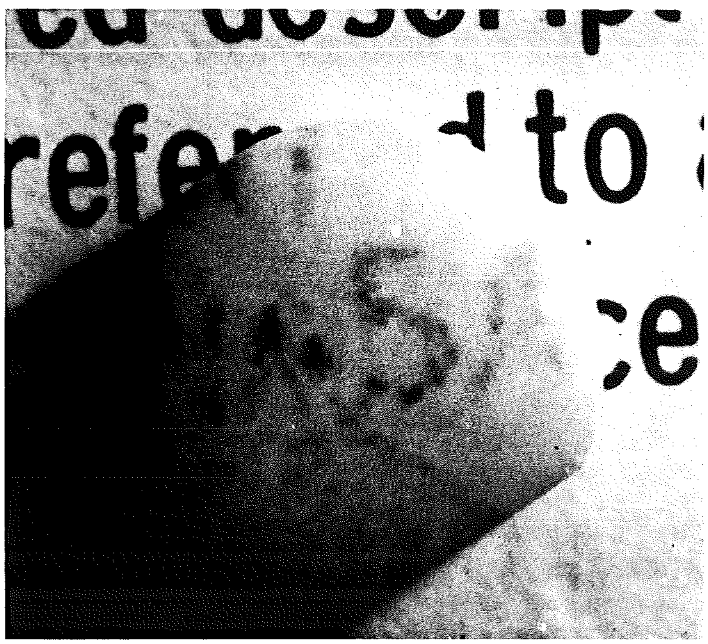


(b) One g (A141).

Figure 6.- Comparison of ASTP and GBT-2 AlSb microstructure.



(a) Space-grown.



(b) Earth-grown.

Figure 7.- Optical transparency of samples from halide eutectics experiment M564.