NAS8-30579

(NASA-CR-161966) ZERO-GRAVITY GROWTH OF A SODIUM CHLORIDE-LITHIUM FLUORIDE EUTECTIC MIXTURE Final Report (California Univ.) 27 p HC A03/MF A01 CSCL 07D

N82-17299

Unclas G3/25 11695

SECTION VIII

ZERO-GRAVITY GROWTH OF NaCl-LiF EUTECTIC

EXPERIMENT MA-131

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TABLE OF CONTENTS

	Page
ABSTRACT	VIII-5
INTRODUCTION	VIII-5
OBJECTIVES	VIII-5
EXPERIMENTAL PROCEDURE	VIII-6
RESULTS	VIII-8
Macrostructures and Microstructures	VIII-8
Lithium Fluoride Fibers	VIII-12
Interfiber Spacing	VIII-18
Image Transmission	VIII-18
Optical Property	VIII-20
DISCUSSION	VIII-24
LiF Fibers	VIII-24
Optical Transmittance	VIII-25
CONCLUSIONS	VIII-26
RECOMMENDATIONS	VIII-26
REFERENCES	VIII-27

LIST OF ILLUSTRATIONS

Figure	Title	Page
VIII-1.	Sketch of an ampoule	VIII-6
VIII-2.	Macrophotograph of the three ASTP ampoules	VIII-9
VIII-3.	Macrophotograph of ASTP-grown NaCl-LiF eutectic	VIII-10
VIII-4.	Macrophotograph showing the original shape of the solid-liquid interface of the ASTP-grown NaCl-LiF eutectic	VIII-11
VIII-5.	Enlarged portion at the original solid-liquid interface	VIII-11
VIII-6.	Photomicrographs of a longitudinal section of the NaCl-LiF eutectic showing (a) continuous LiF fibers and (b) discontinuous LiF fibers	VIII-13
VIII-7.	Photomicrograph of the transverse section showing shapes of LiF fibers	VIII-14
VIII-8.	Scanning electron photomicrograph of the Earth-grown LiF fibers	VIII-14
VIII-9.	Scanning electron photomicrograph of the ASTP-grown LiF fibers	VIII-15
VIII-10.	Macrophotograph of the transverse section of the NaCl-LiF eutectic showing grains and subgrains, sample No. 10	VIII-15
VIII-11.	Macrophotographs of (a) ASTP-grown and (b) Earth-grown NaCl-LiF eutectic	VIII-16
VIII-12.	ASTP-grown ingot with continuous LiF fibers	VIII-17
VIII-13.	Discontinuous and randomly oriented fibers of the Earth-grown ingot end	VIII-17

LIST OF ILLUSTRATIONS (Concluded)

Figure	Title	Page
VIII-14.	Image transmission macrophotograph of (a) ASTP-grown and (b) Earth-grown NaCl-LiF eutectics, 2 mm sample thickness	VIII-19
VIII-15.	Far-field infrared transmission curves of transverse sections of NaCl-LiF eutectics grown on Earth and in space, 0.0211 in. sample thickness	VIII-20
VIII-16.	Far-field infrared transmittance curves of transverse sections of NaCl-LiF eutectics with different thickness, sample No. 131-03	VIII-21
VIII-17.	Far-field infrared transmittance curves of transverse sections of NaCl-LiF eutectics with different thickness, sample No. 131-08	VIII-22
VIII-18(a).	Far infrared transmittance curve of a longitudinal section of NaCl-LiF eutectic grown on Earth, electric field perpendicular to fiber axis	VIII-22
VIII-18(b).	Far infrared transmittance curve of a longitudinal section of NaCl-LiF eutectic grown on Earth, electric field perpendicular to fiber axis	VIII-23
VIII-19(a).	Far infrared transmittance curves of a longitudinal section of NaCl-LiF eutectic (131-07) of varying thicknesses, electric field parallel to fiber axis	VIII-23
VIII-19(b).	Far infrared transmittance curves of a longitudinal section of NaCl-LiF eutectic (131-07) of varying thicknesses, electric field perpendicular to fiber axis	VIII-23
VIII-20.	Diagram illustrating quantities discussed in analyzing scattering measurements for transmission through a longitudinal sample section	VIII-25

ABSTRACT

Continuous and discontinuous lithium fluoride fibers embedded in a sodium chloride matrix have been produced in space and on Earth, respectively. The production of continuous fibers in a eutectic mixture was attributed to the absence of convection current in the liquid during solidification in space. Image transmission and optical transmittance measurements of transverse sections of the space-grown and Earth-grown ingots were made with a light microscope and a spectrometer. It was found that better optical properties were obtained from samples grown in space. This was attributed to a better alignment of lithium fluoride fibers along the growth direction.

INTRODUCTION

When certain binary eutectic mixtures solidify, one of the two phases can form fibers or platelets in a matrix of the second phase. For example, when an eutectic liquid of sodium chloride (NaCl) and sodium fluoride (NaF) solidifies, fibers of NaF [VIII-1] form a matrix of NaCl. Similarly, when a liquid of NaCl and lithium fluoride (LiF) solidifies, LiF will form the fiber phase in the NaCl matrix.

Fiberlike and platelike eutectics produced on Earth are limited in perfection by the presence of a banded structure [VIII-2, VIII-3], discontinuity [VIII-4], and faults [VIII-5, VIII-6] due, at least in part, to vibration and convection currents in the melt during solidification. The presence of these defects renders the solid-state eutectic devices inefficient and useless [VIII-7].

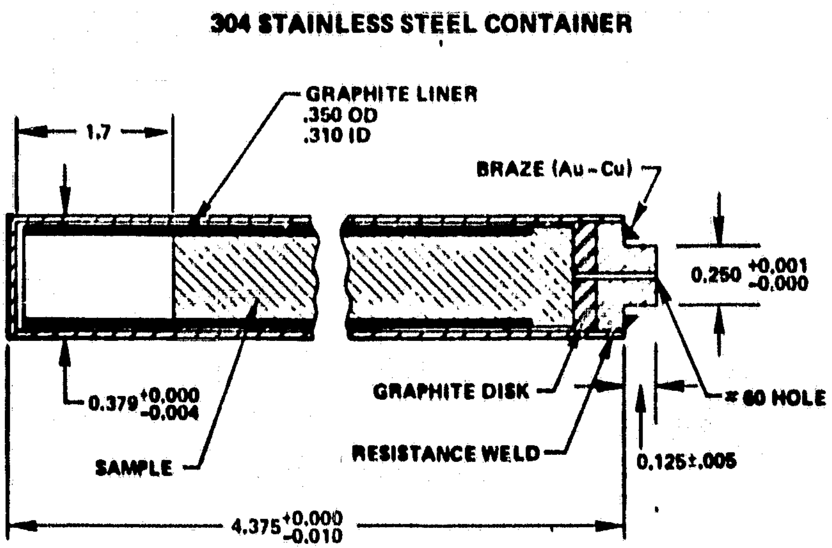
If the solidification process is performed in a space environment, where there are no vibration and convection currents in the melt, there is reason to believe that continuous fiberlike eutectic microstructures can be produced. The electric, thermomagnetic, optical, and superconducting characteristics of such eutectics will be strongly anisotropic, which suggests the possibility of exciting device applications. A Skylab experiment [VIII-1] of NaCl-NaF eutectic demonstrated that continuous fibers of NaF embedded in an NaCl matrix have been grown by the directional solidification technique in space.

OBJECTIVES

The objectives of this project were to: (1) prepare, in a space experiment, fiberlike LiF-NaCl eutectic with continuous LiF fibers embedded in the NaCl matrix (this eutectic of continuous LiF fibers cannot be produced on Earth because of the presence of convection currents and vibration in the melt during solidification); (2) extract a few fibers from an ASTP-grown NaCl-LiF eutectic ingot and also from Earth-grown ingots and determine whether the extracted fibers were continuous or discontinuous; (3) calculate the interfiber spacing of the LiF fibers; and (4) measure transmittance versus wavelength curves of ASTP-grown and Earth-grown ingots, both along and perpendicular to the fiber axes.

EXPERIMENTAL PROCEDURE

NaCl, 29 wt.% LiF eutectic ingots were prepared from 99.96 percent NaCl and 99.99 percent Lif obtained from Research Organic-Inorganic Chemical Corporation, Sun Valley, California. The salt ingots were melted and solidified in an induction furnace under an argon atmosphere. After solidification, each ingot was machined to the shape and dimensions depicted by the sample in Figure VIII-1.



THE SOLIDIFICATION EXPERIMENT WAS CARRIED OUT IN A MULTIPURPOSE ELECTRIC FURNACE [VIII-8] AT A FREEZING RATE OF 0.6°C/min AND A TEMPERATURE GRADIENT OF 50°C/cm. THE PROCEDURE FOR CONDUCTING THE EXPERIMENT IS AUTOMATIC.

Figure VIII-1. Sketch of an ampoule.

To hold the salt ingot, a graphite crucible was machined from a high purity graphite rod, 0.5 in. in diameter and 12 in. long, obtained from Ultra Carbon Corporation, Bay City, Michigan, to the dimensions of 4 in. in length, 0.350 in. outside diameter, and 0.310 in. inside diameter. Since graphite is a very fragile material, a special method was employed to machine the crucibles. Beginning with a 0.5 in. rod mounted in a lathe, the 0.310 in. hole was drilled so as to be concentric with the graphite rod. Then a 0.31 in. brass rod was inserted into the 0.310 in. hole to give the graphite tube support, while the outer diameter was machined from 0.5 in. to 0.35 in. in diameter. The graphite crucibles were cut to 4.375 in. in length and calcined in vacuum at 950°C for 25 h.

The salt ingot-graphite crucible assembly was then loaded into a 304 stainless steel container having the dimensions shown in Figure VIII-1. The steel container was made from 0.375 in. outside diameter and 0.355 in. inside diameter stainless steel tubing obtained from Tube Sales Co., Los Angeles, California, with a stainless steel plug heliarc-welded to one end of the tube. After loading the ingot-crucible assembly, the inner wall of the stainless steel container was coated with a graphite paste where the salt ingot is exposed to the container. Then a calcined graphite disk (0.010 in. thick) was placed over the end of the ingot. The graphite paste and disk were employed to prevent a reaction between the container and the salt when the salt was later remelted and resolidified. The container was then sealed by heliarc welding a cap in place, with a No. 60 vent hole as shown in Figure VIII-1.

There were three groups of samples involved. All were prepared initially at the University of California, Los Angeles (UCLA). The first set was retained at UCLA for later comparison (group A). The second set was sent to Westinghouse Laboratories to be regrown in a prototype furnace for comparison with the space-grown samples (group B). The third set of the three samples went from UCLA to the Apollo-Soyuz space vehicle for growth in space (group C).

The resolidification experiment in the Apollo-Soyuz vehicle was carried out in a multipurpose electric furnace [VIII-8]. One-half of the eutectic sample in this experiment adjacent to the graphite disk was left unmelted, and the remaining portion of the sample was melted and resolidified unidirectionally toward the empty space of the ampoule, as indicated in Figure VIII-1.

RESULTS

The experimental results are presented in five parts. The first part concerns the macrostructure and microstructures of the samples that were grown in the Apollo-Soyuz vehicle and on Earth. The second part concerns the extraction of continuous LiF fibers embedded in an NaCl matrix. The third part calculates the interfiber spacing of the LiF fibers of the ASTP-grown NaCl-LiF eutectic. The fourth part concerns the image the transmission of the NaCl-NaF eutectic, and the fifth part concerns the optical transmittance.

Macrostructures and Microstructures

After the space experiment the three ampoules were brought to us by Dr. Ang of Aerospace Corporation and were given three identity numbers (131-07, 131-08, and 131-09).

Figure VIII-2 is a macrophotograph showing the appearance of the three ampoules after the space experiment. The surfaces of the stainless steel cylinders and plugs were in perfect condition.

Figure VIII-3 is a macrophotograph of the three ASTP-grown samples taken out of the ampoules by grinding off the welded ends of each stainless steel cylinder. A careful inspection of the surface of the samples revealed no reaction between the NaCl-LiF eutectic and the graphite container. In sample 18, one transverse fracture surface occurred at the head portion of the sample, as revealed in Figure VIII-3. However, the fracture, which occurred after resolidification, did not interrupt the growth pattern of the sample.

Figure VIII-4 is a macrophotograph of the solidified sample, 131-09, showing the shape of the melt-back interface. The resolidified portion of the sample is on the right of the interface, and the unmelted portion of the sample is on the left of the interface. In this particular sample, the remelt-back interface is 0.28 cm (0.11 in.) from the left end of the sample, whereas the same melt-back distances of the other two samples are approximately 0.51 cm (0.20 in.). This may indicate a slight offset in the positioning of the heat zones in the furnace. An enlarged portion of the solid-liquid interface is given in Figure VIII-5, which shows the beginning of the solidification process. The elongated LiF phases grew in a direction perpendicular to the conical solid-liquid interface. This indicates that the direction of heat extraction during the onset of solidification was not parallel to the growth direction as intended. At a distance not far away from the initial solid-liquid interface as evidenced in

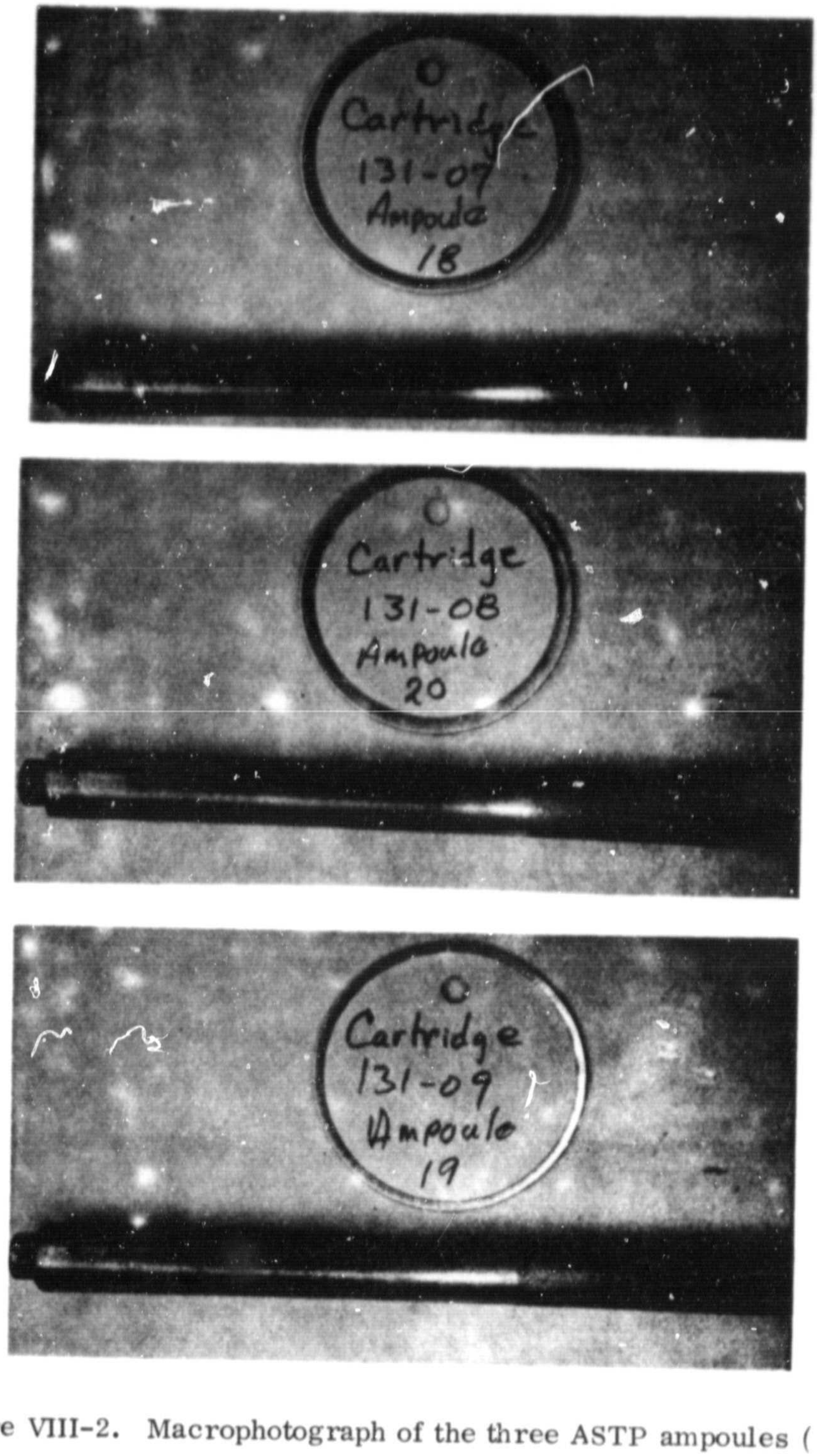


Figure VIII-2. Macrophotograph of the three ASTP ampoules (1X).

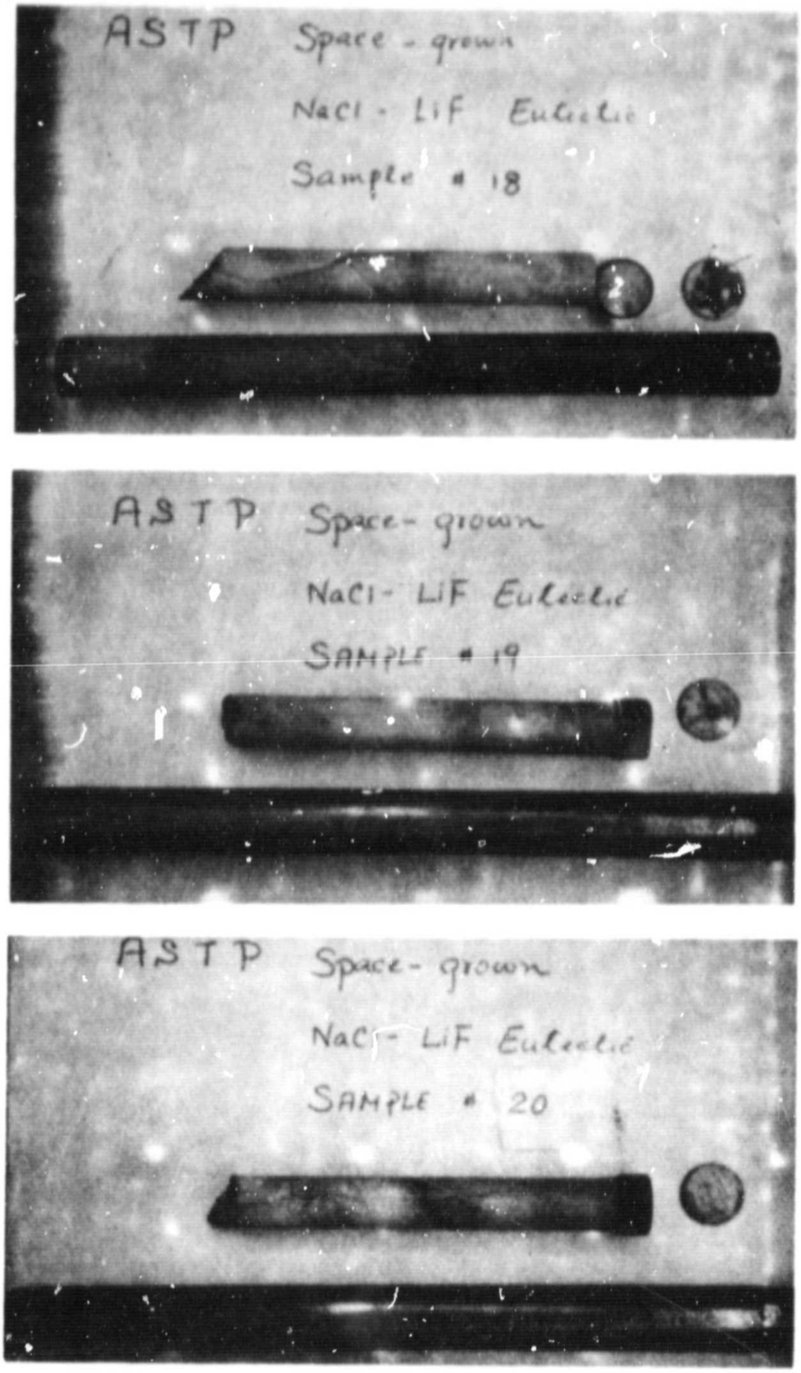


Figure VIII-3. Macrophotograph of ASTP-grown NaCl-LiF eutectic.

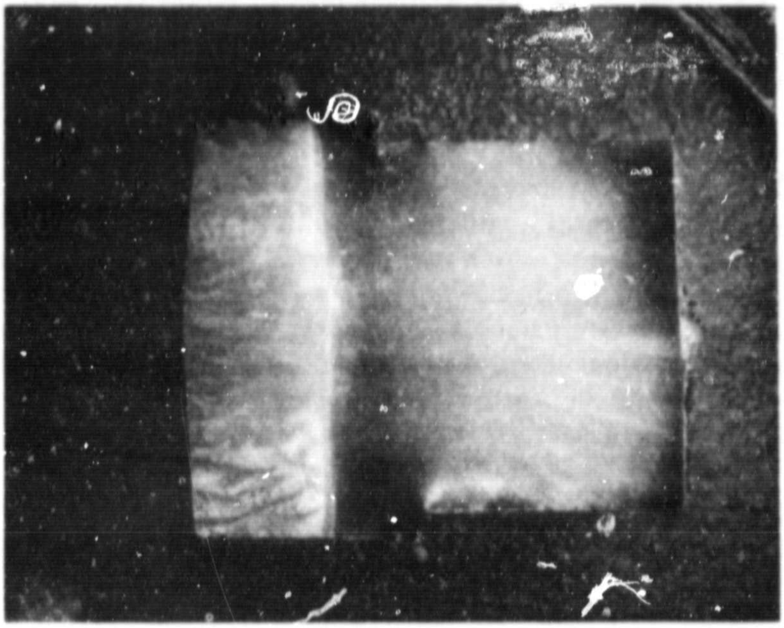


Figure VIII-4. Macrophotograph showing the original shape of the solid-liquid interface of the ASTP-grown NaCl-LiF eutectic (6.4X).

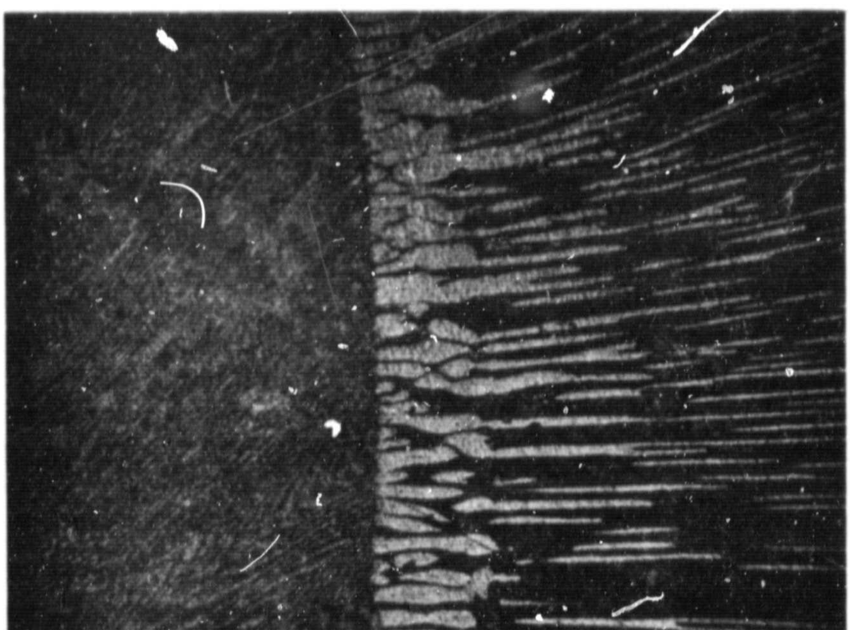


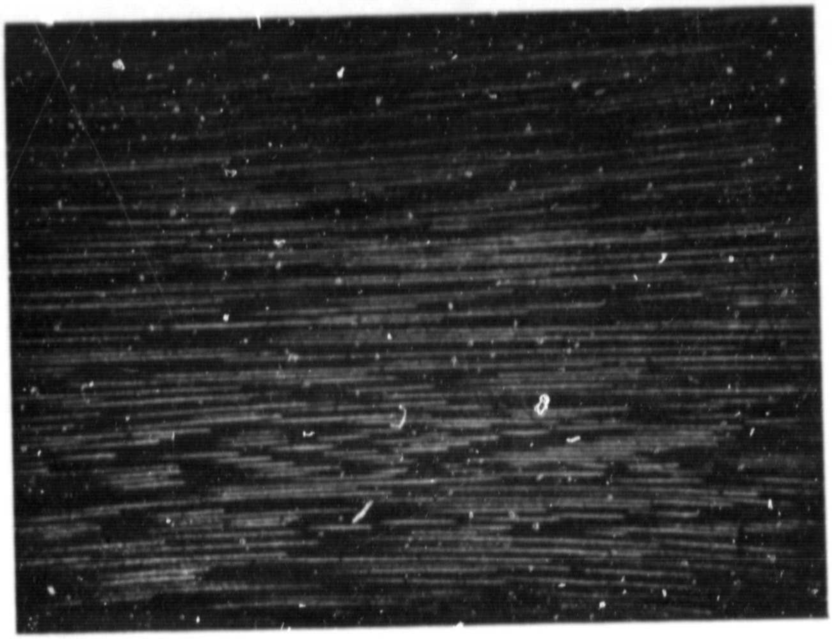
Figure VIII-5. Enlarged portion at the original solid-liquid interface (80X).

in Figures VIII-4 and VIII-5, the LiF fibers began to divert toward the periphery of the ingot. Figure VIII-6(a) is a representative photomicrograph of a longitudinal section of 131-09 showing the long continuous fibers. The eutectic microstructure that resulted when the NaCl-LiF eutectic was grown on Earth with convection current is shown in Figure VIII-6(b), which shows that the solidified LiF fibers are short and discontinuous. A representative photomicrograph of a transverse section of a sample is shown in Figure VIII-7, which reveals the shapes of the fibers which are preferentially round. A scanning electron photomicrograph (SEM) showing the shapes of LiF fibers grown on Earth protruding from the continuous NaCl matrix is given in Figure VIII-8, which is the perspective view of the round LiF fibers. Figure VIII-9 shows a corresponding SEM view of the ASTP-grown ingot showing that all the LiF fibers are aligned in one direction.

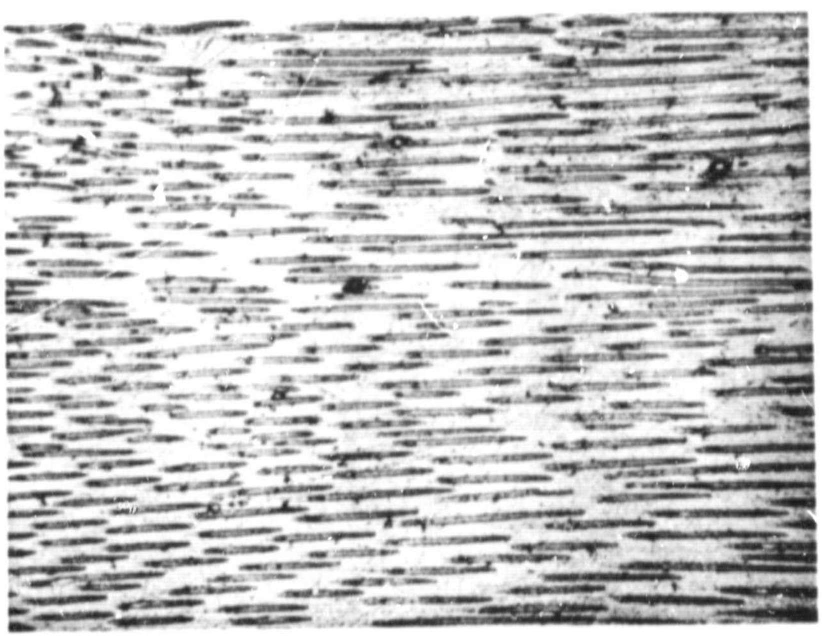
Single-grain eutectic was not produced in the presence of microgravity in space as evidenced in Figure VIII-10, which is a transverse section of sample 131-07. Many grains are present throughout the entire cross section. Evidence supporting this is given in Figure VIII-11(a), which is a picture taken from sample 131-07 grown in space. Filtered light from a Bausch and Lomb microscope was directed at the lower end of the sample. Because of good alignment of LiF fibers along the sample axis, light was transmitted from the lower end and propagated through the ingot at a longer distance with a much stronger intensity than the sample grown on Earth, as evidenced in Figure VIII-11(b). Light was not transmitted through the upper portion of the ASTP sample, because in that portion of the sample, the fibers did not align with the sample axis.

Lithium Fluoride Fibers

Figure VIII-12 is a photograph of an ASTP-grown ingot taken when the LiF fibers were suspended in a methyl alcohol. From Figure VIII-12, it is evident that the LiF fibers are continuous and straight. When the Earth-grown NaCl-LiF eutectic ingot was immersed in the same solution, LiF fibers could also be extracted from the NaCl matrix. However, the extracted fibers were short and discontinuous as indicated in Figure VIII-13, which agreed with predictions (sample from group A). In view of this analysis, it is tentatively concluded that continuous LiF fibers have been produced in space.



(a)



(b)

Figure VIII-6. Photomicrographs of a longitudinal section of the NaCl-LiF eutectic showing (a) continuous LiF fibers (56X) and (b) discontinuous LiF fibers (210X).

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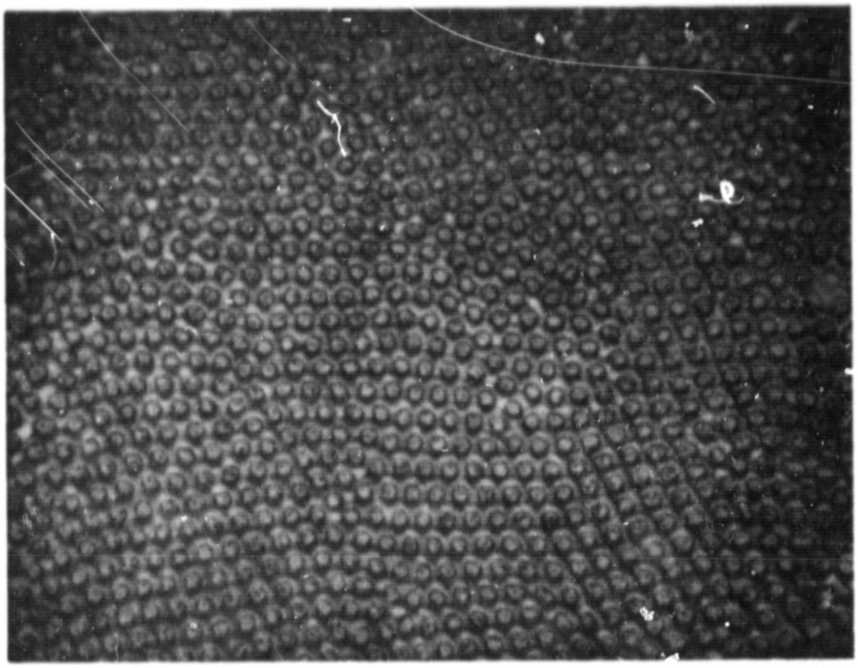


Figure VIII-7. Photomicrograph of the transverse section showing shapes of LiF fibers (410X).

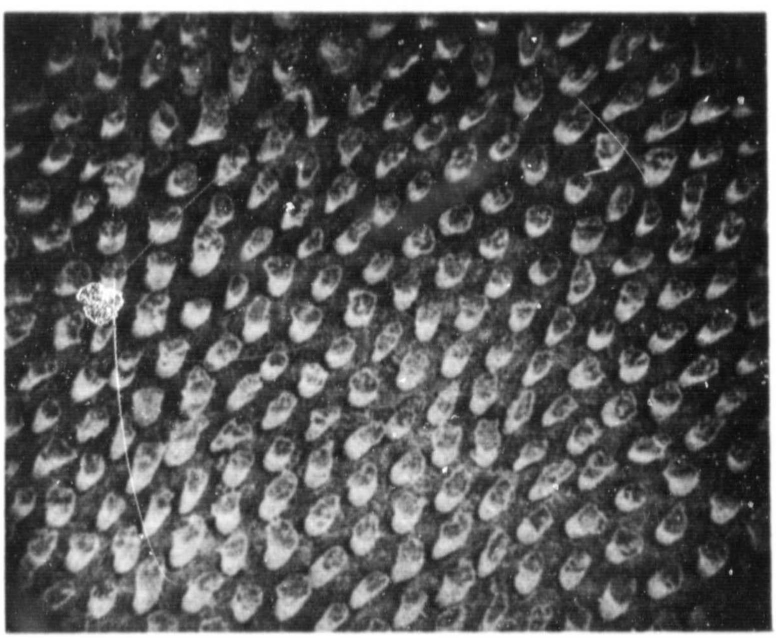


Figure VIII-8. Scanning electron photomicrograph of the Earth-grown LiF fibers (940X).

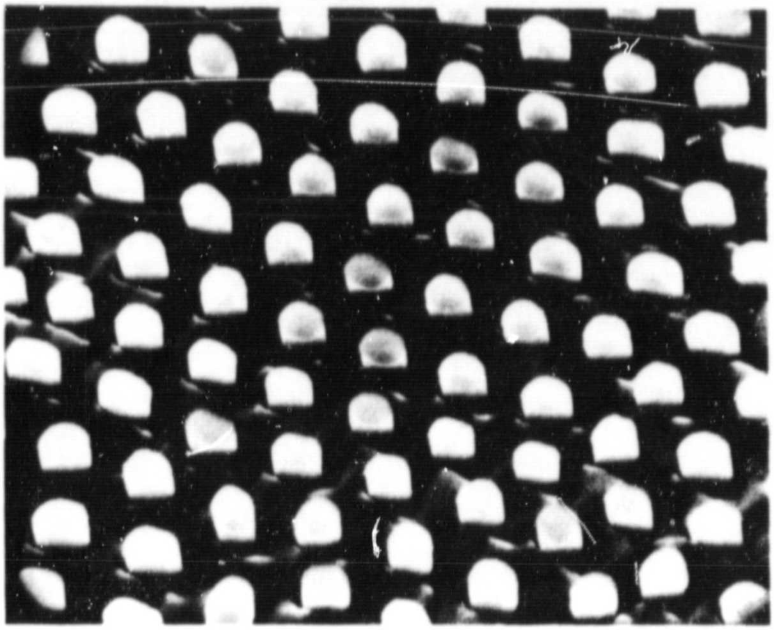


Figure VIII-9. Scanning electron photomicrograph of the ASTP-grown LiF fibers (1900X).

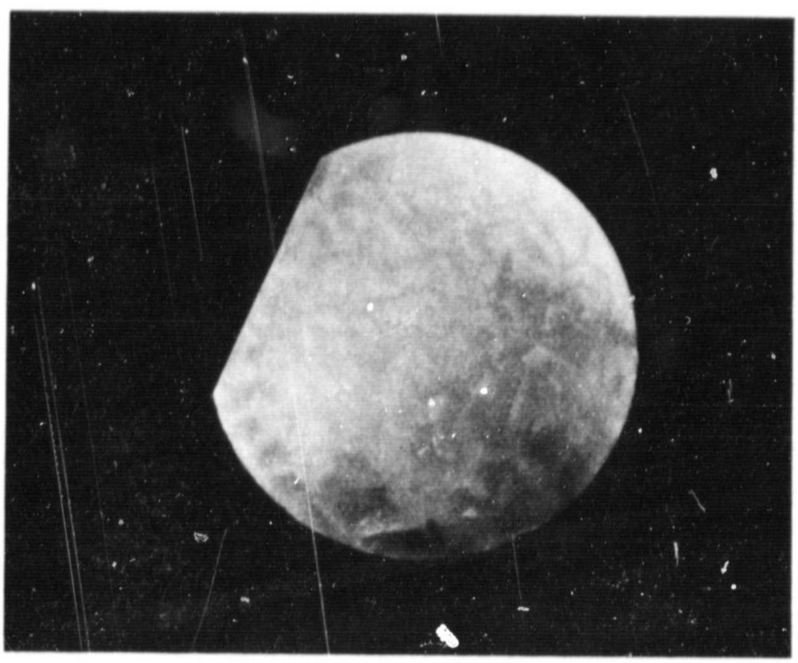
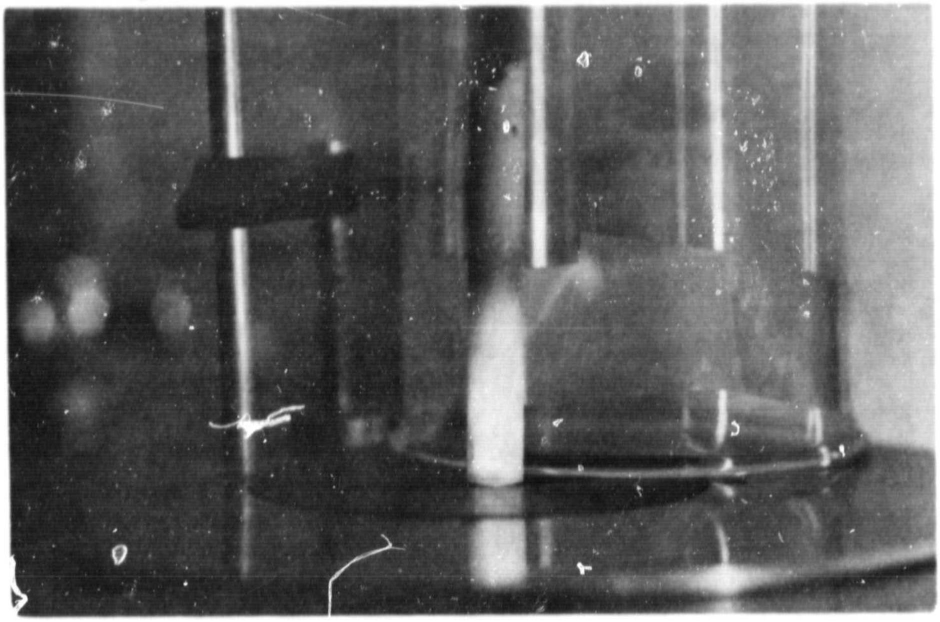
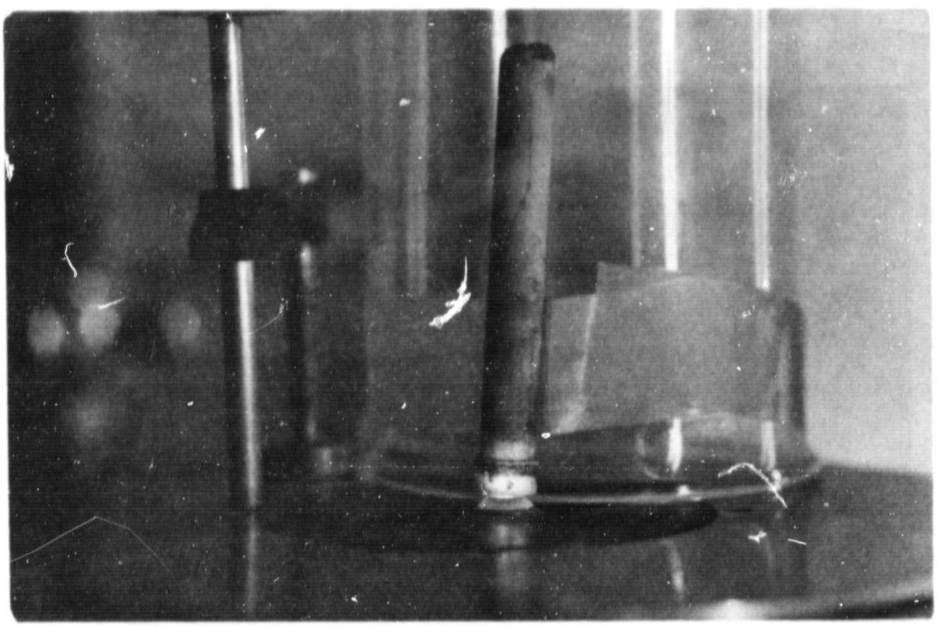


Figure VIII-10. Macrophotograph of the transverse section of the NaCl-LiF eutectic showing grains and subgrains, sample No. 10 (7.2X).



(a)



(b)

Figure VIII-11. Macrophotographs of (a) ASTP-grown and (b) Earth-grown NaCl-LiF eutectic.

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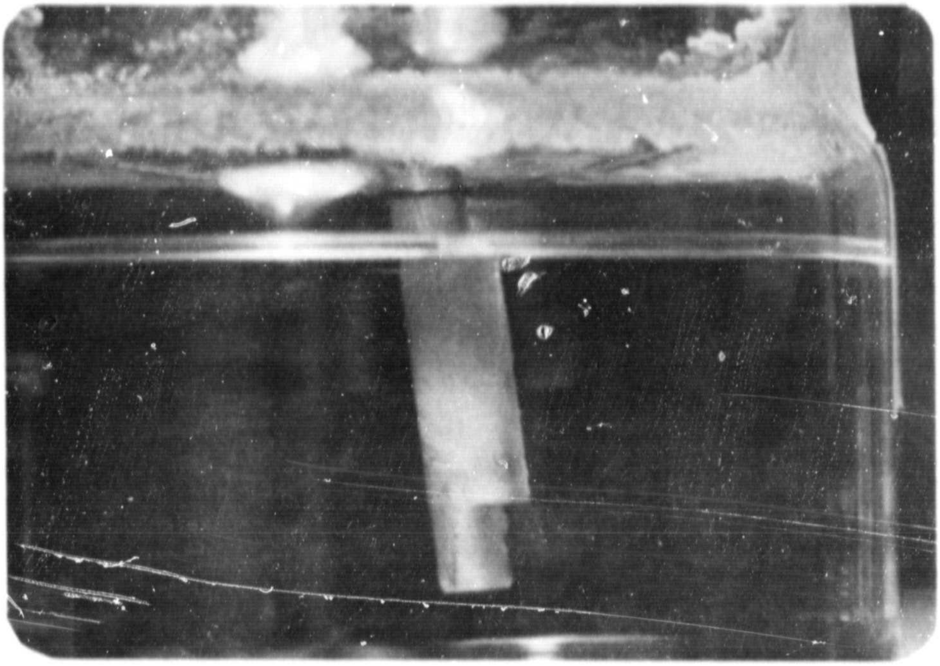


Figure VIII-12. ASTP-grown ingot with continuous LiF fibers (2X).

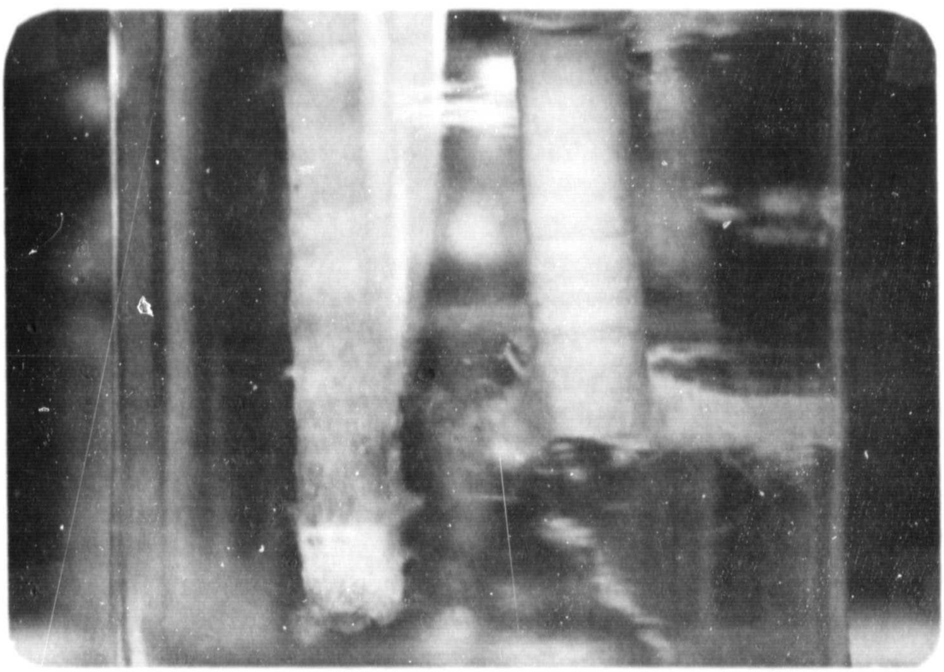


Figure VIII-13. Discontinuous and randomly oriented fibers of the Earth-grown ingot end (2X).

Interfiber Spacing

The measured interfiber distances in ASTP-grown, phototype furnace-grown, and Earth-grown ingots at growth rates of approximately 4.5 μm/s are as follows:

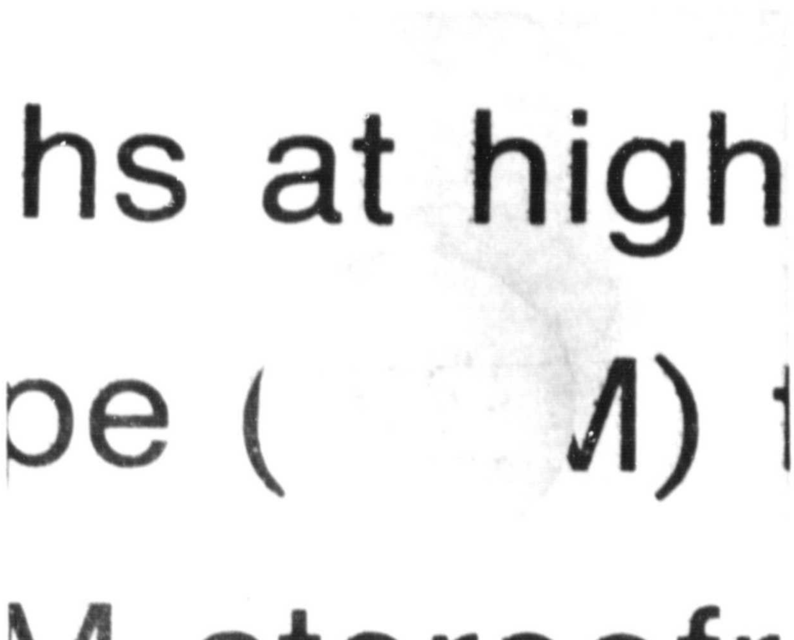
Interfiber Distance (μm)		
ASTP-Grown	Prototype Furnace-Grown	Earth-Grown
6.68 (131-07)	6.92 (131-01)	4.5 (UCLA)
5.41 (131-08)	7.35 (131-02)	
6.41 (131-09)	6.70 (131-03)	

The average diameter of the fiber of prototype furnace-grown and ASTP-grown ingots is 3.6 μm. These measurements indicate that there is no significant difference in interfiber spacing between the ASTP-grown and the prototype furnace-grown ingots. The interfiber spacing of the Earth-grown eutectic is smaller than that of the ASTP-grown ingot because the Earth-grown eutectic had a faster freezing rate during growth.

Image Transmission

Image transmission properties similar to those of fiber optic materials have been obtained by others with an NaCl-LiF eutectic [VIII-9]. Since the eutectic in that case had discontinuous LiF fibers, far better results will be obtained if the same eutectic can be produced in space with continuous fibers. Two cylindrical sections 2 mm long were cut from ASTP-grown and Earth-grown (group A) samples. Image transmission experiments were made on these two samples. The results are given in Figure VIII-14. Figure VIII-14(a) shows that an image was transmitted from a source (a sheet of paper containing the word "solar") through the length of the sample (2 mm) to its surface. The transmitted image has the same dimensions as the source, indicating that the LiF fibers are perpendicular to the plane of the paper. However, the transmitted image is not as clear as the original image of the source, indicating that there is a loss through transmission. Some loss is common to all fiber optics materials [VIII-10]. A transmitted image was also observed from a sample grown on Earth, as seen in Figure VIII-14(b). However, the image is unclear and the dimensions of the letters "S" and "E" are larger than those of the source, indicating that the fibers are diverged away from the source.

(a)



(b)

Figure VIII-14. Image transmission macrophotograph of (a) ASTP-grown and (b) Earth-grown NaCl-LiF eutectics, 2 mm sample thickness (5.3X).

Optical Property

Image transmission properties similar to those of fiber optic materials were obtained with an NaCl-NaF eutectic [VIII-9]. This eutectic was found to be a far-field infrared transmitting medium for wavelengths longer than the interfiber distance. Since the NaCl-NaF eutectic used for optical measurement has discontinuous NaF fibers embedded in an NaCl matrix, better results should be observed if continuous fibers are obtained. This was indeed the case as demonstrated in the Skylab experiment [VIII-1]. Similar experiments comparing transmission properties of NaCl-LiF eutectic grown in space and on Earth were performed for ASTP. The far-field infrared transmission of transverse sections of three NaCl-LiF eutectic samples is compared in Figure VIII-15. The ASTP space-grown sample is shown to have the highest transmittance over nearly the entire wavelength range, indicating that it has the highest percentage of fibers parallel to the growth direction. The transmittance result also shows that the sample grown in the Westinghouse prototype furnace has a lower transmittance than the eutectic sample grown at UCLA over part of the wavelength range.

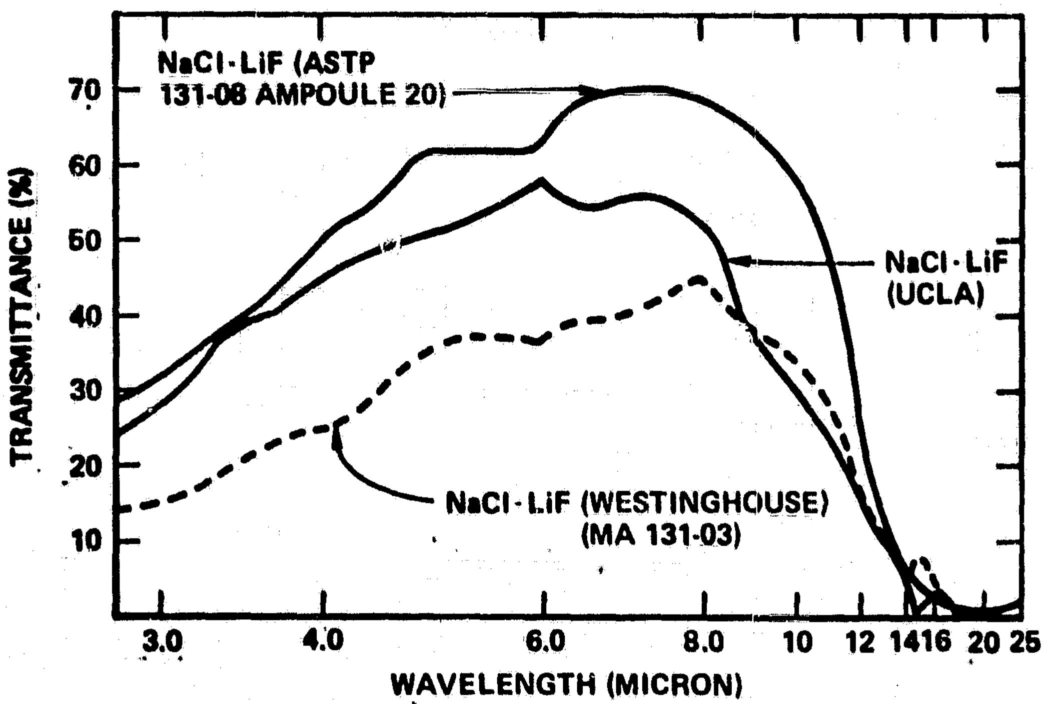


Figure VIII-15. Far-field infrared transmission curves of transverse sections of NaCl-LiF eutectics grown on earth and in space, 0.0211 in. sample thickness.

The effect of thickness on the transmission curve is shown in Figures VIII-16 and VIII-17 for the Westinghouse prototype furnace and the ASTP space-grown samples, respectively. In both cases, the thinner the sample is, the higher the transmittance is for a fixed wavelength. This observation is in agreement with absorption laws, as expected.

Figures VIII-18(a) and VIII-18(b) show plots of the transmittance versus wavelength of a longitudinal section of a UCLA-grown sample (group A) using polarized light with an electric field parallel and perpendicular to the fiber axes, respectively. Figures VIII-19(a) and VIII-19(b) show the same curves for the ASTP-grown sample (131-07). In both cases, the transmittance is lower over a range of wavelength measured. It is difficult to explain this peculiar phenomenon at the present time.

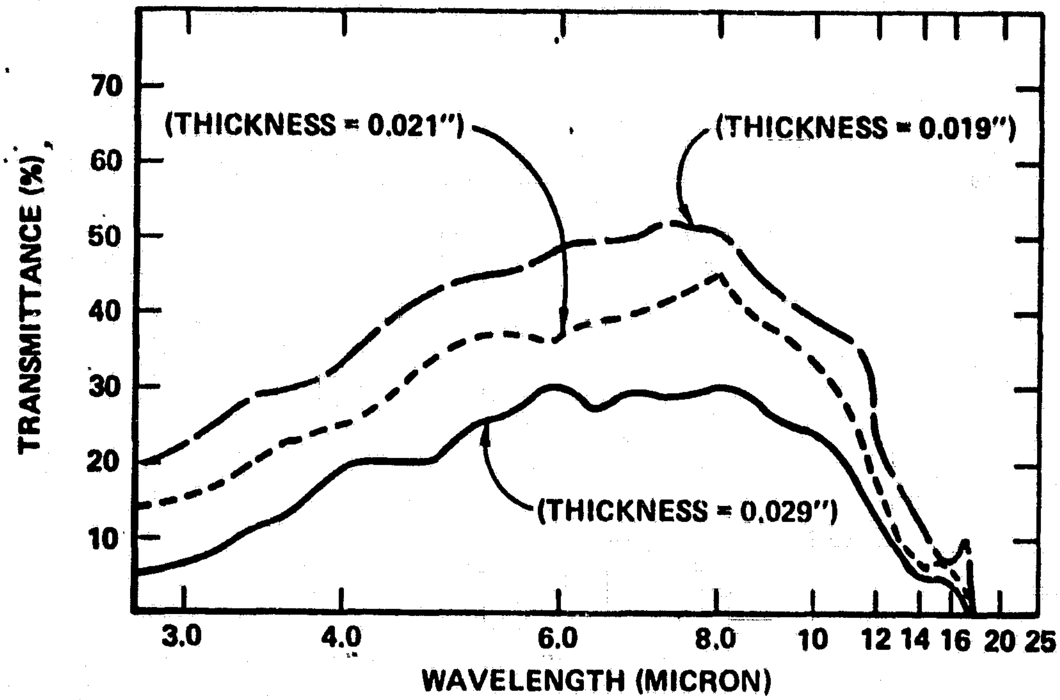


Figure VIII-16. Far-field infrared transmittance curves of transverse sections of NaCl-LiF eutectics with different thickness, sample No. 131-03.

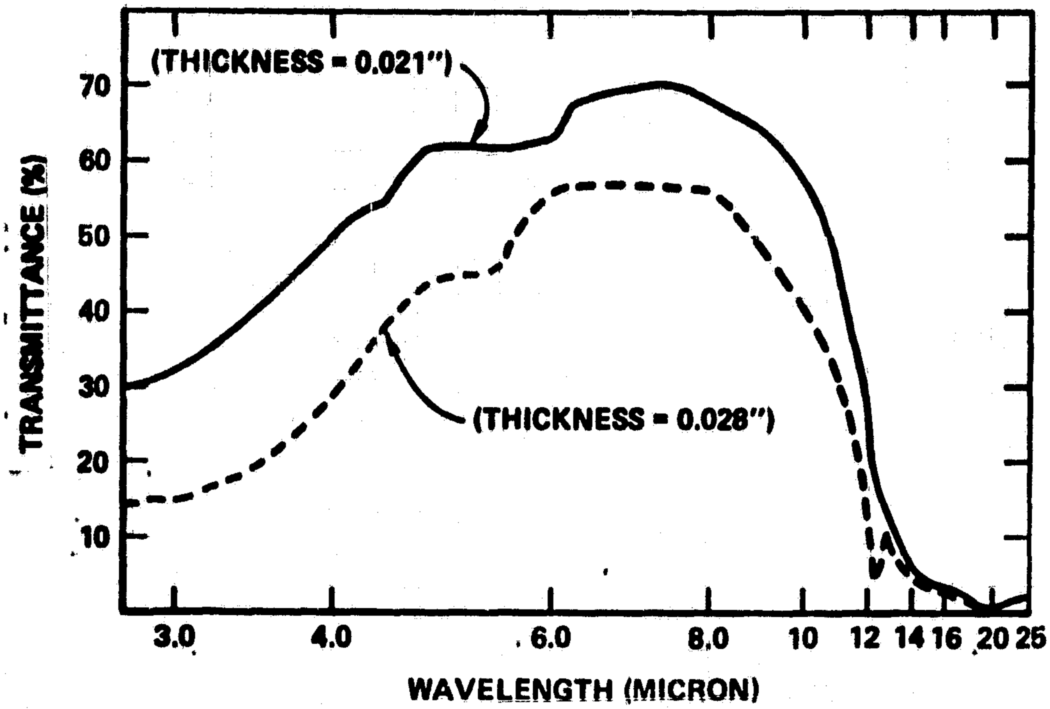


Figure VIII-17. Far-field infrared transmittance curves of transverse sections of NaCl-LiF eutectics with different thickness, sample No. 131-08.

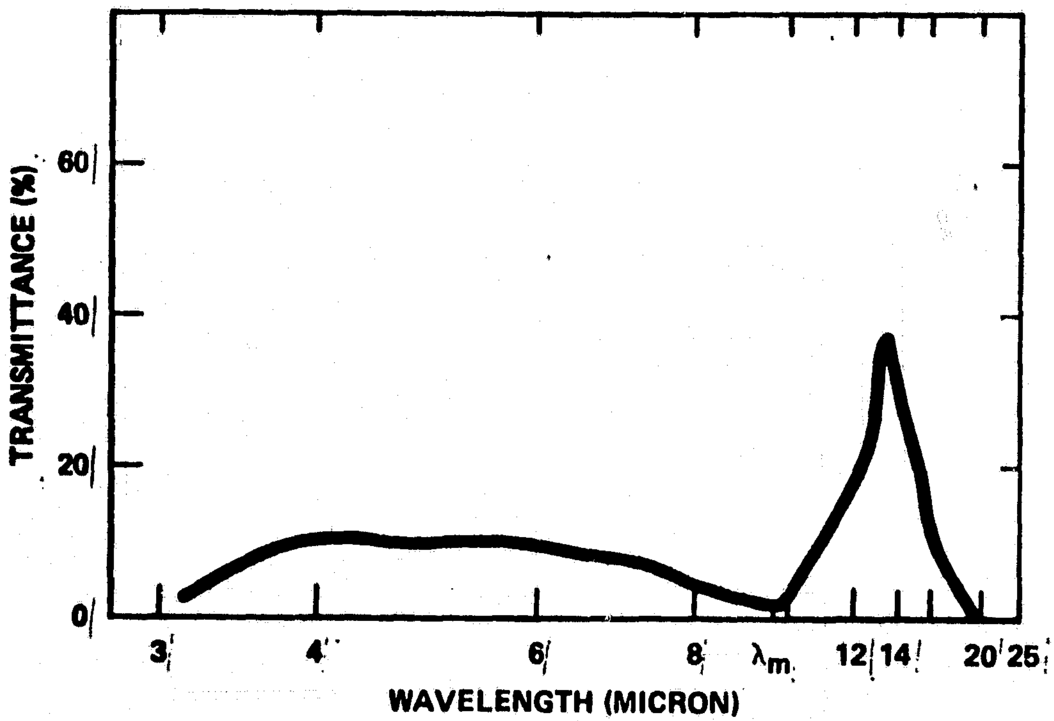


Figure VIII-18(a). Far infrared transmittance curve of a longitudinal section of NaCl-LiF eutectic grown on Earth, electric field parallel to fiber axis.

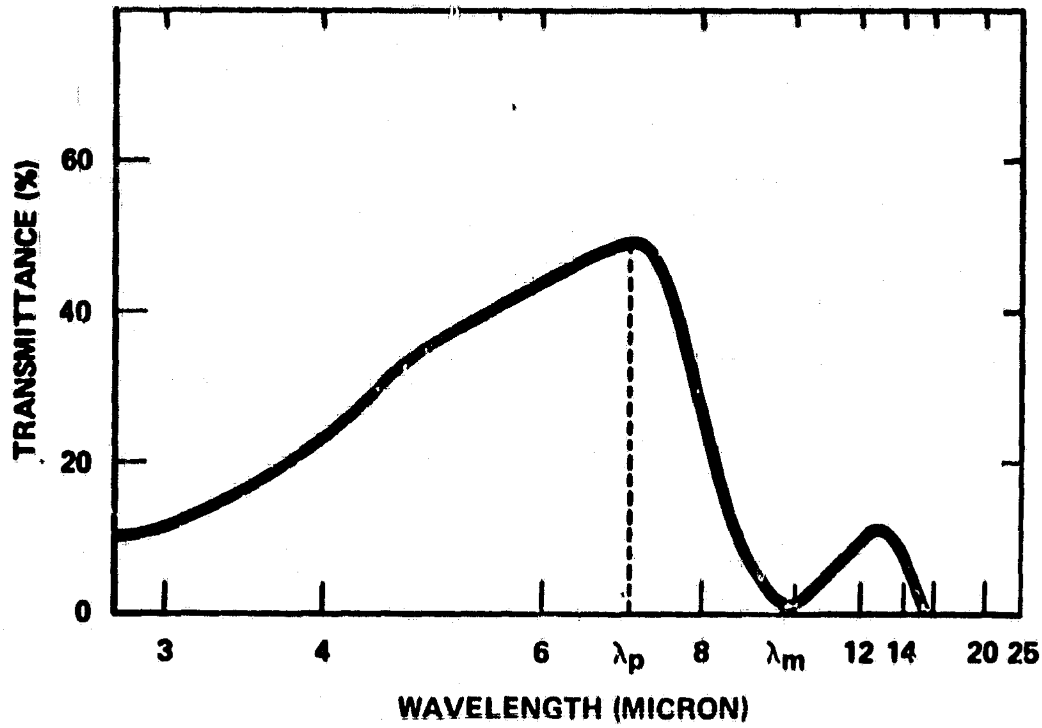


Figure VIII-18(b). Far infrared transmittance curve of a longitudinal section of NaCl-LiF eutectic grown on Earth, electric field perpendicular to fiber axis.

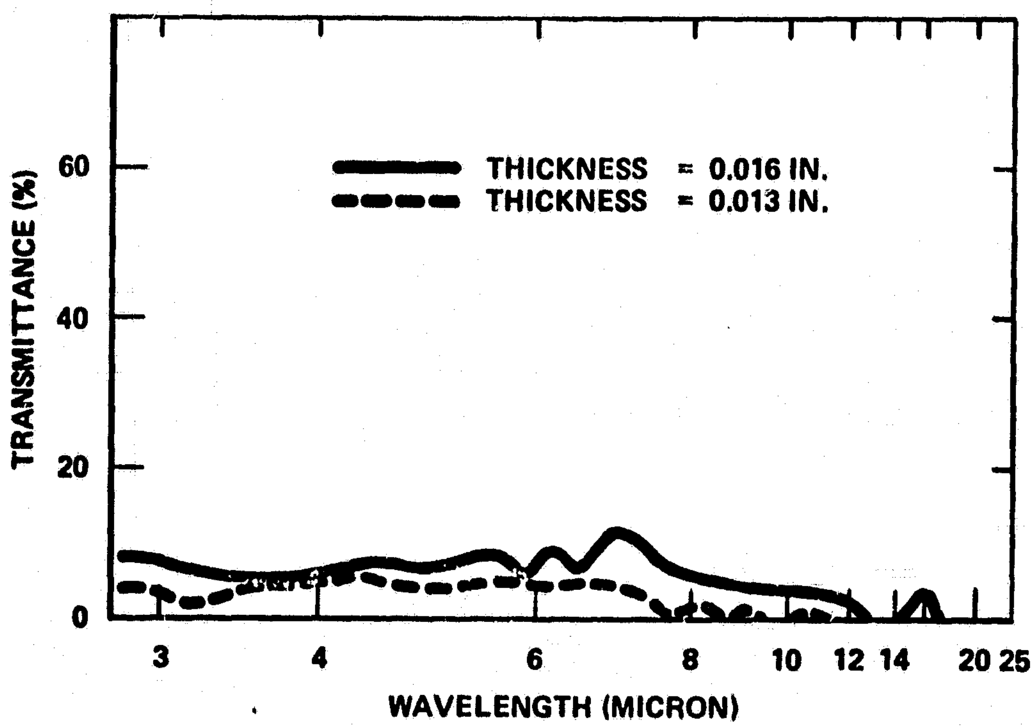


Figure VIII-19(a). Far infrared transmittance curves of a longitudinal section of NaCl-LiF eutectic (131-07) of varying thicknesses, electric field parallel to fiber axis.

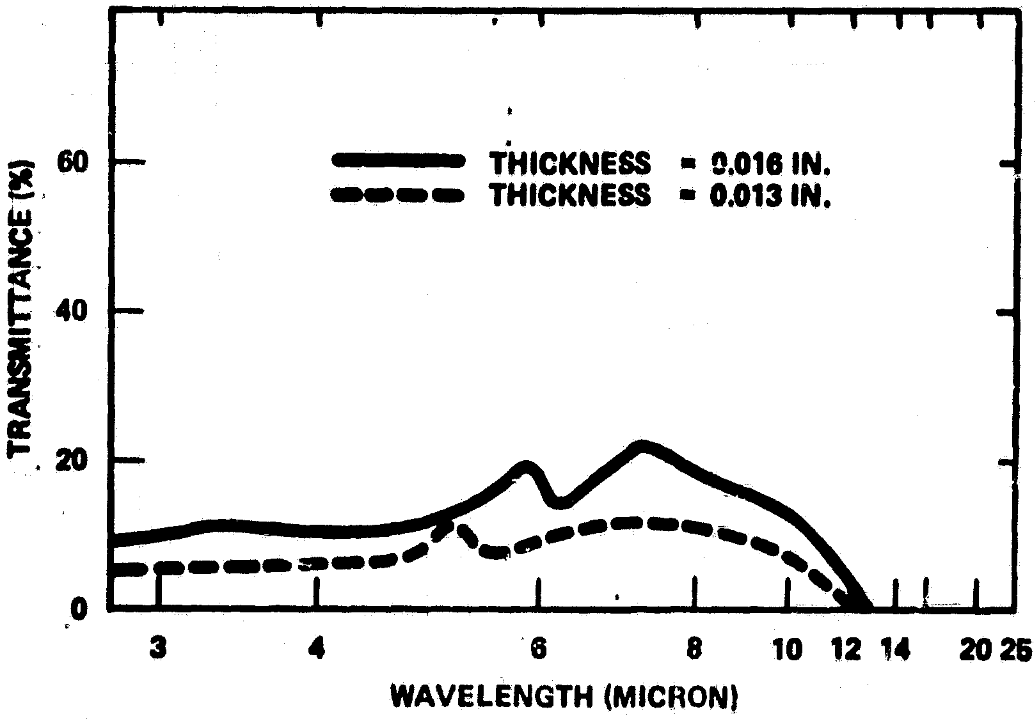


Figure VIII-19(b). Far infrared transmittance curves of a longitudinal section of NaCl-LiF eutectic (131-07) of varying thicknesses, electric field perpendicular to fiber axis.

DISCUSSION

LiF Fibers

In a zero-g environment, there is no gravity-driven convection current in the liquid during solidification and there is no difficulty in mixing two liquid phases of different densities. Furthermore, vibration levels in the spacecraft will be far lower than those on Earth. Consequently, a homogeneous eutectic mixture consisting of continuous fibers can be produced in a space environment, and microstructure sensitive to convection currents and vibration can develop undisturbed. Accordingly, the success in producing continuous LiF fibers as evidenced in Figure VIII-12 is primarily due to the absence of convection current in the melt during resolidification in space.

Optical Transmittance

Pure sodium chloride and sodium fluoride crystals, as is known from previous work [VIII-1], have approximately 95 percent transmittance per centimeter of thickness, up to 15 and 9 μm respectively, in the far infrared wavelength region. Beyond these ranges of wavelengths, these crystals have zero transmittance because of the optical modes of lattice vibration of ionic crystals. If a thin piece of NaCl-LiF eutectics is cut along its fiber axis (longitudinal section), as illustrated in Figure VIII-20, and is measured from 2 to 20 μm wavelength with an infrared spectrometer, typical transmittance versus wavelength curves with a polarized beam parallel and perpendicular to the fiber axes are as given in Figures VIII-18(a) and VIII-18(b), respectively. For the electric field parallel to the fiber axes, the transmittance is near zero over a range of wavelength approximately less than λ_m (10 μm), a maximum wavelength. When the electric field is perpendicular to the fiber axes, the percentage of transmittance increases over a range of wavelengths less than λ_m and reaches a maximum value at a wavelength of 7.2 μm, which is designated

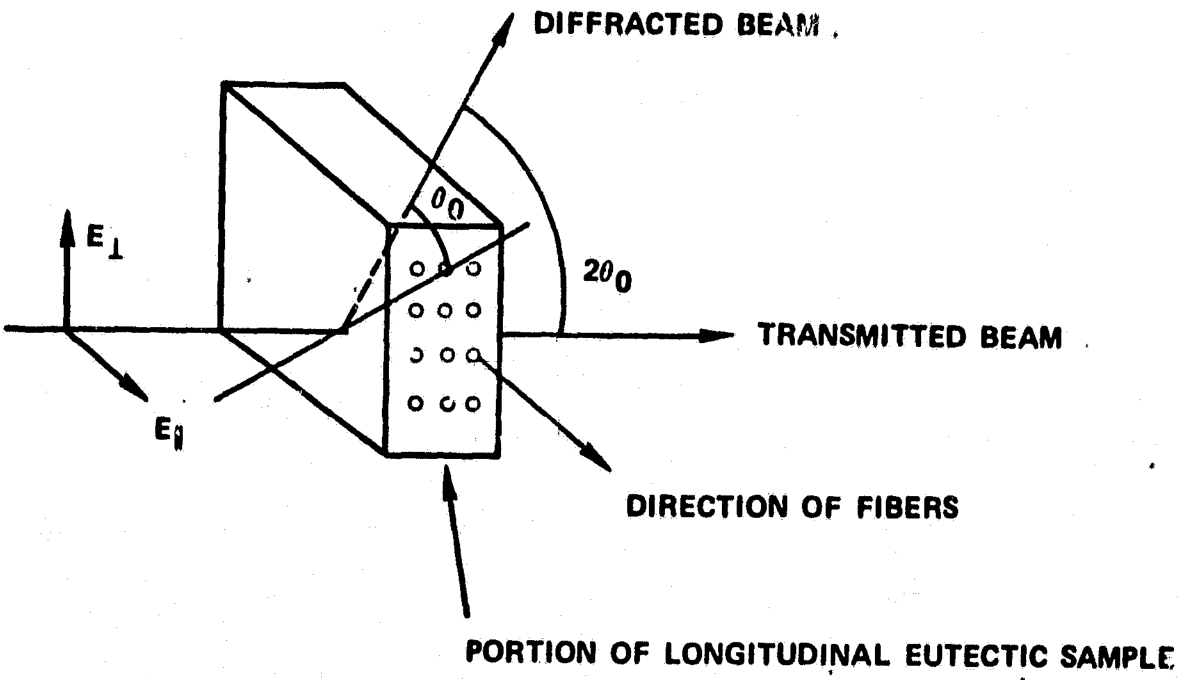


Figure VIII-20. Diagram illustrating quantities discussed in analyzing scattering measurements for transmission through a longitudinal sample section.

λ_p, a peak wavelength, as indicated in Figure VIII-18(b). Since a thin NaCl-LiF eutectic sheet has these characteristics it can be used as a polarizer and a modulator. It can switch on and off the light at a specified wavelength when the electric field is rotating. An explanation has been advanced to account for this phenomenon in terms of two-dimensional Bragg scattering and the polarization effect of Rayleigh scattering [VIII-11].

CONCLUSIONS

The following conclusions can be drawn from this report:

1. Continuous LiF fibers regularly arranged in a portion of the NaCl matrix which has been resolidified unidirectionally in a space environment have been produced.

2. Larger transmittance over a wider wavelength and better image transmission were obtained for transverse sections of the ASTP-grown ingots.

RECOMMENDATIONS

The following recommendations are made:

1. To design decanting experiments to study the interface morphology in the scientific laboratory to be used in the early flights of the Space Shuttle.

2. To etch out LiF fibers from the NaCl matrix for the study of multiple channel electron intensifiers.

3. To make an extensive study on the composition of LiF and NaCl phases using an electron microprobe.

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