CR-161674

(NASA-CR-161674) SPAR 6 EXPERIMENT REPORT CONTAINERLESS PROCESSING OF GLASS EXPERIMENT 74-42 Progress Report (Rockwell International Corp., Downey, Calif.) 54 p HC A04/MF A01 CSCL 22A G3/12
N81-20102
Unclas
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SSD 80-0045

SPAR VI EXPERIMENT REPORT CONTAINERLESS PROCESSING OF GLASS EXPERIMENT 74-42

March 1, 1980

Contract NAS8-32023

By

Ralph A. Happe

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ILLUSTRATIONS

SUMMARY

The first containerless glass melting experiment, NASA experiment 74-42, was flown on the SPAR VI sounding rocket on October 17, 1979.

This report describes pertinent portions of the ground-based research, including experiments leading to selection of the flight sample composition, a silica-modified gallia-calcia glass of the composition, in mol percent, 39.3 Ga_2O_3 : 35.7 CaO : 25.0 SiO_2. Included are details of the preparation of an approximately one-fourth-inch-diameter flight sample.

During the flight experiment, a single sample of the silica-modified gallia-calcia glass was containerless-melted and cooled in a single-axis acoustic positioning apparatus built under contract to NASA by Intersonics, Inc. Since this was the first containerless glass flight, the principal objective of the experiment was to determine the functioning of the flight experimental hardware under actual flight conditions. The flight hardware included a silicon carbide element furnace equipped with a single-axis positioning device designed to prevent contact of the molten sample with other than the gaseous (approximately one-ground-level air) atmosphere during the 4 minutes of low-gravity flight. At the completion of the melting and soaking portion of the processing cycle, which was performed at a nominal temperature of 1575 C, a massive copper cooling shroud was introduced into the hot zone of the furnace to radiation-cool the sample. An injection cage fashioned from platinum and 30-percent rhodium alloy was used to inject the sample into the sonic well, and a single motion-picture camera was provided to observe the flight sample during the entire processing cycle. The hardware package included necessary electronics, timers, temperature control equipment, and mechanical devices, the last for operation of the injection mechanism, furnace wall gates, and cooling shroud.

The sample was completely melted during the flight and was cooled to a clear glass. Injection into the furnace hot zone was accomplished. After it touched the cage four times in the first 9 seconds, the sample remained in suspension 27 seconds, at which time it drifted to the injection cage and attached itself to one of the platinum alloy wires making up the cage. It remained attached and centered on the cage wire during the remainder of the processing cycle.

During the post-flight evaluation, the sample was found to be free of unmelted, crystalline, material. It contained three small bubbles near the platinum wire. The shape of the sample was spherical except for projections caused by wetting the wire by the glass at either pole where the platinum wire emerged from the sample. Unexpectedly, the surface of the sample was found to contain numerous small crystal rosettes not large enough to be seen by the

unaided eye. Analysis of the crystal rosettes with a scanning electron microscope showed them to be of approximately the same composition as the glass. Platinum (with some rhodium present) was found to be the probable cause of crystal nucleation. The principal constituent of the rosettes was found by indirect methods to be $Ca_2Ga_2SiO_7$. The most probable causes of the platinum contamination of the surface are thought to be either (1) mechanical transfer from the loose-fitting injection cage during lift-off of the rocket or (2) surface diffusion from the platinum-rhodium wire which was in contact with the glass sample during most of the flight experiment cycle.

It is felt that, if during the next flight experiment the platinum cage grips the sample firmly to eliminate or substantially reduce rattling and the acoustic positioning device prevents physical contact with the injection cage during the entire processing cycle (eliminating the possibility of surface diffusion), the chances for platinum contamination of the surface and consequent crystal nucleation will have been substantially reduced.

INTRODUCTION

If the promise of containerless melting and cooling, made possible by space processing, is realized fully in the years that lie ahead, an important new area of optical glasses will become a reality. In part, this new area may be visualized by referring to the schematic of Figure 1. The ordinate is the index of refraction, and the abscissa, the Abbe number (ν), an inverse measure of dispersion. The higher Abbe numbers, to the left, of the diagram indicate a low dispersion (i.e., a flatter slope of the index versus wavelength curve). The lower Abbe numbers, to the right, have high dispersion (steep index versus wavelength curve). A century ago flint glasses were developed. This permitted construction of the first achromatic, or color-corrected, multi-element lenses. Responding to the demands for better quality lenses, the optical glass industry developed more glasses with properties between those of the crown and flint glasses. More recently glasses have been developed to fill out the vertically hatched commercial glass area. The trend has been to push the area up and to the left with glasses of complex compositions.

If glasses beyond the reach of current terrestrial technology could be prepared from the more reluctant glass forming oxides, the area of useful

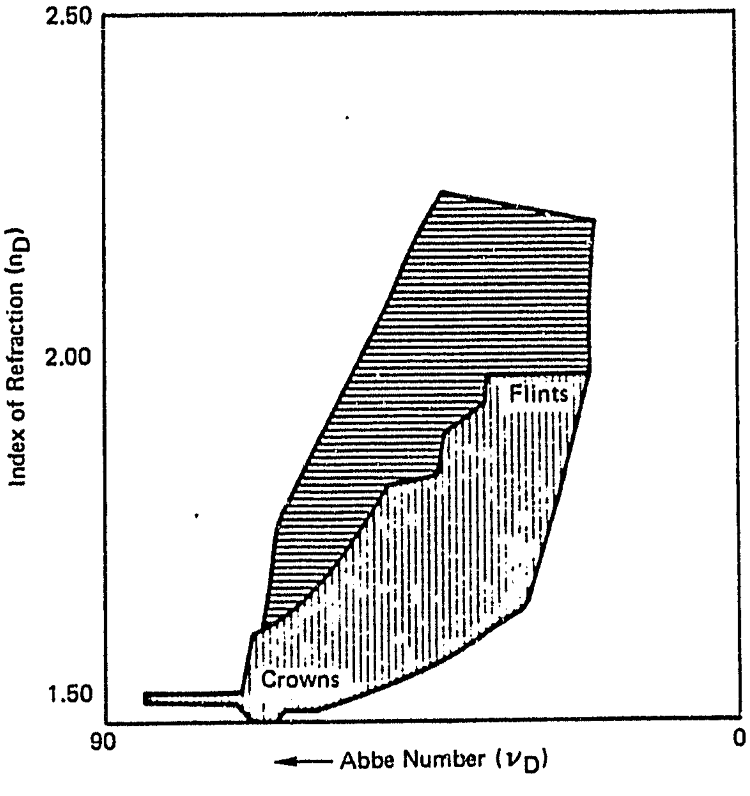

Figure 1. n-ν Diagram for Optical Glasses

properties could be expanded significantly. The expansion would occur by the addition of space-prepared glasses (horizontally hatched area of Figure 1) to the terrestrial base.

THE RATIONALE FOR SPACE PROCESSING

For most of the past decade, the Principal Investigator has been investigating possibilities for producing new optical glasses by containerless melting and cooling utilizing the near-zero-gravity environment available in earth orbit. The paragraphs that follow cover the technical thinking behind the concept of containerless processing in space, a summary of experience to date, and reasons for the interest in space processing of optical glasses.

When a molten oxide is cooled slowly enough to approach equilibrium conditions, it crystallizes near its crystalline melting point. In the case of the conventional glasses, usually based on oxides such as SiO_2, P_2O_5, B_2O_3, or GeO_2, the viscosity of the molten glass is very high. With this high viscosity, the molecular mobility is very low. Thus, when such substances are cooled from the molten state, it is difficult for the molecules to rearrange themselves into the orderly state of the crystalline lattice. Because of this sluggishness, the movement of the molecules into the crystalline lattice positions is incomplete on cooling with normal cooling rates. In these cases, the semi-random molecular arrangement of the liquid state is essentially preserved on cooling, and the substance remains amorphous, the resulting product being called a glass.

The crystallization phenomenon may be considered to occur in two stages: (1) nucleation and (2) crystal growth. In conventional glasses the sluggishness effectively inhibits both of these processes, especially the latter. Therefore, even if the substance manages to nucleate on cooling from the melt, the crystal growth rate is so slow that the nuclei remain, for practical purposes, undetectable in the glass. There are, however, only a few oxides that have sufficiently high viscosities to permit glass formation under normal circumstances.

For the past two decades, glasses have been made terrestrially in the laboratory from some of the less viscous oxides. Invariably, the technique used for preparing them involves extremely high cooling rates from the liquid state. The familiar splat-cooling technique (Reference 1) is a case in point. While such techniques yield valuable research information about the nature of the glasses so prepared, their application for commercial purposes is extremely limited. By the nature of the technique, only very thin films can be prepared. With this technique the liquid, as a very thin layer, is cooled in contact with a chill plate, usually of copper. While the copper provides numerous nucleation sites, the very rapid cooling effectively suppresses crystal growth.

Containerless melting in space offers the first practical opportunity to prepare glasses in massive form from the large number of oxides whose liquid viscosity* is not high. If nucleation can be prevented on cooling, then crystal growth obviously cannot occur, and a glass should result.

It is generally recognized that there are two kinds of nucleation: (1) heterogeneous and (2) homogeneous. Heterogeneous nucleation results from contact of the cooling liquid with crystalline material. Such a material may be entirely different in chemical composition from the melt. Common container wall materials are cases in point. Of course, it can also be of the same or similar composition, for example, unmelted portions of the bath or cool seed crystals of similar composition deliberately introduced into the cooling melt. In practice it is very difficult, or virtually impossible, to eliminate heterogeneous nucleation sites with conventional, terrestrial practice. Normally, a crystalline container must be used both for melting and for cooling. Further, the impingement of cool dust particles on the cooling melt may be enough to cause heterogeneous nucleation, and if the viscosity remains low enough in the supercooled liquid, crystal growth rates will be high and the glassy state will not be obtained.**

Homogeneous nucleation is another matter. Theoretical studies (Reference 2) have shown that homogeneous nucleation rates for oxide glasses are much slower than for heterogeneous nucleation. Experimentally, it is difficult to determine whether nucleation is truly homogeneous. There are those who believe that it may never truly have been observed in an oxide glass. Since only a few molecules of a heterogeneous nucleator need be present, the detection of such a small amount is a formidable technical problem. Thus, the assumption, *a priori*, that nucleation which occurs, for example, throughout the mass of a cooling substance is homogeneous may be erroneous. One can always argue that an undetectably small amount of a crystalline substance was present at the nucleation sites. At any rate, if heterogeneous nucleation can be effectively prevented, it is probable that homogeneous nucleation, if it can occur, will not occur unless the cooling rate is quite slow.

Over the past several years, the writer has successfully prepared numerous approximately 6-mm-diameter (about 3/4 gram) glass boules of roughly spherical shape from several oxide compositions that have low viscosity in the molten state. That work is covered in detail in References 3 and 4. Among the compositions prepared are the gallia-calcia eutectic at approximately 19 weight percent calcia, an alumina-calcia composition with 30 weight percent calcia, and a ternary, 40 weight percent lanthana--40 weight percent alumina--20 weight percent calcia composition. The alumina-calcia composition is well outside the reported glass-forming region based on 20 mg melts (Reference 5). The gallia-calcia composition had been reported to be a glass former in the laboratory, but in sizes less than 40 mg (Reference 6). Furthermore, water

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*It is recognized that the slope of the viscosity versus temperature curve below the crystalline melting point (i.e., in the supercooled region) is very important to the glass formation process. However no such data exist for the oxides proposed here. It is probable that the general tendencies of viscosity change in the supercooled region can be inferred from future terrestrial and space studies.

**The presence of insoluble crystalline material in the melt could also cause heterogeneous nucleation. Fortunately oxides are very good solvents. It therefore follows that with enough melting time this problem should be held to a minimum.

quenching was required to achieve the glassy condition. Thus the preparation of crack-free boules with 50 times the mass of those of the earlier work represents a significant technical achievement. A glass of the ternary composition, to the writer's knowledge, has never been reported in the literature.

The method for preparing the 6-mm boules is described in detail in Reference 3. Briefly, the samples in contact with a silica (glass) sting are suspended in a vertical air column. The energy for melting comes from a CO_2 laser beam aimed at one side of the boule. The silica sting was found necessary to stabilize the motion of the melt and is a definite convenience for getting the process started. The oxide is transferred to the sting from a laser melted area of well-mixed powders of the desired composition. While the technique developed by the writer is excellent for demonstrating that new glasses can indeed be prepared with containerless melting and cooling techniques, it does suffer from several limitations, as follows:

1. Because of the relationship among viscosity, surface tension, and mass, 6 mm is very near the maximum sized boule that can be prepared in this fashion.

2. A small amount of silica sting material is continuously dissolving into the sample while the latter is being melted and held at superheat temperature. While the residual silica content of the boules averages well under one percent, an amount which may be considered too small to significantly affect conclusions regarding glass formation behavior, it apparently is not distributed uniformly, a possible explanation for the presence of marked striae in the glass boule.

3. It is very difficult to eliminate dust in the air from the wind tunnel. Thus the molten sample can be considered to be continually bombarded by dust particles while it is cooling. For this reason the technique may be unnecessarily restrictive compared with the more favorable conditions expected to accrue from space melting.

Space melting promises to eliminate or significantly reduce all three of the shortcomings inherent in the air suspension/laser melting equipment. Very large boules should ultimately be possible if enough power for melting can be made available. The silica sting will not be required with the acoustic positioning technique being developed by NASA. Space melting can be accomplished in a static atmosphere, significantly reducing the possibilities for dust-caused nucleation. It is entirely possible that some of the compositions that failed to form glass in our terrestrial experiments may prove to be glass formers under space melting and cooling conditions.

THE SOUNDING ROCKET PROGRAM

The Space Processing Applications Rocket (SPAR) program is considered a precursor to the Shuttle and later manned orbital programs. While conditions are not ideal for glass melting aboard a sounding rocket, largely because of the rather short melting time (less than five minutes) available, the program does afford a good opportunity to gain early experience with glass melting within the limitations and, more importantly, with space glass melting equipment development.

The gallia-calcia composition mentioned in the previous section of this report was originally chosen as a suitable composition for two sounding rocket experiments and for early Shuttle experiments using the NASA-provided Materials Experiment Assembly (MEA). The composition is a eutectic between the compounds $CaO:Ga_2O_3$ and $CaO:2Ga_2O_3$ and its composition, under equilibrium conditions, is approximately 19 wt. % CaO, balance Ga_2O_3 (approximately 56 mol % CaO). The phrase diagram for the binary gallia-calcia system is shown in Figure 2.

This particular composition was chosen for the following reasons:

1. It has the lowest melting temperature of any of the new optical glass compositions studied by the principal investigator prior to the initiation of the SPAR program.
2. It is a relatively good glass former in the 1/4-inch (approximately 0.6-cm) (about 0.8 gm.) size under terrestrial containerless melting conditions.
3. It potentially has optical properties of interest to the optics industry.
4. Prior to our terrestrial melting work, it had not been prepared in a size exceeding 50 mg (0.050 gm).

OBJECTIVES AND RATIONALE OF THE SPAR VI EXPERIMENT

The 74-42 SPAR VI flight experiment was the first SPAR experiment to deal with sonic positioning coupled with very high temperature (approximately 1575 C). The prime objective of the experiment, then, was to learn as much as possible about the functioning of the flight equipment, built under contract to NASA by Intersonics, Inc. of Northbrook, Illinois, during actual flight conditions.

A silica-modified gallia-calcia composition was flown on SPAR VI to gain as much information as possible about the functioning of the flight hardware. One such composition had been developed by the writer earlier in the program for other reasons. The reason for modifying the composition is to increase the glass formation tendency so that a glass would result when it is melted and cooled in contact with a container. The binary gallia-calcia composition has such low viscosity that it cannot be prepared in sizes of interest to this program as a glass when it is in contact with virtually any crystalline material. Thus, for example, if such a material is incompletely melted and allowed to cool, the residual crystalline material would initiate crystallization on cooling so that the entire mass would be returned from flight in the 100 percent crystalline condition. By contrast, the silica-modified composition can be incompletely melted, and the molten portion is

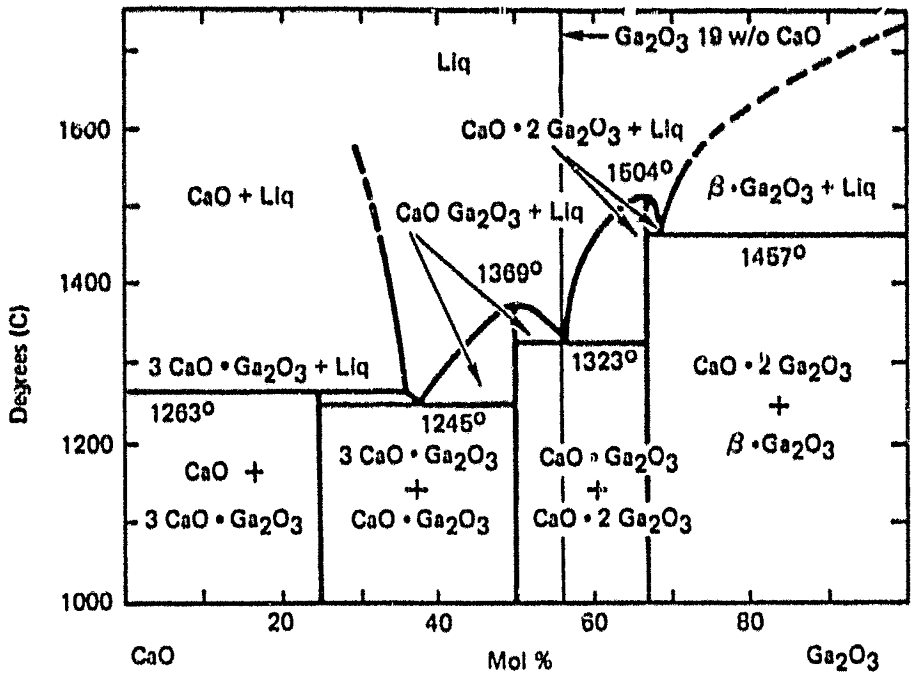


Figure 2. The CaO - Ca$_2$O$_3$ Phase Diagram (From Reference 2)

retained as a glass on cooling. Thus it is possible to detect from examination of the returned specimen whether complete melting had indeed occurred, the unmelted portion being clearly visible as nontransparent crystalline material within the body of the transparent glass. Had the binary gallia-calcia composition been flown in the first experiment and the sample returned in the crystalline condition, it would have been difficult, if not impossible, to determine if melting had been complete or, alternatively, if the crystallization had occurred during cooling because the melt had been contacted by a foreign crystalline material. Occurrence of such an event would have left us in the position of not knowing which of two entirely different mechanisms had caused failure and, consequently, not knowing how to correct the situation in future flights.

It was also decided to reduce the size of the first specimen from the earlier planned 1/2-inch diameter to 1/4 inch. This improved the chances for success by (1) increasing the probability of obtaining complete melting during the short time available during a sounding rocket flight and (2) reducing the mass that must be positioned by the single-axis sonic system.

Subsequent sections of this report describe in more detail the experiments to determine the flight composition and the preparation of the flight samples.

PREPARATION OF SAMPLES FOR FLIGHT EXPERIMENT

SELECTION OF FLIGHT SAMPLE COMPOSITION

During earlier work a ternary composition, 69 wt. % Ga_2O_3-19 wt. % CaO - 12 wt. % SiO_2 (41Ga_2O_3: 36 CaO : 23 SiO_2, in mol %) was used to study the effects of time at various furnace temperatures above the melting point on the progress of melting 6 gram melts. The silica addition stabilized the melt so that only the unmelted portion would be crystalline upon removal from the furnace and rapidly cooling to room temperature. The melted portion is retained as a clear glass.

It was decided to develop a silica-modified composition with similar characteristics for use on the SPAR VI flight, as discussed in the previous section. To obtain optical properties as close as possible to those of the binary gallia-calcia composition, it was desirable to utilize a silica content as low as possible.

Lacking a published ternary phase diagram for the Ga_2O_3 - CaO - SiO_2 system, it was assumed, as an expedient, that an eutectic valley existed in the ternary system which extended from the 56 wt. % gallia eutectic composition in the binary gallia-calcia system (Figure 2) to the 62 mol % silica eutectic in Figure 3.

A series of compositions made from high purity oxides was prepared with varying silica contents near the postulated eutectic valley. Nominal compositions are given in the following:

SiO_2, mol % (wt. %)	Ga_2O_3, mol % (wt. %)	CaO, mol % (wt. %)
5 (2.3)	57 (81.4)	38 (16.3)
10 (4.8)	53 (78.8)	37 (16.4)
15 (7.4)	49 (75.9)	36 (16.7)
20 (10.4)	45 (72.7)	35 (16.9)
22 (12)	40.6 (69)	37.4 (19)
25 (13.8)	39.3 (67.8)	35.7 (18.4)
33.3 (19.8)	33.3 (61.7)	33.3 (18.5)

After the pure oxide powders were weighed and blended by tumbling, 100-gram (approximately 40 cc) melts were prepared in platinum crucibles and held molten at least one hour. The crucible was then removed from the furnace, placed on a relatively massive aluminum slab, and allowed to cool. The appearance of the higher silica-content cooled melts can be seen in Figure 4. It was observed that in those compositions that crystallized completely, i.e. 5, 10, 15, and 20 mol % silica, the crystal growth rate slowed perceptibly with increasing silica content, an indication of the expected increase in viscosity.

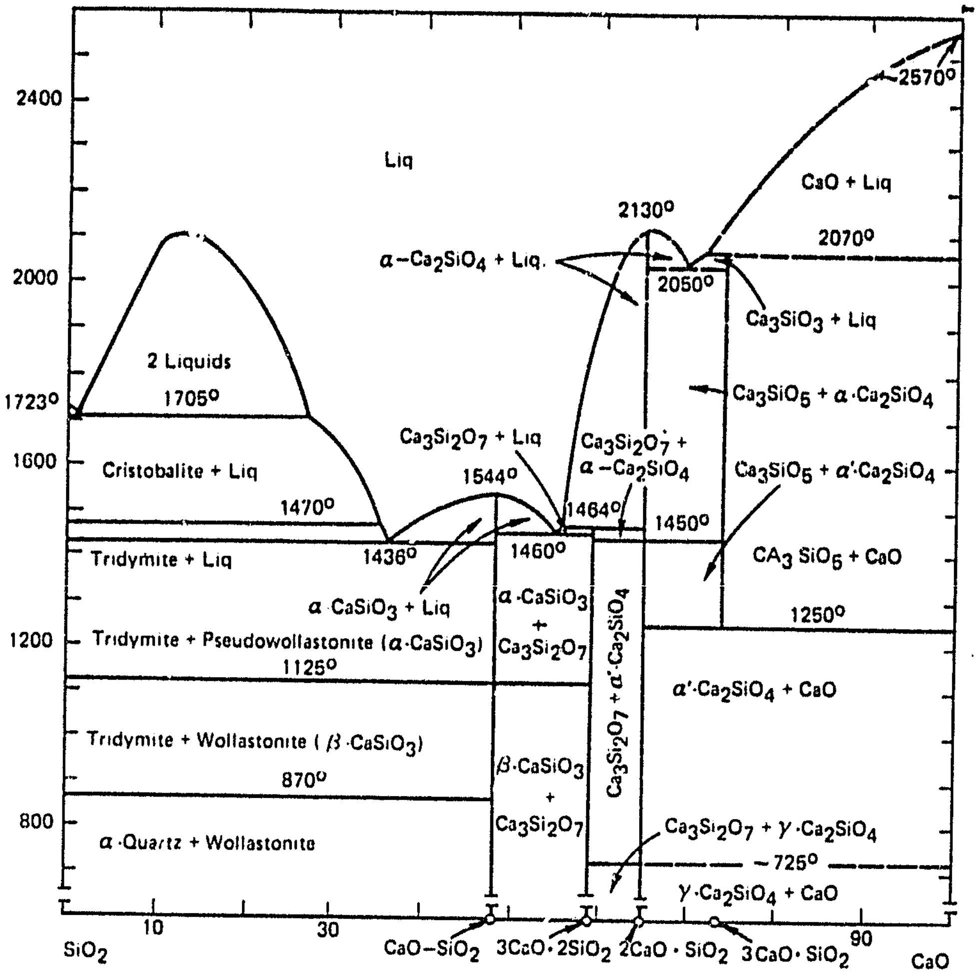
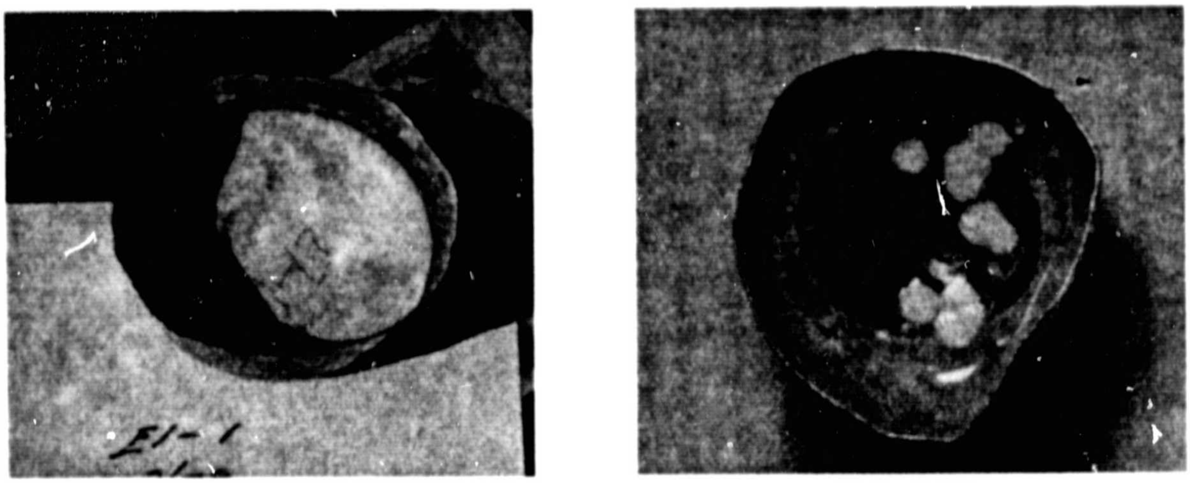
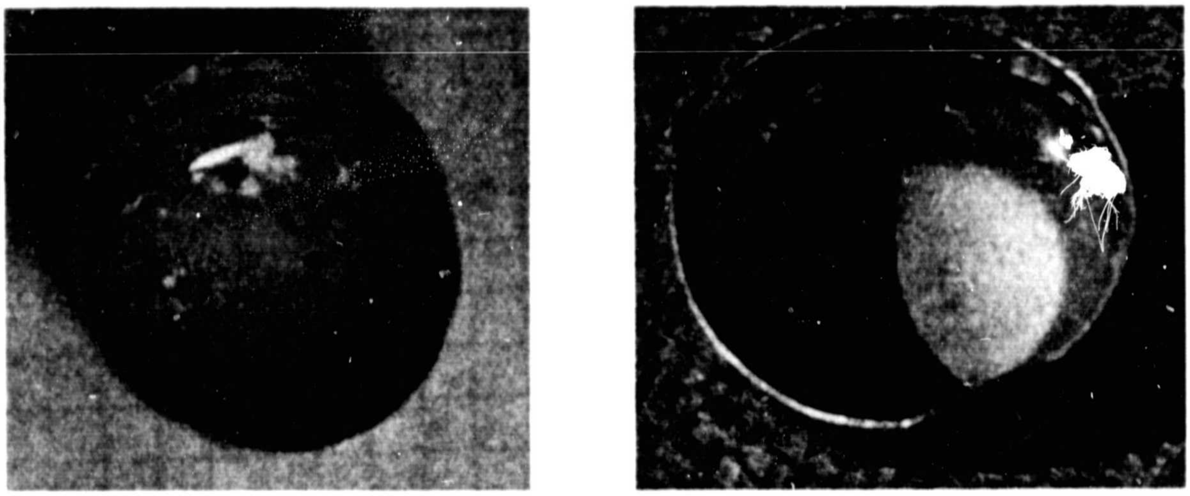


Figure 3. The CaO-SiO$_2$ Phase Diagram (From Reference 9)



20 Mol % SiO_2

22 Mol % SiO_2



25 Mol % SiO_2

33.3 Mol % SiO_2

Figure 4. Cooled Melts, 20 to 33 Mol % SiO_2

After photographing as shown in Figure 4, the melts were remelted and heated to 1550 C and held molten approximately one hour. Each crucible was again removed from the furnace and a "loaf"* casting was poured. The loaf castings are shown in Figure 5,** and the furnace and chill mold for preparing the castings are shown in Figure 6.

The behavior with the more rapid cooling conditions of the loaf castings was similar to that of the cooled melts, with the exception that the 20 mol % silica composition was glass plus crystal in the case of the loaf casting, while it appeared to be 100-percent crystal in the case of the cooled melt.

Based on these results, it was decided to select the 25 mol % silica content composition for preparing the flight samples. The 20 mol % silica content was the lowest in silica that showed any glass retention on cooling, and glass retention appeared to increase rapidly between 20 and 25 mol % silica.

PREPARATION OF FLIGHT SAMPLES

A loaf casting was prepared from the 39.3 Ga_2O_3: 35.7 CaO : 25.0 SiO_2 (mol %) composition with the procedure shown in the flow chart of Figure 7. Both the gallia and calcia powders used were of 99.999-percent purity and were manufactured by Johnson Matthey Chemicals Limited of England under their trade name "Puratronic." The silica was of 99.9-percent purity and was obtained from the Spectrum Chemical Manufacturing Corp. of Los Angeles.

The platinum crucible was embedded*** in a castable, high alumina refractory to (1) increase the thermal mass and slow cooling after removal from the furnace and (2) stiffen the crucible so that it could be handled repeatedly with tongs without crushing. Two such crucibles are shown in Figure 8. Figure 9A shows a print made of the "flight" loaf casting. The print was made by placing the casting, after polishing of opposite faces, on photographic print paper and exposing by means of a light placed above the sample. Note the improvement in striae as compared with Figure 9B, one of the earlier experimental castings made with less stirring with a silica rod. The flight casting was poured near one end, as can be seen by the crystalline material (white in the print). The left one centimeter of the casting is being held in reserve for possible future optical properties measurements. Five of the flight samples were taken from the area to the left of the crystal zone, and one (number 1) was taken from the area to the right bottom.

For convenience for suspending in the flight acoustic positioner, the flight samples were hand-diamond-ground to a roughly spherical shape at a lapidary shop. Figure 10 shows the six samples so prepared. Figure 11 shows them after devitrifying by heating 10 minutes at 1250 C.

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*So named because their shape resembles that of a common loaf of bread.

**Regrettably, the loaf casting for the 20-percent silica content was lost in a spill during a remelting experiment before it was photographed. It appeared to be about 20 percent glass and 80 percent crystal.

***At the suggestion of Dr. D. Day of Missouri School of Mines, Rolla.

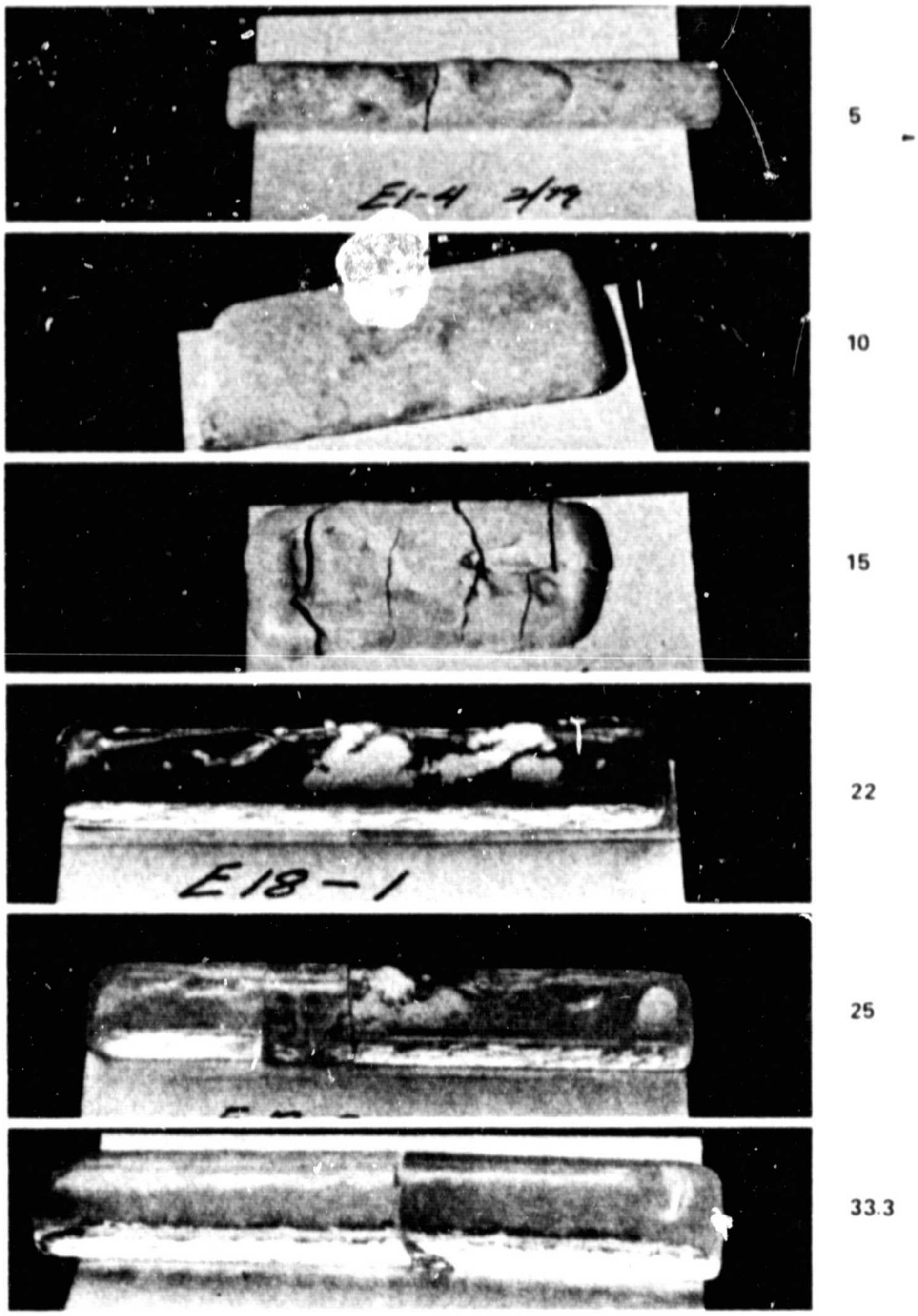


Figure 5. Loaf Castings of 5, 10, 15, 22, 25, and 33.3 Mol % SiO_2 Experimental Compositions

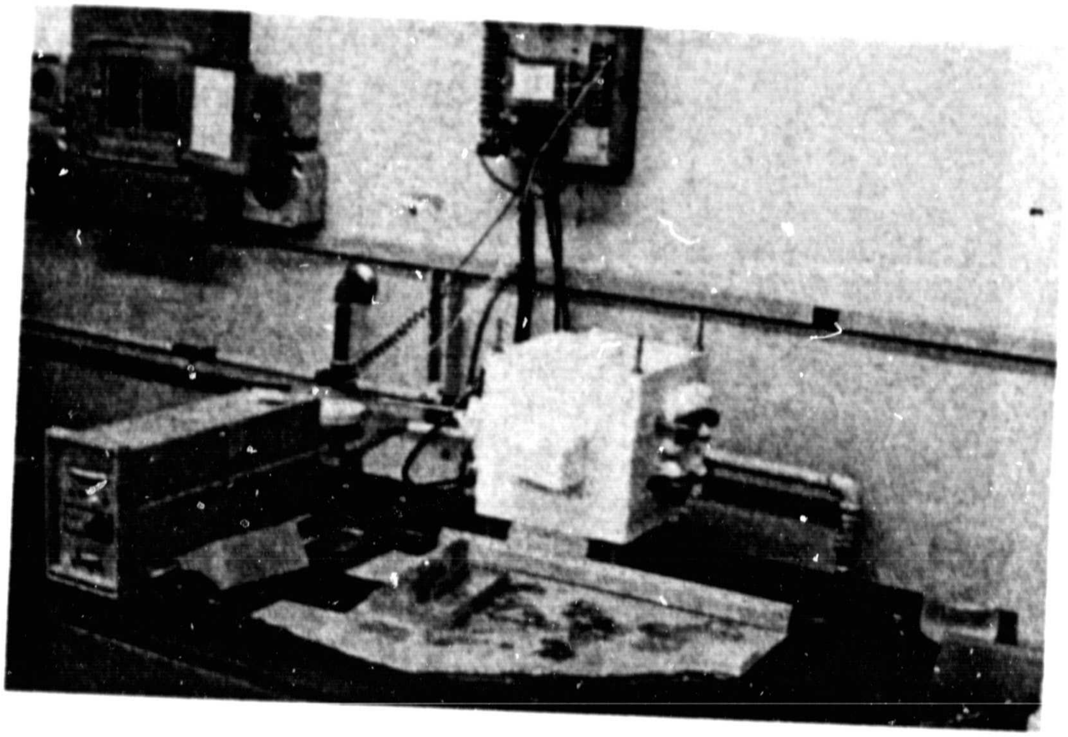


Figure 6. SiC-Element Furnace (Mold for Preparing "Loaf" Castings Shown in Front of Furnace)

Data on the six flight samples are given in the following:

Sample No.	Wt (gm)	Diameter (in.)		
		Average	Max	Min
1	1.2578	0.3364	0.3390	0.3305
2	-	0.3180	0.3242	0.3117
3	0.8972	0.2866	0.3014	0.2790
4	0.8975	0.2972	0.3163	0.2825
5	0.8965	0.2875	0.3079	0.2746
6	0.5778	0.2544	0.2670	0.2473

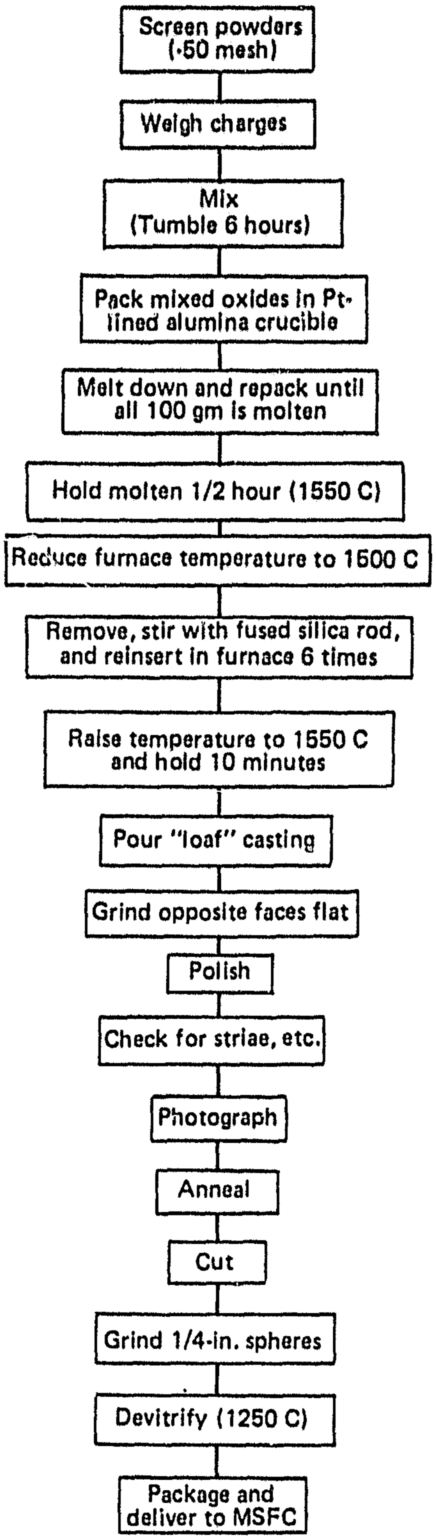


Figure 7. Preparation of Flight Samples

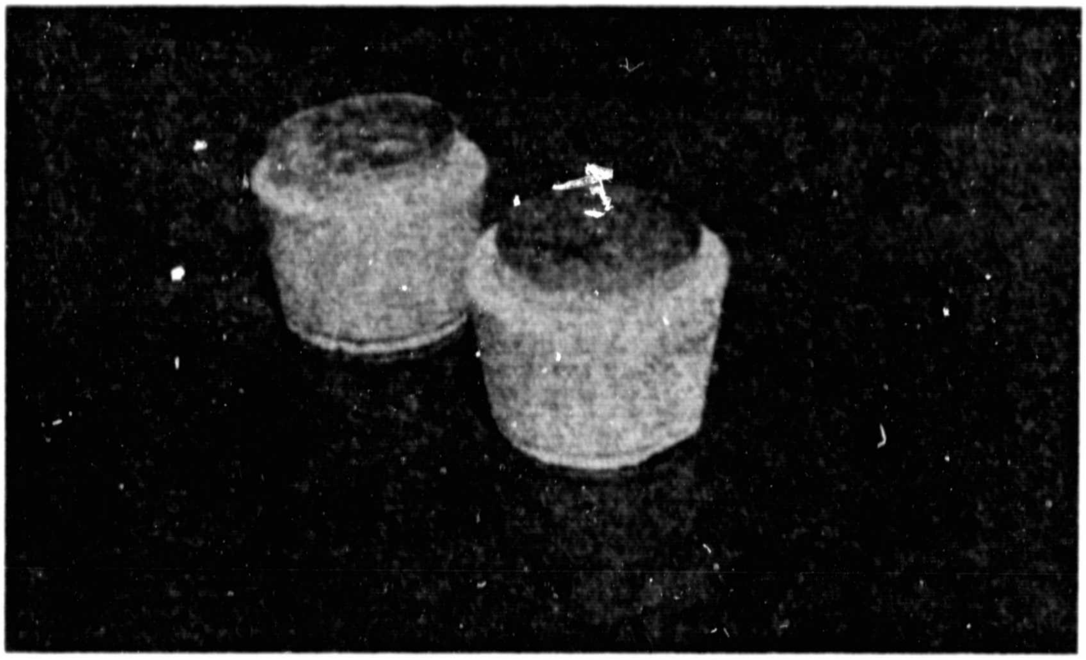
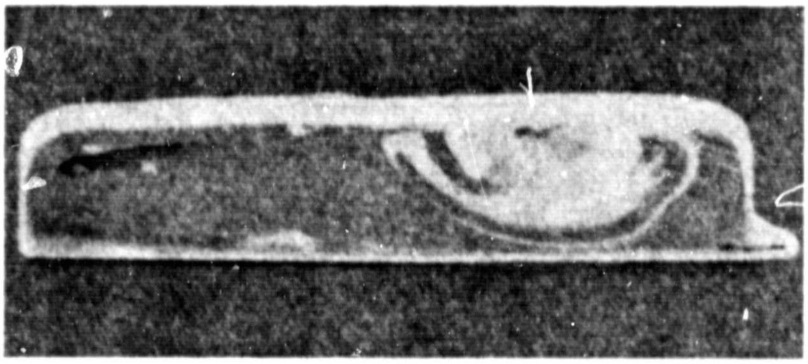


Figure 8. Pt Crucible With Alumina Castable



A. Flight Casting

B. Experimental Casting

Figure 9. Contact Prints of "Loaf" Castings

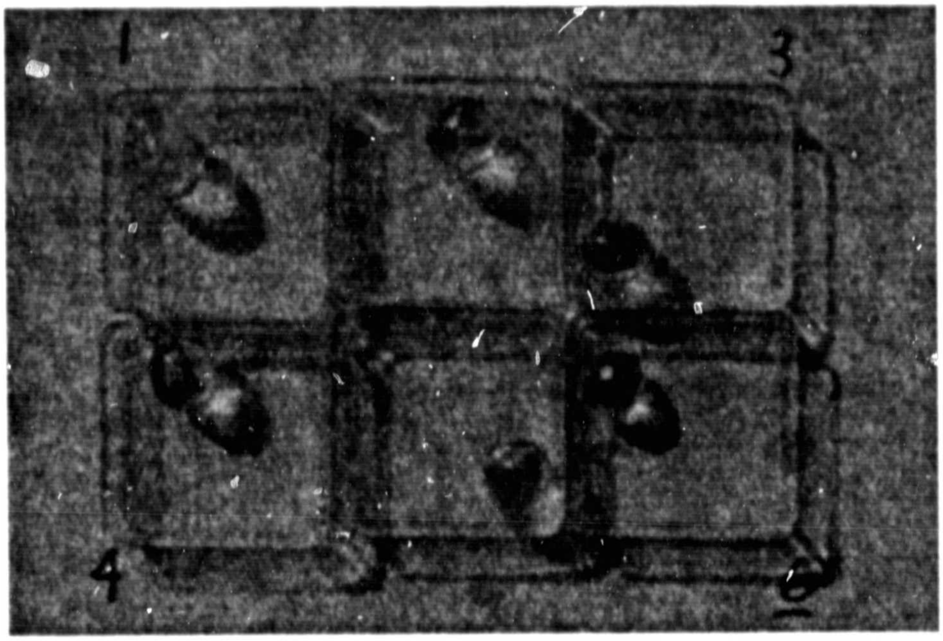


Figure 10. Flight Samples Before Devitrifying

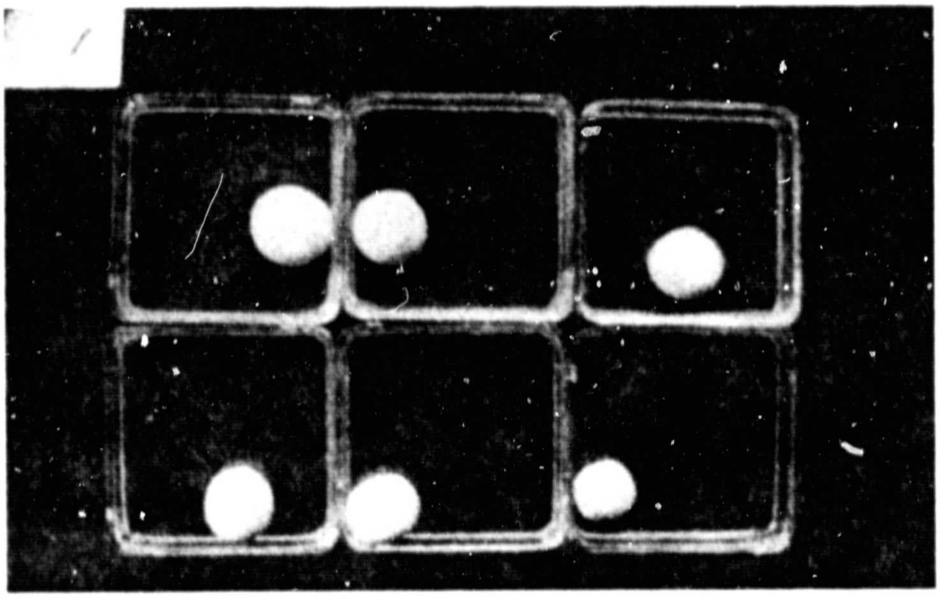


Figure 11. Devitrified Flight Samples

FLIGHT EVENTS

In this section the writer will attempt to give only details pertinent to an understanding of the flight sample examination, which follows later. A detailed reporting on the flight hardware will be issued by Intersonics after the second SPAR flight containing the glass melting experiment, 74-42.

SPAR VI, with flight sample Number 6 loaded in the injection mechanism, was launched at 6:40 a.m., MST at the White Sands Missile Range, New Mexico, on October 17, 1979. The payload suffered a rough landing as discussed in more detail in Appendix A.

Shortly after the payload was returned to the Vertical Assembly Building at White Sands, the access doors on experiment 74-42 were removed, and the injection mechanism containing the flight sample was removed from the furnace. Figure 12 shows the injection unit with the platinum wire cage and glass sample immediately after removal from the flight furnace. During the landing, the copper cooling shroud inside the furnace was torn from its moorings and crashed into the injection cage. The damage to the cage is apparent in Figure 12B.

The glass sample was found "impaled" on one of the finer platinum wires of the cage. Figure 13 shows two closeups of the cage with the glass sample. The latter photographs were taken four days after the rocket flight. Note that the glass sample appears to be quite well centered on the platinum wire. Distortion of the platinum wires near the sample is further evident in Figures 13A and B.

FLIGHT MOTION PICTURES

Appendix A discussed in detail the processing of the flight motion picture film developed under the supervision of the writer's organization. The timeline for the flight experiment beginning with the "camera on" after low g is achieved is given in the following tabulation:

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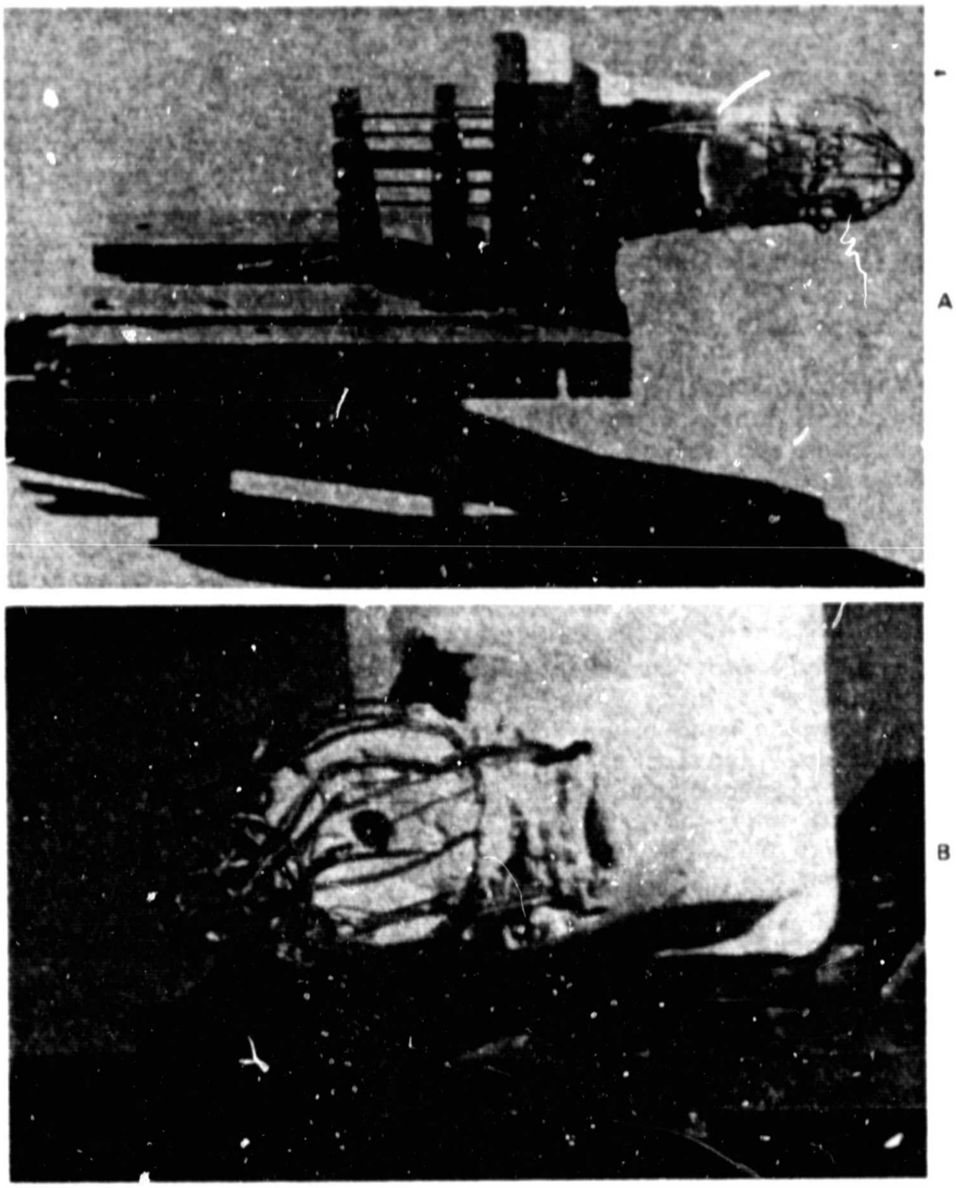

Figure 12. Two Views of Injection Mechanism With Flight Glass Sample (Photographed October 17, 1979, Immediately After Removal From Payload)

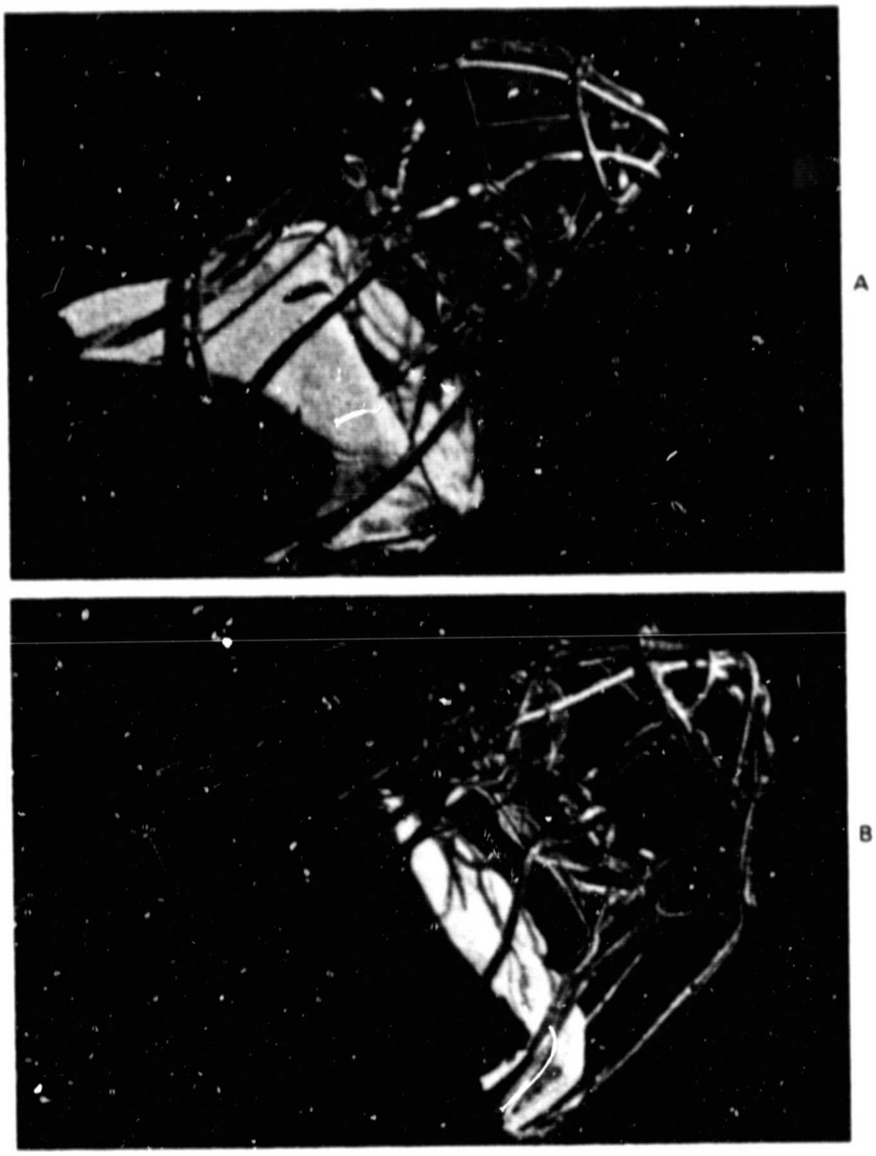

Figure 13. Two Views of Injection Cage With Flight Glass Example (Photographed October 21, 1979)

Event	Cumulative Time (sec)
Camera on	0
Injection into furnace hot zone and start of heating sample	5
Cooling shroud gate open and start of insertion of cooling shroud	160
Cooling shroud fully inserted	170
End of sample cooling and sample retraction	243
Camera off	248

Examination of the flight film showed that immediately after the injection mechanism released the sample, it moved around inside the cage and touched the cage wires four times during the first 9 seconds after injection. During this time sample melting had not begun. It remained in suspension in the sonic well without touching for another 27 seconds, at which time it contacted the cage at 36 seconds after injection. Since the sample was, by this time largely molten (to be discussed in more detail later), it stuck to one of the cage wires. As best as can be judged by appearance of the sample in the film, it seemed to be largely molten about 30 seconds after injection, or about 6 seconds before it contacted the cage wire for the last time.

Table 1 shows significant events on the flight film, and Figure 14 shows prints of selected frames from the flight film. The contrast is low in the early frames because the exposure settings of the flight camera were chosen so the furnace wall, at a nominal 1575 C, would be on the verge of overexposure. This permitted maximum tracking during the cooling cycle. Of course, as the sample and cage approach the furnace wall temperature, the contrast would be very low regardless of exposure setting. In frame 1 the injector has just appeared, and the sample is trapped between the outer and inner (to the right) portions of the cage. In frame 4 the inner portion of the cage is retracting (moving to the right), the outer portion is moving to the left, and the sample has been released. In frame 11 the cage has fully opened, and the sample can be seen near the bottom of the cage just before it contacts the cage in the next frame. In frame 721, 30 seconds after injection, the sample is suspended near the center of the cage. In frame 860, 36 seconds after injection, the sample has moved upward and is just making contact with a cage wire. Five frames (0.2 seconds) later the sample has centered itself on the platinum wire. By frame 1303, 54 seconds after injection, the sample and cage have both closely approached the furnace wall temperature and are almost invisible. In frame C5, which is the fifth frame after the cooling shroud gate opened, a portion of the glass sample can be seen clearly still on the cage wire. In this frame the hot sample and injection cage can be seen in sharp contrast to the cold background of the cooling shroud. In the 316th frame (13 seconds) after the cooling shroud gate opened, the sample and cage can be seen to have cooled considerably. By frame C914 (not shown), 38 seconds after the gate opened, the sample and cage have merged with the cool-

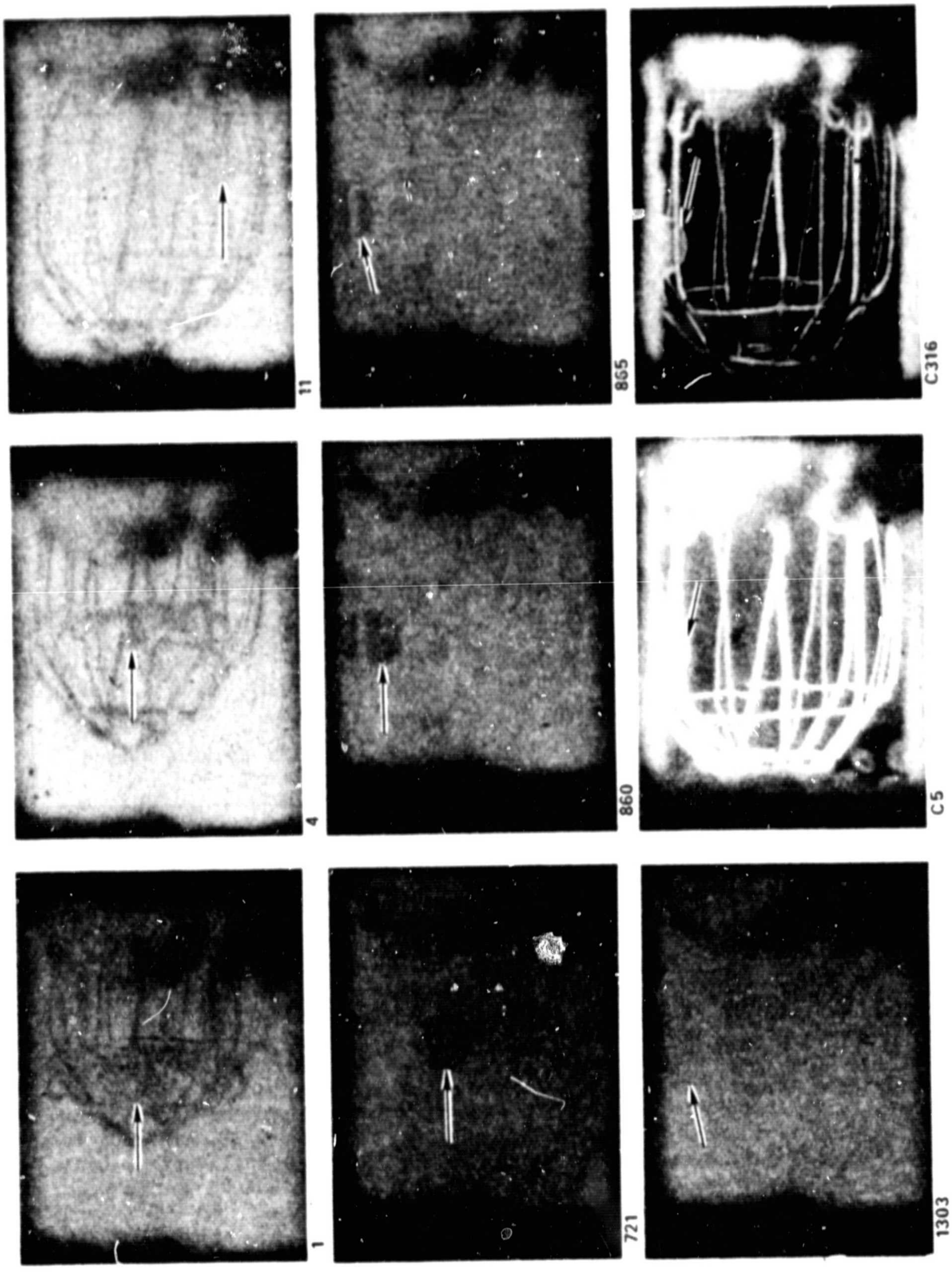

Figure 14. Selected Frames From Flight Motion Pictures

Table 1. Significant Events on Flight Film

Frame Number	Seconds*	Event
0	0	Clear
1	0.042	Injection cage first visible
4	0.17	Cage begins to open
11	0.46	Cage fully open
12	0.50	Sample touches bottom of cage (1st touch)
41	1.7	Sample touches top of cage (2nd touch)
124	5.2	Sample touches right side (inner cage)(3rd touch)
207	8.6	Sample again touches right side (4th touch)
530 ± 10	22.1 ± 0.4	Contrast has increased, sample appears darker than before
577	24.0	"Jiggling" (or rotation of sample) first noticed
780	32.5	Contrast best (sample appears darkest) since frame 530
859	35.8	Sample begins to move upward
860	35.8	Sample appears to touch wire (5th touch)
865	36.0	Sample appears centered on wire, no further motion
C2	0.08	Cooling shroud gate fully open
C914	38	Sample appears to have reached cooling shroud temperature (everything is dark)

*All of the times given here necessarily assume that the flight camera was running at exactly 24 frames a second during the entire flight.

ing shroud background and can no longer be seen in the film. After about 21 seconds (in the vicinity of frame 500) the sample has blended with the background and cannot be seen. This is not shown in Figure 14. At about frame 530 (22 sec) the sample appears to begin to become darker than the background again. This darkening of the sample with respect to the background continues, and maximum contrast is achieved again by frame 780, 8 seconds later.

It is the writer's belief that the reversal in contrast, beginning in the vicinity of frame 530, is indicative of the formation of the liquid phase on the surface of the sample. It is to be expected that the phase change from solid to liquid would be accompanied by a sudden change in the sample's optical properties and would create the illusion that the temperature of the sample had dropped. The fact that the sample quickly centers itself (in 0.2 seconds) after contacting and wetting the cage wire indicates that the sample must have been largely molten at that time.

ANALYSIS AND EVALUATION OF RETURNED FLIGHT SAMPLE

Magnified views of the flight sample are shown in Figures 15 and 16. Note that the sample is entirely free of internal* crystalline material. This indicates that melting was complete during the flight. Any unmelted material would have remained as opaque or translucent material within the body of the glass sample.

It is also apparent from Figures 15 and 16 that the sample is well centered on the 0.014-inch-diameter Pt-Rh wire. The diameter of the sample, as measured by micrometer, is 0.2501 ± 0.000[illegible] inches. As measured from 8-in. by 10-in. photographs, the centering appears to be within 0.002 inches. Good centering is to be expected since the sample, which wets the platinum alloy, would like to spread along the platinum as much as possible. The maximum length it can achieve along the platinum is at the centered position in the absence of significant sonic forces at that location. The wetting angle between the glass and the platinum was measured at about 50 degrees.

In Figure 15, three bubbles can be seen in proximity to the wire. It is suspected that the bubbles result from entrapment of air caused by localized zones of slower wetting of the platinum alloy by the glass as the glass spreads along the wire. A similar effect has been frequently noted by the writer in brazed and soldered metallic joints where spreading of the brazing alloy or solder through the joint is not uniform, resulting in formation of voids within the joint.

Figure 17 shows two end views taken at two different angles to the wire. In both of these views, the sample is circular to within less than 0.0001 inch.

Figure 18 shows the sample under polarized light. It appears to be quite free of residual stress except near the glass/platinum interface. This is not unexpected because the glass and the platinum alloy undoubtably have different thermal expansion coefficients. Since the glass wets the wire, a stress field must therefore be set up as the sample and the wire cool.

An attempt was made, without success, to measure the index of refraction of the flight sample by immersing it in a series of fluids with indices that varied in 0.005 steps. Subsequently, a small chip was removed from the loaf casting from which the flight sample had been removed and was, therefore, of the same composition, i.e., 35.7 CaO : 39.3 Ga_2O_3 : 25 SiO_2, in mol % as the flight sample. The index, measured with sodium D illumination, was determined as falling between 1.710 and 1.715.**

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*In Figure 16 particularly, surface crystallites may be seen out of focus. This subject is discussed in more detail later.

**This compared with a reported value in Reference 7 of 1.712 for the $Ca_2Ga_2SiO_7$ glass. The composition of the latter, in mol %, is 50 CaO : 25 Ga_2O_3 : 25 SiO_2.

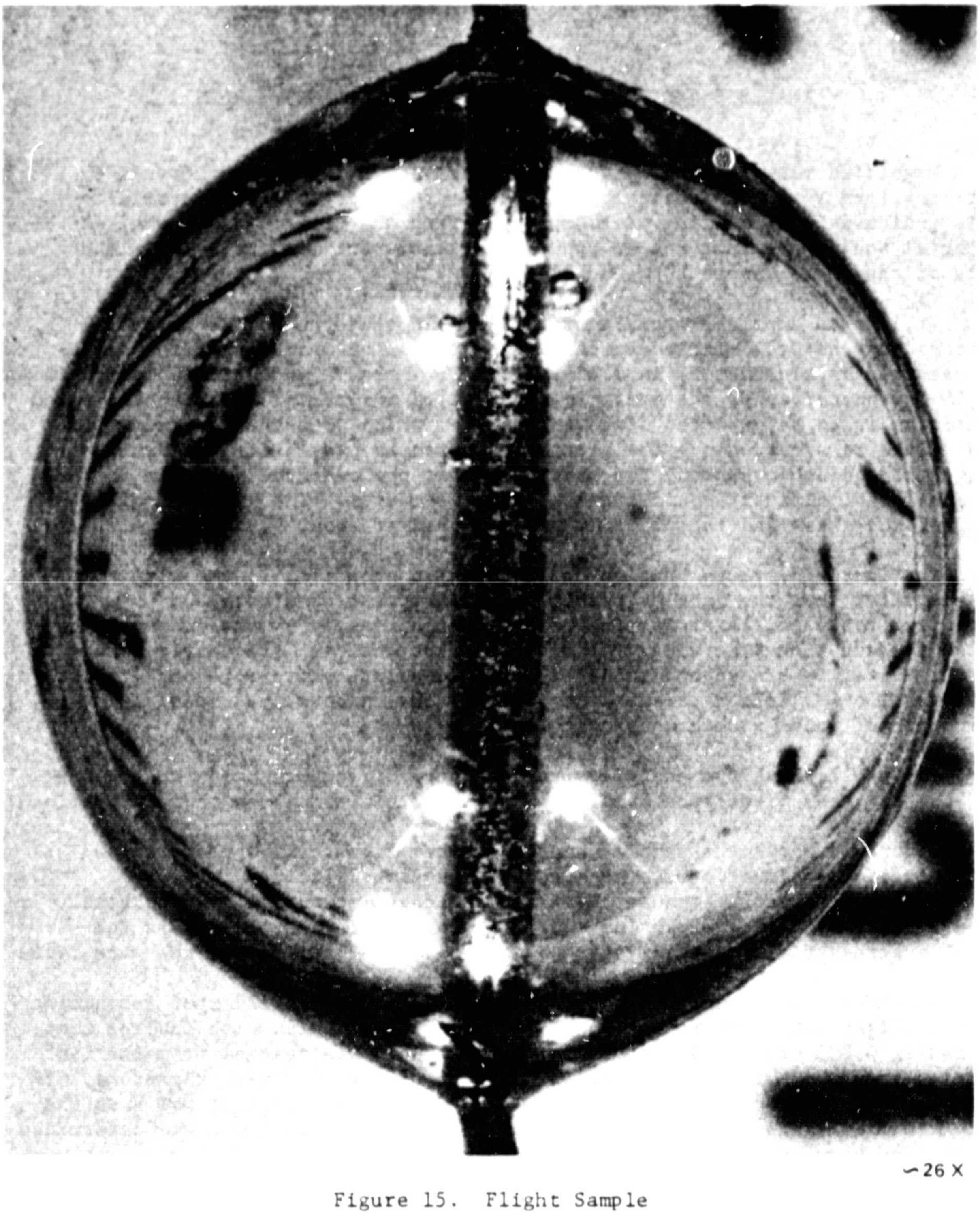


Figure 15. Flight Sample

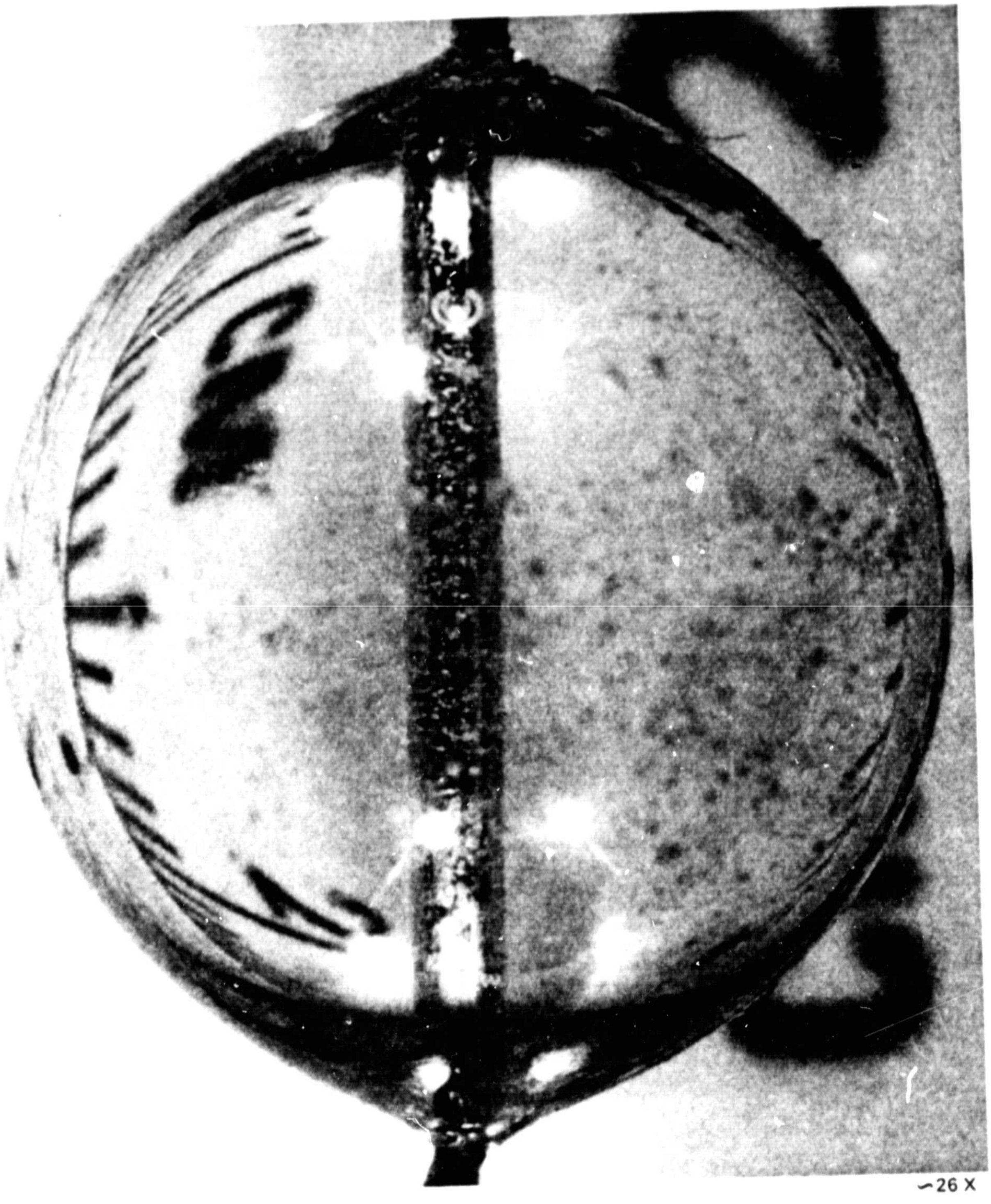


Figure 16. Flight Sample, Opposite Side From Figure 15

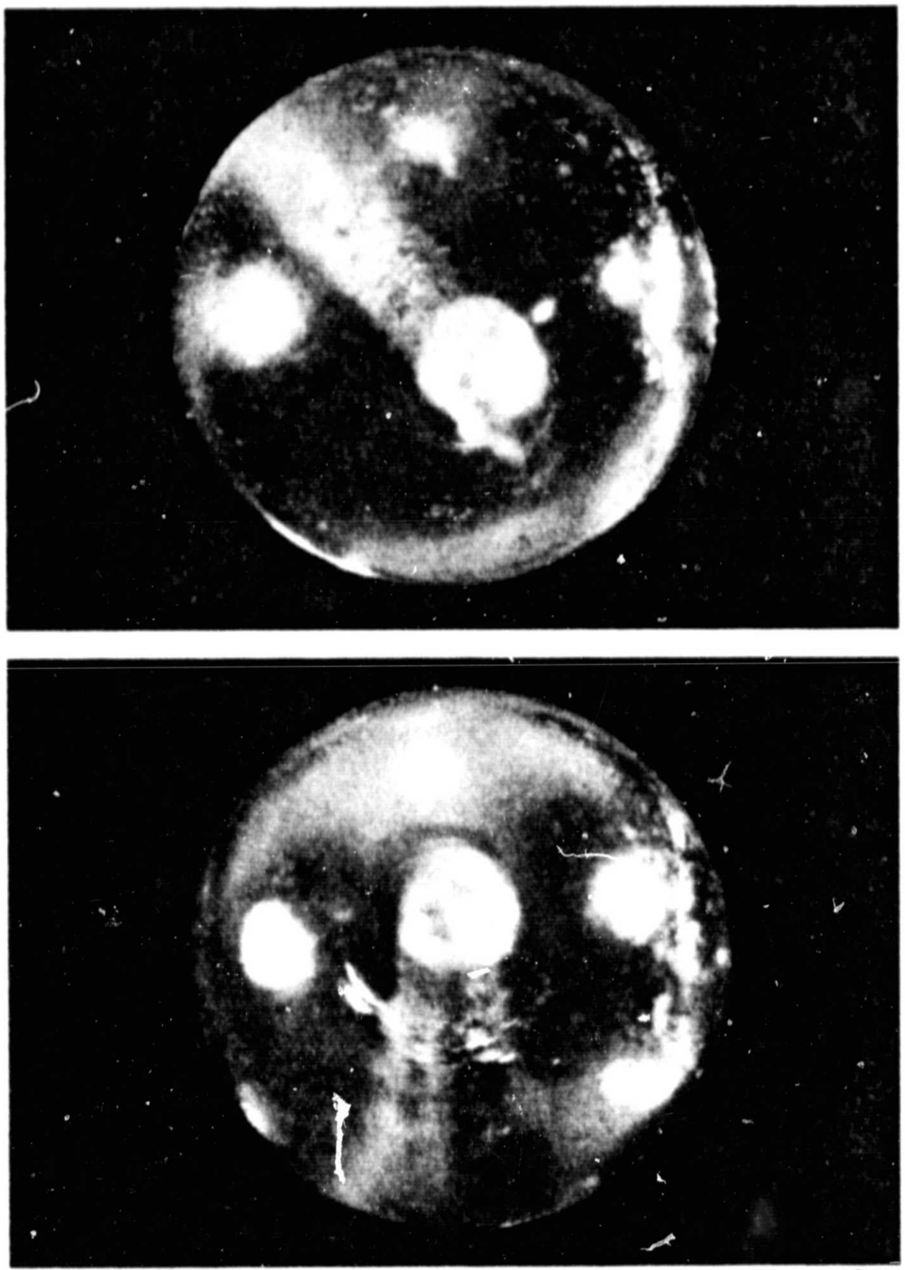


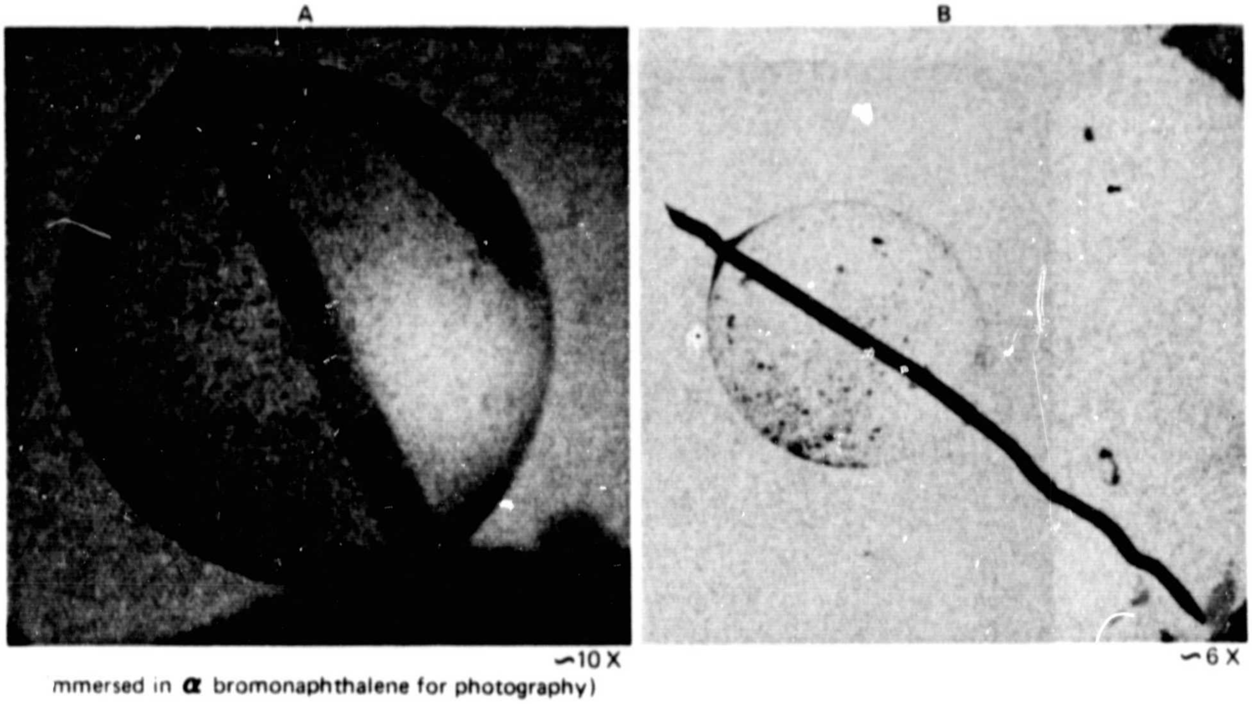
Figure 17. Two End Views of Flight Sample

(Sample immersed in α-bromonaphthalene and photographed by transmitted light with the two polarizers at 90° to each other.)

Figure 18. Stress in Flight Sample - Polarized Light Photograph

SURFACE CRYSTALS

After the flight sample had been in the possession of the writer about four weeks, it was noted that the surface of the sample contained numerous surface crystal rosettes not visible to the unaided eye. They are shown in Figure 19. There appears to be a greater concentration of crystals in one hemisphere than in the other (Figure 19 B). Similar appearing, but much larger, crystals had been noted on the free surface of the cooled melt (Figure 4) and on the free surface (only) of the loaf casting (Figure 5). Figure 20 shows a portion of the free (upper) surface of the loaf casting from which the flight material was removed. There are two forms of crystals present on the casting's free surface. The larger white rosettes are similar to those predominating on the surface of the flight sample. The smaller, darker appearing crystals are invaribly roughly rectangular in shape when examined at high magnification. This subject will be addressed again later.



mmersed in α bromonaphthalene for photography)

Figure 19. Two Views of Flight Sample Showing Surface Crystals

Figure 20. Crystals on Top Surface of "Loaf" Casting

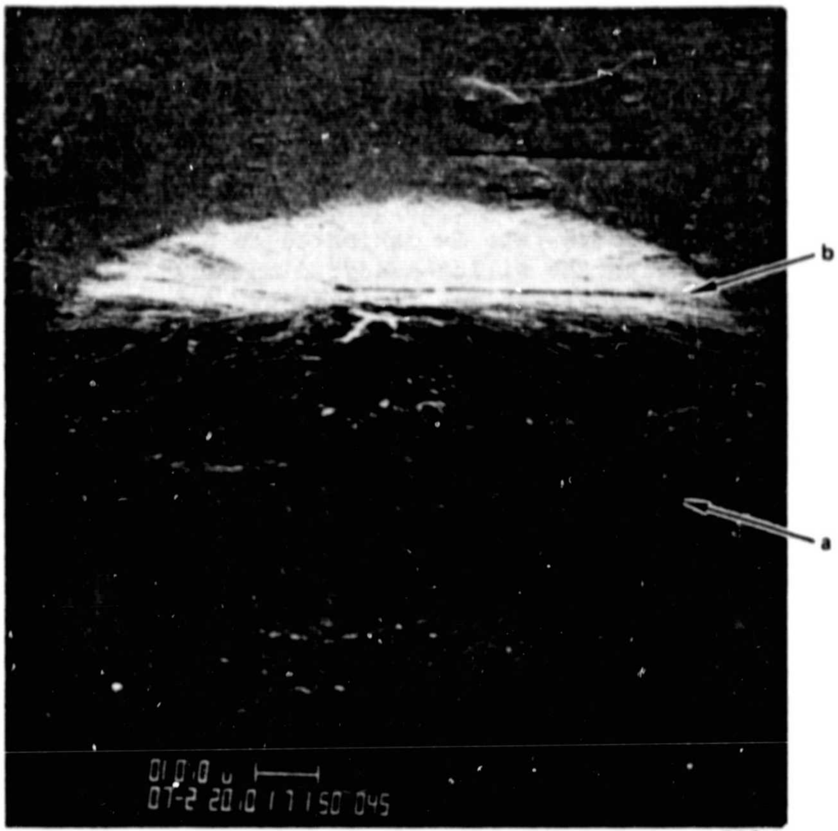
The finding of crystalline material on the flight sample's surface was of concern to the writer because of the possible ramifications in connection with the next SPAR flight experiment. Because of the relatively high silica content of the SPAR VI material, crystal growth is quite slow so that any crystal growth is arrested during cooling from the melt. However, in the binary gallia-calcia material being considered for the next SPAR flight experiment, the crystal growth rate is estimated to be several orders of magnitude greater than with the silica-modified material. Therefore, it is probable that such crystallization, if it occurs on the surface of the gallia-calcia flight sample, would result in conversion of the entire mass to the crystalline condition upon cooling. It then becomes imperative to learn as much as possible, within time and funding limitations, about the small crystallites on the surface of the present flight sample.

A portion of the flight loaf casting was remelted with the laser/air suspension unit (Reference 3). The surface of the sample so prepared was found to be free of crystallites. A sample of the binary gallia-calcia glass prepared also by laser/air suspension melting was also found to be entirely free of crystallites.

The remelted (laser) sample was immersed for four weeks in the same fluid, (α bromonaphthalene) that the flight sample had been exposed to prior to noting of the crystallites. After exposure the sample was still free of crystallites. This convinced the writer that the crystals must have formed on cooling during the SPAR VI flight.

Next, the flight sample was gold-coated and examined with a scanning electron microscope. Results are shown in Figures 21 through 28. Figure 21 shows the appearance of a typical crystallite at about 700X magnification. EDAX (energy dispersive analysis by X ray) analysis (Figure 22) of the rosette material showed it to be of about the same composition as the surrounding glass. Examination of the center of several rosettes failed to reveal a compositional difference that could give a clue to the nucleating material if indeed it is different from the crystallite composition.

The sample was then etched by immersing several hours in dilute KCN. The sample was again gold-coated, and about a dozen rosettes were examined with the SEM (scanning electron micrograph). In about one-third of them, the etching had left a different material in relief at the center. Two such rosettes are shown in Figures 23 and 25. The EDAX analysis of Figure 26 (a and b) shows that the material in the center is largely platinum. Since about one-third of the rosettes examined after etching were found to contain platinum in the center, it would appear that platinum is the nucleating agent. Failure to find platinum in the center of the remaining two-thirds of the rosettes examined probably indicates that etching had not been carried far enough to expose the underlying platinum.



Unetched, Au-Coated ~700X

Figure 21. Scanning Electron Micrograph (SEM) of Typical Crystal Rosette on Surface of Flight Sample

The finding that platinum is most probably the nucleating agent for the crystals leads one to speculate about how the platinum got there. Four different mechanisms may be considered, as follows:

1. Migration of platinum by diffusion from the wire embedded in the molten flight sample over the surface.

2. Vapor transport from the platinum wire to the surface of the flight sample.

3. Migration by diffusion of the small amount of platinum dissolved in the glass from the terrestrial melting in platinum.

4. Mechanical transfer of platinum from the cage to the sample during early portions of the flight.

With respect to the fourth mechanism, the sample was apparently a rather loose fit in the cage. Vibration at launch could have caused it to rattle around between the inner and outer portions of the cage. Since the cage material is a platinum and 30 per cent rhodium alloy, one would expect the rhodium to be transferred along with the platinum. There are strong

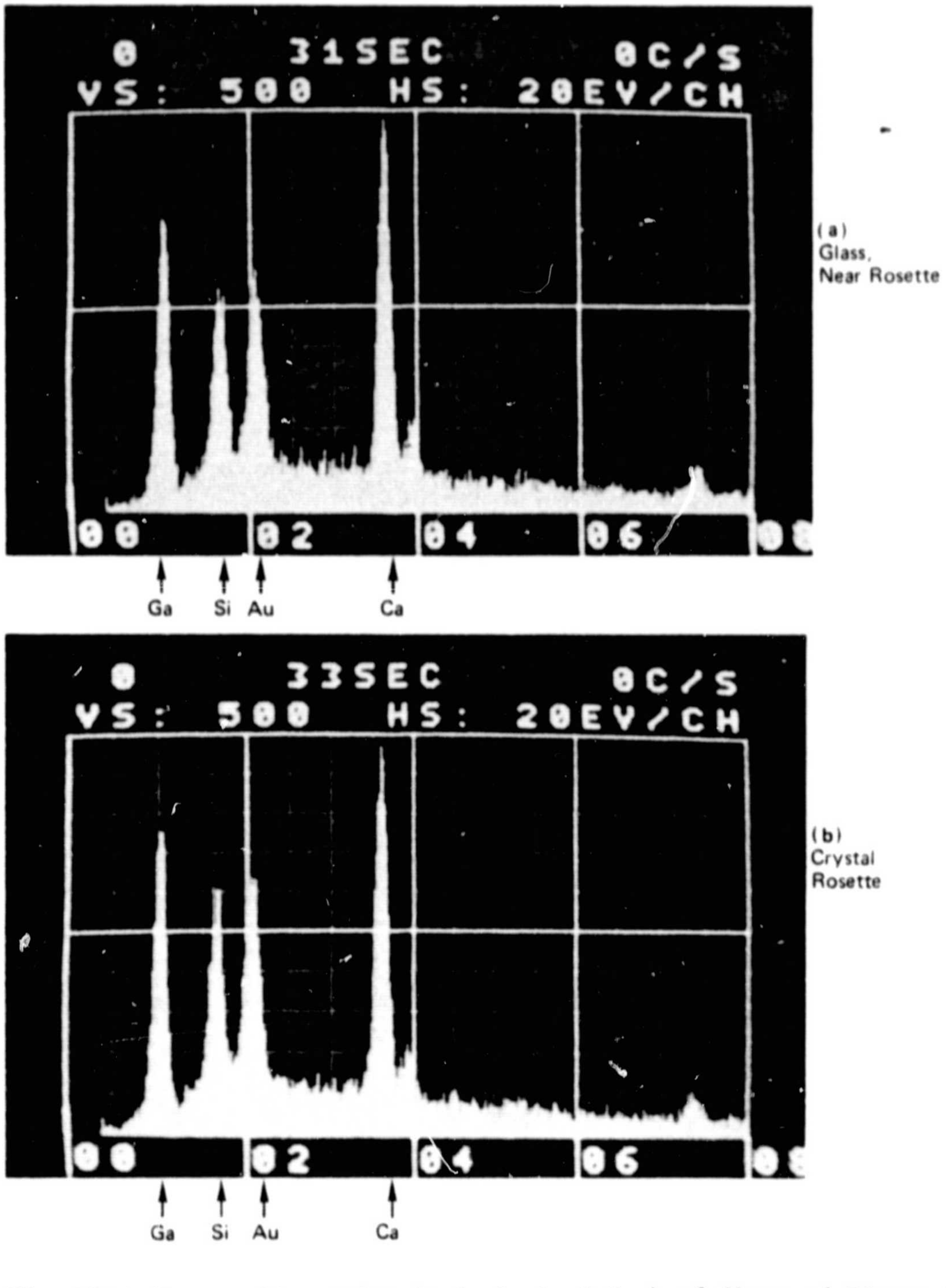
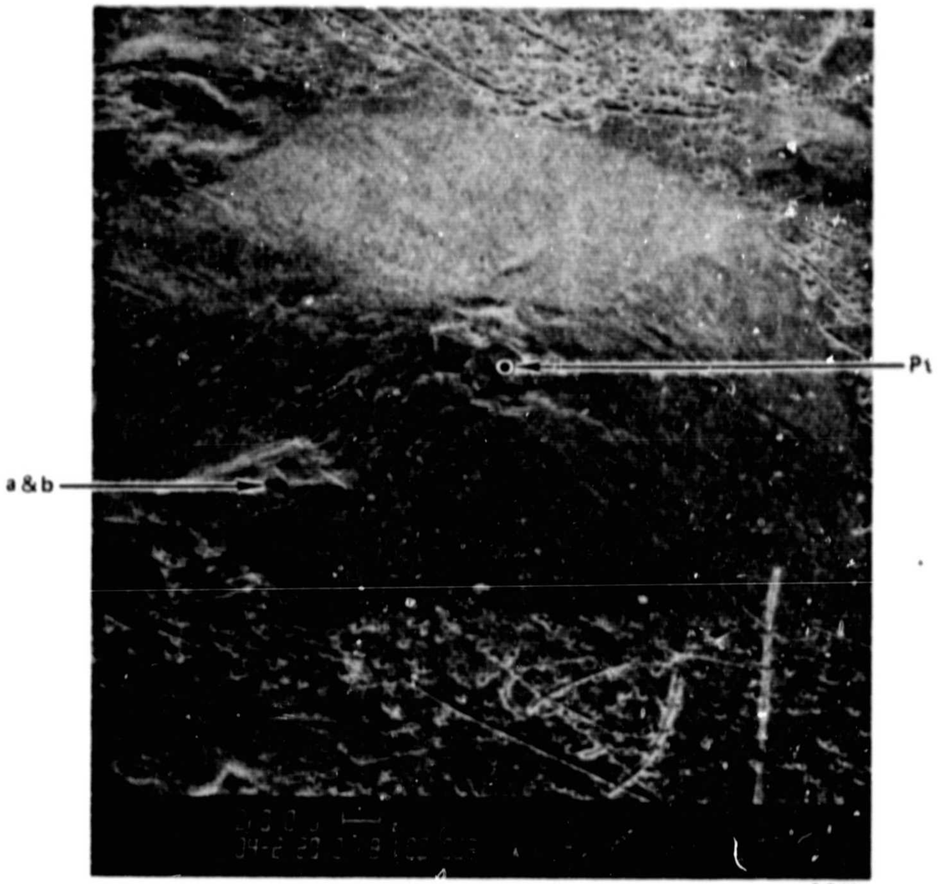


Figure 22. EDAX (Energy Dispersive Analysis by X Ray) of Glass of Figure 21, Near Rosette (Top) and Rosette Material (Bottom)



Etched, 2 hr dil KCN Au-coated ~400 X

Figure 23. SEM Photograph of Rosette on Surface of Flight Sample

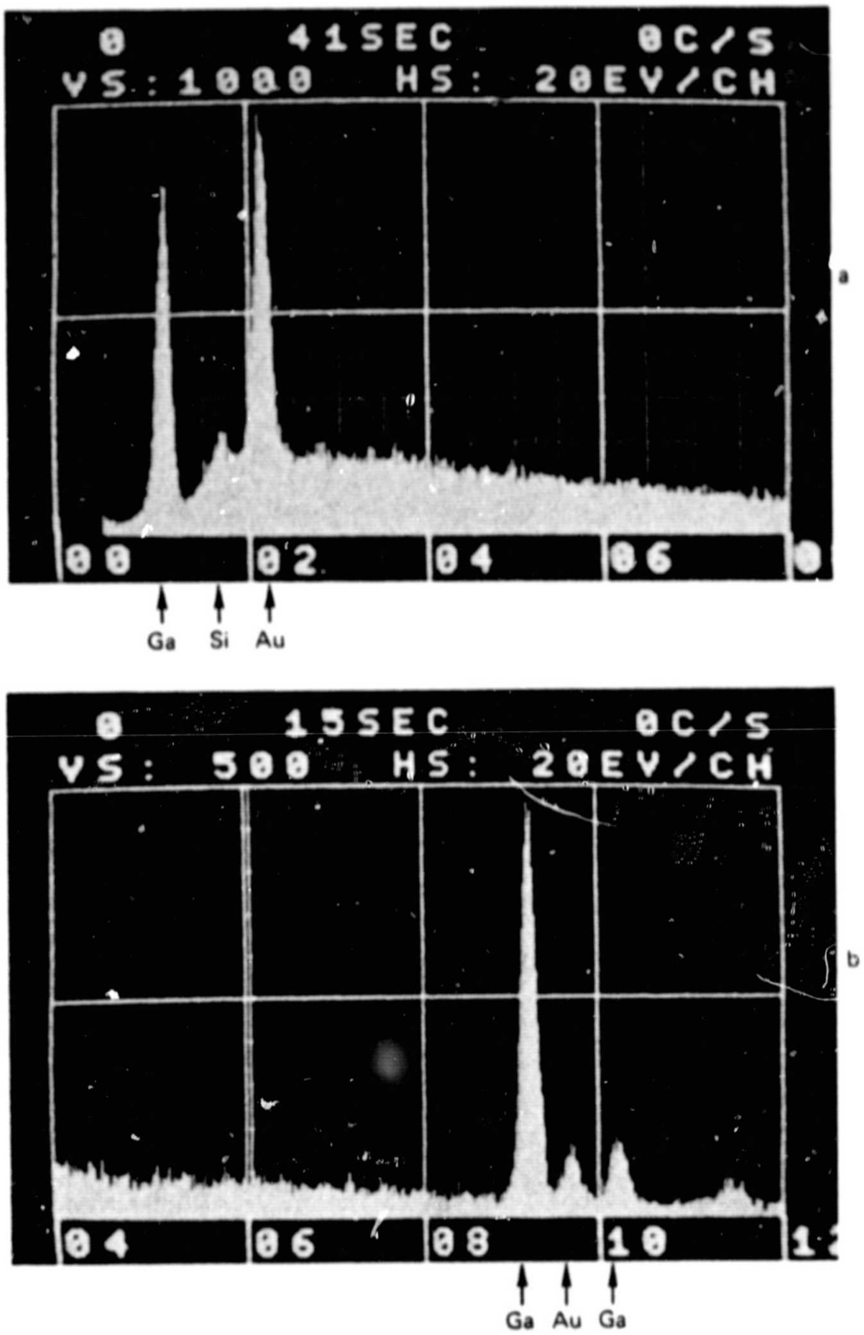
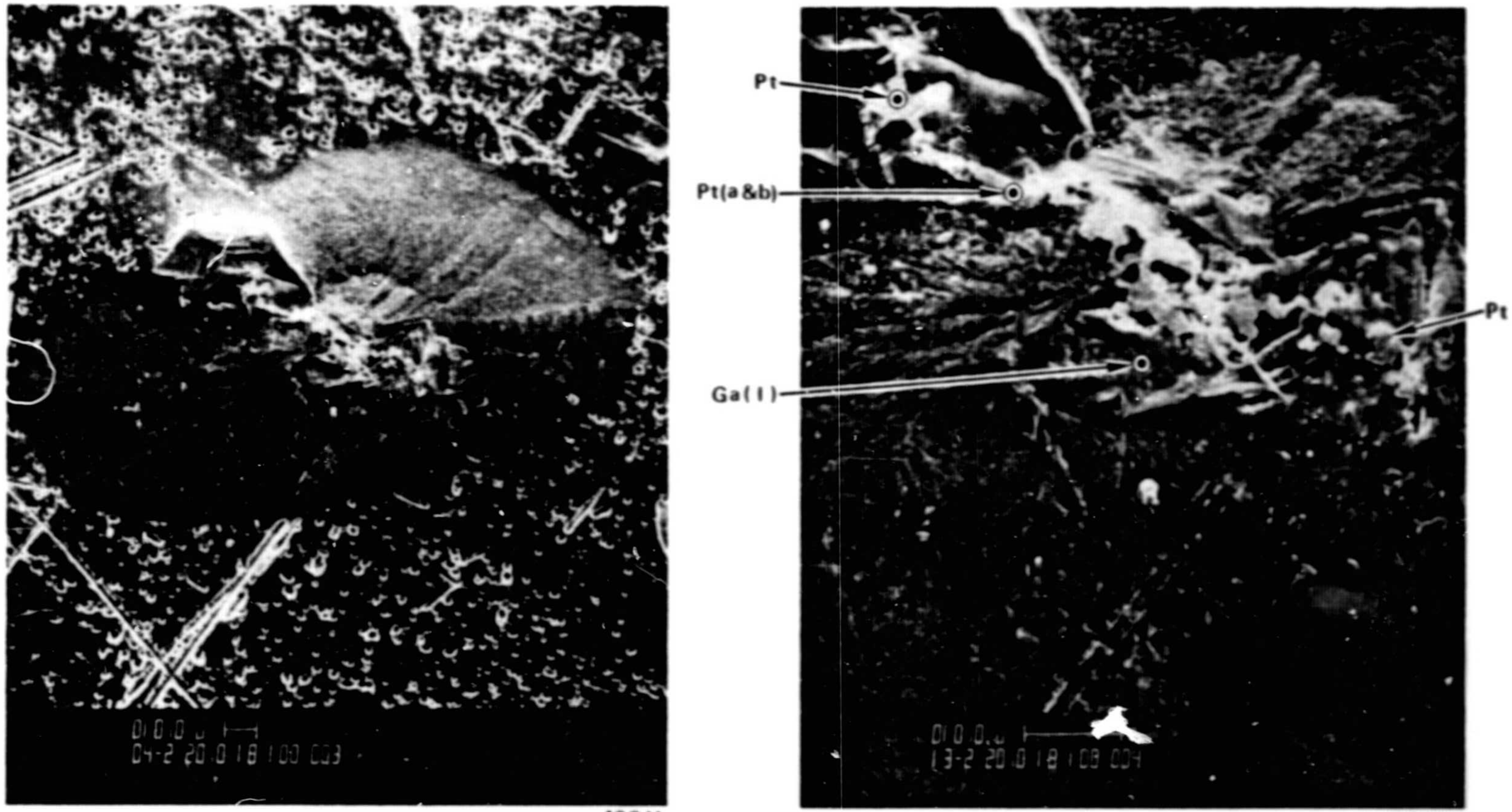


Figure 24. EDAX of Location a, b of Figure 23



~400 X

B. Closeup of center of rosette of A. ~1300 X

Etched 2 hours in dilute KCN, Au-coated

Figure 25. SEM Photographs of Rosette on Surface of Flight Sample

rhodium lines at 2.7 and 2.8 Kev. Examination of the EDAX of Figure 26b shows a small peak in this area, an indication that rhodium may well be present. The third mechanism would not appear to be likely since the laser-remelted sample did not contain surface crystals, as noted earlier.

The second mechanism cannot be positively eliminated at this time. However, a vapor transport mechanism would seem to require that, at the time of transport, the surface temperature of the glass sample be significantly lower than that of the platinum wire so that the platinum would condense there. There should have been only a small temperature difference between the two during the entire flight experiment schedule. If anything, during the heating phase the platinum would be expected to be at a lower temperature than the surface of the sample because it has a higher reflectivity (lower absorptivity). During cooling, the reverse should be the case because the glass has a much higher emissivity than the platinum. However, such a condition exists for only a few seconds of the total schedule. During the longest part of the schedule, i.e. the soak, the temperature of the glass and the platinum should be very nearly the same. If there is a difference, one would expect the platinum to be at a slightly lower temperature than the glass surface because it is in contact with much cooler portions of the hardware and is a relatively good thermal conductor.

It is the writer's opinion, lacking further evidence, that the first and fourth mechanisms are the strongest possibilities of the four. Dr. Housely of Rockwell's Science Center states that he has frequently encountered surface migration of platinum in contact with non-metallic melts of other compositions.

If the writer's reasoning is correct, the lack of physical contact with platinum during melting, which is planned for during the next SPAR experiment, should eliminate the possibility of transfer by surface diffusion.

Ensuring that the sample is held firmly in the cage before launch should substantially reduce the possibility of mechanical transfer. The next SPAR experiment, if it is carried out as planned, should cast further light on the subject.*

Returning to the SEM photographs, it was found that the roughly rectangular-shaped crystals are gallium oxide as is shown by the EDAX of Figures 26 (c) and 28 (c).

EDAX of the white rosettes on the surface of the loaf casting (Figures 27 (b) and 28 (b) showed them to be of approximately the same composition as those found on the flight sample. Accordingly some of the material was filed off and examined by X-ray diffraction. The diffraction pattern showed that the major constituent is $Ca_2Ga_2SiO_7$ ($2CaO : Ga_2O_3 : SiO_2$), Reference 7. This phase has a tetragonal structure and cannot therefore be isomorphous with those found in the binary gallia-calcia system, i.e.,

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*At this writing, a decision has not been made whether to fly the binary gallia-calcia or the silica-modified composition on the next SPAR experiment. In the event the latter is chosen, it will be possible to check for platinum contamination if crystallites are found on the surface.

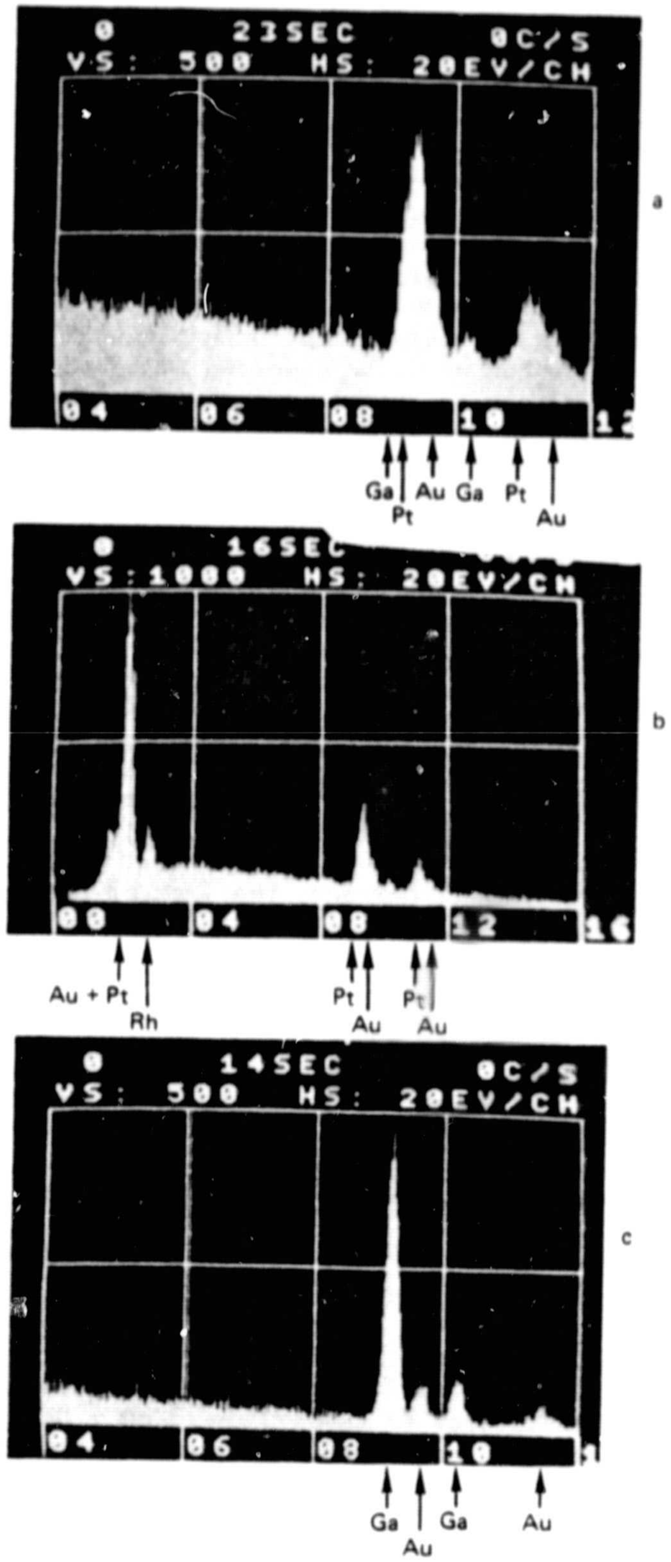
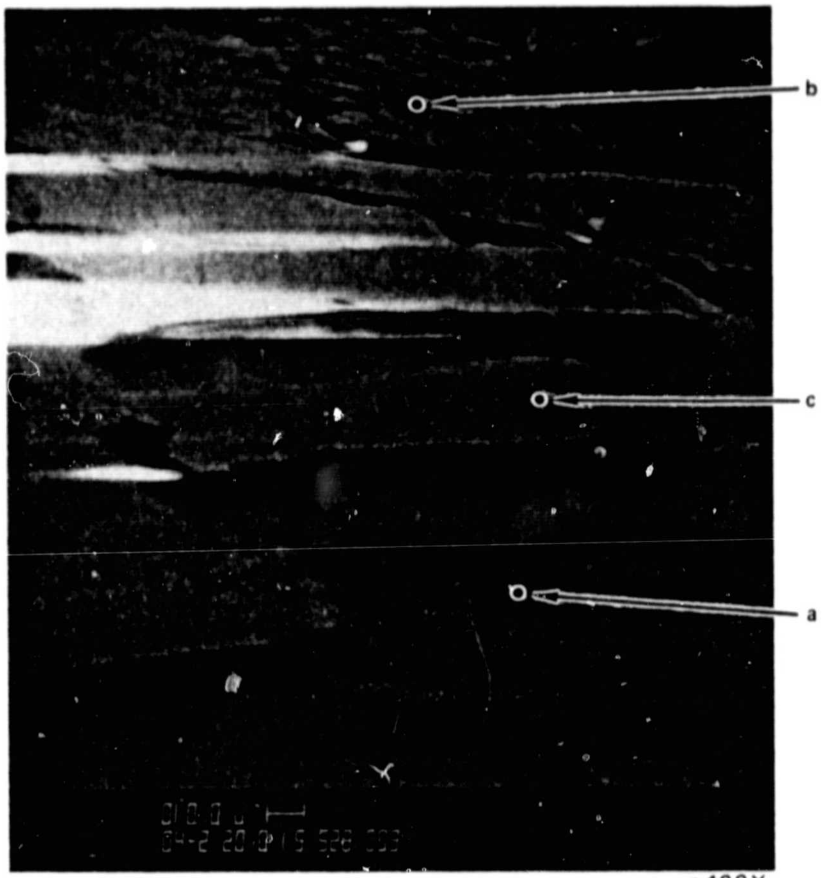


Figure 26. EDAX of Locations a, b, and c of Figure 25



Unetched, Au-Coated ~400X

Figure 27. SEM of Top Surface of Terrestrially Prepared "Loaf" Casting

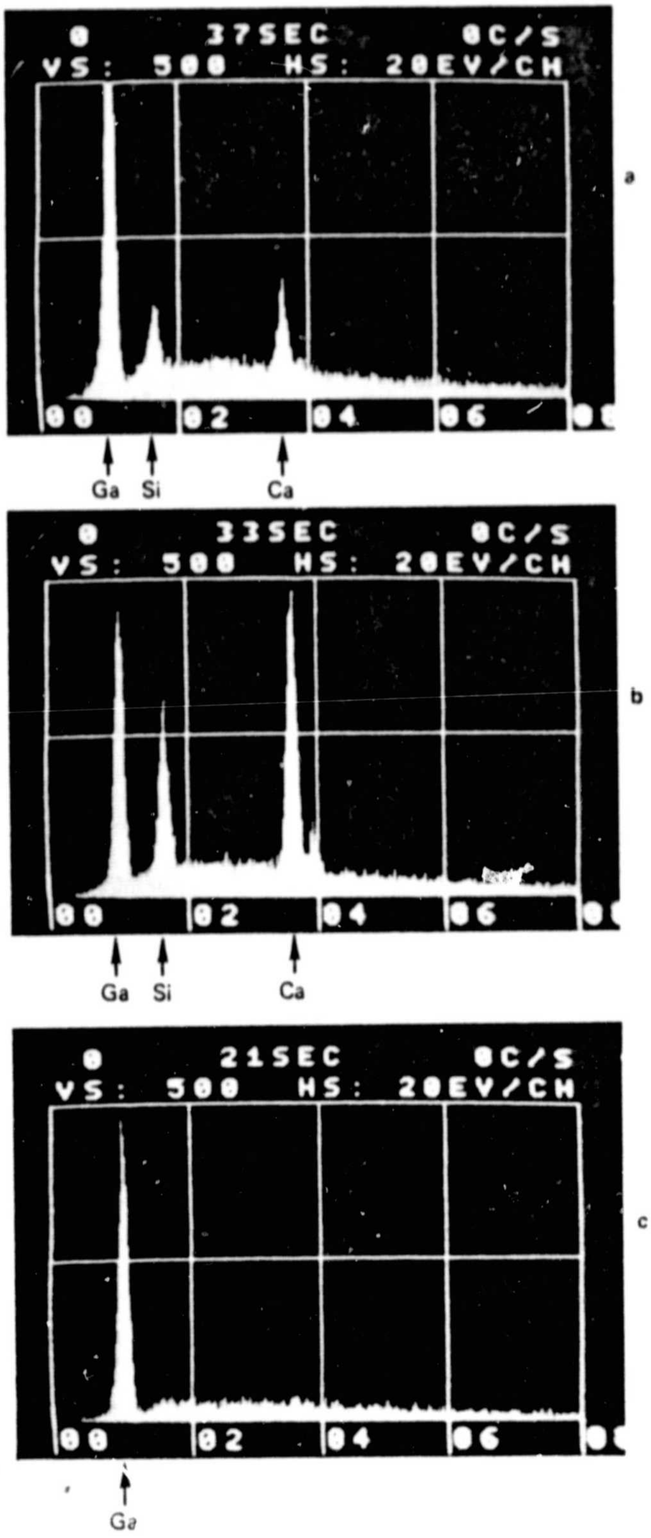


Figure 28. EDAX at Locations a, b, and c of Figure 27

CaO : Ga_2O_3 (orthorhombic) and CaO : 2 Ga_2O_3 (monoclinic, Reference 10). There is no 2CaO : Ga_2O_3 compound in the gallia-calcia system according to Reference 8 (refer also to Figure 2).

There are also five unidentified weak lines in the diffraction pattern which do not fit the $Ca_2Ga_2SiO_7$ pattern. This indicates that there are minor other constituent(s) present also.

In summary, the finding of the silicate phase, not isomorphous with any of the possible calcium gallates, indicates that the rosettes found on the surface of the flight sample are unique to the ternary system. The silicate cannot appear in the binary gallia-calcia glass planned for the next flight experiment.

CONCLUSIONS AND DISCUSSION

Despite the fact that the sonic positioning device did not prevent the sample from contacting solid objects within the furnace during the entire processing cycle as planned, the SPAR VI flight experiment may be considered a qualified success.

SCIENCE

The flight sample, which was returned in very good condition, had obviously been completely melted during the flight processing cycle. It therefore follows that the flight processing temperature-time cycle was adequate.

An unexpected development was the finding of numerous small crystallites of the compound $Ca_2Ga_2SiO_7$ which formed during cooling on the sample's surface. During the post-flight evaluation the crystals were identified and their presence almost certainly linked to the presence of platinum (and rhodium) on the surface of the glass sample. The platinum was found to be, with very little doubt, the nucleation sites for the crystallites. It was resulted either from (1) mechanical transfer from the loose-fitting platinum alloy injection cage during the highest vibration portion of the flight, i.e. during injection of the missile into its trajectory, or (2) surface diffusion of the platinum (and rhodium) from the platinum alloy wire impaling the specimen while it was molten.

FLIGHT HARDWARE

The flight experiment hardware functioned essentially as planned, with the exception that the sample contacted and wet the platinum alloy cage 36 seconds after sample injection into the hot zone of the furnace. Among the hardware functions that occurred successfully were:

1. The furnace and heating elements survived the launch environment and held to a nominal temperature during the entire processing cycle well enough to permit complete melting of the sample.

2. The acoustic positioning device was able to capture the sample, despite a rough injection. After several contacts with the cage during the first nine seconds, the acoustic positioner satisfactorily prevented contact of the sample with furnace components for 27 seconds before it contacted the cage wire.

3. The copper-rod cooling shroud was successfully deployed and cooled the sample approximately to the shroud temperature in the allotted time.

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4. The flight motion picture camera functioned flawlessly, exposures were as planned, and the returned film, after development, was invaluable as an aid to understanding flight events.

While the writer, from his study of the flight motion pictures, is convinced that the sample must have been largely molten at the time of contact with the cage wire, there is room to argue that the successful suspension of a liquid glass has not yet been conclusively demonstrated. It has not been possible to demonstrate that the sample was <u>completely</u> molten at the time of contact. The first evidence that the sample appeared to have undergone a phase change occurred only six to twelve seconds before the sample contacted the wire. It is not known whether the, presumably, molten condition of the sample contributed to its escape from the sonic well. The fact that on its fifth and final contact with the platinum cage the sample stuck to the wire is strong evidence that the surface was molten at that time. The further observation that the sample essentially centered itself on the platinum wire only 2/10 of a second after making contact would appear to indicate that the surface melting must have extended to a depth of <u>at least</u> one-quarter diameter at the time of contact.

While the cooling shroud functioned as planned, the ability of the sonic system to prevent contact with the cage during and after deployment of the shroud was, of course, not demonstrated because the sample's wetting the platinum wire overrode the sonic positioning forces.

It is the writer's firm conviction that, despite the experiment's lack of total success, it was a valuable experience for all concerned and must be considered a major contribution to the technology of glass-making in space.

ACKNOWLEDGMENTS

The writer wishes to express his thanks to those in the Rockwell International organization who assisted in the post-flight evaluation portion of this investigation. Dr. L. E. Topol of the Environmental Monitoring and Service Center offered valuable suggestions in a consulting capacity. Dr. R. Housely of the Science Center performed the SEM work and is reponsible for the identification of the nucleation sites of the surface crystallites. Dr. M. D. Lind of the Electronics Research Center performed the X-ray diffraction analysis of the crystallite material. C. Parker did the still optical photography* of the specimen after removal from the injection cage. A. R. Sale was responsible for motion picture film processing and assisted the writer in analysis and interpretation of the flight motion pictures. C. N. Scully, the discoverer of the surface crystal rosettes, did the index of refraction measurements. The preceding three are all with the Space Operations and Satellite Systems Division of Rockwell.

In addition the writer held numerous valuable conversations with Intersonics personnel C. Ray, R. Whymark, and J. Yearnd.

Thanks are also extended to the members of the USRA Glass Committee, who have offered many valuable suggestions.

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*All photography in the preflight sections and the flight samples with the injection cage was performed by the writer.

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APPENDIX A

PRINCIPAL INVESTIGATOR'S "QUICK-LOOK" REPORT

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Space Division
12214 Lakewood Boulevard
Downey, California 90241

**Rockwell
International**

Nov 9, 1979

In Reply Refer to 79MA5121

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

Attention: David Schaefer, LA11

Subject: Experiment 74-42, Principal Investigator's "Quick Look" Report - SPAR VI - NAS8-32023

Launch Information

SPAR VI Launched at 6:40 a.m., MST - White Sands Missile Range New Mexico, October 17, 1979.

Payload returned to Missile Assembly Building at White Sands at approximately noon, October 17, 1979.

Flight experiment injection cage and specimen removed from the furnace - early afternoon, October 17, 1979.

Payload reported to have landed in a gully, toppled down-hill, slid over 30 feet into the gully before coming to rest. There was a large dent, with a fracture, in the casing surrounding the General Electric (G.E.) experiment at the upper and near the nose cone. Experiment 74-42 was the second from the nose cone. There were numerous scratches in the payload outer skin in the 74-42 section.

Flight Experiment Sample

The gallia-calcia-silica sample was found impaled on a fine platinum wire forming part of the wall of the outer injection cage of the flight hardware. The sample was clear, 100% glass, almost colorless, and appeared to be quite spherical in shape except for small projections caused by wetting the platinum wire at either end where it emerged from the sample. One or two small bubbles can be seen inside the glass sphere and in contact or close proximity with the platinum. Except for these, the sample appears entirely free from bubbles as determined by approximately 10X examination with a hand glass.

Flight Experiment Sample (cont'd)

The platinum wire appears to be very well centered in the sphere. The flight sample and the Pt cage, along with part of the injection mechanism, are in the possession of the writer.

Flight Motion Pictures

The flight film was signed over to the writer and returned to Los Angeles without exposing it to x-ray examination. It measured 159 feet in length. In order to cover the complete time line of the flight, the length would have to have been 149 feet. It was apparent, then, that the returned flight film contained all of the flight information, plus 10 feet of leader and ends.

The time line for the flight camera was as follows:

Event	Time,Sec.	Cumulative Time, Sec.
Camera on	0	0
Sample injection	5	5
Soak	155	160
Cooling shroud gate open		160
Cooling shroud insertion	10	170
Cooling	73	243
Sample retraction		243
Camera off		248

Camera Exposure Information:

-F/8, 140° shutter, 24 frames/second.

Flight Motion Picture Processing and Evaluation

A 21 foot section was cut from the film starting 42 feet (70 seconds) from the head end. The end of the cut was 45 seconds later. This piece is being stored for possible future image enhancement experiments. An additional length of the same type of film (EK Linagraph Shellburst 2476) was exposed outdoors using the manufacturer's ASA rating. This piece was developed along with the two pieces of flight film. Normal results were obtained.

Flight Motion Picture Processing and Evaluation (cont'd)

The flight film and a print were received on October 29, 1979, and examined using a photo-optical data analyzer.*

It was revealed that the specimen contacted the Pt wire, where it stuck, 860 frames (36 seconds) after sample injection. The specimen appeared to have centered itself on the wire 5 frames (0.21 seconds) later. The specimen touched the cage, while still solid, three times during the first 8.5 seconds after injection. It went through various motions without touching the cage for 27 seconds before touching and wetting the Pt wire. The sample appeared to be fully molten about 29 seconds after injection, or 7 seconds before it stuck to the wire.

When the cooling shroud gate opened near the tail end of the film the cage with the sample impaled on it could be seen clearly. It had cooled to a temperature where nothing more could be seen on the film (all black) 914 frames (38 seconds) after the cooling shroud gate opened.

Tentative Conclusions

Everything functioned as planned during the entire experiment with the exception that the specimen contacted and stuck to the platinum wire cage soon after it had melted. The following were accomplished successfully:

1. The furnace reached and held a high enough temperature to permit complete melting of the flight sample in the time available.
2. All mechanical functions appear to have been accomplished as planned. The only exception to this was that the specimen injection gate did not close completely (as shown by the telemetry data). This could have been caused by the failure of the injection cage to retract completely because of a clearance problem created by the specimen's projecting beyond the cage.
3. The flight specimen was recovered in excellent condition - was free of scratches and nicks and in good condition for completing the science evaluation.

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* L-W Photo, Inc. - Type 224-A T-V.

Tentative Conclusions (cont'd)

4. Flight motion pictures were properly exposed according to the Principal Investigator's (P.I.) pre-planning. No camera jamming or other malfunction appeared to have occurred. Resolution is good enough to permit detailed study of the sample's behavior in the sound field.

5. The experiment exceeded the science minimum success criteria established before flight and met the science objectives.

Further Work Required

During the next few weeks, the following work needs to be done before the Final Post Flight Analysis Report is prepared:

1. Experiment Equipment Function

   It is important that the exact cause of the specimen's contacting the platinum cage be understood so that corrective measures, if indicated, can be taken before the next flight experiment. This will involve:

   a. Further detailed study of the flight film by Intersonics, Incorporated.

   b. Careful comparison of the flight data obtained with accelerometer data to be provided by MSFC.

   c. Analysis of pertinent results of other experimenters on the same flight; especially those of G.E. and J.P.L., and comparison with the 74-42 flight information.

2. Flight Sample Evaluation

   The sample will be cut from the cage, after which the following is planed to be accomplished:

   a. Sample dimensions and degree of sphericity will be measured using photographic techniques.

   b. The index of refraction will be determined using immersion fluid techniques.

2. Flight Sample Evaluation (cont'd)

   c. An immersion fluid with matching index of refraction will be prepared and the interior of the sample will be examined for:

      1. Striae, and compared with pre-flight starting material.

      2. Stress using polarized light.

   d. Centering of the platinum wire will be measured using photographic techniques along with measurement of the wetting angles of the glass to the platinum.

If all of the above can be accomplished successfully, it will be unnecessary to cut or grind the sample and it can be preserved intact as a space artifact.

ROCKWELL INTERNATIONAL CORPORATION
Satellite Systems Division

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Experiment 74-42
Contract NAS8-32023

gk

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