RESEARCH STUDY ON MATERIAL PROCESSING IN SPACE SKYLAB EXPERIMENT M553 – SPHERE FORMING

FINAL REPORT

by

Philip C. Johnson, Edward T. Peters, Alfred E. Wechsler

December 1973

PREPARED UNDER CONTRACT NO. NAS8-28723

by

ARTHUR D. LITTLE, INC.
CAMBRIDGE, MASSACHUSETTS

for

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

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FOREWORD

This final report summarizes the results of the overall performance of Contract No. NAS8-28723. This effort was performed by personnel in the Materials Section, Physical Sciences Group and the Engineering Sciences Section for NASA-Marshall Space Flight Center.

The work was performed under the direction of Mr. E.A. Hasemeyer of NASA-MSFC, S+E-PE-MW.

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ABSTRACT

A research program was conducted to study the solidification of metals in the form of small spheres both in the one gravity environment of the earth laboratory and the low gravity environment of KC-135 trajectory flights and the Skylab 1/2 mission. The program had three phases.

PHASE A - A plan was formulated for ground-based studies. This included selection of potential metals and alloys, critical evaluation of the experimental design and procedures to point out areas requiring further refinement as well as theoretical analyses, and selection of the sphere characterization procedures to be utilized and evaluated in the ground-based studies.

PHASE B - Specimens of a number of alloys were melted in KC-135 trajectory flight and ground-based experiments. These were characterized to serve as a basis for the final selection of metals and alloys for the Skylab 1/2 mission. During melting of the ground-based specimens, thermal measurements were made and the data analyzed to provide information as to the thermal cycles experienced by the various alloys.

PHASE C - Six flight specimens were characterized. Arthur D. Little, Inc., prepared a summary report on the pure nickel specimens, based on observations on two specimens evaluated by Arthur D. Little and four pure nickel specimens evaluated by other M553 contractors. These were compared to the ground-based samples prepared in Phase B. Gravity effects were noted for this materials. The results of the evaluation of the other three samples (nickel alloys) were communicated to the appropriate contractors for preparation of their Phase C summary reports. Information was also communicated to the various contractors responsible for preparing discipline reports for all of the materials processing experiments performed on the Skylab 1/2 mission.

The details of the results of this program are contained in interim reports prepared at the conclusion of each of the three phases. This final report is intended to summarize the efforts and results described in detail in each of these interim reports, with particular emphasis on the differences observed between the ground-based and Skylab flight specimens.

I. INTRODUCTION

The apparatus for the M553 sphere forming experiment consisted of two 15-specimen indexing pinwheels mounted perpendicular to an electron beam heat source. The specimens were cylindrical with beveled edges, dimensioned so that when melted and resolidified, they would result in spheres 0.535 cm (0.250 inch) in diameter if fully dense. The first specimen on each wheel was a tungsten cylinder used to adjust the electron beam position and parameters. The subsequent 14 specimens were then rotated into position in turn and melted by the electron beam. The first three pecimens were mounted on large diameter supports (stings) such that they would melt and solidify but be retained on the sting. The final eleven specimens were mounted on aluminum oxide pedestal devices and spring loaded so that the specimens were detached upon melting of small diameter stings. These would then cool and solidify while floating in the work chamber.

The objectives of the M553 sphere forming experiment were to evaluate solidification behavior of metals and metal alloys in the absence of containers which could act as sites for heterogeneous nucleation; in the absence of gravity-induced fluid flow, either thermal or constitutional; and under conditions in which surface tension is the remaining primary driving force for fluid flow. Ideally, it was anticipated that solidification phenomena might be studied under conditions in which the specimens nucleated homogeneously and in which fluid flow and segregation due to gravity might be minimized.

II. PHASE A - GROUND-BASED STUDY PLANS

A. SUMMARY

The efforts and results of this portion of the program are detailed in the Phase A Interim Report.[1]

A series of metals and alloys which would provide a basis for selection of optimum materials for the Skylab mission were proposed for study in ground-based experiments. A number of meetings were held with personnel from NASA-MSFC and the various other contractors involved in the M553 program to define those aspects of the flight experiment which required further experimental or theoretical ground-based study to optimize the expected flight results. A list of those characteristics of the spheres expected to require evaluation was prepared, along with the most promising techniques for making those evaluations.

B. SELECTION OF CANDIDATE MATERIALS

Iron, nickel and cobalt-base alloys have been undercooled by as much as 300°C in the ground laboratory. The selected systems, listed below, were intended to permit the investigation of many of the factors that affect the way the solidification process occurs. All have similar thermal propoerties, simplifying the experimental requirements.

1. High Purity Nickel - This is representative of unalloyed metals having a single (invariant) melting point. Constitutional effects during solidification are minimized to the extent that the nickel is pure. It has well known thermal, magnetic, mechanical and structural properties. The problem of a surface oxide layer which might act as a nucleating site is minimal. Its melting point (as for the other materials selected) is low enough (1455°C) to permit efficient melting in the M553 apparatus, yet high enough to get rapid solidification and thus minimize problems of contact of free float samples with the chamber walls prior to solidification.

2. Nickel - 1 w/o Silver - This is representative of alloys having a narrow melting range (~5°C) and which core upon solidification, permitting relatively simple structural analysis for comparison with the pure nickel. The densities of the two elements are not greatly different.

3. Nickel - 5 w/o Aluminum - This is a companion to the nickel-1 w/o silver alloy in that it also has a narrow melting range (~10°C), but the two elements have a large density difference in the melt, permitting evaluation of convection effects in the melt by comparison with the alloys which do not have this density difference.

4. Nickel - 30 w/o Copper - This alloy has a fairly wide melting range (~50°C) but almost no difference in densities of the two elements. It is a continuous solid solution in the solid state.

5. Nickel - 12 w/o Tin - This alloy has a very wide (~250°C) melting range but very little difference in density between the two elements.

6. Commercial Alloys - One of the original objectives of the M553 materials processing experiment was to produce spheres having a very high as-solidified hardness. Two materials were considered:

   a) a complex cobalt-base alloys, Star J, a proprietary alloy of the Stellite Division of Cabot Corp., and

   b) 350T maraging steel.

These materials would allow investigation of solidification of systems which:

1. Include metals which have invariant, narrow and wide melting ranges, with a liquidus temperature between about 1245°C and 1455°C, simplifying the experimental procedure.

2. Include a pure metal and alloys which have both large and small density differences between the constituents.

3. Would allow investigation of the effects of low gravity on magnetic properties.

C. EXPERIMENTAL DESIGN

In Phase A, calculations were made relative to the source and magnitude of the expected forces on the spheres during the experimental cycle. These are summarized in Table I. In addition, it was concluded that it was desirable to conduct preliminary temperature measurements in ground-based tests to determine optimum heating parameters for the specimens, and to determine if a color-temperature calibrated film or other suitable techniques could be incorporated into the M553 experiment with no significant hardware changes. These efforts were conducted during the Phase B portion of this program and are summarized in the Phase B section of this final report.

D. SPECIMEN EVALUATION

For the purposes of assessing the differences between ground-based and flight specimens, the specimen characteristics listed in Table II were proposed. The techniques utilized are detailed in the interim reports. Some evaluations were the responsibility of NASA-MSFC and others of Arthur D. Little, Inc., and are so indicated in Table II. A critical evaluation of the utility and sensitivity of the various characterization

TABLE I

FORCES ON SPECIMENS AND THEIR EFFECT ON VELOCITIES AND IMPACT TIMES

TYPE	FORCE (Dynes)	DURATION (Seconds)	VELOCITY* (cm/sec)	IMPACT TIME (Seconds)
ELECTRON BEAM	4.6	0.1	0.4	57
VAPORIZATION				
Nickel	1.3-29	<2	1-24	20-1.4
Nickel-Tin	10-116	<2	8-97	3.5-<1
RESIDUAL G	0.01-0.1	Entire	0.02-.2	60-20
PHOTON	10^{-3}	<2	.002	>60
SPRING	?	Short	5?	5?
SURFACE TENSION GRADIENT	?	<2	?	?

TABLE II

SPECIMEN EVALUATION

1. Visual Examination - Photography (ADL and MSFC)
2. Internal Quality (Macrovoids) - Radiography (MSFC)
3. Preferred Orientation by Laue X-ray Mapping (ADL)
4. Magnetic Properties (MSFC)
5. Sphericity (ADL and MSFC)
6. Density (ADL and MSFC)
7. Topography - SEM (ADL and MSFC)
8. Surface Topography after Light Etch (ADL)
9. Internal Structure - Metallography (ADL)
   a. Microsegregation
   b. Macrosegregation
   c. Dendrite Arm Spacing
   d. Second Phase Distribution and Identification
   e. Grain Size and Morphology
10. Microhardness (ADL)
11. Microchemical Analysis and Survey (ADL)
12. Bulk Chemistry (ADL)

procedures and techniques are described in detail in the Phase B and Phase C Interim Reports submitted by Arthur D. Little, Inc.[2,3] Commentary on the utility of selected evaluations and characterization techniques are summarized at the conclusion of the section dealing with Phase C of this final report. Not all of the suggested evaluations were actually employed on the samples.

III. PHASE B - GROUND-BASED EXPERIMENTS

A. SUMMARY

The efforts and results of this portion of the program are detailed in the Interim Reports on evaluation of specimens processed in KC-135 trajectory flights and the M512 apparatus at NASA-MSFC.[2,4]

Various metals and alloys which had been melted in the earth laboratory and on KC-135 trajectory flights were evaluated in order to select and refine the characterization techniques and the choice of alloys for the Skylab mission. The ground-based samples served as comparisons for those melted in low gravity. Thermal measurements and calculations were made to predict the expected heating and cooling cycles of the specimens under various conditions.

B. SELECTION OF MATERIALS FOR THE FLIGHT EXPERIMENTS

On the basis of KC-135 trajectory flights and ground-based experiments, the metal and metal-alloy systems selected in concurrence with NASA-MSFC and the other contractors associated ith the M553 experiments were:

1) pure nickel
2) nickel - 1 w/o silver
3) nickel - 12 w/o tin
4) nickel - 30 w/o copper

The nickel - 5 w/o aluminum alloy proved to be unacceptable due to excessive vaporization of aluminum. The alloys which were intended to have high hardness in the as-cast condition (Stellite "Star J" and 350T maraging steel) proved to be extremely complex systems for which solidification phenomena were extremely difficult to interpret in the context of the differences between one gravity and low gravity solidification.

C. EXPERIMENTAL DESIGN - TEMPERATURE MEASUREMENTS

1. Introduction and Techniques

Several series of ground-based experiments for the M553 Sphere Forming Experiment were conducted to demonstrate the operation of the experiment's hardware to obtain data for the refinement of the flight experiments and to obtain specimens which could be used as comparisons for specimens processed in space. Theoretical studies by several investigators suggested that it would be desirable to know the temperature distribution of the specimens during and after the melting process. These data were useful in determining vaporization losses, estimating surface tension effects and understanding the release and movement of the specimens.

The objectives of this work were to obtain an indication of maximum and average surface temperature of the various M553 experiment specimens during the melting process and to determine the initial cooling rates of the specimens when the electron beam was turned off. The temperature measurements were of secondary importance to the preparation of ground-based specimens and had to be conducted on a non-interfering basis. Thus, optimum temperature measurement techniques could not be used; rather they were more of an expedient and exploratory nature.

The M553 experiment was set up in the M512 facility in a manner similar to that used in previous ground-based tests. The facility and specimens duplicated most of the conditions of the flight experiment; a principal difference was the vertical orientation of the specimens in the ground-based experiment. This permitted the "released specimens" to remain on the ceramic support after melting occurred, rather than having them release and fall under the action of gravity.

The melting procedure was similar to that anticipated in the flight experiments; however, the chamber was opened to permit cleaning of the viewing window after groups of four or five samples had been melted in sequence. Cleaning was necessary because of vaporization of the specimens during melting and deposition of a condensed film on the interior of the viewing window. The film made it difficult to view and photograph the specimens and, as described later, to obtain accurate temperature measurements. Specimens were collected when the chamber was opened and when tests on the first pinwheel were completed. A second duplicate pinwheel was installed and the above sequence was repeated.

Temperature measurements were made with a Micro-Optical pyrometer. The pyrometer was placed horizontally at a distance of approximately 24 inches from the specimen; a rear surface mirror was used to deflect the viewing beam onto the top of the specimen. The pyrometer was focused on the center top of the specimen, at approximately 90° to the impingement of electron beam. The filament of the optical pyrometer lamp was superimposed on the center third of the specimen. Thus, the temperature that was measured can be best described as an "average surface temperature of the top portion of the specimen." As each specimen was melted, attempts were made to obtain a measurement of the maximum upper surface temperature of the melted specimen. Manual optical pyrometer measurements were made by matching the pyrometer filament brightness (color) with that of the specimen. This was done by setting the pyrometer filament temperature (brightness) at a value near the expected melting temperature for each specimen, and rapidly adjusting the filament brightness to achieve a color match in the one to two second period in which the specimen was melted. After this temperature was recorded, the pyrometer filament temperature (brightness) was readjusted to indicate 100 to 200°C lower than the highest temperature observed and the time required for the specimen to cool to this temperature was determined. Thus, an indication of the cooling rate was obtained.

Temperature measurements for each specimen were made in this manner; however, due to the condensation of films on the chamber window and the rapid temperature changes during the melting and cooling processes, the measurements could not be made easily and reliably, and for some specimens we were unable to achieve adequate color matches and good temperature measurements.

The presence of the chamber window, external mirror, and the condensed film on the chamber window were sources of measurement error and required that a special calibration procedure be adopted during the measurements.

A discussion of these calibration procedures is necessary to a better understanding of the results and possible errors in measurement, and is detailed in the Phase B Interim Report in this program.[2]

The errors inherent in the measurements are:

E_{pyro} = error in pyrometer instrument temperature measurement

E_{window} = error caused by optical absorption of the window

E_{mirror} = error caused by incomplete reflection and absorption of the mirror

E_{film} = error caused by absorption of the film on the window

$E_{emittance}$ = error caused by emittance of melted specimen being different from unity

To summarize the method and calibration used, the maximum average upper surface temperature of the specimens was measured as they were melted, and corrections were added for window film error, window/mirror error, and pyrometer instrument error to obtain the best estimate of the specimen temperature. The uncertainty in the specimen temperature is estimated to be as much as 100°C and is attributable primarily to the uncertainty in the amount of film present on the viewing window during the measurement of each specimen. In addition, the measurements may be biased by 50 to 100°C on the low side because of sample emittance effects.

2. Results and Discussion

a. Qualitative Visual Measurements

Notes were taken during the melting of most of the specimens; the following general remarks summarize our observations.

The specimen temperatures seemed to be much higher (several hundred degrees by visual estimate) near the impingement of the electron beam than at other points on the specimen. It was not possible to measure these other temperatures with the optical pyrometer because of the high

localized brightness. These hot temperature zones seemed to decrease very rapidly with distance from the impingement area so that most of the specimen came to a more uniform temperature (to within about 100-150°C) as the specimen became molten. For some samples, melting was observed to occur locally near the impingement of the electron beam, and then proceed across the specimen, with a well-defined temperature gradient between the molten and unmelted zones. After the electron beam was turned off, the temperature of the molten specimen appeared to equalize rapidly, so that when surface solidification began to occur, temperature gradients were not very large. Specimens which remained on the stings were observed to cool more quickly than others; specimens which rested on the ceramic support cooled slowly and had a nearly uniform temperature distribution throughout during the latter stages of cooling. Except at the location of electron beam impingement, superheating of the specimens was not very great, perhaps 50-100°C above the melting point.

b. Quantitative Temperature Measurements

- Specimen "Average" Temperatures

Table III presents the "average" specimen temperatures measured as soon as possible after turnoff of the electron beam. The values given include the corrections for all the errors other than the specimen emittance error.

- Cooling Rates

Average cooling rates were measured for the majority of the specimens. The values obtained should be subject to smaller errors than the absolute measurements because some of the errors (window, pyrometer and emittance) tend to cancel; that is,

$$\frac{dT}{dt} = \frac{T^{(1)}-T^{(2)}}{\Delta t} = \frac{1}{\Delta t}\left(T^{(1)}_{measured}-T^{(2)}_{measured}-(E^{(1)}_{pyro}-E^{(2)}_{pyro})-(E^{(1)}_{WM}-E^{(2)}_{WM})-(E^{(1)}_{film}-E^{(2)}_{film})\right)$$

Errors caused by window fogging may not cancel because of the vaporization and coating of the windows during measurements. The cooling rates indicated on Table IV represent an average cooling rate, i.e., measurements were made of the time required for a sample to cool from some temperature which was above or near the melting point to a temperature several hundred degrees lower. Thus, this cooling rate includes some surface solidification and cooldown.

TABLE III

MAXIMUM "AVERAGE" SPECIMEN TEMPERATURES

Specimen			Temperature (°C)		
No.	Material	Type	Expected Melting Range	Pinwheel No. 1	Pinwheel No. 2
1	Tungsten	Sting	--	--	--
2	Nickel	Sting	1450	1500	1500
3	Nickel-12% Tin	Sting	1220-1370	1410	<1555
4	Nickel-1% Silver	Sting	1450	1380	----
5	Nickel	Release	1450	1415	1380
6	Nickel	Release	1450	1445	1390
7	Nickel-12% Tin	Release	1220-1370	1390	1350
8	Nickel-1% Silver	Release	1450	1270	1425
9	Nickel-30% Copper	Release	1330-1380	1260	1355
10	Nickel-30% Copper	Release	1330-1380	1180	1315
11	Nickel-1% Silver	Release	1450	1320	1390
12	Nickel-12% Tin	Release	1220-1370	1280	1390
13	Nickel	Release	1450	1325	1400
14	Nickel-12% Tin	Release	1220-1370	1330	1340
15	Nickel-1% Silver	Release	1450	1375	1410

Note: ---- indicates when viewing window was cleaned

TABLE IV

"AVERAGE" COOLING RATES

Material	Specimen No.	Initial & Second Temperatures (°C)		Time Between Temperature Measurements (sec)	Cooling Rate (°C/sec)
Nickel	1-6	1445	1030	14	30
Nickel	1-13	1325	925	8	50
Nickel	2-2	1500	1140	10	36
Nickel	2-5	1380	980	15	27
Nickel	2-13	1400	1150	10	25
Nickel-1% Silver	1-15	1375	1095	12	23
Nickel-1% Silver	2-8	1425	1310	10	12
Nickel-1% Silver	2-11	1390	1210	7.5	24
Nickel-1% Silver	2-15	1410	1270	7	20
Nickel-12% Tin	1-12	1280	865	20	21
Nickel-12% Tin	1-14	1330	1060	18	15
Nickel-12% Tin	2-3	<1555	1075	17	<28
Nickel-12% Tin	2-7	1350	1170	15	12
Nickel-12% Tin	2-12	1390	1130	13	20
Nickel-12% Tin	2-14	1340	1165	14	13
Nickel-30% Copper	1-9	1260	905	12	30
Nickel-30% Copper	2-9	1355	1105	11	23
Nickel-30% Copper	2-10	1315	1005	9.5	33

The cooling rates for the nickel were generally the highest, ranging from 25 to 50°C/sec. These rates were higher than others, presumably because the higher average temperature gives a greater radiation heat loss and perhaps because of the higher emittance of the specimen. The nickel-1% silver specimens had cooling rates of 20 to 24°C/sec, with one value (Specimen 2-8) considerably lower. (Window fogging may have been significant in this specific test.) The nickel-12% tin specimens had cooling rates of 12-21°C/sec with one value (Specimen No. 2-3, sting-type) considerably higher. This higher rate may be the result of conduction down the sting. The nickel-30% copper cooling rates were in the range of 23-33°C/sec, higher than the other alloys, possibly due to the higher conduction in the specimens. These results indicate that cooling to the point of surface solidification occurs within several seconds after the electron beam is turned off.

D. KC-135 AND GROUND-BASED SPECIMEN EVALUATIONS

1. KC-135 Trajectory Specimens

Two specimens were evaluated from among those melted during the KC-135 trajectory flights of the week of 12 July 1972. The details of these evaluations are given in a separate report.(4) Specimen D was a Ni-Sn specimen that was only partially melted. Specimen 24 was a small portion of a Star J cobalt alloy specimen which had broken up into a number of droplets upon melting. Neither specimen exhibited solidification behavior which could be attributed to low gravity. The primary purposes of these experiments were to refine the hardward and techniques, develop specimen evaluation methods, and to make decisions as to which alloy would be melted on the Skylab 1/2 mission. As has been mentioned above, the Star J alloy was deleted from the list of candidates because it is an extremely complex system. The nickel-5w/o aluminum specimens were evaluated by other contractors, and this alloy was deleted from the list of candidates because of excessive vaporization of the aluminum.

E. EVALUATION OF GROUND-BASED SAMPLES

Five samples were received which had been processed in the M512 facility on the ground. The sample numbers, nominal composition, and specimen types submitted to ADL were:

Specimen No.	Composition	Type
2-2	Ni	Captive
2-6	Ni	Release
2-8	Ni-1 Ag	Release
2-9	Ni-30 Cu	Release
2-14	Ni-12 Sn	Release

Several unusual solidification features were observed on some of these samples. These are more appropriately discussed in the next section of this report, dealing with Phase C, so that direct comparisons can be made with flight specimens.

IV. PHASE C - COMPARISON OF FLIGHT AND GROUND-BASED M553 SPECIMENS

A. SUMMARY

Five specimens melted on the Skylab 1/2 mission were evaluated and compared to the ground-based specimens. Arthur D. Little, Inc., prepared a Phase C summary report detailing the results and differences between all the pure nickel ground-based and flight samples, and communicated its results of evaluations of three of the nickel alloy flight samples to the contractors responsible for each of the three respective nickel alloy Phase C summary reports. Conclusions on thermal analysis, metallography, chemistry, physical properties, solidification theory and physical forces were communicated to those contractors responsible for preparing the discipline reports on these subjects.

In this section, only a summary of the more important differences between the ground-based and flight samples will be presented, since these evaluations have been described in detail in the various Phase C reports prepared by the M553 program contractors.

Pure nickel; Arthur D. Little, Inc., Ref. 3
Nickel-1w/o silver; University of Connecticut, Ref. 5
Nickel-12w/o tin; Grumman Aerospace Corp., Ref. 6
Nickel-30w/o copper; Georgia Institute of Technology, Ref. 7

Five samples process on the Skylab 1/2 mission were evaluated by Arthur D. Little, Inc.:

SL-1.4	Nickel-12w/o tin
SL-1.12	Nickel-1w/o silver
SL-2.2	Pure Nickel
SL-2.6	Pure Nickel
SL-2.7	Nickel-12w/o tin

B. PRINCIPAL DIFFERENCES BETWEEN FLIGHT AND GROUND-BASED SAMPLES

1. Independent Surface Nucleation and Growth Events

Localized areas of two-dimensional structure were observed for many of the specimens and for at least one specimen of each of the four materials. These areas generally occurred at the end of the specimen opposite the sting and represented a variety of morphologies. They usually represented areas of much finer structure than the remainder of the specimen surface. It has been postulated that these were regions which nucleated and grew independently as a result of radiational heat loss. In the case of the alloys, this cooling was abetted by vaporization of the alloying elements. For the alloys, these "cap" regions were similar in both the ground and flight samples. Typical examples for the alloys, all ground samples, are shown in Figures 1a (nickel-12w/o tin), Figure 1b (nickel-30w/o copper) and Figure 2a (nickel-1w/o Ag, in cross section). In the case of the nickel-1w/o silver specimen, considerable coarsening of the cap

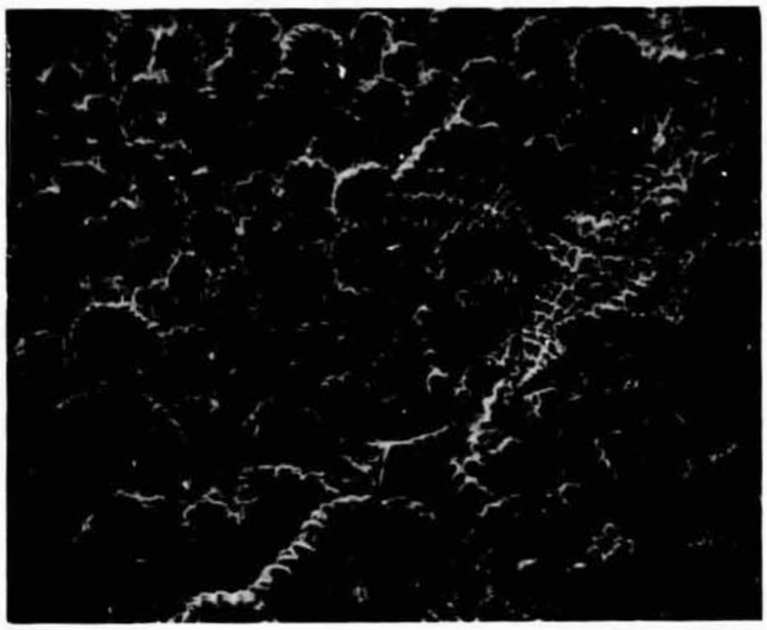

Figure 1a Cap Area on Surface of Nickel-Tin Ground Specimen 2-14
SEM 115X

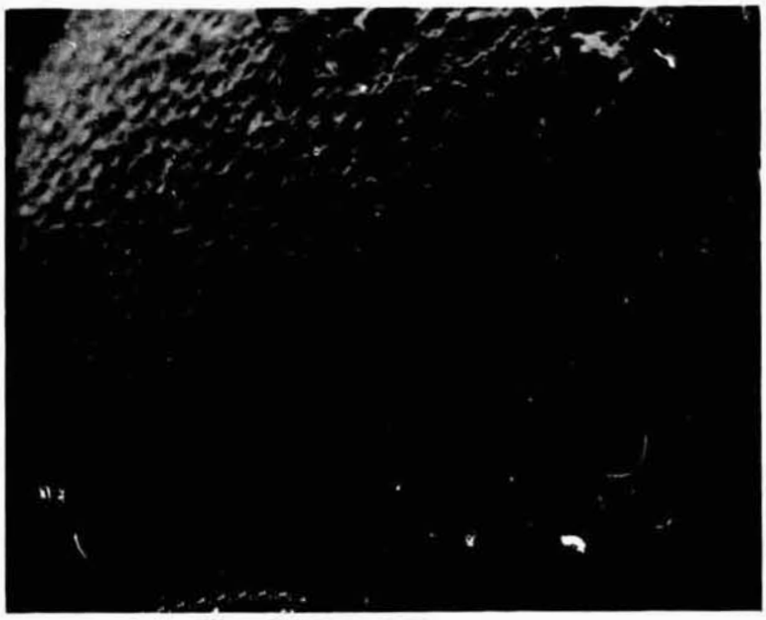

Figure 1b Cap Area on Surface of Nickel-Copper Ground Specimen 2-9
SEM 50X

Figure 2a Cross Section of Nickel-Silver Ground Specimen 2-8 Showing Cap 12.7X

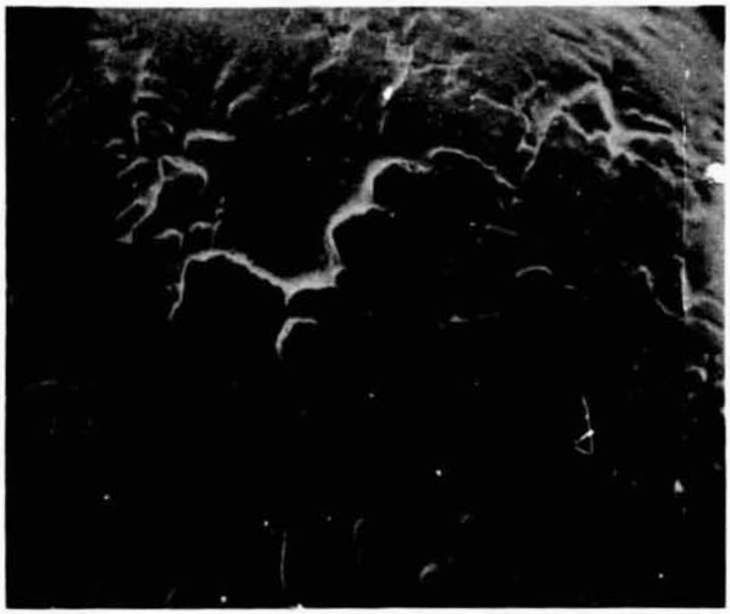

Figure 2b Typical Two Dimensional Surface Dendrites on Pure Nickel Ground Specimen 2-6 SEM 25X

region has occurred which was not evident in the topographical examination. Shown in Figure 2b is a typical surface topography for a pure nickel ground sample, consisting of relatively large two-dimensional dendrites. However, three out of the six pure nickel flight specimens evaluated had localized areas of much finer structure, similar to that observed on both the ground and flight alloy specimens but not observed on any of the pure nickel ground specimens. Two of these specimens were evaluated by Georgia Institue of Technology. Specimen SL-1.9 has a cap consisting of a mixture of fine equiaxed grains surrounded by a ring of fine dendrites. Specimen SL-2.5 appears to have a cap consisting entirely of extremely fine dendrites. Specimen SL-2.6 was evaluated by Arthur D. Little, Inc., and has a cap consisting entirely of fine equiaxed grains, Figures 3a and 3b. It has been postulated that these fine grain areas are chill areas resulting from loss of radiant heat leading to independent surface nucleation. Despite the flight film evidence of considerable turbulence and fluid flow, it may have been enough less than that for the ground specimens to permit this phenomenon to occur on three out of the six pure nickel flight samples.

2. Bulk Macroporosity

Bulk or internal macroporosity was observed by Arthur D. Little, Inc., on two of the alloy flight specimens on a scale much greater than that observed on any of the ground specimens. Cross sections of a nickel-1w/o silver specimen (SL-1.12) and a nickel-12w/o tin specimen (SL-2.7) are shown in Figures 4a and 4b. This has been attributed by the Grumman Aerospace Corporation (Ref. 6) to non-equilibrium solidification with respect to pressure such that a gas phase is formed which leads to large spherical voids. Terrestrially, the large density difference between the liquid and the vapor would result in the vapor being emitted. However, in zero gravity there is no such density difference and the vapor is trapped.

C. CRITIQUE OF EVALUATION TECHNIQUES

The majority of the evaluation techniques used to make the specimen evaluations listed in Table II provided useful information. However, some proved less useful or provided ambiguous results, and comment on these is included here for the purposes of future similar experiments which might be performed.

1. Preferred Orientation by Laue X-ray Mapping

The depth of penetration of the beam (approximately 0.10 mm) was too great to analyze the majority of the surface effects seen. Moreoever, the Xrays in most cases reflected recrystallized structures in the bulk of the sample rather than the original solidification structures.

Figure 3a Fine Equiaxed Grain Cap on Pure Nickel Flight Specimen SL-2.6 25X

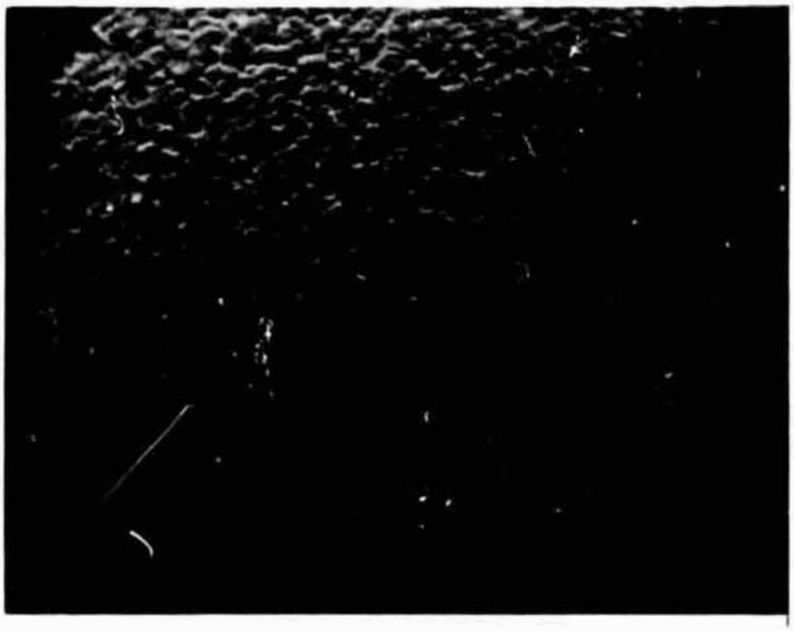

Figure 3b Fine Equiaxed Grain Cap on Pure Nickel Flight Specimen SL-2.6 120X

Figure 4a Gross Macroporosity in Nickel-Silver Flight Specimen SL-1.12 10X

Figure 4b Gross Macroporosity in Nickel-Tin Flight Specimen SL-2.7 10X

2. Internal Porosity by Radiography

The shape of the samples made it very difficult to obtain useful radiographic information except in the few specimens having very large pores.

3. Density

Considerable difficulty was encountered in obtaining meaningful or reproducible density measurements. A part of this is ascribed to the technique used by Arthur D. Little, Inc., (differential weight in air and water). Additional problems were undoubtedly the results of sample porosity in which a path reached the surface. Some of these pores filled with fluid and others did not. Pycnometry, utilized by some of the other contractors, appears to have provided somewhat better results, but the problem of surface-fed porosity remained a problem.

V. REFERENCES

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