PREPARATION OF AMORPHOUS FERROMAGNETIC MATERIALS THROUGH CONTAINERLESS SOLIDIFICATION

Contract No. NAS8-31972

FINAL REPORT

January 27, 1978

Prepared for

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

Prepared by

GENERAL ELECTRIC COMPANY
Space Sciences Laboratory
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Abstract

Reported herein is the ground based research, the work in preparation for the flight experiment, the results of the flight experiment, and the failure analysis for Experiment 74-49, "Preparation of Amorphous Ferromagnetic Materials through Containerless Solidification."

The experiment was flown on the NASA SPAR IV sounding rocket in the Electromagnetic Containerless Processing Payload. Due to a failure to melt, the experiment hypothesis was not tested on this flight.

1.0 INTRODUCTION AND SUMMARY

The first strongly ferromagnetic metallic glass was reported in 1967[1]. It was the alloy $Fe_{83}P_{10}C_7$. The preparation and characterization of many other ferromagnetic metallic glasses has since been described[2, 3, 4]. These generally have a chemical composition of the form $(T'_{1-x}T''_x)_{80}(M'_{1-x}M''_x)_{20}$, where T' and T'' are metals such as Ni, Co, Fe, Pd, Cu and M' and M'' are metalloids such as Si, C, P or B. Typical of this form is the ferromagnetic metallic glass having the composition $Fe_{40}Ni_{40}P_{14}B_6$ (Allied Chemical's METGLAS 2826). The amorphous metallic alloys (metallic glasses) were first prepared by SPLAT cooling[5], then by roller quenching[6], and finally (for a few selected compositions) by quenching in water[7]. The first two methods require very high quenching rates (10^4 to 10^6 °C/sec). The last method utilizes modest quenching rates (10^2 to 10^3 °C/sec).

In light of the above considerations, it was hypothesized that it might be possible to prepare ferromagnetic metallic glasses by containerless cooling at slower cooling rates yet ($< 10^2$ °C/sec), due to elimination of nucleation sites present with container walls. Experiment 78-49 was to test this hypothesis by melting a specimen of METGLAS 2826 composition in the NASA Electromagnetic Containerless Processing Payload (ECPP) during the NASA SPAR IV sounding rocket flight and allowing it to cool while levitated. Due to a failure to melt the specimens, for reasons discussed below, the experiment was not performed and the hypothesis was not tested on this flight.

Ferromagnetic metallic glasses (amorphous ferromagnetic alloys) are "soft" magnetically. They can be magnetized very easily due to low coercive force and high permeability. They also share with other non-ferromagnetic metallic glasses the properties of high strength and hardness. These desired

properties arise from the lack of gross crystallinity and homogeneous structure, both effects, for example, allowing domain walls to move easily. These materials are, hence, interesting from both a scientific and technological viewpoint.

The objectives of the sounding rocket experiment were: (1) to obtain a large amount of supercooling of a containerless melt of composition $Fe_{40}Ni_{40}P_{14}B_6$ (in atomic percent); (2) to prepare a bulk specimen of the ferromagnetic glass of this composition by supercooling to the glass temperature, Tg, thus bypassing crystallization. An alternative objective, in case (1) was achieved but not (2) was: (3) to examine the crystallization of a (possibly) highly supercooled, containerless melt of this composition.

The choice of the METGLAS 2826 composition was made for the following reasons: (1) examination of the many candidate compositions indicated that the prospects for success were good, because of the high reduced glass temperature (ratio of glass transition temperature to melting temperature); (2) the ferromagnetic metallic glass produced by roller quenching in the form of a "tape" has been well characterized; it has not yet been prepared in bulk form, either by water quenching or any other technique.

As reported, the experiment was not performed due to a failure to melt the specimen. The work reported herein, then, consists of: (1) the ground based experiments; (2) the analysis of the ground based specimens; (3) the flight experiment; (4) failure to melt analysis; (5) recommendations and conclusions. The ground based experimental work yielded new results in terms of understanding the visco-elastic properties of these glasses and the variation of viscosity through the glass transition temperature from metallic glass to crystalline solid[6]. This last result is of importance in predicting what glasses can be produced at lower quench rates ($\sim 10^2$ °C/sec) and hence will be discussed further below in greater detail.

2.0 GROUND BASED EXPERIMENTS

2.1 SPECIMEN SELECTION

A survey was made of the known metallic glasses. The results of the survey are shown in Appendix A. Compositions selected for further investigation in our laboratory are shown in Table I. A very important parameter for successful production of a metallic glass is the reduced glass temperature. A reduced glass temperature above 0.5 was recommended (by Dr. David Turnbull of Harvard University, who consulted on this experiment with the Principal and Co-Investigators). METGLAS 2826 with a melting temperature of 1223°K (approximately) and a glass transition temperature of 693°K (approximately) has a reduced glass temperature of 0.57, satisfying the above criterion. Hence, along with the fact that it was the most studied ferromagnetic metallic glass, it was selected, for the above reason, as the flight composition.

2.2 SPECIMEN PREPARATION

Techniques for preparing specimens of selected compositions for ground based experiments were examined. These included: (1) melting the elemental powders; (2) vacuum hot pressing; (3) vacuum melting and casting using glass tapes as the starting material. This last technique was adopted for the preparation of ground based and flight specimens. Pieces of METGLAS 2826 glass tape were compacted in a crucible and vacuum melted to produce specimens approximagely 0.922 centimeters in diameter. The specimens produced were crystalline in nature. The objective was to melt these and by containerless cooling produce a bulk ferromagnetic glass specimen.

2.3 BREADBOARD EXPERIMENTS

Experiments were conducted in the Breadboard Facility with the following objectives: (1) to quantify the performance of the Breadboard and the Flight Unit; (2) to determine the specimen preparation procedures to be used in fabricating

Table I. Compositions Selected for Laboratory Investigations with Melting Points

Composition	T_ℓ (°C) ±20°	T_g
$Fe_{32}Ni_{36}Cr_{14}P_{12}B_6$ (2826A)	940*	paramagnetic at R. T.
$Fe_{78}Mo_2B_{20}$ (2605A)	885	420
$Fe_{29}Ni_{49}P_{14}B_6Si_2$ (2826B)	940*	
$Fe_{40}Ni_{40}P_{14}B_6$ (2826)	955	410
$Co_{72}Fe_3P_{16}B_6Al_3$	950	
$Ni_{60}Fe_{20}B_{20}$	940*	
$Fe_{70}Co_{10}B_{20}$	940*	
$Fe_{83}B_{17}$	1050*	

The starred values listed for melting points T_ℓ and glass temperature T_g were measured by the authors.

the flight specimen; (3) to specify the chamber preparation, specimen insertion, gas purification, and filling procedures for the flight experiment; (4) to conduct specimen melting studies. It was recognized at the outset that, whereas the Breadboard Facility was designed only for the purposes of testing the conceptual design of the ECPP, its performance would be different from that of the ECPP. The functional operation of the experiment could be tested in the Breadboard Facility, and, with the proper scaling ratios, estimates of the melting time of the specimens in the selected gaseous environments could be predicted for the flight experiment in the ECPP.

Breadboard calibration tests were performed in Argon, using the same #316 stainless steel as used in the flight unit (ECPP) tests, discussed below. A calibration factor between the performance of the breadboard and the ECPP with regard to heating was determined. This is discussed below in the failure analysis, where the experiment log summary is given.

Breadboard tests were conducted with specimens of the METGLAS 2826 composition, having an average diameter of 0.922 centimeter diameter. Heating and melting tests were conducted in 1 atmosphere Argon, 10 torr Argon, 1 atmosphere Helium, 10 torr helium, 800 torr of 50% He and 50% A mixture, 100 torr Helium.

Analysis of the data from these tests showed: (1) that the specimen would not cool in vacuo to 400^{o}C within the 200 seconds allotted for the experiment; (2) that the specimen would not melt in pure helium in the flight unit (using the calibration factor); (3) that the specimen would melt and cool within 200 seconds in a helium-argon mixture having the molar ratio 0.65 He to 0.35 argon. Accordingly this mixture was adopted for the flight experiment.

2.4 GROUND BASED EXPERIMENTS AT DREXEL UNIVERSITY

The research program at Drexel University, conducted by the Principal Investigator, had these objectives: (1) the preparation of metallic glasses; (2) the characterization of their properties; (3) devising testing techniques to characterize the ground based and flight specimens (which are not tapes but bulk specimens, spheroidal in shape). Research was conducted in the following areas: (1) the effects of hydrostatic pressure on the magnetic properties of metallic glasses such as METGLAS 2826[5]; (2) the glass-crystalline phase transition and properties at the transition of such metallic glasses[6]; (3) acoustic emission generated during viscous flow of metallic glasses[7]. This work has great relevance in predicting what magnetic glasses can be produced in bulk form as proposed. In particular it was found[6] that the viscosity at the glass transition temperature (Tg) of METGLAS 2826 is about 10^8 poise, which is an order of magnitude lower than observed at the Tg for non-ferromagnetic metallic glasses[8, 9] such as $Pd_{77.5}Si_{16.5}Cu_{6.7}$. This indicates that an important factor is this viscosity as well as the reduced glass temperature. This finding came late in the research work and is new and unexpected. It may well be that both a high reduced glass temperature and a high transition viscosity are required to prepare a bulk specimen of ferromagnetic glass by the technique proposed. This will be considered before the following flights. During this work, the Pd Si Cu metallic glass mentioned above was routinely prepared in bulk form in the laboratory.

The following characterization techniques were established to investigate the properties of the ground based and flight specimens: (1) Magnetic Measurements. A vibrating reed magnetometer was constructed to characterize the magnetic properties of the specimens produced. It was tested with nickel and crystalline alloy prepared by crystallizing METGLAS 2826 tape. B-H curves through a closed magnetic loop were also obtained and the technique perfected for the

ground based and flight specimen geometries, also an induction method was developed for use with the long thin tapes and also for use in certain measurements of the bulk samples; (2) X-Ray Characterization. Back reflection and diffractometry were performed both at Drexel University and GE on METGLAS 2826 tape, crystalline tape, and the ground based specimens. This would have served as the comparison between the flight specimen produced and the glass tape produced terrestrially; (3) Differential Scanning Calorimetry. Phase transitions in METGLAS 2826 were investigated using differential scanning calorimetry. This technique again would have served as a comparison between the terrestrially produced tape and the flight specimen produced; (4) metallographic work was performed on the terrestrially produced tape, the ground based specimen, and the unmelted flight specimen (discussed below). This included electron probe microanalysis.

Preparation for examination of the flight specimen was completed prior to launch. The post flight analysis showed that the specimen had not melted, but was near the melting point. Hence the post flight examination could have no bearing on the experiment hypothesis.

2.5 FLIGHT EXPERIMENT

Analysis of the flight experiment data indicated that the specimen was not melted during the flight. The input power to the specimen was too low (see failure analysis below) and the specimen load was not matched properly to the r.f. tank circuit. Metallographic analysis confirmed this failure to melt. The specimen had reached a temperature near melting and incipient melting had begun on the surface but had not progressed inward during the experiment.

3.0 FAILURE TO MELT

A memo, appended to this report, was issued August 30, 1977, explaining the failure to melt the specimen during flight. Appended also to this report is a memo on the Ground Based Preparations for Experiment 74-49, which constitutes a summary of the log book records in preparation for the flight. There has been no change in the conclusions reached in the failure analysis presented since that time.

CONCLUSIONS

The testing of the hypothesis that bulk specimens of ferromagnetic metallic glasses may be produced by containerless cooling was not performed on the flight experiment 74-49. Further flights should utilize a quench gas (helium) introduced after melting to rapidly cool the levitated specimen. The test documentation should be improved to insure that the procedural error committed in final adjustment of the flight equipment is not repeated. Careful attention should be paid to the fabrication of the flight specimen as discussed in Appendix A. Testing of the performance of the flight unit (ECPP) should be conducted with a flight specimen rather than #316 stainless steel specimens.

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APPENDIX A

RESULTS OF LITERATURE SEARCH ON GLASS FORMING COMPOSITIONS

Alloy	Tg (°C)	Ref.	Tx	Ref.	Tℓ	Ref.
$Fe_{39.8}Ni_{40.6}P_{14}B_6$	404	1	412	1		
$Fe_{41.3}Ni_{40.4}P_{13.7}B_6$	408	1	418	1		
$Fe_{40.1}Ni_{39.1}P_{12.2}B_6$	404	1	417	1		
$Fe_{38.8}Ni_{38.1}P_{12.1}B_6$	408	1	418	1		
$Fe_{40.2}Ni_{38.2}P_{.5}B_{20}$	442	1	451	1		
$Fe_{51.8}Ni_{30.5}P_{14.7}B_6$	418	1	423	1		
$Fe_{50}Ni_{30}B_{20}$	444	1	455	1		
$Fe_{73}P_{18}B_6Al_3$	---	2	415	2		
$Fe_{75}P_{16}B_6Al_3$	---	2	427	2		
$Fe_{77}P_{14}B_6Al_3$	---		445	2		
$Fe_{79}P_{12}B_6Al_3$	450	2	445	2		
$Co_{73}P_{18}B_6Al_3$	---	2	479	2		
$Co_{75}P_{16}B_6Al_3$	---	2	399	2		
$Co_{77}P_{14}B_6Al_3$	---	2	381	2		
$Co_{79}P_{12}B_6Al_3$	---	2	392	2		
$(Co_{80}Fe_{20})_{75}P_{16}B_6Al_3$	470	2	477	2		
$(Co_{60}Fe_{40})_{75}$ " "	457	2	479	2		
$(Co_{40}Fe_{60})_{75}$ " "	456	2	472	2		
$(Co_{20}Fe_{80})_{75}$ " "	462	2	452	2		
$Fe_{75}P_{16}B_5Al_3$ " "	---		427	2		

Alloy	Tg	Ref.	Tx	Ref.	Tℓ	Ref.
$(Fe_{80}Ni_{20})_{75}P_{16}B_6Al_3$	---		427	2		
$(Fe_{60}Ni_{40})_{75}$ " "	439	2	437	2		
$(Fe_{40}Ni_{60})_{75}$ " "	420	2	437	2		
$(Fe_{20}Ni_{80})_{75}$ " "	414	2	435	2		
$Ni_{75}P_{16}B_6Al_3$	417	2	427	2		
$(Ni_{80}Co_{20})_{75}P_{16}B_6Al_3$	427	2	447	2		
$(Ni_{60}Co_{40})_{75}$ " "	---		425	2		
$(Ni_{40}Co_{60})_{75}$ " "	---		371	2		
$(Ni_{20}Co_{80})_{75}$ " "	---		394	2		
$Co_{75}P_{16}B_6Al_3$	---		387	2		
$(Fe_{60}Co_{40})_{79}P_{12}B_6Al_3$	437	3				
$(Fe_{60}Co_{40})_{77}P_{14}B_6Al_3$	447	3				
$(Fe_{60}Co_{40})_{75}P_{16}B_6Al_3$	457	3				
$(Fe_{60}Co_{40})_{73}P_{18}B_6Al_3$	467	3				
$(Pd_{80}Ni_{20})_{83}P_{17}$	319	3	329	3		
$(Pd_{80}Ni_{20})_{82}P_{18}$	317	3	330	3		
$(\quad)_{81}P_{19}$	314	3	351	3		
$(\quad)_{80}P_{20}$	317	3	354	3		
$(\quad)_{79}P_{21}$	325	3	356	3		
$(\quad)_{78}P_{22}$	334	3	355	3		
$(\quad)_{77}P_{23}$	339	3	349	3		
$(Pd_{50}Ni_{50})_{83}P_{17}$	311	3	323	3		
$(\quad)_{82}P_{18}$	311	3	323	3		
$(\quad)_{81}P_{19}$	310	3	323	3		

Alloy	Tg	Ref.	Tx	Ref.	Tℓ	Ref.
$(Pd_{50}Ni_{50})_{80}P_{20}$	310	3	377	3		
$(\quad)_{79}P_{21}$	315	3	377	3		
$(\quad)_{78}P_{22}$	320	3	377	3		
$(\quad)_{77}P_{23}$	331	3	377	3		
$(\quad)_{76}P_{24}$	342	3	377	3		
$(\quad)_{75}P_{25}$	353	3	372	3		
$(Pd_{20}Ni_{80})_{84}P_{16}$	---		332	3		
$(\quad)_{83}P_{17}$	327	3	357	3		
$(\quad)_{82}P_{18}$	321	3	344	3		
$(\quad)_{81}P_{19}$	327	3	352	3		
$(\quad)_{80}P_{20}$	329	3	365	3		
$(\quad)_{79}P_{21}$	335	3	372	3		
$(\quad)_{78}P_{22}$	348	3	367	3		
$(\quad)_{77}P_{23}$	349	3	360	3		
$(Pt_{80}Ni_{20})_{80}P_{20}$	207	3				
$(\quad)_{77.7}P_{22.3}$	207	3				
$(\quad)_{75}P_{25}$	207	3				
$(Pt_{75}Ni_{25})_{80}P_{20}$	207	3				
$(Pt_{60}Ni_{40})_{80}P_{20}$	222	3				
$(Pt_{50}Ni_{50})_{80}P_{20}$	247	3				
$(Pt_{40}Ni_{60})_{80}P_{20}$	267	3				
$(Pt_{30}Ni_{70})_{80}P_{20}$	277	3				
$(Pt_{20}Ni_{80})_{80}P_{20}$	297	3				

Alloy	Tg	Ref.	Tx	Ref.	Tℓ	Ref.
$(Pd)_{80}P_{20}$	334	4				
$(Pd_{80}Ni_{20})_{80}P_{20}$	317	4				
$(Pd_{60}N_{40})_{80}P_{20}$	312	4				
$(Pd_{40}N_{60})_{80}P_{20}$	315	4				
$(Pd_{20}N_{80})_{80}P_{20}$	329	4				
$Ni_{80}P_{20}$	345	4				
$(Pd_{85}Fe_{15})_{80}P_{20}$	370	4				
$(Pd_{80}F_{20})_{80}P_{20}$	357	4				
$(Pd_{75}Fe_{25})_{80}P_{20}$	344	4				
$(Pd_{70}Fe_{30})_{80}P_{20}$	339	4				
$(Pt_{80}Ni_{20})_{75}P_{25}$	212	4				
$(Pt_{70}Ni_{30})_{75}P_{25}$	---	4				
$(Pt_{60}Ni_{40})_{75}P_{25}$	229	4				
$(Pt_{50}Ni_{50})_{75}P_{25}$	247	4				
$Pd_{79.5}Cu_6Si_{16.5}$	363	4				
$Fe_{80}P_{13}B_7$			410	5		
$Pd_{82}Si_{18}$	362	6	367	6		
$Pd_{79.5}Au_4Si_{16.5}$	372	6	402	6		
$Pd_{77.5}Cu_6Si_{16.5}$	373	6	413	6		
$Pd_{79.5}Ag_4Si_{16.5}$	367	7		7		
$Pd_{81}Si_{19}$	377	8				

Alloy	Tg	Ref.	Tx	Ref.	Tℓ	Ref.
$Au_{31}Si_{19}$	17	9	5	9	363** 1127*	9
$Au_{77}Ge_{14}Si_9$	12	9	24	9	352** 1077*	9
$Fe_{75}P_{15}C_{10}$	---		347	9	937** 1567*	9
$Pt_{66}Sb_{34}$	---		212	9	612** 1657*	9
$Pd_{80}Si_{20}$	382	9	394	9	827 1527*	9
$Nb_{40}Ni_{60}$	---		697	9	1147** 1837	9
Pd (pure)	277	9	---		1557*	9
$Ni_{72}P_{14}B_6Si_3Al_5$	437	10	442	10		
$Ni_{72}P_{14}B_7C_2Al_5$	---		432	10		
$Fe_{72}P_{16}C_5Si_2Al_5$	457	10	467	10		
$Fe_{75}P_{15}C_6Al_4$	440	11	454	11		
$Fe_{76.6}P_{14.2}C_{1.2}Al_{3.2}B_{4.8}$	447	11	457	11		
$Fe_{77}P_{17}C_4Al_4$	441	11	451	11		
$Fe_{38.5}Ni_{38.5}P_{18}B_2Al_3$	409	11	426	11		
$Fe_{38.5}Ni_{38.5}P_{14}B_6Al_3$	415	11	434	11		
$Fe_{38.5}Ni_{38.5}P_{14}B_6Si_3$	435	11	449	11		
$Fe_{40}Ni_{40}P_{14}B_6$	390	11	400	11		
$Fe_{37.5}Ni_{37.5}P_{16}B_6Al_3$	428	11	441	11		
$Ni_{75.3}P_{16}B_{5.7}Al_3$	420	11	437	11		
$Fe_{29}Ni_{49}P_{14}B_6Al_2$	403	11	431	11		

* Avg. melting temperature of components
** Eutectic

Alloy	Tg	Ref.	Tx	Ref.	Tℓ	Ref.
$Fe_{38.5}Ni_{38.5}P_{20}Al_3$	405	11				
$Fe_{38.5}Ni_{38.5}P_{16}B_4Al_3$	412	11				
$Fe_{38.5}Ni_{38.5}P_{15}B_5Al_3$	413	11				
$Fe_{38.5}Ni_{38.5}P_{13}B_7Al_3$	415	11				
$Fe_{38.5}Ni_{38.5}P_{12}B_8Al_3$	416	11				
$Fe_{38.5}Ni_{38.5}P_{10}B_{10}Al_3$	418	11				
$Au_{77}Ge_{13.6}Si_{9.4}$					352	12
$Au_{81.4}Si_{18.6}$					363	12
$Ni_{72}P_{18}B_7Al_3$	442	13	434	13		
$Ni_{49}Fe_{29}P_{14}B_6Al_2$	403	13	431	13		
$Fe_{76}P_{16}C_4Si_2Al_2$	457	13	461	13		
$Fe_{80}P_{16}B_1C_3$	100	14				
$Fe_{78}Mo_2B_{20}$	440				(2605A)	
$Co_{72}Fe_3P_{16}B_6Al_3$	390					
$Fe_{40}Ni_{40}P_{14}B_6$	400				(2826)	

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APPENDIX B

FAILURE TO MELT ANALYSIS OF EXPERIMENT 74-49

AUGUST 30, 1977

TO: FRED A. REEVES/FA21
NASA-MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA 35812

SUBJECT: FAILURE TO MELT ANALYSIS OF EXP 74-49

SUMMARY: Evidence of incipient melting on the surface of the flight specimen indicates that the melting temperature of the specimen was attained, but that the net power, above radiation and conduction losses, available to furnish the latent heat of fusion was too small to appreciably melt the specimen during the 118 second period of high power. This was due to a procedural error committed during the performance of the ground based reference experiments. This led the Principal and Co-Investigators to incorrectly conclude before the flight that sufficient net power would be available to heat and melt the flight specimen with the experiment package configuration used.

The procedural error committed was the performance of these experiments utilizing a matching transformer having a different turns ratio than that of the matching transformer used in the flight apparatus. This caused an increase of the heating efficiency of the breadboard apparatus above its nominal reference power level. Had this error not been committed, it would have been realized that not enough power was available to heat and melt the specimen during the time available in the flight unit with the particular chamber gas mixture and transformer turns ratio employed. As it was, the specimen was melted in one atmosphere of helium in the breadboard apparatus in the available time for melting. Applying the calibrated power scale factor between the breadboard apparatus and the flight apparatus, then led to the incorrect conclusion that the specimen would heat and melt with the mixture of helium and argon used.

The heating efficiency depends very sensitively upon the specimen radius, and to a lesser extent upon its electrical resistivity. The flight specimen is 3.58% smaller than the stainless steel (#316) calibration specimen used in testing the flight package. This was due to the lack of provision in grinding the specimen resulting in flats. The maximum radius is 0.465 cm, the minimum is 0.433 cm, and the average is 0.4445 cm (determined by 10 micrometer readings). The stainless steel ball is precision ground with a radius of 0.461 cm. This reduction in size led to an estimated 16% reduction in absorbed power.

The resistivity and temperature coefficient of resistivity of the stainless steel material matches that of the flight specimen material to within $\sim$ 10% over the required temperature range. Further there is a void structure on the equator plane with a string of voids running from the surface to the center. These effects together led to an estimated 8% reduction in power, so that the total reduction in net power to the flight specimen was 24%. This corresponds to a calculated 45 watts absorbed power instead of the 59 watts expected.

The measured absorbed power in the flight specimen, from the flight pyrometer data is 46 watts. Due to uncertainties in reading temperatures accurately from the flight pyrometer data (because of uncertainties in specimen emissivity), the absorbed power is uncertain to $\pm$ 20%, so that the corresponding absorbed power may be 46 ± 9.2 watts. For a sphere of radius 0.4445 cm, the estimated loss due to radiation and conduction through the chamber gas mixture at 950^{o}C is 38 watts, for an emissivity of 0.8 (estimated from the appearance of the specimen). Due to the irregular shape of the specimen this could be as much as 10% high so that the loss may be 38 + 3.8, -0 watts.

In light of the above information, failure to melt in the allotted time may be explained. The absorbed power at 950°C was nearly equal to the radiation losses, leaving a negligible amount of available power for melting. The melting time was greatly extended and, in the time allotted, only incipient melting on the surface was observed. This is in contrast to the situation planned for the experiment, where 59 watts was estimated to be the absorbed power and the loss due to conduction and radiation was 40.5 watts, leaving 18.5 watts available for melting. Under those circumstances, melting would have occurred in 43 seconds. A margin of 22 seconds was allowed to account for a 10% difference in absorbed power. In the actual experiment the additional losses due to the undersize specimen, the slight difference in resistivity, and the presence of voids, coupled with the uncertainty in actual absorbed power added up to cause a failure to melt in the time allotted.

The recommendations to correct this situation so that failure to melt shall not occur again are:

(1) Introduction of a quench gas to cool the specimen after melting instead of allowing this gas to be present from the outset, which greatly reduces the net melting power margin available.

(2) Insure that the turns ratio on the matching transformer in the breadboard facility is the same as the flight apparatus of each experiment by proper documentation.

(3) Fabricate flight specimens so that they have the correct average radius instead of the correct maximum diameter and eliminate voids.

(4) Perform some calibration tests with the flight specimen in the flight apparatus. If it is determined that melting is required, the flight apparatus would have to be replaced prior to flight. An additional recommendation is to calibrate the flight pyrometer for at least three points with any flight specimen material. These should be the point where the flight pyrometer begins indicating, the melting temperature and one point between these. That will enable absorbed power and loss calculations to be performed with much greater accuracy.

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APPENDIX C

GROUND BASED PREPARATIONS FOR EXPERIMENT 74-49

The laboratory test plan for ground based tests using the breadboard processor and the flight processor was prepared on February 4, 1977 and had the following objectives:

1 - Quantify the performance of the breadboard and the flight unit.

2 - Specimen mounting, clean up, pump down and back fill of breadboard unit.

3 - Melting run using one of the ferromagnetic ground truth specimens.

In implementing the tests use was made of a 316 stainless steel specimen exclusively in the flight unit because it was the nearest material to the Metglas alloy. The flight Metglas specimens were not available for test during the flight unit preparation prior to delivery.

Breadboard tests were run using the same 316 stainless specimen used in the flight unit tests. These tests were followed by the tests using the Metglas alloy.

After the original objectives were met, further tests were deemed essential because of the decentering toward the coil of the magnetic specimen during heating until the curie temperature is reached. Also, the final cooling calculations showed that Argon as used in the 74-48 flight would not cool rapidly enough and if pure Helium was used the specimen may not melt in the flight unit. These tests were all done with the ground truth Amorphous Ferromagnetic specimen.

TEST SEQUENCES

Jan. 18, 19 — Black Body tests of solid state radiometer

Jan. 21-23 — Flight unit tuning, loading and trimming of servo using 316 stainless ball, 0.92 CM dia.

Jan. 25 — 316 S.S. heating test, flight unit; 1 ATM Argon, 1 ATM Helium. Recorded solid state radiometer.

Jan. 26 — 316 S.S. heating tests recording heat sink temp., water temp., and solid state radiometer. 1 Torr Argon, 1 Torr Helium (T.C. gauge pressure).

Feb. 1 — Final flight unit test, no specimen, recorded all T/M lines.

All further tests were with the breadboard set-up.

Feb. 4 — 316 stainless ball, 0.92 CM dia. heating and cooling tests, 1 ATM Helium, 1 ATM Argon. 12:5 matching transformer turns ratio.

Feb. 11 — 316 stainless ball, heating and cooling, 10 Torr Argon, two tests, first with 11:5 turns ratio and next with 12:5 turns ratio on the matching transformer. Proper match was 12:5 ratio*.

* Heating was 15% better at 11:5 but amplifier was saturated so servo would not damp adequately at high power.

Feb. 11 — 316 run with 10 Torr Helium, 12:5 turns ratio on matching transformer, heating and cooling test.

Feb. 15 — Calculation of relative average heating power, breadboard and flight unit.

At this point (from Feb. 11 to Feb. 17) the breadboard was being equipped

for testing the Metglas alloy. No further tests were made using 316 stainless material. Immediately, difficulties arose which we found were due to the magnetic properties of the Metglas alloy. Initial loading was severely mismatched and there was fear the low amplifier efficiency would cause overheating. Experiments were made by varying the transformer turns ratio to try to improve the match. Finally, it was determined that the time to reach the curie temperature was not long enough for the amplifier to overheat and that the match would have to be what was originally determined by the stainless. At this point the 12:5 ratio was supposed to have been restored. Subsequent post flight review of the breadboard equipment disclosed the transformer was marked 12:5 ratio but an actual turn count showed 11:5.

The conclusion is that all tests with the Metglas alloy were done with the 11:5 ratio and its 15% higher heating capability.

Feb. 17 Heated the 2.95 gm Metglas ball to melting.

Mar. 8,9 Replaced defective D.C. to D.C. regulated power supply in flight unit and retested without specimen.

Apr. 18 Breadboard heating test, Metglas alloy, vacuum

Apr. 25 Breadboard heating and cooling tests, Metglas vacuum, 100 Torr Helium, 760 Torr Helium, 100 Torr Argon, 760 Torr Argon

Apr. 26 Breadboard heating and cooling test, Metglas near coil top, 50% He, 50% Ar Mixture

May 5 Breadboard heating tests, Metglas, vacuum, low battery voltages

May 6 Breadboard heating and cooling tests, Metglas, 760 Torr Helium, 100 Torr Helium

May 6 Breadboard melting of Metglas specimen, 100 Torr Helium

No further tests were performed until after the 74-49 experiment flight