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ALLOY UNDERCOOLING EXPERIMENTS

Final Report
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(NASA-CR-199682) ALLOY UNDERCOOLING EXPERIMENTS Final Report (MIT) 11 p

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A. Tasks Accomplished in Present Grant Period

The research accomplished during 1995 can be organized into three parts. The first task involves analyzing the results of microgravity experiments carried out using TEMPUS hardware during the IML-2 mission on STS-65. The second part was to finalize ground-based experimentation which supported the above flight sample analysis. The final part was provide technical support for post-flight mission activities specifically aimed at improving TEMPUS performance for potential future missions.

Analysis of Microgravity Experiments

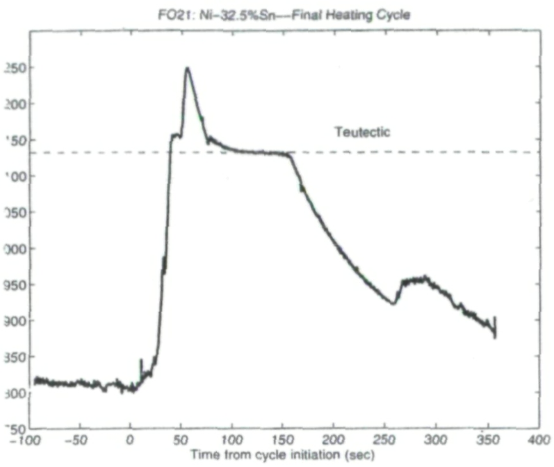
Undercooling studies in microgravity were performed using the TEMPUS facility during STS--65 IML-2 mission. Experiments were conducted by successfully processing samples of hypoeutectic Ni-25 Sn, eutectic Ni-32.5 Sn, and pure nickel under conditions of microgravity. Undercooling of the molten samples was observed to be on the order of 10 degrees when processed with a superheat of between 20 and 200 degrees above the liquidus. A sample of hypoeutectic composition was also quenched by contact with the containment wire cage. Solidification structures were thus obtained for various nickel-tin alloys processed under conditions of microgravity for comparison to terrestrially produced materials.

The results of the metallographic analysis of the flight samples are presented in Figures 1 and 2. A typical flight thermal profile for each sample is shown along with the resultant solidification microstucture from both the sample surface and from metallographic sectioning of the sample interior. Surface contamination from the containment cage holder acted as heterogeneous nucleation sites and limited the undercooling which was achieved. A comparison to terrestrially produced material is shown in Figure 2.

Ground-based Experimentation

The surface condition of flight samples was analyzed at the NASA Marshall Space Flight Center Failure Analysis Branch Facilities in Huntsville, Alabama. Using ESEM and ESCA facilities, the surface contamination was analyzed and the source identified. It was determined that pieces of the sample holder had adhered to the surface and served as heterogeneous nucleation sites. Flight conditions were subsequently simulated in a ground-based levitation facility at MIT. The surface oxide conditions were reproduced and terrestrial microstructures produced for comparison to the flight samples at comparable undercoolings as produced in flight. These results are shown in Figure 2.

Figure 1 : IML-2 eutectic sample Ni - 32.5 wt% Sn
(a) Flight thermal profile
(b) Surface morphology
(c) Nucleation initiation site on surface
(d) Metallographic section showing lamellar structure



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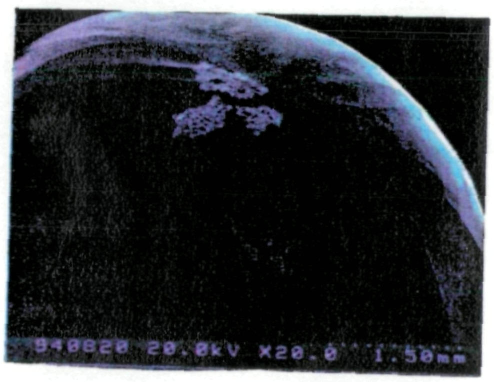

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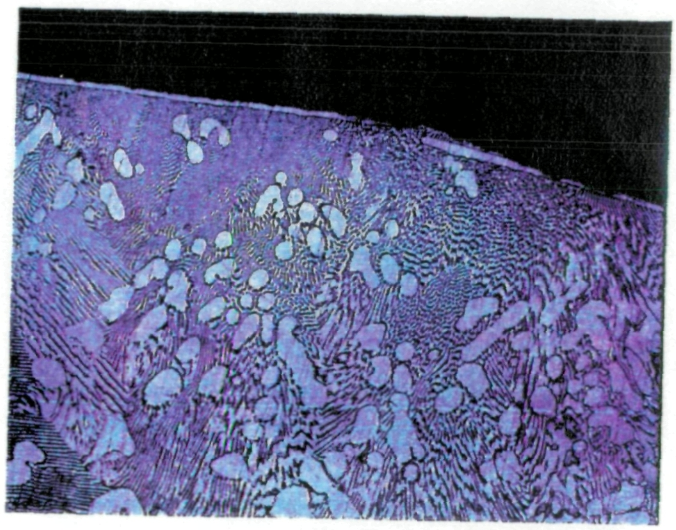
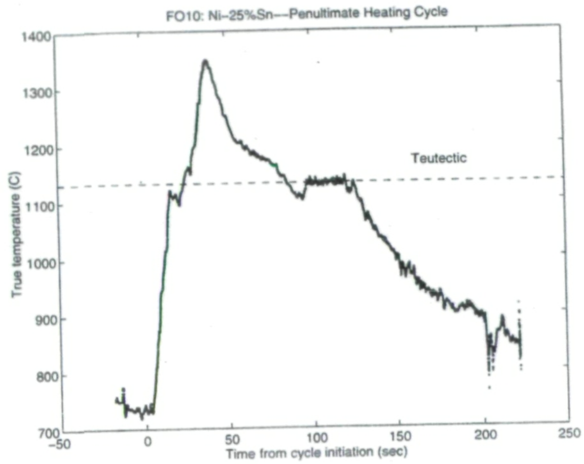


50 µm

(d)

Figure 2 : IML-2 hypoeutectic sample Ni - 25 wt% Sn
(a) Flight thermal profile
(b) Micrograph of surface rosette dendrite structure
(c) DAS comparison between Flight and Ground-based samples
(d) Micrograph of exposed dendrite structure



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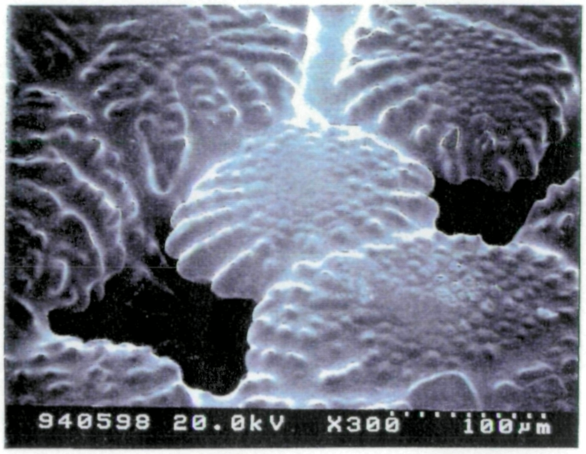

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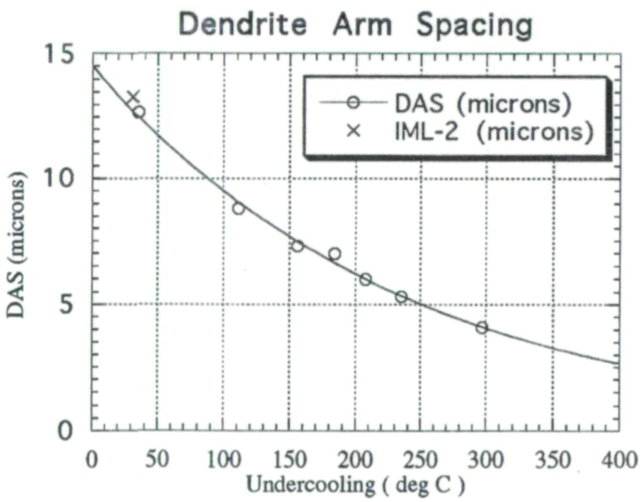

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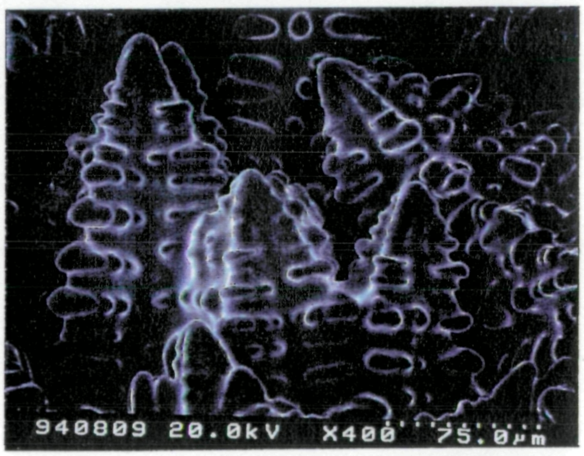

(d)

Technical Support

Recommendations for Assuring Future Mission Success

In our experiments on IML-2, we relied on the simplicity of the requirements of our approach to minimize risk. We expected to encounter unanticipated challenges, as is true in all space-born experimentation, and structured our goals accordingly. Our accomplishments were significant - we successfully processed solidification samples in TEMPUS and attained undercooling data in microgravity - but below expectations. Any perception that the hardware failed to perform fully should be tempered with the acknowledgment that the maturity of the development of the TEMPUS experiment platform has significantly improved as a result of the maiden voyage.

A major unforeseen difficulty which affected our experiment on IML-2 involved severe sample contamination. We consider solution of this problem to be in the enabling category; it must be solved before proceeding to additional flight missions. Three issues relate to the contamination issue. First, samples could not be baked-out as is done on earth as the sample levitation coils were not shielded from evaporation and thus samples could not be recovered if only slight contamination was present. Second, some types of materials could not be processed in TEMPUS as excessive evaporation would build-up on the coils and material would flake off when a critical layer thickness was achieved; fears of sample-to-sample contamination required careful orchestration of sample order and resource allocation. Third, the sample holder was inadequately protected by wire bumpers that were designed to keep the samples from striking the ceramic pedestal during ground transport and during launch and re-entry. Thus motion of the unrestrained sample abraded excessive quantities of the sintered alumina pedestal from exposed surfaces which subsequently acted as heterogeneous nucleation sites and preventing attainment of high undercoolings.

The TEMPUS team has instituted a series of design modifications which will satisfactorily prevent contamination problems from occurring during the MSL-1 mission. The ceramic pedestal has been replaced with a quartz holder while the wire containment cage has been replaced with a quartz evaporation shield. Access slots will be provided to lock the sample in place for ground transport and launch/recovery. By providing an evaporation shield, the samples may be baked-out (in our case other investigators have requested to use this time to conduct thermophysical property evaluations) and processing of more volatile component materials is now possible. These recommendations are summarized in Table I.

Resource allocation is also simplified in this regard in that once the evaporation shield becomes opaque due to the deposition of material from the sample, the test must stop as pyrometry and video data is unavailable. The individual PI will assign priority for experiments on proposed samples instead of negotiating with all other teams; volatile materials will have a limited cycle life which may be monitored real-time. Note that excessive flaking could still be a problem and ground-based tests will easily identify acceptable limits as indicated by changes to the transmissivity of the optical path.

A second enabling category from IML-2 centers around sample stability. The coil system, as-installed, was radially asymmetric with the heater and positioner coils having unmatched centers of symmetry. This resulted in two apparently unrelated problems; the sample spins excessively and the equilibrium position is skewed toward one wall of the wire enclosure. This problem was exacerbated by a requirement for violent heating cycles to minimize coil deposition by limiting the time above the melting point. As a result, wild surface oscillations were induced during superheating of the liquid and, with inadequate damping, the probability that a sample would strike the wall was significantly increased . Although only one of our four samples suffered this fate, a complete coil redesign effort has been initiated and several coil designs have been flown on the KC-135 parabolic flight campaign in Houston. We supported these test activities and plan to assist in further optimization of coil design.

Table I : Key Hardware Modifications

Issues	Solution	Impact	Status
Sample stability			
Cage impact	Coil redesign	Moderate	Complete
	Software algorithm	Moderate	Complete
	Reduced Sample Size	Small	In-progress
Coil geometry	Field Mapping	Large	Complete
Oscillation damp	Magnet / new coil	Small	Complete
Transformation oscillation	Lower heating rates(3)	Large	KC-135
Sample rotation / spin	Magnet / new coil	Moderate	KC-135
Video coverage	Dedicated s-band video	Small	In-progress
Contamination			
Cross sample contam.	Evaporation shield (1)	Large	Complete
Pedestal contam.	Sample holder redesign	Large	Complete
Sample abrasion(2)	Locking device pre-mission	Large	Complete
Vapor deposition			
Coil degradation	Evaporation shield	Large	Complete
Gas environment	RGA	Moderate	Complete

Notes:

(1) Quartz containment represents an order of magnitude improvement over the IML-2 wire containment system as coil deposition becomes a non-issue. This allows for expansion into alloy systems which previously could not be run.

(2) Contamination was observed on the surface of flight samples and traced to the pedestal used to position the sample in the levitation field. Use of an evaporation shield requires locking the sample in place during all ground-based transfer and during both launch and landing. This problem has been solved as part of the sample carousel redesign to accommodate the new quartz shields.

(3) Sample oscillations upon heating, and subsequent cage contact, were a direct consequence of a lack of evaporation shielding available on IML-2. The new design allows for benign heating during melting and will reduce the tendency for evolution of uncontrolled surface motion with a concomitant reduction in risk of sample loss.

This new design addresses asymmetry, sample spinning, field strength, and with the addition of an evaporation shield, more benign heating cycles will reduce the potential for developing uncontrolled surface oscillations. Smaller samples are also under investigation to increase motion tolerance while not seriously reducing the ability to observe surface oscillations during thermophysical property evaluations.

In addition to these two major issues, several secondary problems surfaced during the IML-2 mission which have been addressed for MSL-1; solution of these issues is considered to be in the efficacious category for the success of our experimental program. The gas environment during one of our functional objectives was compromised by a leaking gas valve. Although no contamination is attributed to this event, an RGA system has been installed to monitor real-time the environment within the test chamber. Software changes have also been implemented which allow entry into any processing step following a HOLD command and have greatly increases operational flexibility.

Several systems which were operational during IML-2 were never fully checked out due to the problems attaining reproducible undercooling. These systems should be verified early in the MSL-1 mission and thus are addressed in our first sample. They include demonstrating the ability to discern the thermal front accompanying recalescence using the video system, calibration of the pyrometer as a velocity measurement instrument, and triggering nucleation at desired undercooling using the non-intrusive method proposed earlier and using physical nucleation triggering with a stimulation needle. The recalescence detection software has also been upgraded for MSL-1 and a successful demonstration of its use is of prime importance to the investigation of the effects of microgravity on nucleation and growth through analysis of double recalescence behavior using our later samples. An investigation into including dedicated video coverage for facility health monitoring is also recommended as a desired improvement but would not be required to assure mission success. A summary of the TEMPUS modifications, their impact on our proposed test series, and the degree of implementation is shown in Table I.

In summary, the issue of sample contamination from the pedestal, from the coils, and between samples was the primary reason for reduced return of science from IML-2. Contributing to these problems, but not as severe and impact to our experiments, was a lack of lateral stability due to less than optimal coil design, as well as concomitant sample off-set, spin and oscillatory motion. The lack of a proper evaporation shield was also a limiting factor in that sample selection was restricted while the method used to reach the desired superheat was unduly violent and possibly resulted in excessive surface oscillations which subsequently lead to the cage wire contact observed during the processing of one of our specimens. We feel that the hardware problems have satisfactorily been addressed and improvements are being incorporated into the design of the TEMPUS experimental platform to assure mission success.

We have initiated model development tasks and conducted experimentation to anchor the numerical analysis and computer model results applicable to both flight and ground-based experiments. We have extended the application of the model to include data from the ternary Fe-Ni-Cr system used to demonstrate phase selection during undercooled solidification processing. We have successfully modeled microstructural evolution during recalescence under both nucleation controlled and growth controlled solidification. Phase selection is possible in either case but the metastable phases differ. In the first case, cooling conditions corresponding to adiabatic droplet solidification result in metastable BCC. Double recalescence was observed and the decomposition of the metastable phases have been investigated. In the latter case, chill casting applications involving massive heat extraction techniques resulted in the formation of metastable FCC with practical processing ramifications for space-based fabrication facilities.

We have submitted suggestions for facility upgrades required for future missions to allow an increased flexibility and more capabilities for space-based undercooling experiments. We continue the evaluation of furnace designs for implementation of rapid solidification processing.

Direction for Future Work

The investigations of rapid recalescence in highly undercooled alloys carried out in previous years under NASA sponsorship will be continued, using video and film imaging techniques. Enhanced imaging results could provide direct observation of recalescence at the extremely high growth rates typical of alloy undercooling experiments and would allow comparison of experiments and dendrite growth theories under rapid solidification growth rate conditions.

The effort in developing and evaluating physical and numerical models of solidification will also be continued. The heat flow and solute redistribution model developed during the past grant period will continue to be improved and extended, and will be used to calculate solidification behavior under different undercooling and heat extraction conditions. The results from these calculations will be compared with data from pyrometric measurements and microstructural studies.

Technical Meetings

At the Mission Hardware readiness review at NASA Headquarters, we presented our plans to build on our IML-2 experience by conducting experiments using selected ternary steel alloys on the upcoming MSL-1 mission. This work is based on successful ground-based experimentation into the feasibility for evaluating phase selection using physical triggering as conducted as a part of flight support activities for IML-2. As part of our flight support activities we also attended working group meetings at Jackson to define sample selection for MSL-1 and attended the Huntsville IWG for MSL-1 crew selection.

B. Statement of Work and Timetable

The Statement of Work and Timetable included in the following pages is the same as was included with the previous proposal submitted in January of 1993. Work has progressed as planned with the exceptions noted in the previous proposal. With the extension of the contract to allow input into the planning of subsequent missions, as detailed in tasks §2.2, §2.3 and §3.3, the work under this contract is complete as of the end of the grant period. Items shown in this statement of work as in-progress will be completed at that time.

		1993	1994	1995
1.0	Definition and Feasibility			
1.1	Define and refine science requirements			
1.1.1	Goal identification	completed		
1.1.2	Approach	completed		
1.1.3	Experimental Protocol (will continue as needed)	completed		
1.1.4	Equipment Requirements (will continue)	completed		
1.2	Feasibility experiments (will continue)			
1.2.1	Controllably achieved high undercoolings in ternary Fe-Ni-Cr	completed		
1.2.2	Controllably achieve slow recalescence slow dendrite velocity in ternary droplets	completed		
1.2.3	Controllably interrupt solidification by quenching	completed		
2.0	Ground-based Data Preparation (will continue)			
2.1	Experiments on the solidification of highly undercooled Ni-Sn, Fe-Ni, and Fe-P alloys and pure Ni			
2.1.1	Solidification of undercooled sample in glass	completed		
2.1.2	Solidification of levitated undercooled samples in He/Ar with no glass encapsulation	completed		
2.1.3	Rapid solidification of highly undercooled samples	completed		
2.1.4	Microstructural and chemical analyses of the structures produced in § 2.1.1 to 2.2.3	completed		
2.1.5	Cinematography and video recording of the samples processed in § 2.1.1 and 2.2.2	completed		
2.1.6	Determination of dendrite arm spacing versus solidification time relations	completed		
2.2	Develop and evaluate physical and numerical methods of solidification by correlation with prior and concurrent experimental results	in-progress		

2.3	Evaluate the results of new ground-based experiments in terms of processing in a space environment, and evaluation of furnace designs for space implementation in relation to high undercooling experiments	in-progress
2.4	Collaborate with NASA, DLR, and other Principal Investigators on space experimentation	
2.4.1	Attend IWG and other NASA meetings	completed
2.4.2	Assist in experiment documentation	completed
2.4.3	Assist in training of flight personnel and participate in training and simulations at MSFC	completed
2.4.4	Prepare samples for MUSC tests	completed
2.4.5	Prepare and deliver samples for flight experiments	completed
2.4.6	Monitor flight experiments	completed
2.4.7	Collect samples for analysis	completed
3.0	Analyze flight data and samples	
3.1	Analyze pyrometric and video data, flight records	completed
3.2	Analyze microstructures using optical metallography, SEM, EMPA, TEM, and STEM	completed
3.3	Prepare reports, papers, presentations, and recommendations for follow-up experiments and processing	in-progress

C. Publications

The following publications have been produced as a result of activity during the present grant period in support of the IML-2 mission :

T. Koseki and M.C. Flemings, "Thermal behavior during solidification of undercooled Fe-Cr-Ni alloys", accepted Metallurgical Transactions, 1995.

T. Koseki and M.C. Flemings, "Microstructural evolution during undercooling and rapid solidification of Fe-Cr-Ni alloys", submitted to Metallurgical Transactions, 1995.

T. Koseki and M.C. Flemings, "Rapid dendritic growth during chill casting", Proceedings, 2nd Pacific Rim International Conference on Modeling of Casting and Solidification, Sendai, Japan, 1995.

T. Koseki and M.C. Flemings, "Effect of external heat extraction on dendritic growth into undercooled melts", ISIJ International, Vol. 35, 1995, pp. 611-618.

J. Lum, D.M. Matson, A. Shokuhfar, M.C. Flemings, "High speed imaging and analysis of the solidification of undercooled nickel melts", submitted to Metallurgical Transactions, 1995.

D.M. Matson, J. Lum, A. Shokuhfar, T. Koseki, and M.C. Flemings, "Imaging the double-recalescence behavior of undercooled Fe-Cr-Ni ternary alloys using a high speed video technique", accepted to JIM Materials Transactions, 1995.