NASA/CR-97- 206192

S. W. Johnson
A/C 61296

NASA FINAL REPORT

NASA Grant No. NAG8-954

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September, 1996

TABLE OF CONTENTS

I. SUMMARY

The AC Modulation Calorimetry experimental method (ACMC) was implemented on the TEMPUS facility in low earth orbit during the IML-2 flight. The ACMC technique was originally developed by two of the authors, Fecht and Johnson, as a method of measuring the heat capacity of a liquid drop under containerless conditions in high vacuum using electromagnetic heating of the droplet. Two sets of samples were investigated during IML-2. These included samples from the Fecht/Wunderlich group (TU Berlin) and the Johnson/Lee group (Caltech). The technique proved to be robust and provided valuable information on the heat capacity to total emissivity ratio of molten metallic alloys. The amount of undercooling achieved was less than hoped for due to sample contamination problems arising from facility limitations. These limitations have been addressed and the experiment is currently scheduled for reflight on the MSL-1 mission in early 1997.

Samples flown on the IML-2 mission included pure Zr metal, $Zr_{76}Ni_{24}$, $Zr_{64}Ni_{36}$, $Nb_{40}Nb_{60}$, and $Zr_{72}Fe_{28}$. Stability problems with the samples in TEMPUS during IML-2 limited the available processing time for samples. Reasonable amounts of ACMC data were obtained on the first three binary alloys. Little or no data was obtained on Zr or the Zr-Fe alloy. We briefly summarize some of the most complete results on one of the alloys below:

$Zr_{64}Ni_{36}$ (Other alloy results reported in "Highlights & Results")

Temperature (C)	**Mod. Frequency (Hz)**	**Total Hemisph. Emissivity (at T_m)**	**Heat Capacity (J/mole-K)**
1215	0.05	0.37	43.7 +/- 0.8
1160	0.08	0.35	44.5 +/- 1.2
	0.10		43.2 +/- 1.0
1038	0.05	0.33	43.9 +/- 1.0
	0.08		44.2 +/- 1.0
1008	0.05	0.32	44.0 +/- 1.0
	0.10		44.6 +/- 1.0
980	0.05	0.32	45.5 +/- 1.2

The results on this alloy are the most extensively analyzed of the IML-2 results. The alloy could not be significantly undercooled during the IML-2 mission due to contamination problems which arose with the samples during the flight. As such, data were limited primarily to the equilibrium liquid region (this eutectic alloy has a eutectic melting temperature of 1010 C). Data for slight undercooling to 980 C was the best obtained. These contamination issues have been extensively addressed and the TEMPUS sample holders and containment system has been modified to eliminate such problems during the MSL-1 mission. This should result in far more extensive undercooling of the samples to be studied.

The problems with sample stability during the IML-2 flight were analyzed and determined to have arisen from a misalignment of the heating and positioning coils. Corrective measures have also been taken to ensure that these problems do not occur during the MSL-1 mission.

II. PERSONNEL

Professor William L. Johnson - Principal Investigator, 9/91 - end of grant

Dr. David S. Lee - Member of the Professional Staff, Co-Investigator, 11/92 - end of grant

Dr. Y.J. Kim - Post-doctoral Research Fellow, Academic Year '92-'93

Dr. Joseph C. Holzer - Post-doctoral Research Fellow, 9/91 - 11/92

Jian Li - graduate student, 9/91 - end of grant

III. HIGHLIGHTS & RESULTS

1. Introduction

The measurement of the specific heat of liquid and undercooled metals and alloys provides important information regarding the thermodynamics of glass formation and metastability. For example, the Gibbs free energy difference between the metastable liquid phase of a material and its stable solid phase can be determined experimentally by the following equation:

$$\Delta G(T)=(\Delta H_t)+\int_T^{T_l}\Delta C_p dT-T*\left(\Delta S_t+\int_T^{T_l}\frac{\Delta C_p}{T}dT\right), \quad (1)$$

This quantity can be used to construct a metastable phase diagram and thus determine metastable equilibrium between the phases. The excess thermodynamic quantities (ΔG^{lx}, ΔS^{lx}, ΔH^{lx}) can be calculated from measurement of the undercooled liquid specific heat, allowing determination of the reduced glass transition temperature - a measure of the glass-formability of an alloy. Extrapolation of the liquid and solid phase entropies allows determination of the isentropic Kauzmann temperature. The free energy difference is also used in classical nucleation theory, which, when combined with viscosity data, predicts the nucleation rate of a stable solid solution from the undercooled liquid. Unfortunately, the specific heat of undercooled and stable liquid metal alloys are not generally available.

The noncontact AC calorimetry technique uses a modulated radio frequency field to inductively heat the sample under UHV conditions. The specific heat and, under certain conditions, the thermal conductivity of the sample can be obtained from the pyrometrically measured temperature response of the sample to this field.

2. The Technique of AC Calorimetry

On TEMPUS, heating and positioning fields are controlled independently by two RF power supplies: one operating at 400kHz with its coil in a dipole field geometry for heating of the sample and one operating at 100kHz with its coil in a quadrupole field geometry for positioning of the sample within the dipole field. Other features include sample processing up to 2300K, UHV and/or inert gas processing, high speed video, optical pyrometers (100Hz) operating in the visible (650nm) and infrared (1.0-2.5μm and 3.0-4.0μm) and high speed pyrometry for recalescence detection and analysis (1Mhz). This is shown schematically in Figure 1.

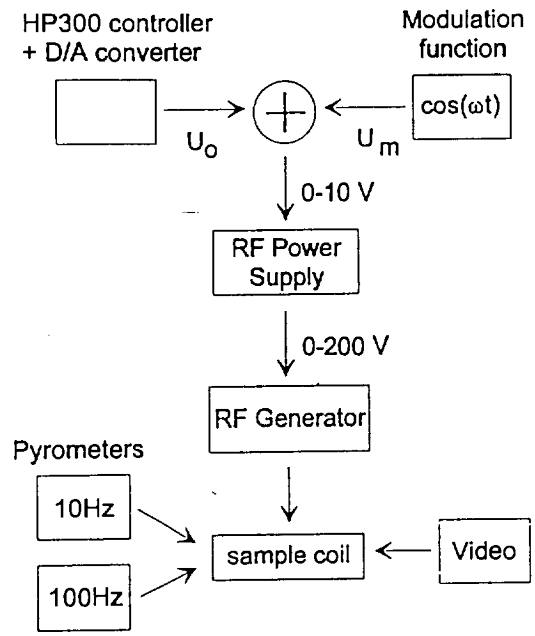

Figure 1. Schematic of the experiment setup.

Typical modulation frequencies used were in the range of 0.05Hz to 1.0Hz. A signal in the range 0-10V defines the output control voltage to the RF circuits. This signal is composed of a DC component from the facility controller and the superimposed AC signal from a function generator. This voltage is applied to the RF generator, producing an RF output signal with an 0-200V amplitude of the form:

$$U_\omega = U_o + U_m \cos(\omega_m t) \tag{2}$$

where U_o is the DC bias voltage, U_m is the modulation amplitude and ω_m is the modulation frequency - all user-controllable quantities. In the experiments, modulation amplitude was varied between 0.1 and 2.0V. Since $P_\omega \sim U_\omega^2$, the modulation of RF amplitude results in a modulation of the RF heating power:

$$P_\omega = (cpl)*\left\{U_o^2 + \tfrac{1}{2}U_m^2 + 2U_oU_m\cos(\omega_m t) + \tfrac{1}{2}U_m^2\sin(2\omega_m t)\right\} \tag{3}$$

where *(cpl)* is the coupling coefficient between the RF coils and the sample, and is dependent on the sample resistivity and the mutual inductances of the circuit. As we can see from the above equation, this type of power modulation will generate an increase of the average sample temperature superimposed over a periodic temperature modulation.

The Fourier solution of the heat flow equation of this problem is:

$$P_{tot} = P_o + \Delta P_{av} + P(\omega)\cos(\omega t) + P(2\omega)\cos(2\omega t) + \mathrm{K} + O(higher) \tag{4}$$

where ΔPav is the increase in average DC power absorbed by the sample when the modulation is turned on, P(ω) is the power component at frequency ω, P(2ω) is the power component at frequency 2ω, etc., and P_o is the constant power absorbed by the sample in the absence of any modulation. A one-to-one correspondence exists between the leading terms in this equation and the power modulation equation in (3). P_o is related to the sample bias temperature by the Stefan-Boltzmann law, and in the absence of any modulation, can be written as:

spectral response is flat from 0.6μm to 40.0μm. In the temperature range from about 700K to 2500K, this detector will measure better than 98% of the greybody spectrum from the radiating sample. Direct measurement of P_o has the advantage that the evaluation of Cp is much less susceptible to the accuracy of the T_o measurement.

Note also that the modulation calorimetry technique provides an intrinsic measurement of temperature which can be used to verify the accuracy of the pyrometry. Combining Equations (5), (6), (7) and (10) we obtain:

$$T_o = \frac{1}{2} f(\omega_m, \tau_1, \tau_2)^2 (\omega_m \tau_1)^2 \left\{ \frac{\Delta T_m^{\ 2}}{\Delta T_{av}} \right\} \tag{13}$$

so the sample temperature corresponding to an input DC power of P_o can be measured just by measuring ΔT_m and ΔT_{av}. Data from IML-2 is shown in Figure 2.

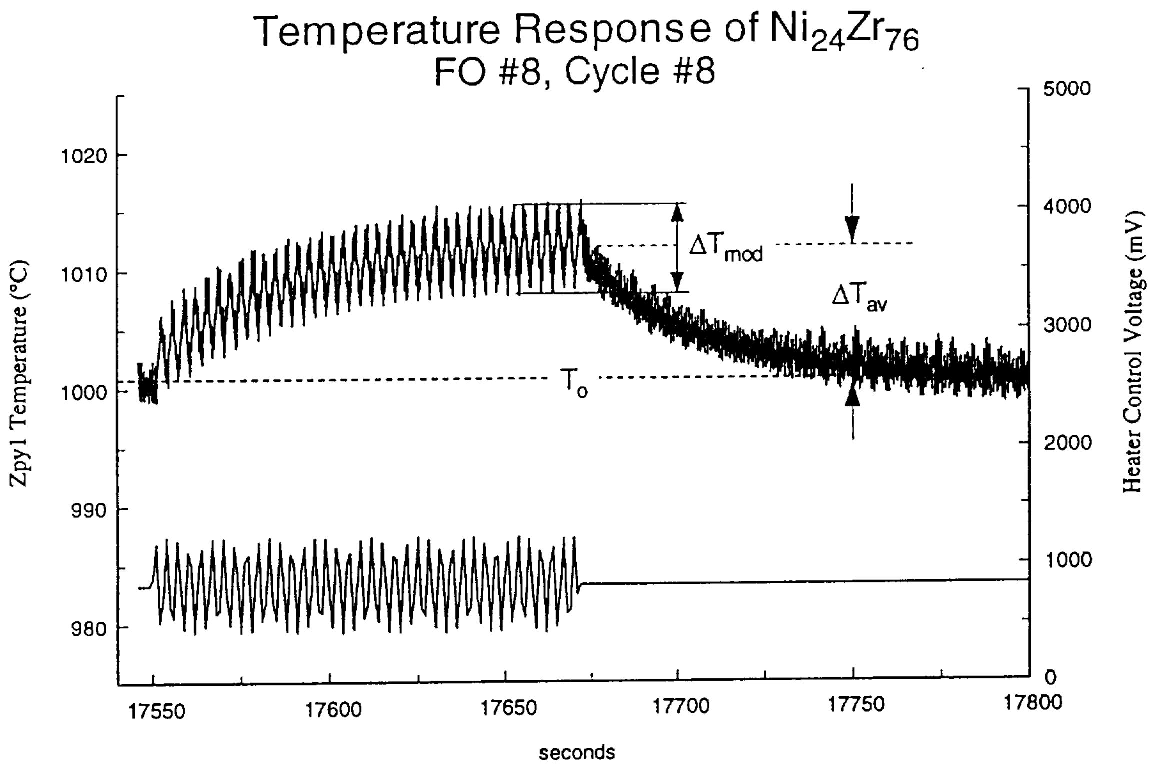

Figure 2. Temperature response of $Ni_{24}Zr_{76}$ sample to RF power modulation.

3. Results and Discussion

The dynamic temperature response of the sample upon application of heating power modulation is shown in Figure 2. The upper trace shows the RF control voltage modulation according to Equation (2) and the lower trace shows the temperature response of the sample. This temperature response can be written as follows:

$$T(t) = T_o + \Delta T_{av} \left[1 - \exp\left(-\frac{t}{\tau_1} \right) \right] + \Delta T_m \cos(\omega_m t) \tag{14}$$

As can be seen from Figure 2, the increase in average temperature, ΔT_{av}, is independent of the modulation frequency, ω_m. No modulation component is seen in the raw data at frequency (2ω) because the ratio of $P(\omega)/P(2\omega) = (4\sqrt{2})U_o/U_m$. For the control voltages used in this experiment, this ratio is ~50, and because the amplitude of temperature modulation is inversely proportional to the modulation frequency, the modulation term at 2ω contributes less than 1% of the observed temperature modulation. In fact, in the FFT spectrum of the data shown in Figure 3, we see the peak associated with the 2ω modulation term. The peaks in the 1.5Hz and 3Hz regions correspond to center of mass motion in the radial and axial directions, caused by misalignment of the heater and positioner coils on TEMPUS. A 12th order Butterworth filter positioned to rolloff at 0.8Hz results in the backtransformed signal shown in Figure 4a. The filter shifts the phase of the signal very slightly, but is positioned well outside of the modulation frequencies of interest.

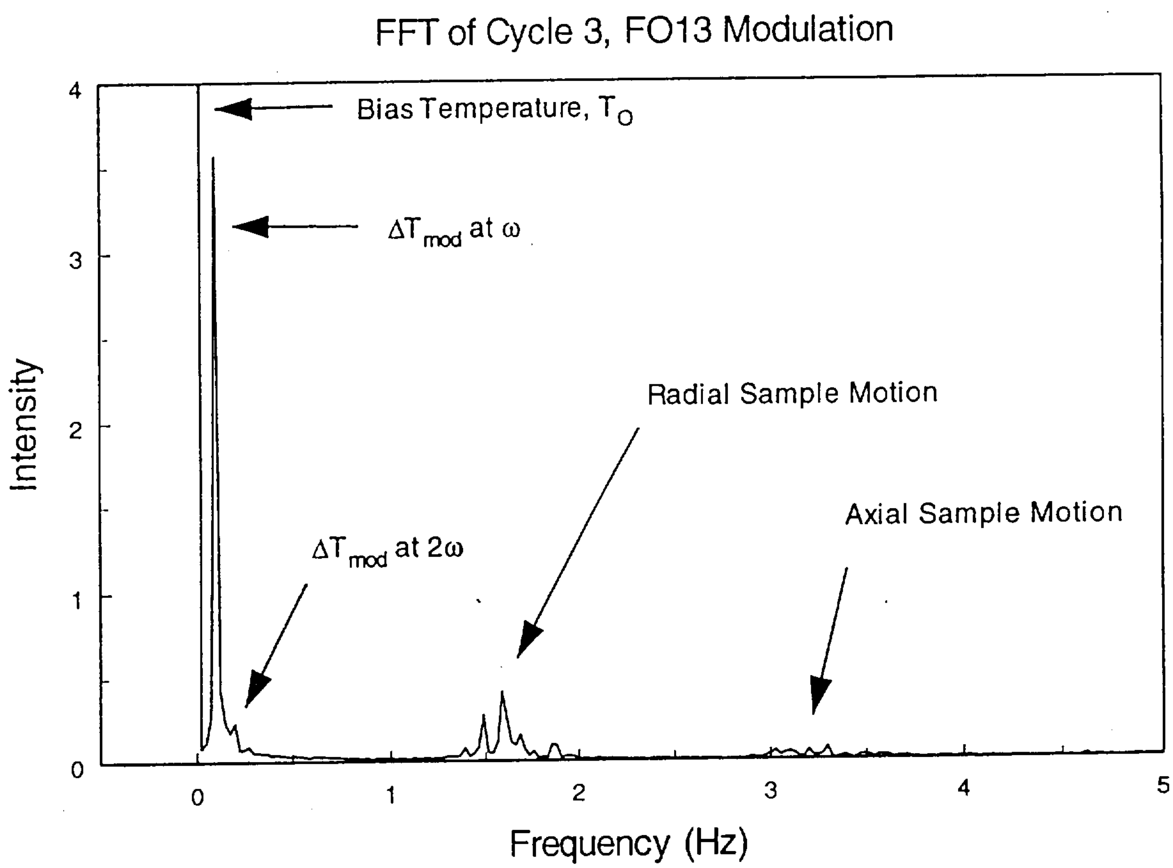

Figure 3. FFT spectrum of a NiNb modulation cycle.

Evaluation of Cp requires knowledge of the frequency dependent correction factor, $f(\omega, \tau_1, \tau_2)$, and the DC power absorbed by the sample, P_o. Because τ_1 is typically greater than τ_2 by a factor of 50, the relaxation times are well separated in frequency. At lower frequencies, the function f is dominated by the $(\omega\tau_1)^{-2}$ term, allowing f to be determined solely from measurements of τ_1, either from the temperature decay of the sample in response to a small step change in DC power or from the time dependence of the increase in ΔT_{av} with heating power modulation. The time constants measured agree to better than 2% and show a purely exponential temperature dependence that is independent of modulation frequency. From these measurements of τ_1, we can determine τ_2 at higher modulation frequencies. Note also that there is always a range in modulation frequency for which the correction function $f(\omega)$ is unity (to better than 1%). For this range of frequencies and slower, only τ_1 enters into the equations and Cp can be determined with only measurements of ΔT_m, ΔT_{av}, and T_o, and a knowledge of ε. In fact, for the range in which f is approximately unity, Cp can be determined to better than 1% with no knowledge of τ_1 (with proper temperature and power calibration). At higher modulation frequencies, a determination of τ_1 from lower frequencies allows us vary τ_2 as a free parameter and determine the thermal conductivity of the sample by fitting f to the theoretical model. As will be shown, in the stable liquid state, electromagnetic stirring forces may preclude measurement of the intrinsic thermal conductivity. Higher viscosity, undercooled liquids are required for this measurement. Nonetheless, the effects of both relaxation times are well-separated in frequency, allowing determination of C_p without knowledge of τ_2.

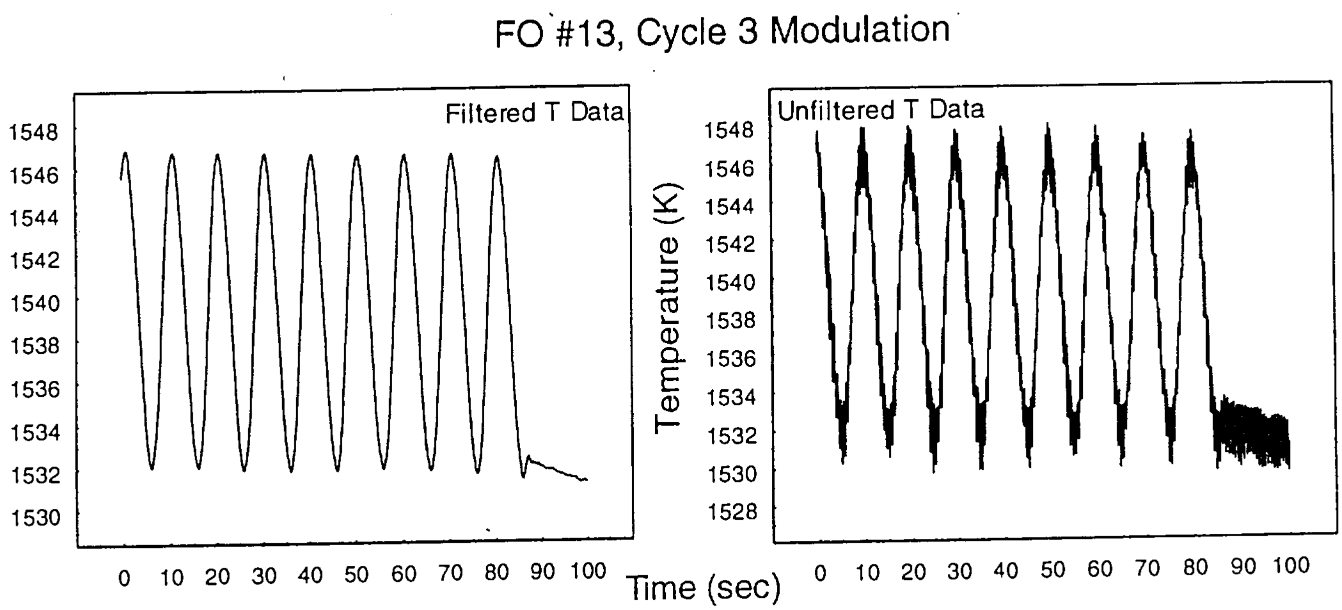

Figure 4a. Filtered data after applying 12th order Butterworth. The sample is $Ni_{60}Nb_{40}$ and the modulation frequency is 0.1Hz (the corresponding FFT spectrum is shown in Figure 3). Note the slight phase shift in the signal (compare to 4b) resulting from the filter.
Figure 4b. The original down-linked data for comparison.

For evaluation of the heat capacity, T_O, ΔT_m, ΔT_{av}, and τ_1 were measured for different modulation frequencies and samples over the temperature range 1465K to 1606K for $Ni_{60}Nb_{40}$ and over the range 1160K to 1295K in the stable liquid for $Zr_{76}Ni_{24}$.

Table I - C_p/ε Values for $Ni_{60}Nb_{40}$

T_o (K)	ω_m (Hz)	C_p/ε - measured (J/K-mol) ±3%	
1465.3	0.1	154.8	
1477.0	0.1	157.1	
1497.6	0.1	159.3	
1498.0	0.05	160.0	
1529.1	0.1	162.8	
1558.9	0.1	170.8	
1606.3	0.1	166.7	

Table II - C_p/ε Values for $Zr_{76}Ni_{24}$

T_o (K)	ω_m (Hz)	C_p/ε- measured (J/K-mol)
1160	0.1	153.0
1248	0.1	153.3
1295	0.1	150.7

We have used a drop calorimeter to measure enthalpies of liquid $Zr_{76}Ni_{24}$ and $Ni_{60}Nb_{40}$. By differentiating the enthalpy curves, it is possible to estimate (±10%) the specific heat. The sample's temperature is measured by a pyrometer only while it is in the levitation coils. Unfortunately, during the time in which the sample falls from the levitation coils to the copper block, the sample is cooled both radiatively and conductively (if a gas is used) and the amount of cooling not measured directly. Thus, the actual sample temperature is the largest error in the experiment. Moreover, the data then needs to be differentiated to obtain the specific heat, propagating and increasing sensitivity to temperature measurement error. Our drop calorimetry data is shown in Figure 5. We have fixed the value of the heat of fusion to match our results from drop calorimetry. Doing so allows us to refine Cp/ε by matching the duration of the recalescence plateau to the total heat of fusion for the sample. We have also plotted our values for Cp, choosing an ε to agree with the drop calorimetry data. This is done only to show that the temperature dependence of Cp in both experiments is similar, and *not* to imply a specific value.

Combined Cp Measurements on $Ni_{60}Nb_{40}$

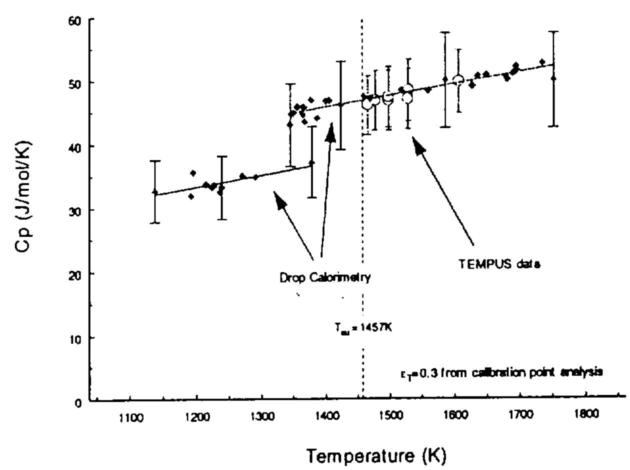

Figure 5. Drop calorimetry data plotted with data from IML-2.

The correction function f can be calculated for $Zr_{76}Ni_{24}$ from modulation data taken at T_O=1304K, using U_m= 0.401V modulation. τ_2 was varied as the free parameter to fit equation (9) for 4 different modulation frequencies, ω_m. The best fit gives $\tau_2 = 0.18$s which in turn gives a measured "thermal conductivity" of $\kappa_{th} \approx 0.14$ W/cm-s; this seems high. In comparison, the thermal conductivity for liquid Cu is 0.05 W/cm-s. We attribute this to the fact that the stable liquid has quite a low viscosity and is thus stirred significantly by the RF coil forces. Calculations performed by Szekely et al. estimate the sample to be in the turbulent flow regime. Thermal conductivity measurements are likely only possible on highly viscous, deeply undercooled liquids, as was originally intended.

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FY96 Data Update Form
Microgravity Science & Applications Program
PI Index: Task Description/Bibliography Database

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did this really expire June 1995?
NO - 3/96

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NAG Number: NAG8-954

Monitoring NASA Center:	MSFC	NRA
Task Type and Discipline:	Flight	Materials Science
Task Subdiscipline:	Metals and Alloys	
Task Identification Number:	FY 95	963-35-10
Task Initiation/Expiration:	mo/yr 2/92	mo/yr 6/95

1995/06

Degree Kind	Task-Funded Students	Task-Funded Degrees Granted
BS	0	0
MS	2	0
PhD	2	1
TOTALS	4	1

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Thermophysical Properties of Metallic Glasses and Undercooled Alloys

TASK OBJECTIVE (FOR ENTIRE LENGTH OF TASK)

☑ *NO CHANGES* MAY NEED ONLY MINOR EDITS

The objective is to study thermophysical properties of undercooled alloy melts and how they relate to glass formation. Toward this end, we have developed non-contact calorimetric methods to investigate the specific heat and thermal conductivity of these melts, both in the liquid and undercooled region. These quantities are essential for the development of newer, more advanced processing technologies for both existing and future materials.

TASK DESCRIPTION (FOR ENTIRE LENGTH OF TASK)

☑ *NO CHANGES* MAY NEED ONLY MINOR EDITS

Non-contact AC calorimetry was successfully demonstrated on the IML-2 flight in July, 1994. We obtained information on the specific heat and thermal conductivity of liquid and undercooled $Zr_{76}Ni_{24}$ and $Ni_{60}Nb_{40}$ melts using TEMPUS. This data is currently being analyzed to calculate entropy and free energy functions for these melts. We will compare these quantities to their values for the corresponding equilibrium and metastable crystals to compare the relative stability of the phases. Also, we will determine the Kauzman isentropic temperature of the alloys and compare it to the observed glass transition temperature.

In addition, the ground-based total radiance bolometer is currently being integrated onto a UHV levitation chamber for total hemispherical emissivity measurements. Measurement of temperature-dependent total hemispherical emissivity functions will allow us to unwind specific heat from undercooling data in an unambiguous manner.

TASK SIGNIFICANCE (FOR ENTIRE LENGTH OF TASK)

☑ *NO CHANGES* MAY NEED ONLY MINOR EDITS

The non-contact AC calorimetry experiment is significant for many reasons. First, the thermodynamic properties of these advanced materials are a prerequisite to the development of processing technologies for them. Without knowledge of heat capacities and thermal conductivities, it is not possible to define, for example, how much power is needed to melt and cast the materials. In addition, the specific materials chosen for our experiment are the parent compounds for a new class of bulk metallic glasses that have recently been discovered by our group here at Caltech. By studying the properties of these parent compounds, we hope to better understand the bulk metallic glasses and how they form. These materials will revolutionize metallic processing technologies with their novel, superior properties. These materials can be engineered to be more ductile, slipperier, harder, lighter and more corrosion resistant than the typical materials used today. It is essential that the processing technologies for these materials be developed as quickly as possible and that, therefore, the thermophysical properties be measured.

TASK PROGRESS (FOR FY96 ONLY)

☐ *NO CHANGES* (NEW TASKS REQUIRE NEW PROGRESS TEXT)

Task Progress for 1996

The final report for this project was submitted in September 1996. The flight data obtained on TEMPUS during the IML-2 mission have been analyzed and the results of ACMC measurements carried out during IML-2 have been reported in a number of meetings and submitted for publication in Journals as reported in the bibliography section below. In addition to the IML-2 results, we have carried out ground base studies of the glass forming liquid alloys using the High Vacuum Electrostatic Levitation method in collaboration with Dr. W.Q. Rhim's group at JPL. This work was carried out by Dr. Y.J. Kim and was used to provide ground base support for the flight experiments. In particular, undercooling studies, and crystal nucleation kinetics vs. undercooling have been measured for the glass forming alloy samples of this study using the ground based Electrostatic Levitation facility at JPL, while ground base heat capacity measurements (using DSC at Caltech), were carried out to provide a data base for comparison with results of the flight experiments.

The ACMC method is constantly being refined. Better filtering and analysis routines have been developed and used to analysis data from IML-2. A ground base electromagnetic levitation facility for use in measuring total hemispherical emissivity was built under support of this grant and is operational. It is being used to charaterized the flight samples for both the IML-2 and upcoming MSL-1 missions.

We summarize the heat capacity data obtained from the IML-2 flight experiment on one of the best analyzed IML-2 binary alloy samples, $Zr_{64}Ni_{36}$, in the table below. The data were analyzed using the method of sample impedance change during melting as described by Wunderlich et. al. (Phys. Rev. B, Rapid Comm., in press, 1996) to determine the power coupling constant to the sample.

Table. Specific heat data for liquid $Zr_{64}Ni_{36}$ obtained from IML-2 mission.

Temperature (C)	Mod. Freq. (Hz)	Total Hemispherical Emissivity	Heat Capacity (J/mole-K)
1215	0.05	0.37	43.7 +/- 0.8
1160	0.08	0.35	44.5 +/- 1.2
	0.10	0.35	43.2 +/- 1.0
1038	0.05	0.33	43.9 +/- 1.0
	0.08	0.33	44.2 +/- 1.0
1008	0.05	0.32	44.0 +/- 1.0
	0.10	0.32	44.6 +/- 1.0
980	0.05	0.32	45.5 +/- 1.2

The development of the ACMC method is being continued and refined as part of another round of experiments to be carried out on the TEMPUS facility during the upcoming MSL-1 shuttle flight. The continuing work is [illegible] under NASA Grant No. NAG9-1192.

PUBLICATIONS

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