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Containerless Processing in Space—Thermophysical Property Measurements Using Electromagnetic Levitation[1]

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Electromagnetic levitation is a novel tool for measuring thermophysical properties of high-temperature metallic melts. Contamination by a crucible is avoided, and undercooling becomes possible. By exploiting the microgravity environment of an orbiting spacecraft, the positioning fields can be further reduced and undesired side effects of these fields can be minimized. After two successful Spacelab flights of the electromagnetic levitation facility TEMPUS, an advanced electromagnetic levitation facility is presently being studied for accommodation on the International Space Station, ISS. Due to the permanent nature of the ISS, an operational concept must be defined which allows the exchange of consumables without exchanging the entire facilty. This is accomplished by a modular design, which is presented. For all experiments, like measurement of specific heat, of surface tension and viscosity, of thermal expansion, and of electrical conductivity, noncontact diagnostic tools must be either improved or developed. Such tools are, for example, pyrometry, videography (high-speed and high-resolution), and inductive measurements. This paper summarizes the scientific results obtained so far and deduces some lessons learned that will be incorporated into the new design and will lead to both new results and a higher precision of the data.

KEY WORDS: containerless processing; electromagnetic levitation; liquid metals; microgravity; thermophysical properties.

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1. INTRODUCTION

The precise knowledge of thermophysical properties becomes increasingly important as progress is made in numerical simulations of complex processes. With the improvement of the models, their reliability is limited by the accuracy of the input parameters characterizing the material under study. For high-temperature melts, such as liquid metals, containerless methods are the best choice for their measurement. Among the containerless techniques, electromagnetic levitation is especially suitable for the study of metallic melts. It can be used to obtain high temperatures, up to 2400°C, to levitate bulk samples of a few grams, and to maintain the undercooled state for an extended period of time (up to hours).

In addition to containerless positioning and melting, noncontact diagnostic tools also have to be applied or developed. For example, temperature is measured by the well-known principle of radiation pyrometry. In other cases, the necessary information is obtained by optical imaging or by inductive methods.

Some limitations exist for electromagnetic levitation when used on the ground; the required high electromagnetic fields deform the shape of a molten sample and induce turbulent currents inside the sample. In addition, the required fields are so strong that the samples must be cooled convectively using a high-purity inert gas, such as He or Ar. All these drawbacks can be avoided if electromagnetic levitation is performed in microgravity (μg). Under such conditions, the positioning forces are reduced by at least a factor of 100, and the sample is essentially force-free during the cooling phase. (The fields required for inductive heating are, of course, the same in $1g$ and in μg.)

2. TEMPUS SPACELAB FACILITY

In microgravity, only restoring forces are required to counteract the effect of the random g-jitter and residual accelerations. The TEMPUS facility [1] uses a quadrupole field for positioning, which is established by sending an electrical current in opposite directions through two sets of parallel coils of identical dimensions. For such a configuration, the stabilizing field gradients are very strong, and the power absorption is very low. TEMPUS provides stable positioning against $10^{-2}g_0$, reducing the power absorbed in the sample by a factor of 100, compared to the $1g$ case. Heating is accomplished by a high-efficiency dipole field. The TEMPUS design allows control of the heating and positioning of the sample independently, providing a quiescent sample during cooling. Temperature can be controlled over a wide range by adjusting the heating power, from approximately 400 to 2400°C, depending on the material being processed.

The experiment unit of TEMPUS consists of an ultrahigh-vacuum chamber which surrounds the levitation coils. Attached to the chamber through an axial and radial window are pyrometers and videocameras. Evaporation shields are located in the optical path between pyrometers and sample and protect the pyrometers from contamination due to the evaporation from hot samples. The samples are contained in sample holders, which are either wire cages or ceramic cups. These are loaded in the sample storage carousel. A special videocamera with high spatial resolution can be optionally mounted to the radial window. The experiment chamber is connected to the vacuum and gas systems.

3. THE MSL-1 EXPERIMENTS

3.1. Overall Performance

For the MSL-1 mission, nine science teams prepared 22 experiments on 18 samples. One of the major advantages of TEMPUS is the fact that a single sample can be used for several experiments, either in parallel or sequentially. For example, data for the measurement of electrical conductivity can be collected during an ongoing experiment concerning the specific heat. The investigated sample systems and the different thermophysical properties measured are shown in Table I.

One major concern for a successful mission was the careful monitoring of facility contamination due to evaporation from the liquid samples. To

Table I. Sample Systems Used for Experiments Performed During the MSL-1 Mission

Sample system	Specific heat	Surface tension	Viscosity	Density	Electrical resistivity
Zr		×			
$Zr_{65}Cu_{17.5}Al_{7.5}Ni_{10}$	×			×	×
$Zr_{60}Al_{10}Cu_{18}Ni_9Co_3$	×			×	
$Zr_{11}Ti_{34}Cu_{47}Ni_8$	×	×		×	
$Zr_{57}Cu_{15.4}Ni_{12.6}Nb_5Al_{10}$	×			×	
$Fe_{72}Cr_{12}Ni_{16}$		×			
$Co_{80}Pd_{20}$		×	×		×
$Pd_{82}Si_{18}$		×	×	×	
$Pd_{78}Cu_6Si_{16}$		×	×	×	

this end, the samples were categorized according to their vapor pressures. Samples with a high vapor pressure were processed in cup-shaped sample holders under an inert gas atmosphere, provided that the scientific objectives were not compromised. As is well known [2], the presence of a gas atmosphere reduces the evaporation losses by a factor of approximately 100. In addition, strict management of the admissible contamination of the two critical subsystems, coil and pyrometer, was introduced. A layer thickness of 10 μm on the coil was defined as the upper limit of acceptable contamination for the entire mission, and this resource was distributed among the individual experiments. The total amount of evaporated material during each experiment run depends on both the total processing time and the processing temperatures. Using the planned temperature–time profile, the expected contamination from each experiment was calculated preflight. During the mission, this calculation was repeated in realtime, using the actual temperatures. By taking these precautions, it was possible to process even materials with high vapor pressures, such as the FeNiCr-steels, for many hours.

In the following, no comparison is made between the results of the microgravity experiments and conventional ground-based results. This is due to the fact that, except for a few exceptions, no such data exist for the alloys and temperatures considered here. For surface tension measurements, it has been shown earlier that the microgravity data eliminate the spurious mass dependence found in previous terrestrial experiments [3]. In the case of viscosity, the data obtained during MSL-1 confirm one set of terrestrial data and help to rule out a second, much discussed data set [4].

3.2. Thermophysical Property Measurements

3.2.1. *Specific Heat*

The experiments of this class were concerned with the measurement of the specific heat of a number of glass-forming alloys. These systems do not crystallize even at cooling rates as low as $5\ \mathrm{K \cdot s^{-1}}$. They have melting points below 1000°C and glass transition temperatures around 400°C. Therefore, they cannot be undercooled in terrestrial electromagnetic levitators under UHV conditions.

A noncontact method developed by Fecht and Johnson [5] was used in these experiments. It is a variant of noncontact modulation calorimetry, normally used in low-temperature physics. The heater power is modulated according to $P_\omega(t) = \Delta P_\omega \cos(\omega t)$, resulting in a modulated temperature response ΔT_ω of the sample. If the modulation frequency ω is chosen appropriately, a simple relation for the temperature variation can be derived:

$$\Delta T_{\omega} = \Delta P_{\omega} / (\omega c_p) \tag{1}$$

from which the specific heat, c_p, can be determined.

During MSL-1, a large number of modulation cycles could be performed on different alloys, both in the equilibrium melt and in the undercooled region. The analysis of the data by Fecht and Wunderlich suggests that the specific heat increases nonlinearly in the undercooled regime [6].

3.2.2. Surface Tension and Viscosity

These experiments use the oscillating drop technique [7] to measure surface tension and viscosity. In microgravity, liquid samples perform oscillations around their spherical equilibrium shape: $R(t) = R_0(1 + \delta \cos \omega t e^{-\Gamma t})$. In that case, simple formulae can be used to relate the frequency ω and damping Γ of the oscillations to the surface tension γ and viscosity η, respectively. They read

$$\omega_R^2 = \frac{32\pi}{3} \frac{\gamma}{M} \tag{2}$$

and

$$\Gamma_K = \frac{20\pi}{3} \frac{R_0 \eta}{M} \tag{3}$$

where M is the mass of the droplet and R_0 is its radius. Under terrestrial conditions, the above relations are not valid; corrections have to be made for the external forces, namely, gravity and electromagnetic field [8, 9]. For TEMPUS MSL-1, experiments on Zr, $Co_{80}Pd_{20}$, $Pd_{82}Si_{18}$, $Pd_{78}Cu_6Si_{16}$, $Zr_{11}Ti_{34}Cu_{47}Ni_8$, and on Fe–Ni–Cr alloys were performed. During the cooling phase short heating pulses are applied to excite oscillations. The sample oscillations are recorded with two videocameras, providing axial and radial view. The values for surface tensions measured during MSL-1 are compiled in Table II.

The same data set which is used for the determination of the surface tension can also be analyzed with respect to viscosity, using Eq. (3). The data collected during MSL-1 indicate that this method works remarkably well. Measurements were performed on a wide class of materials including $Co_{80}Pd_{20}$, $Pd_{82}Si_{18}$, $Pd_{78}Cu_6Si_{16}$, and $Zr_{11}Ti_{34}Cu_{47}Ni_8$. For convenience, all viscosity data obtained during MSL-1 are parametrized according to an Arrhenius law, $\eta = \eta_0 \exp(A/T)$, and are listed in Table III.

Table II. Surface Tension of Different Alloys Measured During the MSL-1 Mission, $\gamma(T) = \gamma(T_m) + (T - T_m)(d\gamma/dT)$

Sample	$\gamma(T_m)$ ($\text{mN} \cdot \text{m}^{-1}$)	$d\gamma/dT$ ($\text{mN} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$)
Zr	1512	-0.37
$Fe_{72}Cr_{12}Ni_{16}$	1878	-1.01
$Zr_{11}Ti_{34}Cu_{47}Ni_8$	1400	-0.19
$Co_{80}Pd_{20}$	1675	-0.17
$Pd_{82}Si_{18}$	1740	-0.191
$Pd_{78}Cu_6Si_{16}$	1399	$+0.26$

3.2.3. *Thermal Expansion*

The high-resolution radial videocamera is equipped with telecentric optics, which allows for absolute size measurements of moving objects. It is used to measure the density and thermal expansion of glass-forming alloys in the undercooled regime, in particular, near the glass transition temperature. The visible cross section of the sample is recorded, and, assuming rotational symmetry, the volume is calculated. Since the mass of the sample is known and does not change, this yields the density. To observe any anomalies in the undercooled regime, a resolution of $\Delta V/V = 10^{-4}$ is required. This can be achieved using subpixel algorithms for edge detection, curve fitting of the shape, and statistical averaging [10].

Measurements were made on the following samples: $Zr_{65}Cu_{17.5}Al_{7.5}Ni_{10}$, $Zr_{60}Al_{10}Cu_{18}Ni_9Co_3$, $Zr_{11}Ti_{34}Cu_{47}Ni_8$, $Zr_{57}Cu_{15.4}Ni_{12.6}Nb_5Al_{10}$, $Pd_{78}Cu_6Si_{16}$, and $Pd_{82}Si_{18}$. Results for the volumetric thermal expansion $\alpha = (-1/V)(\partial V/\partial T)$ of these systems are listed in Table IV.

3.2.4. *Electrical Conductivity*

Finally, it was also possible to measure the electrical conductivity of undercooled melts during MSL-1. This was done using a noncontact,

Table III. Viscosity of Different Alloys Measured During the MSL-1 Mission, $\eta(T) = \eta_0 \exp(A/T)$

Sample	η_0 ($\text{mPa} \cdot \text{s}$)	A (K)
$Co_{80}Pd_{20}$	0.15	6790
$Pd_{82}Si_{18}$	0.192	5754
$Pd_{78}Cu_6Si_{16}$	0.126	6104

Table IV. Volumetric Thermal Expansion Coefficient α and Volume Change Upon Melting ΔV of Some Glass-Forming Alloys

Sample	$\alpha = 1/V(\partial V/\partial T)$ (10^{-5} K^{-1})	$\Delta V(T_l)$ (%)
$Zr_{57}Cu_{15.4}Ni_{12.6}Nb_5Al_{10}$	5.9	
$Zr_{65}Cu_{17.5}Al_{7.5}Ni_{10}$	6.8	1.2
$Zr_{11}Ti_{34}Cu_{47}Ni_8$	7.7	2.1
$Zr_{60}Al_{10}Cu_{18}Ni_9Co_3$	5.5	1.0
$Pd_{78}Cu_6Si_{16}$	7.9	
$Pd_{82}Si_{18}$	7.7	

inductive method. The levitated sample influences the impedance of the heating coils. The impedance of the coil can be determined by measuring both the current I and the voltage U simultaneously. Such measurements require that the current through the heating coil is not zero, $I \neq 0$, and therefore cannot be performed during free cooling. A small residual heating field is, however, sufficient. Such conditions were met during the experiments on $Co_{80}Pd_{20}$, and it was possible to measure the electrical resistivity of a liquid metal in the undercooled state [11]. Except for a temperature region near the Curie temperature (1250 K), the resistivity of both the solid and the liquid phases can be given by a linear relation:

$$\begin{aligned} \rho_s(T) &= 56.3 + 0.042T \qquad (\mu\Omega \cdot \text{cm}) \\ \rho_l(T) &= 65.3 + 0.050T \qquad (\mu\Omega \cdot \text{cm}) \end{aligned} \tag{4}$$

As the temperature approaches the Curie temperature, deviations from the linear behavior occur in both phases. This is due to the onset of magnetic ordering, i.e., the relative magnetic permeability is different from unity, $\mu_r \neq 1$.

4. ELECTROMAGNETIC LEVITATION FACILITY FOR THE SPACE STATION

4.1. Hardware Design

The design of an electromagnetic levitation facility to be available eventually on the International Space Station has been studied. The result was a concept where the sample transport container which is needed for keeping the samples in a sealed and inert environment will serve in orbit as the processing chamber. All the experimental capabilities of the Spacelab facility with respect to thermophysical properties and solidification studies and more shall be available within a Space Station facility. After finishing the experiments with a charge of 15 samples, the chamber/transport container will be brought back to the ground.

All devices which will be more or less contaminated by evaporating sample material due to condensation and aerosol formation will thus be replaced by a fresh set for the new sample charge. The exchangeable experiment insert with 15 samples includes the coil system connected to the rf capacitors, together forming the oscillating circuits for heating and positioning. Part of the insert also includes all vacuum windows and the evaporation shields for the optical paths for axial and radial pyrometers and cameras. The pyrometers and cameras stay in orbit but are replaceable by new developments. The insert is sealed by vacuum valves, which are opened only when the insert is connected to the vacuum module of the accommodating facility. Appropriate filtering will keep the contaminants within the insert.

Besides always having a clean processing environment for a new sample charge, the advantage of the exchangeable experiment insert is that it can be equipped with, e.g., dedicated coil systems or additional instruments which are not needed for all experiments. This design gives the flexibility for a usage of an electromagnetic levitation facility over many years.

4.2. Experiment Preparation and User Support

For the flights of TEMPUS, the Microgravity User Support Center MUSC at DLR Cologne has supported the scientists with the preparation and interactive conduct of their experiments under microgravity [12]. In addition, user support has been provided for four parabolic flight campaigns [13].

In the Ground Support Program, an assessment of the compatibility of experiment requirements with the facility specifications has been performed; all experiment procedures and facility control parameters were developed by test and simulation and verified on the TEMPUS ground models. The multiuser and multipurpose character of TEMPUS leads to a complex internal process flow so that over 80,000 process parameters were predefined for 22 experiments. In addition, resource requirements were determined, crew procedures were developed, and astronaut, as well as scientist, training was performed in the lab and during parabolic flights.

In the Mission Support Program, the technical infrastructure for the on-line interactive control of the flight experiments was set up in the Science Operations Area of the NASA Marshall Space Flight Center in Huntsville. The services to the scientists comprised a real-time database system and a variety of customized tools for the monitoring of the experiment data (such as multipurpose data displays and on-line FFT analysis) and of facility resources (such as power consumption or the amount of sample evaporation). With a commanding tool, process parameters could be optimized and prepared for up-link to the Spacelab in a user-friendly way, either before or during an ongoing experiment. With a turn-around time of only a few seconds, the highly interactive performance of the TEMPUS experiments with more than 25,000 commands sent real time has been essential for the mission success.

According to present planning, the new Electromagnetic Levitation Facility for the International Space Station will be developed under the lead of the European Space Agency ESA. For that reason, the operations concept will be similar to that for the other European multiuser payloads. ESA has decided on a decentralized approach to payload operations characterized by assigning the tasks related to the preparation for in-flight operations of their facilities to so-called User Support and Operations Centers. Based on the scientific experience, one of these USOCs is appointed as the Facility Responsible Center for a specific payload. This FRC will then provide the focal point of contact for the interested scientists and provide support for the preparation of flight experiments as well as their management, monitoring, and control during the in-flight performance. To fulfill these tasks, an FRC is equipped with a broad scientific and technical infrastructure ranging from engineering models of the ISS payloads to communications capabilities for data, voice, and video transfer from the ISS to the user, and vice versa. Based on the long experience with the operation of a microgravity electromagnetic levitiation facility, MUSC is prepared to support the operation of the new facility onboard the ISS as a Facility Responsible Center.

5. CONCLUSIONS

The MSL-1 mission has demonstrated that electromagnetic levitation in microgravity is a viable technology which provides insight into the properties of undercooled melts not obtainable by other methods. The results presented here prove the feasibility of the noncontact diagnostic methods developed for containerless processing. In some cases, the applicability of these methods remains restricted to experiments under microgravity and will, therefore, rely on future space experiments. In other cases, results obtained in space can serve as benchmark tests and validation of terrestrial experiments combined with appropriate correction formulae. Although the results presented have revealed some scientifically interesting phenomena, such as the nonmonotonic behavior of the specific heat, the main purpose was a demonstration mission. For the future, systematic studies of scientifically and technologically important alloys are proposed, using an Advanced Electromagnetic Levitation Facility on the International Space Station (ISS).

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