Containerless Processing in Space – Thermophysical Property Measurements using Electromagnetic Levitation [1]

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ABSTRACT

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 minimised.

After two successful Spacelab flights of the electromagnetic levitation facility TEMPUS, presently an Advanced Electromagnetic Levitation facility is 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 will be presented.

For all experiments, like measurement of specific heat, of surface tension and viscosity, of thermal expansion and of electrical conductivity, non-contact diagnostic tools must be either improved or developed. Such tools are, for example: pyrometry, videography (high-speed and high-resolution), inductive measurements, etc. 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 higher precision of the data.

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

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 characterising the material under study. For high temperature melts, such as liquid metals, containerless methods are the best choice for their measurement. Among the different containerless techniques, electromagnetic levitation is especially suitable for the study of metallic melts. It allows 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, also non-contact diagnostic tools have to be applied or developed. For instance, 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 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, like He or Ar. All these drawbacks can be avoided if electromagnetic levitation is performed in microgravity. In 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 reqired 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 through two sets of parallel coils of identical dimension in opposite direction. 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, as compared to the 1-g case. Heating is accomplished by a high-efficiency dipole field. The TEMPUS design allows to control 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°C to 2400°C, depending on the material being processed.

The experiment unit of TEMPUS consists of an ultra high vacuum chamber which surrounds the levitation coils. Attached to the chamber through an axial and radial window are pyrometers and video cameras. 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 carrousel. A special video camera 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, 9 science teams have 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 on-going experiment concerning the specific heat. The investigated sample systems and the different thermophysical properties measured are shown in table 1.

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 crystallise even at cooling rates as low as 5 K/s. 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 non-contact method developed by Fecht and Johnson [2] was used in these experiments. It is a variant of non-contact 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:

$$\Delta T_\omega = \Delta P_\omega / (\omega c_p) \quad (1)$$

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 non-linearly in the undercooled regime [3].

3.2.2 Surface Tension and Viscosity

These experiments use the oscillating drop technique [4] 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 frequency ω and damping Γ of the oscillations to surface tension γ and viscosity η, respectively. They read:

$$\omega_R^2 = \frac{32\pi}{3} \frac{\gamma}{M} \quad (2)$$

and:

$$\Gamma_K = \frac{20\pi}{3} \frac{R_0 \eta}{M} \quad (3)$$

where M is the mass of the droplet and R_0 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 [5,6]. 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 video cameras, providing axial and radial view. The values for surface tensions measured during MSL-1 are compiled in table 2.

The same data set which is used for the determination of the surface tension can also be analysed with respect to viscosity, using eqn (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 3.

3.2.3 Thermal Expansion

The high resolution radial video camera is equipped with telecentric optics which allows for absolute size measurements of moving objects. It is used to measure density and thermal expansion of glass-forming alloys in the undercooled regime, and, 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. In order to observe any anomalies in the undercooled regime, a resolution of $\Delta V/V = 10^{-4}$ is required. This can be achieved using sub-pixel algorithms for edge detection, curve fitting of the shape, and statistical averaging [7].

Measurements were made on 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 shown in table 4.

3.2.4 Electrical Conductivity

Finally, it was also be possible to measure the electrical conductivity of undercooled melts during MSL-1. This was done using a non-contact, inductive method. The levitated sample influences the impedance of the heating coils. The impedance of the coil can be determined by measuring both, current *I* and voltage *U* simultaneously. Such measurements require that the current through the heating coil is not zero, $I_0 \neq 0$, and can therefore not 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 [8]. Except for a temperature region near the Curie temperature (1250 K), the resistivity of both, solid and liquid phase, can be given by a linear relation:

$$\begin{aligned} \rho_s(T) &= 51.9 + 0.044\,T \ \ [\mu\Omega\text{cm}] \\ \rho_l(T) &= 69.8 + 0.058\,T \ \ [\mu\Omega\text{cm}] \end{aligned} \tag{4}$$

As the temperature approaches the Curie temperature, deviations from the linear behaviour 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 r.f. capacitors forming together the oscillating circuits for heating and positioning. Also part of the insert are 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 only opened when the insert is connected to the vacuum module of the accommodating facility. Appropriate filtering will keep the contaminants within the insert.

Beside having everytime a clean processing environment for 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 [9]. In addition, user support has been provided for four parabolic flight campaigns [10].

In the Ground Support Program, an assessment of the experiment requirements compatibility 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 multi-user and multi-purpose 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 built up in the Science Operations Area of NASA Marshall Space Flight Center in Huntsville. The services to the scientists comprised a real-time data base system and a variety of customized tools for the monitoring of the experiment data, (such as multi-purpose data displays, 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 on-going 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 as for the other European multi-user payloads. ESA has decided on a decentralized approach to payload operations characterized by assigning the tasks related to the preparation for and the 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. In order to fulfil 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 on board 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 non-contact 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, like the non-monotonic behaviour 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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Table 1 Sample systems used and experiments performed during MSL-1 mission.

Sample System	*specific heat*	*surface tension*	*viscosity*	*density*	*electrical resistivity*
Zr		x			
$Zr_{65}Cu_{17.5}Al_{7.5}Ni_{10}$	x			x	x
$Zr_{60}Al_{10}Cu_{18}Ni_{9}Co_{3}$	x			x	
$Zr_{11}Ti_{34}Cu_{47}Ni_{8}$	x	x		x	
$Zr_{57}Cu_{15.4}Ni_{12.6}Nb_{5}Al_{10}$	x			x	
$Fe_{72}Cr_{12}Ni_{16}$		x			
$Co_{80}Pd_{20}$		x	x		x
$Pd_{82}Si_{18}$		x	x	x	
$Pd_{78}Cu_{6}Si_{16}$		x	x	x	

Table 2 Surface tension of different alloys measured during MSL-1 mission

$\gamma(T) = \gamma(T_m) + (T-T_m)\, d\gamma/dT$

Sample	**$\gamma(T_m)$ [mN/m]**	**$d\gamma/dT$ [mN/mK]**
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

Table 3 Viscosity of different alloys measured during MSL-1 mission

$\eta(T) = \eta_0 \exp(A/T)$

Sample	**η_0 [mPa 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 4 Volumetric thermal expansion coefficient α of some glass-forming alloys and volume change upon melting Δ*V*.

Sample	α = ***1/V(∂V/∂T)*** **[10^{-5} K^{-1}]**	**Δ*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	