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Containerless processing in reduced gravity using the TEMPUS facility

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ABSTRACT

Containerless processing provides a high purity environment for the study of high-temperature, very reactive materials. It is an important method which provides access to the metastable state of an undercooled melt. In the absence of container walls, the nucleation rate is greatly reduced and undercooling up to $(T_m - T_n)/T_m \sim 0.2$ can be obtained, where T_m and T_n are the melting and nucleation temperatures, respectively. Electromagnetic levitation represents a method particularly well-suited for the study of metallic melts. The TEMPUS facility is a research instrument designed to perform electromagnetic levitation studies in reduced gravity. It provides temperatures up to 2600 °C, levitation of several grams of material and access to the undercooled state for an extended period of time (up to hours).

keywords: containerless processing, undercooling, solidification, levitation

1. INTRODUCTION

Containerless processing is one area of interest in materials science.^{1,2} Levitation, positioning and processing of materials which are not in direct contact with container surfaces provides a unique opportunity to study a number of phenomena. In particular, the lack of contact with container walls reduces the possibility for heterogeneous nucleation in studies of the processing of molten materials allowing access to the undercooled state. The study of the properties and solidification of undercooled materials is, therefore, facilitated by containerless processing. Electromagnetic levitation represents a method well-suited for the study of metallic melts.

Ground-based processing necessitates the use of strong magnetic fields to levitate samples against the force of gravity. Several limitations exist for ground-based electromagnetic levitation:

High electromagnetic fields deform the shape of a molten sample

High electromagnetic fields also induce turbulent flow inside the sample

Required fields are so strong that low melting temperature samples must be cooled convectively with high-purity inert gas

In microgravity, the required electromagnetic fields are greatly reduced. This offers the following advantages:

Very little deformation of the sample; spherical shape is maintained

Reduced opportunity for turbulence in the melt

Positioning field generates greatly reduced heating in the sample, and gas cooling is not required--ultra high vacuum processing is possible

Greatly diminished power dissipation as a result of the reduced positioning field permits access to a wide temperature range and facilitates temperature control.

Terrestrial levitation experiments are essentially restricted to refractory metals and good conductors. In microgravity, processing and undercooling of metals with lower melting points becomes possible. This permits the study of alloys with a deep eutectic temperature and glass forming alloys. The TEMPUS (Tiegelfreies ElektroMagnetisches Prozessieren Unter Schwerelosigkeit) facility is an electromagnetic levitation instrument designed to process samples in the microgravity environment of the Space Shuttle. The equipment was developed by Daimler-Benz Aerospace, with funding from the German

space agency (DARA), cooperation with the Microgravity and Science Applications Division of the National Aeronautics and Space Administration (NASA).

Scientific investigations in TEMPUS are categorized using four experiment classes:

Class A: Studies of undercooling phenomena and the kinetics of nucleation

Class B: Measurement of specific heat in liquid metals using an AC calorimetry technique

Class C: Measurement of viscosity and surface tension of undercooled metals

Class D: Measurement of thermal expansion in undercooled materials

Studies of undercooling and solidification are central to materials science. Certain class A experiments examine the fundamentals of nucleation, while other Class A studies examine growth velocities as a function of undercooling. Information obtained from Class B includes thermodynamic data which to provide insight into the formation of metallic glasses. Metallic glasses are an industrially important new class of materials. Metallic glasses are ductile and corrosion resistant. Metallic glasses are used in magnetic storage media films. Applications for metallic glasses have been limited to the use of materials produced by thermal quench methods which result in thin films and powders. Better insight into the formation of these materials may provide the information needed to better exploit the potential of metallic glasses. Class C and D studies provide important thermophysical property data on undercooled materials, namely viscosity, surface tension, and thermal expansion. Accurate modeling of materials processing requires such data.

2. FACILITY DESCRIPTION

Four subsystems housed in a single Space Shuttle rack comprise the TEMPUS facility.³ The subsystems consist of: the Process control and Data Acquisition Module (PDM), the Experiment Unit (EU), the High Power Supply (HPS), and the Cold plate/ Heat Exchanger (CHEX). The PDM provides process control, data acquisition and transmission, as well as the interface for crew interaction. The CHEX transfers heat generated by TEMPUS subsystems to the Spacelab water cooling loop. The HPS, tuned for operation with the TEMPUS coil system, provides power. The EU houses the processing and diagnostic equipment of the facility. As shown in Fig. 1, the levitation and heating coils are located in the EU. Table 1 summarizes technical data on many of the important capabilities of TEMPUS. A rotating sample exchange mechanism provides access to the up to 22 different samples within TEMPUS. Samples may be processed under vacuum or selected gas environments.

The performance of the coil systems determines the ability of an electromagnetic levitator to provide the magnetic fields required to meet the specified scientific objectives.^{3,4} The coils provide the electromagnetic forces to heat, position and manipulate the samples during experiments. The TEMPUS coil system consists of two differently shaped coils, one heating and one for positioning. Temperature measurements are performed by a number of pyrometers. Hofmeister and Bayuzick⁵ provide a detailed analysis of pyrometry on TEMPUS.

System/Subsystem	Description
Heating	up to 2600 C
RF system/heater (dipole)	1350 W / 360 kHz
RF system/positioner (quadrupole)	1050 W / 150 kHz
Temperature measurement/two color pyrometer/axial view	
Temperature measurement	300 °C–2400 °C
Sensor Elements	Indium arsenide
Wavelength ranges/sampling rate	1–2.5 μm/1 Hz, 100 Hz, and 1 MHz 3–4 μm/10 Hz, 1 MHz
Temperature measurement/silicon pyrometer/axial view	
Temperature range	850 °C–2000°C
Sensor Element	Silicon
Wavelength	632 nm
Sampling rates	1 Hz, 100 Hz, and 1 MHz
Axial pyrometer/evaporation shielding	exchange windows and double mirrors (18)
Axial pyrometer/measurement spot size	< 3mm
Temperature measurement/infrared detector, radial view	
Temperature range	300 °C–2400 °C
Sensor element	Indium arsenide
Wavelength range	1–2.5 μm
Sampling rates	1 Hz, 1 MHz
Evaporation shielding	double mirror
Process environment/Ultra-high vacuum	3×10 ⁻⁸ mbar
Process environment/gas He or Ar	1–400 mbar
Gas purity at filling	99.9999%
Video/observation	axial and radial views (2 cameras)
Video/resolution (non-interlaced)	horizontal/vertical 400/240 lines
Sample storage capacity	22
Sample size	7-10 mm
Magnetic damping	DC magnetic field <2-60 mT (variable)
Process control/facility	automatic by facility computer
Process control/telescience	remote commanding/interactive manual control
Process control	crew interaction

TABLE 1. TEMPUS facility technical data.

3. RESULTS AND DISCUSSION

The first mission for TEMPUS occurred in July 1994 as part of the Second International Microgravity Laboratory (IML-2) Payload on the NASA's Space Shuttle. Four NASA sponsored and four DARA sponsored investigation teams had 22 samples of different compositions with experiments from Classes A, B, and C. During the initial processing studies it became evident that the samples were often unstable. Egry et al.⁶ provide a summary of the findings from IML-2. The samples, metal spheres approximately 8 mm in diameter, were housed in separate assemblies. The assemblies included a wire cage enclosure designed to protect the coils from impact by molten samples. The magnetic field produced by the coil system caused a radial positioning offset during sample processing. The offset reduced the clearance between the sample and the wire cage. Additionally, during the mission, radial translation oscillations had a greater amplitude than anticipated. During several

experiment runs, the amplitude of the oscillations increased sufficiently that molten samples contacted and became stuck to the wire cage. Additional processing of these samples was not possible. The TEMPUS investigation team developed procedures to process samples and collect some data despite problems with the magnetic field. Achievements on IML-2 include: 48 hours of levitation time, melting and heating Zr to 2000 °C, measurement of the specific heat of NiNb and ZrNi in the slightly undercooled state, solidification of NiNb into a possible metastable state and surface tension measurements of liquid Au, AuCu and ZrNi.

Other reported hardware-related problems included generation of particulates and outgassing of polymer equipment components. The contamination compromised the desired high purity processing conditions and the scientific objectives of the IML-2 payload were not fully realized. A major source of the particulates is thought to have been abrasion during launch and transport. Each sample holder assembly included an alumina pedestal to facilitate sample deployment. The samples were not fixed in position during the intense vibrations of the Space Shuttle launch and the subsequent mission operations. Contact between the sample and pedestal or wire cage materials could have generated particles. Post-flight analysis of the samples confirmed the presence of alumina particles, as well as particles traced to many of the flight samples.⁶

Additionally, during IML-2, to obtain stable positioning it was necessary to apply much higher magnetic fields than called for in the nominal experiment protocols. Flows induced by the high magnetic fields precluded the quiescent conditions required by many of the scientific investigations. The design and evaluation of a new coil system represent a major undertaking in the modification of TEMPUS facility for any future Space Shuttle missions. Design efforts included theoretical/modeling studies as well as field mapping and torque measurements on candidate coil systems. TEMPUS is unable to levitate samples against 1-g acceleration; ground-based performance testing is not possible.

Performance tests were undertaken in reduced gravity on NASA's KC-135 aircraft. This KC-135 aircraft is a research platform for reduced gravity experimentation and astronaut training. When flown in a special parabolic trajectory, the plane provides approximately 20 seconds of reduced gravity. Under good conditions, the plane can provide acceleration levels of approximately 0.01g. During turbulent flying conditions, the acceleration environment in the reduced gravity portion of the parabola can vary greatly in both magnitude (up to 0.1g) and direction (up vs. down). This aircraft presented a challenging test environment for the TEMPUS coil systems, designed to operate in the microgravity environment of the Space Shuttle.

Verification studies utilized the engineering development model of TEMPUS system and selected candidate coil systems on the KC-135 aircraft. Testing included procedures to examine the positioning stability of a variety of solid and molten samples. A preliminary campaign completed in June 1995 indicated that the newly designed coil systems provided superior performance when compared to the coil system used on IML-2. A second campaign, in January 1996 demonstrated that the coil system designated E2A3 could stably position molten samples against disturbances on the order of 100 milli-g. The coil was able to melt and stably position a number of materials, including Zr, CoPd, Ni and AuCu.⁸

Other facility modifications include the design of a sample launch-lock mechanism to help eliminate particulate contamination due to sample vibration during launch. A cup-shaped sample holder is proposed for a number of investigations to help shield the coils from the deposition of sample materials due to evaporation. Outgassing tests and careful selection of equipment components should help eliminate other sources of contamination. A residual gas analyzer has been added to the unit to TEMPUS to monitor the composition of gases within the process chamber.

4. FUTURE PLANS

TEMPUS is manifested for a reflight on NASA's first Microgravity Science Laboratory Mission (MSL-1), scheduled to launch in 1997. The payload will support 10 principal investigators, listed in Tables 2 and 3. Twenty two sample systems have been proposed for study, with studies from each of the four experiment classes represented. In many cases the investigation teams will share samples. For example, teams which study thermophysical properties will apply their techniques to samples specified by teams with other research interests. These interactions help maximize the return of scientific data from MSL-1.

Principal Investigator	Institution	Experiment Title	Sample Material at%
Dr. Bayuzick	Vanderbilt Univ.	Effects on Nucleation by Containerless Processing in Low Earth Orbit	Zr
Dr. Flemings	MIT	Alloy Undercooling Experiments	Fe ₇₀ Cr ₁₅ Ni ₁₅ Fe ₇₀ Cr ₁₈ Ni ₁₂ Fe ₇₀ Cr ₁₆ Ni ₁₄
Dr. Johnson	California Institute of Technology	AC Calorimetry and Thermophysical Properties of Bulk Glass Forming Metallic Liquids--A Flight Experiment Employing the TEMPUS hardware	Zr ₆₀ Ni ₂₀ Cu ₂₀ Zr ₁₁ Ti ₃₄ Cu ₃₇ Ni ₈ ZrCuNiNbAl
Dr. Szekely(deceased) Drs. Flemings/Trapaga (continuation PI team)	MIT	Measurement of the Viscosity and Surface Tension of the Undercooled Melts Under Microgravity Conditions and Supporting Magnetohydrodynamic Calculations	Au Au ₅₆ Cu ₄₄ Pd ₈₂ Si ₁₈

Table 2. NASA sponsored investigations.

Principal Investigator	Institution	Experiment Title	Sample Material at%
Dr. Egry	DLR, Institute for Space Simulation	Thermophysical Properties of Undercooled Metallic Melts	Co ₈₀ Pd ₂₀ Pd ₇₈ Cu ₆ Si ₁₆
Dr. Fecht	TU Berlin, Institute for Metal Research	Thermophysical Properties of Advanced Materials in the Undercooled State	Zr ₆₄ Ni ₃₆ Zr ₆₅ Cu _{17.5} Al _{7.5} Ni ₁₀ Zr ₆₀ Al ₁₀ Cu ₁₈ Ni ₉ Co ₃
Dr. Froberg	TU Berlin, Institute of Metallic Materials and General Metallurgy	Measurement of the Surface Tension of Liquid and Undercooled Metallic Alloys by Oscillating Drop Technique	Shares samples with other PI teams
Dr. Herlach	DLR, Institute for Space Research	Comparative Dendrite Velocity Measurement on Pure Ni and a Dilute Ni-C alloy	Ni NiC _{0.6}
Dr. Herlach	DLR, Institute for Space Research	Undercooled Melts of Alloys with Polytetrahedral Short-Range Order	Al ₆₀ Cu ₃₄ Fe ₆ Al ₆₅ Cu ₂₅ Co ₁₀ Al ₆₄ Cu ₂₂ Co ₁₄
Dr. Samwer	University of Augsburg, Institute for Physics	Thermal Expansion of Glass Forming Metallic Alloys in the Undercooled State	Shares samples with other PI teams

TABLE 3. DARA sponsored investigations.

5. ACKNOWLEDGMENTS

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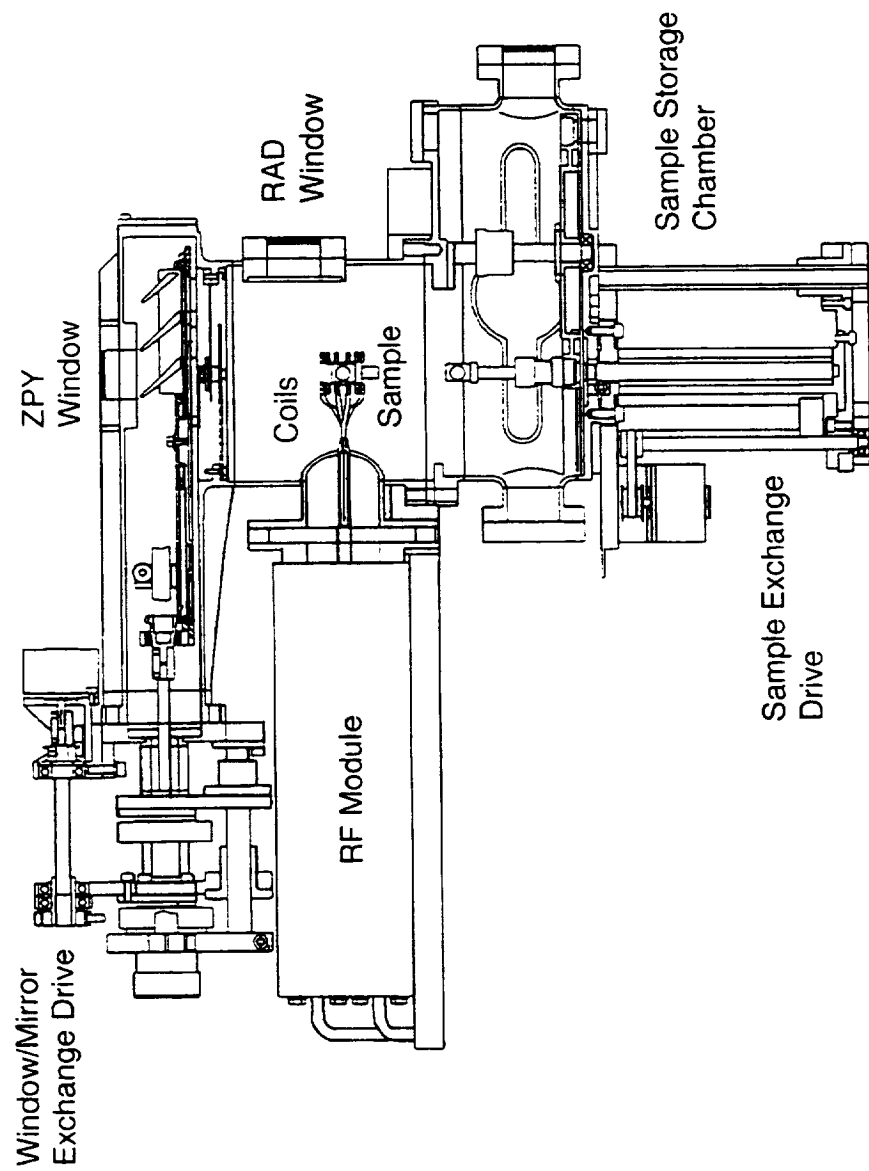


Figure 1. TEMPUS Experiment Unit Side View