Materials Science Investigations using Electromagnetic Levitation

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Abstract. EML on ISS allows levitating liquid samples both above and below their melting points for extended periods under ultra-high vacuum or ultra clean noble gas atmosphere. Various stimuli can be applied to the samples for dedicated experiment objectives. The heat input into the sample can be modulated to induce a thermal response of the sample, short heater pulses can be used to induce surface shape oscillations of the liquid sample, a custom made trigger needle can be driven into the undercooled sample to induce heterogeneous nucleation at a predefined temperature, touching of the sample by a dedicated chill cool plate or application of a forced gas flow can be used to increase the cooling rate of the sample or to simulate convection for reference experiments. Dedicated diagnostics elements are available to measure the physical properties of the sample. Sample temperature is measured by a pyrometer; two video units in orthogonal views provide both high spatial (up to 1 Megapixel and relative size resolution 2 * 10^-4) and temporal (up to 30 kHz) resolution. Additional capabilities are under discussion which would allow to measure the electrical conductivity of the sample from electrical data of the rf coil system, and to determine the residual oxygen content of the process atmosphere.

1. Scientific Objectives

Electromagnetic levitation provides unique opportunities for the investigation of electrically conductive liquid metals, alloys and semiconductors (if pre-heated or doped), both above and below their melting temperatures. In particular, the undercooled regime where the sample is liquid at temperatures below its equilibrium melting temperature is accessible through electromagnetic levitation thus allowing the determination of thermo-physical properties of materials in this particular state as well as studying of nucleation and solidification phenomena.

Thermo-physical properties that can be measured comprise heat capacity, enthalpy of fusion, thermal transport coefficients in the liquid phase, viscosity, surface tension, thermal expansion coefficient, hemispherical emissivity, fraction solid and electrical conductivity. In studies of nucleation and solidification phenomena the nucleation kinetics and solidification velocity can be determined. [1], [2]

The results gathered by this technique can improve our understanding of the solidification process and of the physics of undercooled melts, and are therefore useful for both, pure science and practical applications. For example, in the industrial production a large fraction of all metallic materials is made from the melt. The solidification process in applications like casting determines the material properties

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and product quality; therefore the results of the process are influenced by the properties of the material while it is undercooled. In order to improve the casting process and to avoid high costs due to iterations of the process, numerical simulations of the melt processing including casting and solidification is increasingly applied in industrial production. The quality and predictive power of the simulation critically relies on the availability of reliable thermo-physical data in the liquid phase, however there is a lack of such data due to the high chemical reactivity of high-temperature metals in the liquid phase with any crucible. Containerless processing can provide thermophysical data that are otherwise not available and can therefore help improve the modeling of casting processes.

In a recent industry survey conducted among European industries the need for better thermo-physical properties became evident, in particular in the industries dealing with casting, metal and alloy production and refinement [3].

2. Ingredients
In order to prepare the undercooled state and to perform experiments aiming at the determination of the thermo-physical properties without adverse impacts related to chemical reactions, heterogeneous nucleation, or lack of visibility, the following ingredients are essential:

2.1. Electromagnetic levitation
Electromagnetic levitation allows processing of the samples without contact to a container. A wide range of electrically conductive samples including pure metals, alloys and semiconductors can be processed with this technique. The sample is placed in the center of a coil system which is part of an oscillating RF circuit and generates an RF electromagnetic field. The interaction of eddy currents induced in the sample with the electromagnetic field leads to a displacement force which keeps the sample at the center of the coil system. Heating is achieved by ohmic losses of the eddy currents flowing in the sample. In ground-based experiments the required levitation force and therefore electromagnetic field strength to counteract gravity is so large that many materials are melted just by applying the field to position them. Heating and positioning of the sample are therefore not independent and the undercooled regime of samples with low melting temperatures is not accessible. Furthermore, the electromagnetic pressure exerted by the strong fields leads to strong convection and a deformation of the liquid samples which are not compatible with many experiment objectives such as the determination of viscosity and surface tension. The need for lower electromagnetic field strengths thus becomes evident which means that gravity has to be largely eliminated in order to perform experiments with undisturbed samples over a wide temperature range [4] ... [8].

2.2. Ultraclean process environment:
In order to reduce the potential for heterogeneous nucleation as much as possible an ultra-high vacuum or ultra-pure noble gas process atmosphere is necessary. In particular, the amounts of reactive trace gases, like for example oxygen, need to be minimized.

2.3. Contactless diagnostics
The diagnostic instruments have to be compatible with the containerless processing principle as well, such as pyrometers for measurement of the sample temperature and various dedicated video cameras for specific purposes, e.g. cameras with high spatial or temporal resolution.

2.4. Sample handling
Even though the samples are processed without contact to a container wall they need to be carefully prepared and handled during the experiment. Typical experimental set-ups provide the possibility to store several samples, to select a dedicated sample for processing and to transfer the selected sample into the coil. Therefore, the samples are placed in so-called sample holders made of chemically inert thermal shock resistant material which are connected to a transfer mechanism and provide enough free space for the sample such that the containerless principle is respected during processing.
3. Undercooling at Work

Driven by the size of the RF coils which are optimized for positioning and heating efficiency under microgravity the samples are spherical with diameters typically ranging from 5 to 8 mm. The samples are manufactured by the scientists in their labs and are then stored and transferred under controlled environmental conditions into the experimental setup where they are processed. The process environment for the samples is either UHV or ultra pure noble gas (He or Ar) up to 400 mbar.

A typical experiment consists of several melt cycles. Starting from the solid state the sample is heated up above the melting temperature (overheating of up to 300 K), and then the heater power into the sample is reduced. During the subsequent cooling phase, the majority of the experimental data are gathered. Depending on its properties the sample undercools to temperatures below the melting temperature until solidification sets in rapidly and the heat of fusion is released in a very short time such that the sample temperature rises again to the melting temperature, a phenomenon known as recalescence. During the following cool down of the solid sample the cooling rate can be enhanced by additional convective cooling with the process gas being circulated through a pipe and filter system and released through a nozzle at the sample's vicinity. A typical melt cycle is shown in figure 2.

Due to the small size and related low heat content of the sample the typical duration of a melt cycle is in the order of seconds to minutes, depending on the sample material and the definition of the process, such that many such melt cycles can be performed during a the time frame available for an on-orbit experiment which is in the order of hours.

At high temperatures samples processed in ultra-high vacuum may evaporate some material which leads to a deposition of evaporated material on the inner surfaces of the experiment set-up in a physical vapor deposition process. Elements that are sensitive to deposition like the components of the optical path from the sample to the pyrometer and video cameras have to be protected from deposition.
by e.g. exchangeable double mirror systems. If samples are processed in a noble gas atmosphere the evaporated material does coagulate into microscopic dust particles which are toxic and need to be removed from the experiment setup by a dedicated gas cleaning system.

After processing samples can be retrieved from the facility and returned to ground for further evaluation.

![Figure 2](image-url)

**Figure 2.** Principle sketch of the temperature-time profile of a typical melt cycle in the EML facility. Experiments are typically performed on the cooling flank when the sample is in the liquid state.

4. **EML on ISS: Experiment Classes and Measurement Capabilities**

Based on a long and successful heritage of electromagnetic levitation facilities on various carriers and missions, the electromagnetic levitation facility EML for the International Space Station ISS is currently being developed by Astrium under contracts to ESA and DLR. A comprehensive discussion of the performance and design features of EML is provided in [9].

The EML facility supports 5 classes of experiments which cover a wide range of scientific objectives. An overview of the experiment classes and the main physical parameters that are measured is provided in Table 1 below.
Table 1. The five EML Experiment Classes.

| Class       | Description                                                                 |
|-------------|-----------------------------------------------------------------------------|
| Class A:    | Undercooling and nucleation                                                 |
|             | Solidification front speed, nucleation statistics and phase selection &      |
|             | morphology                                                                  |
| Class B:    | Modulation Calorimetry                                                     |
|             | Heat capacity, heat of fusion, total hemispherical emissivity, thermal      |
|             | transport coefficients, fraction solid                                      |
| Class C:    | Surface oscillations                                                        |
|             | Surface tension, viscosity                                                  |
| Class D:    | Size Measurement                                                            |
|             | Thermal expansion                                                           |
| Class E:    | Electrical Coupling                                                         |
|             | Electrical conductivity                                                     |

4.1. Class A: Undercooling and nucleation

The scientific objectives for this experiment are to:

1. measure solidification front speed
2. perform nucleation statistics (maximum undercooling)
3. study morphology and phase selection depending on undercooling and induced fluid flow [10]

An experiment of class A type is being executed as follows: The molten sample will be cooled by turning off the heater, whereas the cooling rate may be enhanced by dedicated convective cooling per adjustable gas flow. Recalescence may occur either spontaneously or it can be triggered by touching the undercooled sample with a custom-made trigger needle at a predefined undercooling temperature. The recalescence of the undercooled sample is detected automatically by an algorithm that detects the sudden increase of the pyrometer signal upon recalescence. A high-speed video camera is used to observe the sample from a direction perpendicular to the trigger needle such that the nucleation front passing from the nucleation point (trigger needle tip) across the surface of the sample can be observed. Once recalescence has been detected the high-speed video acquisition is stopped. Pre- and post-event storage of video data is facilitated by the use of a ring memory for the video data. See also the first pictogram in table 1.

The positioning field can be varied to determine the effect on the induced fluid flow versus the Marangoni convection prior to solidification. Typical duration of one melt cycle is in the range of 30-60 seconds.

The temperature is recorded with a frequency of 100 Hz; video images are taken with up to 30,000 frames/s @ 256x256 pixel spatial resolution. An example of high-speed video recording is shown in figure 3.

The following equipment is needed for the execution of a class A experiment:

- high-speed and high resolution camera to resolve solidification process, this includes high digitalization of the camera chip to support good thermal resolution of the phases solidifying
- trigger needle to induce nucleation at desired temperatures of the undercooled liquid
- precision pyrometer
4.2. Class B: Modulation Calorimetry

The scientific objectives for this experiment class are to measure thermo-physical properties and if applicable their temperature dependence [3] as e.g.

1. Heat capacity
2. Enthalpy of fusion
3. Total hemispherical emissivity
4. Thermal transport coefficients
5. Fraction solid

The class B experiment is carried out as follows: At a constant base temperature of the molten sample the heater power is modulated sinusoidally and the sample's temperature response is evaluated. The modulation can be repeated at different sample temperatures with different modulation frequencies in order to obtain several data points within one melt cycle. The typical duration of a melt cycle ranges from 10 to 20 minutes. The temperature data will be acquired with 100 Hz; furthermore input data of RF coil system (voltage, current) are needed.

The following equipment is mandatory to perform a class B experiment:
- High precision pyrometer
- Calibrated modulated power input source (amplitude or power modulation is possible)

![Figure 3. Class A Experiment: Sequence of a NiAl sample solidifying from the undercooled liquid captured by high-speed video camera @ 10,000 frames/s during parabolic flight. The total duration of the recalescence event was 29.1ms.](image)

4.3. Class C: Surface Oscillations

The measurement of surface tension and viscosity and their temperature dependence are the main scientific objectives for these experiments.

The experiment starts with heating up the sample slightly above its melting point. During subsequent cooling of the molten sample a short heater pulse is applied which excites surface oscillations due to the dipole nature of the heating field, and the radial force of the field squeezing the sample. The surface oscillations are imaged by two video cameras in orthogonal views. From the
oscillating frequency the surface tension can be derived and from the oscillations decay the viscosity can be derived since this is the only damping mechanism involved for these oscillations. The temperature as well as the stimulus (pulse amplitude & shape) can be altered to achieve various measurement points during one melt cycle. For increasing viscosity the amplitude can be increased to observe a high enough oscillation signal. Instead of an excitation pulse also a frequency sweep can be applied to find the resonance frequency of the sample's surface oscillations. The typical duration of a melt cycle is in the range of 5-15 min.

The video images are usually taken with the radial camera @ 200 frames/s and 600x600 pixel spatial resolution and with axial camera @ 150 frames/s and 350x350 pixel spatial resolution. An example is shown in figure 4. The temperature is recorded with 100 Hz.

To perform a class C experiment the following equipment must be provided:

- fast and high resolution video cameras to resolve shape oscillations such that offline Fourier analysis on the sample diameter can be performed to derive the oscillation frequency and decay time
- high precision pyrometer
- free programmable power input source to apply either short pulses or a user defined signal shape as e.g. frequency sweep

Figure 4. Class C experiment: Induced surface oscillations of a CuSnP sample (TEMPUS, 600x600x pixels @ 200 fps)

4.4. Class D: Size Measurement

The scientific target for this class of experiments is to measure the thermal expansion coefficient and its temperature dependence. It is carried out as follows: The diameter of the solid and molten sample is precisely determined by the two video cameras in orthogonal view along the changing temperature of a melt cycle. For this purpose a high spatial resolution of the camera is necessary which will be even enhanced by a sub-pixel resolution algorithm which is applied to the video images off-line during evaluation. The algorithm requires the image of the sample's edge to be smeared out over a small number of camera pixels such that the algorithm can fit a 50% intensity value, as assumed sample edge, with a precision higher than the pixel resolution. The slightly blurred imaging of the sample's equator is achieved by setting the camera's focal plane slightly in front of it still achieving a sharp picture of the sample's surface. The typical duration of a melt cycle is in the range of 1-5 min. Video images captured by the axial camera are taken with typically 25 frames/s at up to 1000x1000 pixel resolution, whereas the radial camera offers 600x600 pixel spatial resolution. The selected frame rate depends on the temperature gradient and the desired temperature resolution of individual measurement points. The temperature data are recorded with 100 Hz.

The following equipment is needed for the execution of a class D experiment:

- high resolution video cameras to resolve sample diameter
4.5. Class E: Electrical coupling

The measurement of the electrical conductivity and its temperature dependence are the scientific targets for these investigations. A class E experiment is carried out as follows: The electrical data of the RF oscillating circuit are used to derive the electrical conductivity of the sample since the sample itself is changing slightly the characteristics of the RF oscillating circuit by its inductivity, which is determined by its geometry and electrical conductivity. Some calibration measurements on the empty coil have to be performed to distinguish between the coil's inductivity and the one introduced by the sample. The relevant housekeeping data are measured by the levitation power supply which uses them mainly for internal control of the high RF power output. The basic resolution and accuracy can be further enhanced by using dedicated measurement equipment for this task-the so-called SCE which is described later.

The measurement can be performed together with other experiment classes since it requires only the high precision pyrometer as diagnostics, as all the other experiments too, and the RF oscillating circuit data which are part of the regular EML housekeeping data anyway. The acquisition frequency for the temperature data is 100 Hz, the RF oscillating circuit data (voltage, current, frequency) are recorded with 10 Hz.

5. Experiment preparation

Experiment preparation involves all aspects of transforming a scientific objective or idea into a fully developed flight experiment. Since EML provides a wide range of functions related to the definition of the sample processing including diagnostics, a deep understanding of the associated systems, their capabilities, functions, interrelations and constraints is needed in order to develop a successful flight experiment. Support to the scientists for the preparation of their experiments is given in the frame of the Ground Support Program GSP by a joint team consisting of Astrium and the DLR Microgravity User Support Center MUSC. Thereby the experience made in former TEMPUS missions is fully provided to the scientific community. A brief outline of the support tasks is given hereafter.

From the EML facility operation point of view the experiments are organized into melt cycles which in turn are decomposed into process steps. Each process step is characterized by a number of EML facility settings such as heater power, camera speed, etc. These settings are called Experiment Parameters or EP. In order to perform an experiment in the EML facility all EPs belonging to this experiment are needed since the EML facility is being operated using these parameters.

One of the major tasks of the GSP is therefore to translate an experiment idea into an EP set, a task that involves multiple steps and activities (see also figure 5):

- A rough outline of the experiment is developed in standardized form which is already organized with the detailed implementation in mind, i.e. with defined melt cycles, desired temperature-time profiles, diagnostics requirements in each phase etc. The result is captured in so-called "theater scripts". This work is done in close collaboration with the scientists and is supported by a simulator which provides a first indication on how the desired experiment can be implemented.
- In order to design an EML experiment with a sample and to define the heater and positioner settings the coupling of the sample to the rf field, the emissivity of the sample and the cooling of the sample by the gas atmosphere must be known precisely. These parameters are determined by the GSP in dedicated ground facilities using ground reference samples that are provided by the scientists.
- Based on the information contained in the theater scripts and the results of the sample characterization measurements, the detailed experiment parameters for each melt cycle and process phase can now be developed. This step is again performed in close collaboration with the scientists and is supported by the simulator which is now used to determine the heater and positioner settings needed to achieve the desired temperature-time profiles using the results of
the sample characterization measurements as inputs to the underlying thermo physical model. The result of this step is an EP set which is essentially a table of EML facility settings.

- In a next step the EP sets are verified in a representative EML facility. The objective of this step is to verify that the EP set is compatible with the scientific objectives and the EML facility such that sufficient confidence is achieved that the actual flight experiment in the EML facility on ISS can actually be run based on this EP set. After successful verification the EP sets are ready to be loaded into the EML facility.

- The preparation of the flight samples by the scientists is supported by the GSP by providing requirements for the flight samples and support to the verification of these requirements. This aspect includes for example dimensional requirements, verification of the sample composition, and transportation of the flight samples to the integration site in a controlled environment.

- After delivery to the project the flight samples will be integrated into their sample holders and then into the Sample Chamber in a clean environment. The fully integrated Sample Chamber will then be shipped to the launch site, uploaded to the ISS, attached to EML, and is then ready for the execution of the experiments.

**Figure 5.** Preparation tasks for conducting experiments in EML onboard the ISS.

### 6. Advanced experimental techniques

The scientific outcome of the experiments as described in Chapter 4.1 may be significantly improved by implementing the following two upgrades of the EML facility [1], [9]:

- Oxygen control system
- Sample coupling electronics (SCE).

Oxygen represents a serious and potentially harmful contaminant to many materials at elevated temperatures due to its high chemical reactivity. Especially for experiments on the determination of the viscosity and surface tension of levitated metal melts, which are part of the EML scientific programme, the presence of oxygen in the atmosphere might lead to a contamination of the sample with oxygen leading to the formation of a surface oxide layer or to dissolution of oxygen into the liquid sample. This could significantly alter the physical parameters of the sample to be determined and finally the experimental results.

Furthermore, the sample itself may act as an oxygen source or sink, depending on the preparation procedure. Hence, processing the sample in the liquid state could lead to a release or absorption of oxygen to or from the process atmosphere over several orders of magnitude. In both cases, a non-steady condition concerning the process atmosphere is apparent which makes the interpretation of the measured data quite difficult and ambiguous.
For these reasons the precise knowledge of the residual oxygen in the process atmosphere and its control is essential. The oxygen control system as introduced in [11] is suited to measure the partial pressure of oxygen and to control this value within short time constants. Hence, the oxygen concentration in the process atmosphere could be kept constant during the entire experiment within a range of a few percent and a broad range of partial pressures could be adjusted ($p_{O_2} = 1 \times 10^{-19}$ bar), see also figure 6 for reference. This would enable systematic investigations on the surface tension and/or viscosity of samples versus partial pressure of oxygen at a constant temperature [12].

The second major target for expanding the scientific potential of EML experiments in an upgraded facility would be to measure the electrical conductivity of the sample at a much higher precision than possible with the EML baseline feature using the high power RF supply as diagnostic tool. With the help of the dedicated diagnostics SCE the parameter electrical conductivity would complete the list of thermo-physical data for materials in the under-cooled state which are relevant for processing these industrial metallic alloys in terrestrial applications. The measurement principle of the SCE is contactless using induction where the sample changes by its properties the characteristics of the oscillating circuit. Hence the SCE would directly connected to a dedicated interface of the RF oscillating circuit in order to pick up the subtle changes to the field by its sensitive measurement circuitry. Moreover the SCE device as described in [13] allows also measuring the surface tension and viscosity due to its high sensitivity and fast response time such that even sample shape oscillations can be detected. Thus the SCE is a perfect complement to the optical diagnostic tools.

![Graph](image)

**Figure 6.** Range of oxygen partial pressure of oxygen as controlled by oxygen control system.
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