Application of modulated calorimetry to the liquid metals using electromagnetic levitation and static magnetic field

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Abstract
Measurement of the thermophysical properties of liquid metals is challenging because of their high chemical activity and high temperatures. The electromagnetic levitation allows one to hold the electrically conductive liquid sample containerless in an inert atmosphere in thermal equilibrium while measurements on the sample can be taken in a non-contact way followed by extraction of some thermophysical properties. Yet, the electromagnetic forces within the skin layer inside the sample cause convective flow of the liquid thus disabling the data extraction. A static magnetic field imposed over a sample is known to damp the convective flow. With these ideas, an experimental set-up with a DC magnetic field directed perpendicular to the gravity vector was constructed and first experiments were performed with liquid Ni and some other materials. In most of the experiments the instability of the levitated sample during a slow variation of the DC magnetic field was observed which is reported in the present article.

Key words: modulated calorimetry, electromagnetic levitation, DC magnetic field, liquid metals, thermophysical properties

Introduction

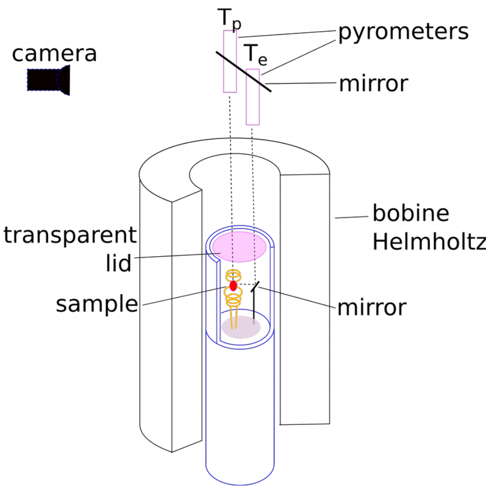
Measuring of thermophysical properties of the most liquid metals and their alloys is a challenging problem, related to their high melting temperatures and chemical reactivities. The latter implies that it is preferably to keep the liquid sample under an inert atmoshpere and without any contact with a container, i.e. in levitation. A good electrical conductivity of the liquid metals allows one to use the electromagnetic levitation in a field created by a high frequency (HF) monophase AC current which circulates in an inductor (e.m. levitator). Simultaneously, due to the Joule heating, the sample can be heated and kept at different temperatures in thermal equilibrium. Further, a modulated calorymetry can be applied which consists in perturbing the thermal equilibrium of the sample by a modulated heating. The latter can be done either using the same e.m. levitator with a specially adapted modulation of a HF current [1] or with use of the laser heating [2]. The “thermal response” of the sample (i.e. the temperature of the latter in a certain zone) to the perturbation is registered and analysed. In a case of a purely conductive heat transfer inside the sample and with use of an appropriate modulation of the heating it is possible to extract the data about the specific heat C_p of the sample and its thermal conductivity κ. Such procedure has been succefully applied to various samples under microgravity conditions on the International Space Station [1] where the electromagnetic levitation is required, in fact, only to stabilize the sample in space, but there is no need to overcome the gravity. That makes possible of a special adaptation of a geometry of the e.m. levitator to minimize the convective flow in the sample which nevertheless remains non-zero.

The possibility of the application of the same measurement procedure in on-ground condition is highly attractive, yet related to many technical difficulties. In fact, the electromagnetic forces required to keep the liquid sample in levitation create strong convection inside the droplet [3] thus making the data processing adapted to the coductive heat transfer invalid and data extraction impossible. However, a static magnetic field (DC field) can be used to damp the convective flow. This idea was realized in [4]-[7] with a DC field parallel to the gravity vector, i.e. directed along the axis of symmetry of the e.m. levitator (with neglecting the helicity of the latter). The intensity of the DC magnetic field necessary to damp any oscillation of the droplet’s shape was found to be higher than 3T. The mesurement procedure developed in [4-6] is based on a modulated laser heating of the sample in its equatorial zone and mesurement of the temperature of the pole of the sample. In principle, such procedure requires preliminary knowledge of the optimal frequency of the modulated heating as well as the size of the equatorial and polar zones within which the temperature is supposed to be uniform.

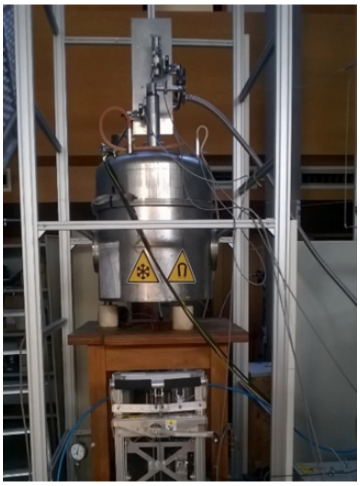
A mesurement protocol, proposed in [8], is similar to the one used at International Space Station and does not require additional equipement (laser) thus it is simpler than the one described above since the heating of the sample is produced by the same e.m. inductor. Another advantage of the proposed protocol is the use of a heating in a form of a white noise with a fixed amplitude instead of its sinusoidal modulation with a predefined frequency. Further data processing based on the theory of the dynamic systems allows one to define all the parameters, namely, C_p, κ, and total emissivity ε provided the power input into the sample is known.

Experimental set-up and measurement procedure

The scheme of the experimental set-up is presented briefly below and can be found elsewhere [9]. A Helmholtz coil (superconducting magnet) and a double wall water-cooled steel chamber are the two main parts of the set-up (Fig. 1). The chamber contains water cooled electromagnetic inductor which is mounted over a base together with a mirror allowing for the observation of the lateral side of the sample. The top of the chamber is covered with a transparent quartz lid through which the sample and the mirror are visbile. Two pyrometers are mounted above the Helmoltz coil and measure the temperature of the polar and equatorial zone of the sample. Another mirror mounted close to the pyrometers allows us to registered the image of the sample in two projections due to its reflection in the lateral mirror. Before the measuring procedure the chamber containig the levitator and the sample is pumped to obtain a low vacuum and is filled with the argon after that.



a



b

Fig. 1: The scheme of the experimental set-up (a) and the photo of the Helmholtz coil with pyrometers mounted above it and a water-cooled chamber inside (b)

The variation of the control parameters during the measurement procedure is shown schematically in Fig. 2, where V is a voltage commanded on the generator to be supplied to the e.m. levitator and B is the variation of the intensity of the DC field inside the supermagnet. At the beginning of the experiment the sample is placed at the bottom of the electromagnetic levitator. At the time t_0 we switch on and increase the voltage supply to the levitator up to a predefined fixed value as fast as possible to make the solid sample levitate and further to melt it already in levitation. The electric current supplied to the Helmoltz bobine is switched on when the sample is liquid, i.e. after the Curie temperature is attained that can be seen duering the experiment due to temperature measurement. The speed at which the intensity of the DC magnetic field increases during the period from t_2 to t_3 is limited by techical conditions. When a required value of a DC field is reached, the modulation of the voltage is performed with a lower (m1) and higher (m2) amplitude. In Fig. 2 only two series of the modulation are shown, but in experiment we can perform more. After the perturbations are done, the magnetic DC field is directed to zero (from time t_2 to t_3) and when $B=0$, the voltage on the e.m. levitator decreases till zero. The temperature of the sample goes down, the latter solidifies and descend back to the bottom of the e.m. levitator. A refractive ceramics at the bottom of the levitator protects it from being melted by the sample which reamins rather hot. At this stage the sample can be contaminated due to its interaction with ceramics, but the measurements are already performed.

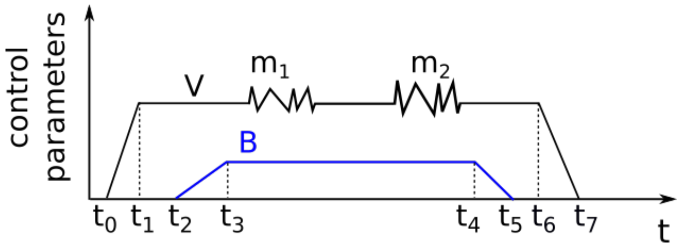


Fig. 2: Protocol for the variation of the control parameters during the measuring procedure with V the voltage supplied to the electromagnetic levitator and B the intensity of the DC field.

Using this (or similar) protocols, we performed experiments with the samples of Ni and a Ni-based high entropy alloy. In some experiments the perturbation of the samples were performed with varios intensity of the DC field in order to observe its effect on the thermal response of the sample, the most strong field which we used was *B=1.2T*. Generally it was found that with a more intense DC field the difference between the temperatures measured at the equatorial and the polar zone strongly decreases. This can be explained by damping of the convective flow which otherwise creates vortices inside the liquid thus promoting and supporting in the sample the existence of the areas with the strong temperture difference.

Yet, due to visualisation of the samples we have strong grounds to believe that non-zero flow remained in the sample, the latter was strongly rotated and shape variation were equally observed. This observation is in agreement with [4-6] where the damping of such effects was reached at B>3T. The Helmolhtz coil which we used, in principle, allowed us to generate the DC field up to 5T but we found the sample extremely unstable when the slow variation of the DC magnetic field was performed in order to reach a preset value. Thes osciallation and displacement of the droplet become more intense with a higher value of B although the speed at which it was varied was kept fixed. An example of these observations is given in Fig.3 with the time interval between the images about 0.1. The image A is due to the observation of the sample from the top (i.e. after 1 reflection in the mirror mounted outside the Helmholtz coil) and the image B is a lateral image of the top part of the sample formed after reflections in two mirrors: the one close to the electromagnetic levitator and another one outside the Helmholtz coil. A droplet contour on the image A is shown with a dashed line. A bright area outside the contour is strong reflection from the ceramics at the bottom of the electromagnetic levitator. The variation of the intensity of the image B can be related with the temperature distribution but this has to be confirmed.

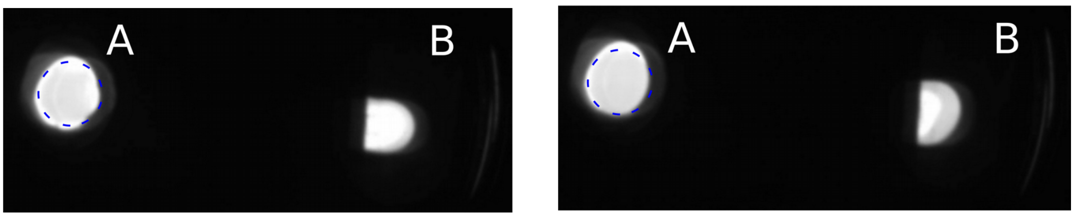


Fig. 3: An example of the observation of the sample during the increase of the intensity of the DC magnetic field from *B=1T* to *B=1.2T*, the time interval between the two images is about 0.1s. The image A is due to the observation of the sample from the top (after 1 reflection in the mirror mounted outside the Helmholtz coil) and the image B is a lateral image of the top part of the sample formed after reflections in two mirrors: the one close to the electromagnetic levitator and another one outside the Helmholtz coil.

These instabilities did not allow us to perform the measurements with a higher DC magnetic field because of the risk to loose the sample that, because of uncontrolled trajectory, could stick to a levitator and melt it.

Conclusion

The effect of the DC magnetic field perpendicular to the gravity vector during the experiments on the application of the modulated calorimetry to the metallic sample in levitation was observed. It was found that a fixed value of the DC did provide the damping effect, but its slow variations required to obtain its preset value caused strong instabilities in droplet motions.

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