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A new device for measuring thermal conductivity and heat capacity of metallic alloys by electromagnetic levitation

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Abstract
Electromagnetic levitation of electrically conductive droplets by alternative magnetic fields is a technique used to measure the physical properties of liquid metallic alloys such as heat capacity and thermal diffusivity, among others. We present a modulated calorimetry technique using electromagnetic induction. This technique is tested on a solid nickel spherical sample. Experimental results are compared with literature. An attempt to deal with liquid sample is presented as prospect.

Key words : electromagnetic levitation, modulated calorimetry

Introduction
Numerical models of casting and solidification are used to optimize industrial processes for the production of metallic alloys. Such models require, as input parameters, reliable thermophysical property values such as heat capacity and thermal conductivity. However, most standard measurement techniques, such as differential scanning calorimetry, require a molten metallic sample to be in contact with a crucible, leading to unavoidable contamination of this sample and difficulties to work in the melting zone range temperature. Based on the early work of Okress [1] on electromagnetic (e.m.) levitation, Egry, see for example [2], developed an electromagnetic levitator (EML), a non-contact technique that when used in microgravity conditions, reduces flows inside the molten sample enabling reliable measurements. This instrument is used in the framework of the Thermolab ESA-MAP project (Fecht [3]).

In this article, we first remind how the experimental device is used to perform calorimetry [4],[5]. Then, we briefly sum up a new modulation calorimetry technique where thermal behavior of levitated sample is deduced from unsteady thermal simulation. Those signals are processed by a signal analysis technique coming from Process Engineering. It couples both a white-noised modulated Joule power input and an identification procedure [6]. This procedure allows to derive relevant transfer functions of the considered system. The eigen-frequencies of those functions are relevant times for external and internal heat exchanges of the sample. An inverse method allows the calculations of the unknowns.

For validation purpose, this sequence is performed on solid spherical nickel sample. Experimental heat capacity and thermal conductivity are compared with literature.

Induction modulated calorimetry
The principle of the calorimetry measure is given by Fecht [4] and Wunderlich [5]. It is sketched on figure 1.

A spherical sample of radius $R$ and electrical conductivity $\sigma$ is located in an inductor powered by a high frequency alternating current: $I_0 cos(\omega t)$, $I_0$ and $\omega$ being the peak current and the angular frequency respectively. This inductor generates an almost bi-polar magnetic field which induces, inside the sample, electrical currents located in a surface layer called the electromagnetic skin depth. These currents generate a Joule heating power $P$ inside the sample.
Modulated calorimetry consists in perturbing the thermal equilibrium of the sample by modulating the total Joule power as $P = \bar{P} + \bar{P}(t)$, where $\bar{P}$ and $\bar{P}(t)$ are respectively the mean value and the time depending parts, and recording the related time-dependent behavior of surface temperatures, i.e. $T_p(t)$ and $T_e(t)$ the polar and the equatorial temperatures.

The 2 zones analytical model [4], [5] gives the following set of equations relating $\bar{P}$ to $\bar{T}_p$ and $\bar{T}_e$.

$$C_p g_e \frac{d\bar{T}_e}{dt} = h_{int}[\bar{T}_p - (1 + s_e B_i)\bar{T}_p] + \bar{P}$$

$$C_p(1 - g_e) \frac{d\bar{T}_p}{dt} = h_{int} [\bar{T}_e - (1 + (1 - s_e)B_i)\bar{T}_p]$$

In those equations $C_p$ and $h_{int}$ are respectively the heat capacity and the internal heat transfer coefficient.

Currently, the shape factors $s_e$ (resp. $g_e$) ratios of heated surface (resp. volume) to total surface (resp. volume) are supposed to be known a priori (for example by calibration). Moreover, when the modulation of the power is harmonic, its modulation frequency as to be chosen as well tuned and a decay time characteristic of the external heat exchange, mainly due to radiation has to be calibrated as well. After all, often, the Biot number $B_i = \frac{h_{ext}}{h_{int}}$ ratio of the external to the internal heat transfers is considered as very low.

On figure 1.b, the functions $H_p$ and $H_e$ are the transfer functions relative to $\bar{T}_p(t)$ and $\bar{T}_e(t)$ the time depending part of the polar and equatorial temperatures respectively. The set of above equation gives the following analytical transfer functions

$$H_p^{ana}(\rho) = \frac{h_{int}}{C_p(1 - g_e) \cdot \rho (1 + (1 - s_e)B_i) h_{int}}$$

$$H_e^{ana}(\rho) = \frac{1}{h_{int} \cdot (C_p(1 - g_e) \cdot \rho (1 + (1 - s_e)B_i) h_{int})}$$

The measure gives the internal transfer coefficient not the thermal conductivity. In order to be coherent with the 2 zones model, we use the following relation to find $\kappa_{th} h_{int} = -\frac{4\pi a - (1 - (g_e)1/3)}{1 + (1 - (g_e)1/3)^{1/2}}$.

In order to dispense the experimenter of all pre-settings, and taking advantage of the similarity between the whole "inductor+ load" (Fig. 1a) and its system representation (Fig. 1b), Schetelat [6] proposed to use as $\bar{P}$ a white-noised input power joined to an identification procedure, followed by an inverse resolution as sketched in figure 2.

<table>
<thead>
<tr>
<th>System Identification</th>
<th>$\bar{T}_e, \bar{T}_p, \bar{P}$</th>
<th>$\bar{P}$</th>
<th>$H_p$</th>
<th>$\bar{T}_p(n)$</th>
<th>$H_e$</th>
<th>$\bar{T}_e(n)$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Indirect Measurement</td>
<td>$H_e^{exp}, H_p^{exp}$</td>
<td>$H_e^{exp} = H_e^{ana}$</td>
<td>$H_p^{exp} = H_p^{ana}$</td>
<td>$C_p, Bi, ge, se, hint$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>$w_{ext}, w_{int}$</td>
<td></td>
<td></td>
<td></td>
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</tbody>
</table>

Fig. 2 – Signal processing of the “white noise” procedure
Experimental facility

The above procedure is tested on a metallic spherical by using an experimental facility comprising 3 parts: an experimental cell, an electro-technical part, and a recording system.

The test cell (figure 3.a) is made of a quartz tube of 140 mm in diameter and closed by two caps. The bottom cap, made of electrically insulating material, contains both the through passes for electrical connections of the inductor. The top cap, made of steel, incorporates vacuum tube and argon supply.

The electrotechnical equipment includes a high-frequency generator made by CELES that powers an oscillatory circuit consisting of an inductor and a set of capacitors. The inductor comprises 2 parallel pancakes of 3-windings each (figure 3.b).

Recording tools. Two bichromatic pyrometers are used to measure temperatures both at the pole and at the equator. A Rogowski probe allows measuring both intensity and frequency of the inducting current. The recording tools are connected to a PC. The recorded signals are analyzed a posteriori following the procedure illustrated on figure 2. The recorded signals are analyzed a posteriori following the procedure illustrated on figure 2.

Results

Experiments are performed using a spherical nickel sample an alternating inducting current carrying in the range from 90 to 123 Aeff, a frequency of 88 kHz, an modulation amplitude of 8%.

Results on both heat capacity and thermal conductivity are plotted on figures 4.a and 4.b.

We see that the measurement and literature values are close. The discrepancy is in the range of 1 to 3 % and of 9 to 19% for the heat capacity and thermal conductivity respectively. The higher discrepancy on the thermal conductivity is related to its indirect measurement.

Prospect

In order to reduce electromagnetic stirring and shaping of the molten sample, experiments are conducted in microgravity. However, first numerical simulations have shown that the use of a DC magnetic field had an effect similar
or better than micro-gravity to reduce/suppress e.m. stirring.

A steel cell with a double-walled cooled by water made has been built (figure 5.a). An electromagnetic levitation inductor is located in this cell and connected to a set of capacitors. The connections for electricity and water are in the bottom of the cell. By mean of a forklift on which both the cell and the capacitors are held, the inductor can to be located in a magnetic field hole of an Helmholtz coil whose intensity can reach 5 Teslas (figure 5.b). The cooled wall protects the supraconductive coil by acting as a shield to both thermal and electromagnetic radiations.

To allow pyrometric measurements, the upper part of the cell is a quartz window. In order to measure the equatorial temperature a golden mirror, 45 °oriented, is placed in the cell. Polar temperature is measured directly.

In this configuration setting and keeping electromagnetic levitation had to be automated. The figure 6 represents the way the inducting current is forwarded during the measuring sequence.

![Fig. 5.a. Drawing of the cell and the inductor](image1)

![Fig. 5.b. Drawing of the coil, the forklift and the cell](image2)

![Fig 6. Time behavior of the inducting current in the levitation coil during a levitation sequence](image3)

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