INFLUENCE OF HEAT CONVECTION ON THE MEASUREMENT OF THE SPECIFIC HEAT CAPACITY OF SOLIDS USING THE TEMPERATURE RELAXATION METHOD

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ABSTRACT
An experiment is described which employs a relaxation method for the measurement of the specific heat capacity at constant pressure of solids at room temperature. The experiment employs measurements of the cooling (or heating) rate of a sample whose temperature differs from that of the surroundings due to light heating. This rate depends on the temperature difference, the heat capacity of the sample and the heat transfer coefficient. The sample is suspended adiabatically in a reservoir in which vacuum can be made. The influence of heat dissipation by convection on the results is discussed for the first time in this kind of experiments.

RESUMEN
Se describe un experimento basado en un método de relajación para la medición de la capacidad calorífica específica a presión constante de sólidos a temperatura ambiente. El experimento se basa en la medición de la velocidad de enfriamiento (o calentamiento) de una muestra cuya temperatura difiere de la ambiente debido a la absorción de luz. Esa velocidad depende de la diferencia de temperaturas, la capacidad calorífica de la muestra y del coeficiente de transferencia de calor. La muestra es suspendida adiabáticamente en un recipiente en el cual se realiza vacío. La influencia de la disipación de calor por convección en los resultados es discutida por primera vez en este tipo de experimento.

I. INTRODUCTION

This work deals with an experimental approach currently used for the measurement of the specific heat capacity, C (units of $Jcm^{-3}K^{-1}$), of small solid samples at room temperature. This magnitude is defined as a product of the specific heat, c ($Jg^{-1}K^{-1}$), and the density, ρ (gcm^{-3}), and express the amount of heat developed per masse unit in a sample of unit volume when its temperature is varied in one degree[1]. The knowledge of C is of great importance in solid state physics due to its intrinsically value, unique for each sample, and due to its sensibility to phase transitions, among others. The measurement of C provides us with a direct mean to test theoretical models of a given physical system.

There are several methods for the practical determination of the specific heat capacity of solids. Some of them are discussed in detail by Touloukian[2], while in the work of Kraftmakher[3] an excellent overview of the most useful calorimetric methods is given. Among them, the temperature relaxation method offers the advantages of inexpensive experimental setup as well as relative simple and understable physical-mathematical formalism. This method was first proposed by Bachman **et al**.[4] for low temperature (1 to 35 K) measurements, later extended to measurements below 1K by Schutz[5] and successfully used, with properly modifications, by several authors in a higher temperature range. Djurek and Baturic-Rubidic[6] have modified Bachman's method for measurements above 35 K. Experiments on tungsten in the range 2400-3600 K were performed by Zinovev and Lebedev[7]. Hatta[8] have designed a relaxation calorimeter, employing for the first time light heating for measurements on small samples in the temperature range around room temperature. Mansanares and co-workers[9] have later developed Hatta's approach for simple measurements of C at room temperature, with the aim of complementing photoacoustic measurements of thermal properties of solids. This variant of the temperature relaxation method, designed by the last mentioned authors as temperature rise method under continuous illumination, was used in the last years for a characterization of different materials such as semiconductors[10], foods[11], wood[12], zeolites[13], clays[14] and ferroelectrics[15] ceramics, among others.

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II. EXPERIMENTAL DETAILS AND THEORY

The experimental setup for the measurement of the specific heat capacity is shown schematically in Figure 1. The samples, in the form of discs of 1cm diameter or 1 cm side squares and about 0.1 cm thick, were suspended adiabatically in a reservoir, which has an optical glass window through which a white light beam is uniformly focused onto one surface of the sample. On the opposite side, a Chromel-Alumel thermocouple is attached mechanically to the sample. Both sample surfaces were painted black to assure good light absorption and an Emissivity factor approximating one, a condition of great importance for accurate measurements, as seen below. The Temperature evolution of the back surface could be monitoring as a function of time using a programmable digital multimeter interfaced to a personal computer via its serial port, and stored by a data acquisition program.

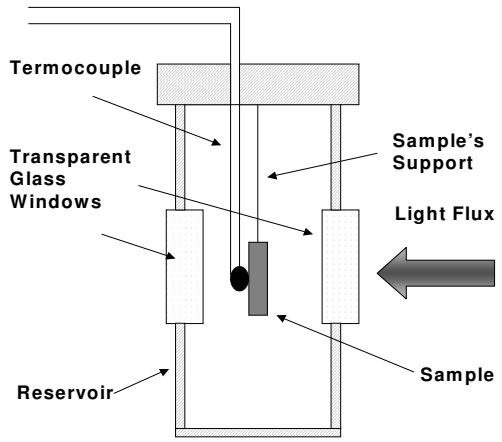

Figure 1. Schema of the experimental setup.

The heat developed in the sample as the function of time due to the absorption of light of incident power P_o is given by

$$\frac{\partial Q}{\partial t} = P_0 - L, \qquad (1)$$

where

$$L = R + K \qquad (2)$$

represents the power lost by radiation (R) and convection (K).

The first term on the right hand side of the above equation is derived from the Stefan-Boltzmann law of radiation and for temperature variations in the sample ΔT much lower than the ambient temperature T_{amb} is given by

$$R \approx 4A\varepsilon\sigma T_0^3 \Delta T \qquad (3)$$

where A is the sample's surface area, $\varepsilon\bullet$ is its the emissivity ($\varepsilon\bullet \approx 1$ if the sample's surfaces are black painted, as in our case), T is the sample temperature, and σ the Stefan-Boltzmann constant. For the K parameter, the term representing heat convection, we use Newton empirical cooling law to write

$$K = hA\Delta T \qquad (4)$$

where h is a characteristic parameter.

On the other hand, the temperature variation within the sample is related to the generated heat through

$$Q = \rho cV\Delta T = CV\Delta T \qquad (5)$$

where V is the sample volume. Differentiation of (5) with respect to the time and substituting in equation (1) leads to:

$$\frac{\partial \Delta T}{\partial T} + \frac{\gamma}{C}\Delta T - \frac{P_0}{C} = 0 \qquad (6)$$

with

$$\gamma = A(4\sigma\varepsilon T_0^3 + h) \qquad (7)$$

The solution of this differential equation, using the condition $\Delta T(0) = 0$ is:

$$\Delta T_{\uparrow}(t) = \frac{P_0}{\gamma}\left[1 - \exp\left(-\frac{t}{\tau}\right)\right], \qquad (8)$$

where

$$\tau = \frac{1C}{2(4\varepsilon\sigma T_{amb}^3 + h)} \qquad (9)$$

and l is the sample thickness. The characteristic time τ can be re-written as

$$\frac{1}{\tau} = \frac{1}{\tau_R} + \frac{1}{\tau_K}, \qquad (10)$$

where $\tau_R = lC/8\varepsilon\sigma T_{amb}^3$ is the relaxation time due to the heat losses by radiation and τ_K = lC/2h is that time characteristic for the convection mechanism.

The sample temperature is saturated at a value $T_0 = P_0/\gamma$ when thermal equilibrium is reached for $L = P_0$. Then, if the illumination is interrupted, one obtains for the temperature cooling the following time dependence:

$$AT_{\downarrow}(t) = \frac{P_0}{\gamma}\left[\exp\left(-\frac{t}{\tau}\right)\right] \qquad (11)$$

III. RESULTS AND DISCUSSION

Suppose that we are capable of perform a vacuum in the measurement cell, so that we can neglect the heat losses by convection. In this case τ_K can be neglected in expression (10) and the specific heat capacity can be obtained from the value of $\tau = \tau_R$ • • after

$$C = \frac{8\varepsilon\sigma T_{amb}^3 \tau_R}{1}, \qquad (12)$$

where we have denoted as τ_R the relaxation time obtained under no-convection conditions.

Now, if τ is the relaxation time obtained in an experiment performed at atmospheric pressure one gets from equations (9) and (12):

$$h = 4\varepsilon T_{amb}^3 \left(\frac{t_R}{\tau} - 1 \right) \qquad (13)$$

Using a reference material, a measurement instrument can be calibrated determining its characteristic constant h. For this purpose, measurements can be performed under different vacuum conditions until a value of $\tau = \tau_R$ is obtained, which satisfies equation (12), leading to a tabulated value of C. This value of τ_R can be then substituted in equation (13) in order to calculate the h value.

The open circles in Figure 2 shows typical normalized heating and cooling curves measured on a typical sample of Si at a vacuum of 10^{-2} Torr obtained using a mechanical pump and measured by a Pirani Vacuum meter. The solid curves are the best fit of the experimental data to expressions (8) and (11) in the case of sample's heating and cooling respectively. The variables $T_0 = P_0/\gamma$ and τ were taking as adjustable parameters. The mean value of the later parameter ($\tau = \tau_R$), determined from the best adjust values to the heating and cooling curves, was then used to calculate the specific heat capacity C of the sample by means of equation (12).

The results of the measurements at atmospheric pressure in the same Si sample are plotted also for comparison purposes in Figure 2 as full circles. As expected, a shift of the curves is obtained with respect to those obtained under vacuum conditions, as a result of the influence of the convection term. The solid curves, as above, represents the best fit to the theoretical expressions, from which a value of τ was obtained as a mean value from those corresponding to the heating and cooling curves. From the above values of τ_R and τ, h was calculated using equation (13).

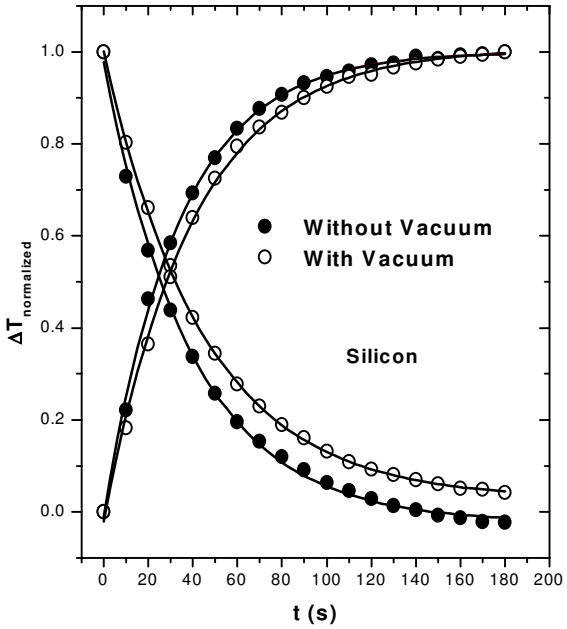

Figure 2. Typical heating and cooling curves for measurements performed under vacuum conditions (open circles) and without vacuum (solid circles), showing the results of the best fit of the data to equations (8) and (11) respectively.

The results of this and similar measurements performed in different kind of materials are summarized in Table 1. The samples have been selected taking into account that the reported values of their specific heat capacity differs appreciable from material to material in a wide range[16]. As can be seen in the table, the measured C values shows a good agreement with the ones reported in the literature, within the range of our experimental error.

Table 1. Experimental results.

Sample	τ (s)	τ_R (s)	τ_R/τ	H (10^{-4} Wcm^{-3} K^{-1})	C (Jcm^{-3} K)	C_{lit} (Jcm^{-3} K)
Si	40.8 ± 0.7	63.5 ± 0.7	1.56 ± 0.06	3.4 ± 0.4	1.6 ± 0.1	1.65
Zn	87 ± 3	144 ± 1	1.6 ± 0.1	4.0 ± 0.6	2.3 ± 0.1	2.77
Cu	32.1 ± 0.5	49.3 ± 0.5	1.53 ± 0.06	3.0 ± 0.4	3.0 ± 0.5	3.45
Al	28.6 ± 0.7	41.6 ± 0.8	1.45 ± 0.09	2.9 ± 0.5	4.0 ± 1	2.43

From the mean values of the convection characteristic parameter we obtained a mean value $h = (3.40 \pm 0.05) Wm^{-3}K^{-1}$. It is worth to notice here, that h is independent on sample's properties and depend only on geometric parameters describing the measurement device and on the properties of the fluid in which the sample is immersed, in our case air. The knowledge of the parameter h is important because it can avoid the use of vacuum apparatuses in the experimental setup. In other words, if the constant h is well known, then, from a relaxation time obtained in a given experiment, one can calculate the specific heat capacity using equation (10), without the need of perform any vacuum in the sample's reservoir.

IV. CONCLUSIONS

In this work we have described an experiment based on a temperature relaxation method for the measurement of the specific heat capacity at constant pressure of solids at room temperature. The knowledge of thermal properties of materials, such as their specific heat capacity is of great importance in many fields of research. For example in the design of electronic devices, it is very important to take into account the thermal properties of the involved materials, because the rate at which the generated heat inside the device dissipates, may determine the device performance and lifetime. However, accurate data on the thermal properties of several materials is scarce[2]. Therefore, the development of techniques for their measurement is always an impetus.

The arrangement described in this paper is technically simple and requires only minimal expenses. The influence of convection on the experimental results is analyzed in this work for the first time. It is shown that the heat transfer coefficient, characteristic for this phenomenon, can be used for determining the specific heat capacity of solids by measurements performed at atmospheric pressure. The obtained results for the specific heat capacity of several materials agree very well with the values reported in the literature for this magnitude.

This work represents a new step in demonstrating the capabilities of the temperature relaxation method to perform the experimental determination of specific heat capacities of solids at room temperature and towards the better interpretation of the physics lying besides this technique.

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