DIFFERENTIAL THERMAL CONDUCTIVITY MEASUREMENTS METHOD AND EXPERIMENTS

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Résumé.- Nous proposons une méthode différentielle de mesure de la conductivité thermique aux basses températures. Cette méthode parait bien adaptée à des mesures dynamiques en champ magnétique ou température variable.
Nous donnons un exemple de mesures faites sur un échantillon d'alumine contenant du manganèse et présentant un effet magnétique, la référence étant un échantillon d'alumine pure.

Abstract.- We propose a differential method of measuring thermal conductivity at low temperatures. This method seems appropriate for dynamic measurements with variable magnetic fiels or temperature. We give an example of measurement concerning a Mn doped alumina sample presenting a magnetic effect, the reference being a pure alumina sample.

INTRODUCTION.- Differential methods are very useful for many experimental measurements but, as we know, they have not yet been used for low temperature thermal conductivity measurements.

We propose here a differential method for this type of measurement. Firstly we have used this method to avoid the problem of magnetic effects on thermometers in thermal conductivity measurements under magnetic field. Also we propose using this method for continuous or semi-continuous measurements of thermal conductivity versus temperature.

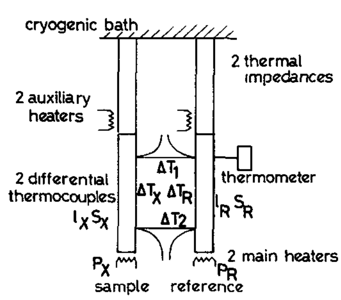

Fig.1 : Principle and experimental arrangement
For $\Delta T_1=\Delta T_2$ we have $\Delta T_X=\Delta T_R$ and $K_X=K_R.(P_X.l_X.S_R)/(P_R.l_R.S_X)$

PRINCIPLE.- In figure 1 we show the principle of the method, using two differential thermocouples mounted between two samples, and using appropriate heaters, we realise the same temperature differences on the samples. On sample is the reference and the other the specimen to be measured. The absolute temperature is measured on one sample.

In the presence of magnetic field since the signal on the thermocouples are zero, we have no magnetic effect, if the sensitivity of the thermocouples is finite and not too small. In this case the absolute thermometer must have zero or negligeable magnetic effect.

As in the standard method we have to use a double heat flow technique to take account of the parasitic heat flux on the samples.

In one case we apply power to the two samples, and at the equilibrium where $\Delta T_R=\Delta T_X$ we have P_R on the reference and P_X on the specimen. In the other case we apply power P_R° or P_X° only on one sample to have the same temperature differences ΔT_R° and ΔT_X°. The thermal conductivity of the specimen K_X is given as a function of that of the reference K_R, from the two conditions by the relations :

$$K_{X(T,H)} = K_{R(T,H)}.P_X.A_X/((P_R-P_R^\circ).A_R)$$

$$\text{or} \quad = K_{R(T,H)}.(P_X-P_X^\circ).A_X/(P_R.A_R)$$

where A_R and A_X depend on the geometric dimensions of the samples.

EXPERIMENTAL ARRANGEMENT.-The experimental arrangement is shown schematically in figure 1. The two samples are cylindrical, and the two main heat flows are supplied by two electrical heaters fixed to the free end of the samples.

The average temperatures are varied with two auxilliary heaters and by two thermal impedances to the bath.

The absolute thermometers are fixed on the reference sample with a collar. These thermometers can be a germanium, carbon or platinum resistor, a capacitor or a gas bulb. The two last are not affected by the magnetic field.

The null differential thermocouples mounted between the two samples are gold iron-chromel thermocouples. They are mounted so that they present the smallest self inductance.

EXPERIMENTS.- Three temperature regulators are used, one to maintain constant the absolute temperature and the others maintain a zero signal on the thermocouples.

A power generator is used for the main heating of the reference.

The two main heating powers are measured or recorded.

Due to the interaction between the different regulators, step measurements are not easy and a dynamic or semi-dynamic method is more appropriate In this sense two types of experiments are possible.

a/ Variable temperature and constant external parameters (H,E,σ). The absolute temperature and the main heat power on the reference are programmed versus the time. The heat power P_X and P_R° or P_X° are recorded versus the temperature. This technique is not working completely, but the first tests are encouraging.

b/ Constant temperature and variable external parameters, for example magnetic field.

To test the method we have used a pure alumina crystal as reference, and a Mn doped alumina crystal, presenting a magnetic effect on the thermal conductivity, as specimen. They were previously measured by the standard method between 1.5 and 30 K.

The absolute temperature is regulated from a carbon resistor, for this test we have neglected the magnetoresistance of the absolute thermometer.

The heating power on the reference in fixed, for each temperature, to obtain an adequate value of the temperature difference, that means a few percent of the absolute temperature.

The heating power P_X, and P_X° in this case are recorded versus the magnetic field provided by a superconducting coil.

The magnetic field is continuously varied from 0 to 5 tesla at a rate of 0.01 tesla/s.

A small difference is observed on the results for increasing and decreasing magnetic fields. This difference is due to the response of the electronic system and can be limited by changing the gain of the regulators, or annulled using a semi-dynamic progression. Evidently a data acquisition system would be very useful.

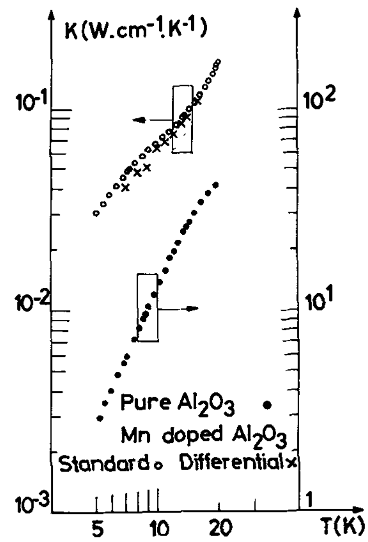

Fig.2 : K(T) in zero magnetic field.

RESULTS AND ACCURACY.- The results in zero magnetic field are plotted in figure 2 along with the results from standard thermal conductivity measurements for the specimen and the reference. The difference observed at low temperatures is due to the fact that the temperature differences along the samples are small-about 0.1K. The sensitivity of the temperature regulators used is about 0.01K, and we deduce that the total error, considering both regulators is $\delta(\Delta T)/\Delta T \simeq 20\%$.

As we can see the accuracy depends mainly on the value of ΔT and on the sensitivity of the regulators. It is possible, by changing the thermal impedances to the bath and the reference, to increase ΔT, and it is also possible to improve the sensitivity of the regulators, which would decrease the error by 10.

In figure 3 we have plotted the magnetic field effect on the thermal conductivity of Mn doped alumina at constant temperature as an example of the use of this technique.

CONCLUSIONS.- The differential thermal conductivity method seems to be very useful for relative thermal conductivity measurements, for example in magnetic field and also for absolute measurements of thermal conductivity. This method is very well adapted for continuous or semi-continuous measurements with a data acquisition system, now in progress, which can also allow us to improve the accuracy of the results.

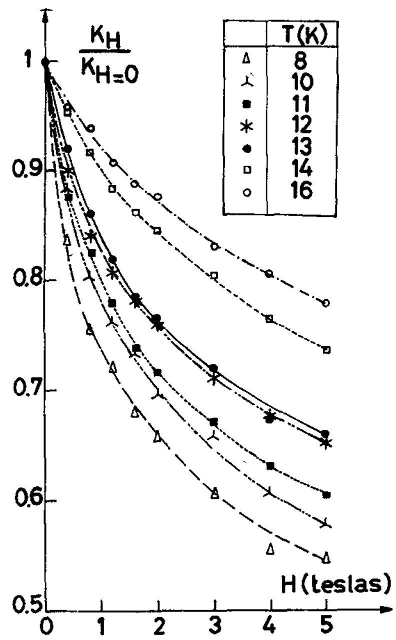

Fig.3 : Magnetic field effect on the thermal conductivity of Mn doped alumina.

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