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THERMAL CONDUCTIVITY MEASUREMENTS ON SAMPLES WITH LOW CROSS-SECTIONS

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A simple, low-cost apparatus has been designed and constructed for measurement of the thermal conductivities of samples with low cross-sections ($\sim 10^{-7}$ m^2). This apparatus has been used to determine variations in the thermal conductivity of the metallic glass $Fe_{80}B_{20}$ (Metglas 2605) in the crystallization process induced by thermal treatment.
In spite of the simplicity, the deviations from the real values of thermal conductivities measured have been lower than 8%, which has proved to be satisfactory for establishing the glass formation, temperature, T_g, from changes in thermal conductivity.

Although there are many methods for the measurement of thermal conductivities [1], difficulties arise when samples with special geometry are to be measured, e.g. samples with low cross-sections ($\sim 10^{-7}$ m^2). In this work, our purpose was to measure the variation in the thermal conductivity of metallic glasses with the crystallization process. Because of the preparation (rapid quenching), the samples usually have small cross-sections. The preparation procedure for the measured samples was the twin-roller technique [2]. Metallic glasses are in a structurally metastable state. Above a characteristic temperature for each glass, the crystallization process acquires an observable velocity, giving rise to the appearance of crystalline phases when heat treatment is applied above this characteristic temperature, called the glass formation temperature, T_g [3]. When crystallization occurs, the physical properties in general show drastic changes. From the measurement of these physical properties, it is possible to determine T_g and additional information about the structural changes.

Measurement of the thermal conductivities of samples with low cross-sections (37 μm × 1 mm, ribbon) has been made by means of a stationary flux method, with a specifically designed and constructed simple and low-cost apparatus, which is described below.

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Experimental arrangement

For samples with uniform cross-sections and unidimensional heat flux, the stationary-state Fourier law can be written as [4]

$$\dot{q} = KS\frac{\Delta T}{l}$$

where $\dot{q}$ is the heat flux, K is the thermal conductivity, S is the cross-section of the sample and ΔT is the temperature difference between two points of the sample separated by a distance l along the heat flux direction.

However, for samples with a lateral surface much greater than the cross-section, it is difficult to have unidimensional heat fluxes.

In fact, in our case, the ribbon-shaped samples strongly condition the design of the conductimeter. The use of a thermal screen is necessary to obtain a nearly linear temperature distribution along the samples. We used a two-block system, the first one heated electrically, with the sample connected to the second one (Fig. 1).

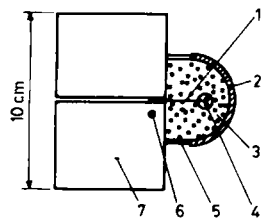

Fig. 1 Mechanical disposition of the sample. 1 Sample, 2 Thermal screening, 3 Fine grain insulation, 4 Small copper block, 5 Plastic support, 6 Thermistor, 7 Heat sink

Taking into account the magnitude of the cross-section of our samples, in front of the lateral surface and the surface of the heated block it is necessary to avoid heat losses to the ambient. For this we have used an aluminium guard placed behind the heated block, maintained at the same temperature as the block by means of a single feed-back system, which, with a thermistor bridge, compares both temperatures.

Plastic slices support this aluminium piece and connect the non-heated block, forming a thermal guard for the sample under measurement. In addition, the space between the sample, the heated block and the thermal guard is filled with insulating material (small-grain styropor) (Fig. 2).

This contributes to minimizing the convective phenomena.

In order to avoid external perturbations, the whole apparatus is shielded with a 5 cm layer of styropor, and a metallic screen Fig. 2. The dimensions should be kept small, in accordance with the sample dimensions and in order to minimize heat

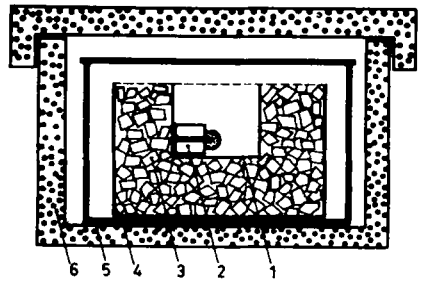

Fig. 2 General mechanical mounting.
1 Small container, 2 Copper blocks, 3 Ice and water, 4 Second container, 5 Metallic screening, 6 Styropor insulation

losses. The device consists of two copper blocks, the "reference" block ($10 \times 5 \times 5$ cm^3) in contact with ice, and the "heated" block ($0.6 \times 0.6 \times 0.6$ cm^3), which includes a heating resistor (metallic film type, 1100 Ω) and a resistance thermometer (Fig. 3).

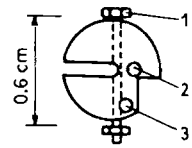

Fig. 3 Heated copper block. 1 Screw, 2 Heating resistor, 3 Thermistor

The temperatures are measured with small thermistors (Miniwatt 2322 627 21104 NTC, 110 KΩ at 20°), after calibration. The thermistors are placed in the copper blocks, next to the sample (< 0.1 cm). The fixed distance between the blocks is 1.5 cm and the sample is in thermal contact with the two blocks. The block diagram is presented in Fig. 4. The experimental procedure we have used to measure thermal conductivities is as follows:

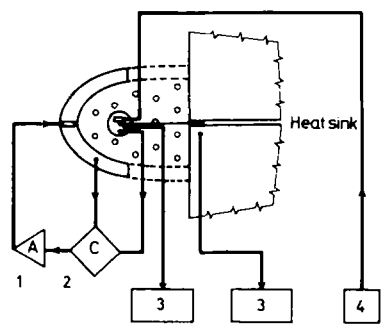

Fig. 4 Block diagram of the apparatus. 1 Power amplifier, 2 Bridge comparator, 3 Meters, 4 Power

1. Mounting of the sample, fitted with screws between the halves of the copper blocks, and with heat conductive paste in the block contact parts.

2. Obtain 0° in all the apparatus with ice and water.

3. Swich on the heating of the small copper block (200 mW), and the guard system.

4. Wait for temperature stabilization (about 3 hours in our case), and read the stationary temperatures and the power dissipated in the heated block.

The waiting time for measurement could be improved by heating in thermostatic mode in place of constant power mode; we used this latter mode for simplicity of instrumentation.

The copper block temperatures reached were of the order of 45° for the heated block, and less than 5° for the other block, next to the sample.

Results

The thermal conductivities of metallic glass ribbons of $Fe_{80}B_{20}$ were measured for different thermal treatments.

The reproducibility of the results, if the sample is dismounted and mounted again, is better than 4%. The estimated error taking into account the geometrical conditions and heat loss evaluation is less than 8%.

For the as-quenched alloy $Fe_{80}B_{20}$, the results we have obtained with the above experimental procedure are in agreement with those of Mizoguchi et al. [5], who gave conductivity values only for the natural state of this metallic glass.

Table 1

Heat treatment $Fe_{80}B_{20}$ (metglas 2605)	Thermal conductivity, W/m °C
Natural	6.5 [2]
2 h to 80 °C	5.5
1 h to 100 °C	5.3
3 h to 100 °C	5.3
1.30 h to 150 °C	5.0
2 h to 200 °C	4.9
1 h to 300 °C	3.9
2 h to 380 °C	8.6
30 min to 500 °C	8.0
30 min to 600 °C	9.0
15 min to 780 °C	10.9
1 h to 780 °C	11.6
2.30 h to 780 °C	12.1

Other attempts to obtain the thermal conductivity of samples of metallic materials with low cross-sections involve measurement of the electrical resistivity and application of the Wiedemann-Franz law. However, thermal conductivities obtained in this way consider only electronic contributions and not lattice contributions (phonons, defects, grain boundaries, etc.), which are important in our samples [6].

We have used this apparatus to measure changes in thermal conductivity in heat-treated samples. In Table 1 we list some results. The changes in thermal conductivity can be used to characterize the crystallization processes, together with other physical properties. In our case, the strong variation of thermal conductivity with treatment at 380° has been correlated with the glass formation temperature, T_g [6]. For treatment above T_g, crystalline phases appear, and increasing structural order means increasing mean free paths of electrons and phonons, giving rise to higher thermal and electrical conductivities.

Some discussions concerning the correlation between thermal and electrical conductivity, X-ray diffraction and Mössbauer spectroscopy measurements on heat-treated metallic glass $Fe_{80}B_{20}$ samples can be found elsewhere [6].

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Zusammenfassung — Eine einfache, billige Apparatur zur Messung der Wärmeleitfähigkeit von Proben mit kleinen Querschnitten ($\sim 10^{-7}$ m^2) wurde entworfen und gebaut. Die Apparatur wurde zur Bestimmung von Veränderungen der Wärmeleitfähigkeit des metallischen Glases $Fe_{80}B_{20}$ (Metglass 2605) während des durch thermische Behandlung ausgelösten Kristallisationsprozesses benutzt. Trotz der Einfachheit betrug die Abweichung von den gemessenen tatsächlichen Wärmeleitfähigkeitswerten weniger als 8%, was sich als ausreichend für die Ermittlung der Glasbildungstemperatur T_g aus Veränderungen der Wärmeleitfähigkeit erwiesen hat.

Резюме — Построен простой и дешевый прибор для измерения удельной теплопроводности образцов с малым поперечным сечением ($\sim 10^{-7}$ м^2). Аппаратура была использована для определения изменений удельной теплопроводности металлического стекла $Fe_{80}B_{20}$ Метгласс 2605) в процессе его кристаллизации, вызываемой термической обработкой. Несмотря на простоту прибора, отклонения измеренных значений от действительных значений удельной теплопроводности составляло менее 8%, что было удовлетворительным для определения температуры стеклования T_g на основе изменений удельной теплопроводности.