

A LASER INTERFEROMETRIC METHOD APPLIED TO THERMAL DIFFUSIVITY MEASUREMENTS OF FERRITES

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A laser interferometric method for the measurement of thermal diffusivity of solid materials is discussed. Measurements on ferrites of different composition are presented.

1. Introduction

A large theoretical and experimental effort has been done in the last years in the realization of new techniques for the measurement of thermal properties of solids. We can add to the classical methods (ASTM C408-58) new techniques utilizing light beams, such as the flash method [1], photothermal deflection spectroscopy [2], and interferometric methods [3,4].

Ceramics are one of the most extensively studied materials for the importance they have both for applied physics and technology. They are replacing the traditional materials for mechanical applications and play a fundamental role for special applications (e.g. when a low electrical conductivity and high thermal conductivity are required). Recently an interferometric method [4] for studying thermal diffusivity of ceramic materials has been developed. We report here an analysis of the method as well as some experimental results.

2. Sample preparation

The samples are ferrites, made of oxides such as Fe_2O_3 , Y_2O_3 , Al_2O_3 , MnO_2 , MgO , Gd_2O_3 and In_2O_3 and mixed (for several hours) in a stainless-steel ball mill containing deionized water. The oxide mixture is then oven-dried and calcined in fireclay crucibles at temperatures in excess of 1000°C for approximately 1 h.

After a second process cycle of ball milling, drying, binder addition (polyvinyl alcohol or acrylic resin), crushing, and sieving, the powder is pressed by an hydraulic press, at pressures of the order of 1 ton/cm^2 .

The organic binder must be eliminated from the pressed pieces to prevent their cracking during the subsequent sintering cycle, and to avoid the carbonization of the polymer with its reducing and damaging effects.

Sintering allows one to obtain a compact ceramic

structure. This operation is carried out in a kiln, in an oxygen flow, and at a final temperature from 1200 to 1400°C for approximately 10 h. The samples of dimension of few centimeters then receive mechanical finish to obtain the wanted size. One of the surfaces is then well polished (maximum roughness less than 3000 Å) in order to reflect the He-Ne laser beam.

3. Experimental arrangement

The interferometer used in the measurements is a Michelson interferometer where the sample under measurement replaces one of the mirrors. The surface of the sample is polished specular for reflection. A CO₂ laser beam of about 0.5 W heats the sample from the back side as shown in fig. 1. A shutter on the laser beam establishes the exact starting time of the heating.

In order to know the exact amount of energy given to the sample from the heating source the reflectivity of the surface has been measured. For the first two samples of table 1 the reflectivity was measured with a pyroelectric detector; for the other samples a Perkin-Elmer 683 infrared spectrometer was used. We can estimate the value of the absorption coefficient ($\alpha = 10 \text{ cm}^{-1}$) from the measured transmission coefficient and from it the exact amount of energy given to the sample can be obtained.

Particular attention must be given to the mechanical coupling of the sample with its support. A num-

ber of measurements with different configurations have shown that the horizontal position of the sample allows free thermal expansion without coupling to the support. In fig. 2, a schematic view of the position of the sample is shown.

4. Theory

Before heating the sample, the He-Ne intensity at the photodetector is given by

$$I_d = 2I_0(1 + \cos \Delta\phi), \quad (1)$$

where I_0 is the intensity of the laser beam and $\Delta\phi$ is the phase shift between the two beams of the interferometer, which is

$$\Delta\phi = (2\pi/\lambda)(d_1 - d_2) + \delta. \quad (2)$$

Here d_1 and d_2 are the two arm lengths, and δ is the phase shift due to the reflection at the sample surface.

When the shutter of the CO₂ laser beam is open the sample begins to heat and undergoes a thermal expansion. The thermal expansion produces a change Δl in the path of the laser beam reflected by the sample, and therefore the phase shift between the two arms of the interferometer changes as given by

$$\delta = (2\pi/\lambda)2\Delta l. \quad (3)$$

This phase shift produces a movement of the fringe pattern on the detector. Therefore the detector output has a temporal dependence which gives the temporal behavior of Δl .

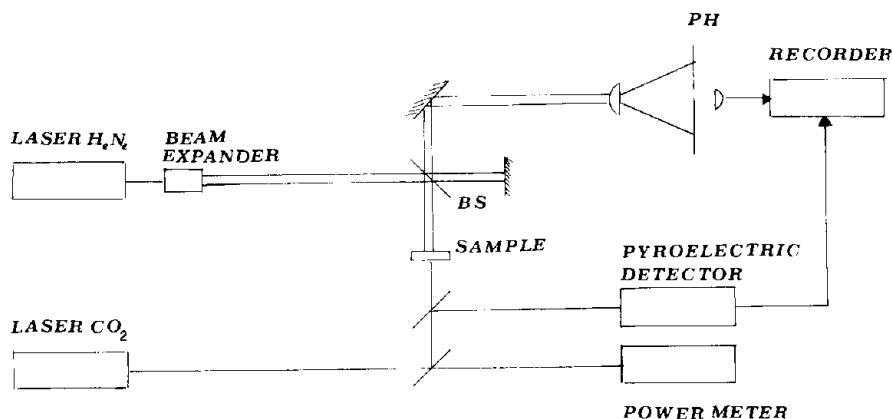


Fig. 1. Experimental arrangement - side view.

Table 1

Sample	P (W)	R	t (s)	l (cm)	χ (cm ² /s)
S2210 (Mg ₁ Mn _{0.1} Fe ₂ O ₄)	0.52	0.07	0.03	0.065	0.0633
G1 (Y ₃ Fe ₄ O ₁₂)	0.6	0.07	0.3	0.43	0.06
ASNICO 15 (NiFe _{1.85} Co _{0.15} O ₄)	0.5	0.12	0.036	0.05	0.007
G6063 (Y _{0.5} Gd _{2.5} Fe _{4.3} In _{0.5} Al _{0.2} O ₁₂)	0.5	0.09	0.016	0.05	0.026
G6063-Be05 (idem Be _{0.005})	0.5	0.085	0.048	0.09	0.021
G6063-Be1 (idem Be _{0.01})	0.5	0.087	0.064	0.09	0.014
G6063-Be1.5 (idem Be _{0.015})	0.5	0.086	0.064	0.1	0.018
G2110 (Y ₃ Al _{0.1} Fe _{4.9} O ₁₂)	1.25	0.084	0.42	0.39	0.025

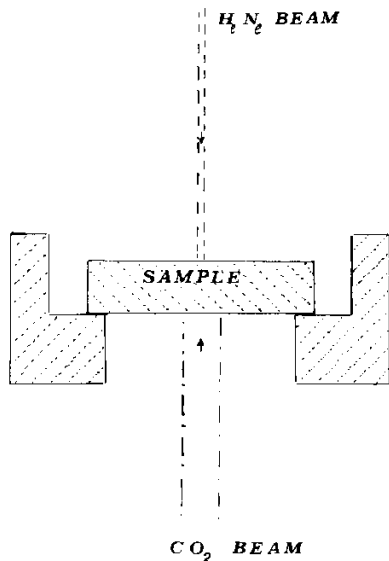


Fig. 2. Sample disposition.

In order to calculate the thermal diffusivity we start from the well known solution of the one-dimensional lossless heat equation, for a semi-infinite slab,

$$\Delta T_c = \Delta T_h \exp(-l^2/4t_s\chi), \quad (4)$$

ΔT_c and ΔT_h being the temperature increase on the rear (cold) and front (hot) surface respectively, l the sample length, t_s the "diffusion time", and χ the thermal diffusivity. The "diffusion time" t_s in this

experiment is defined as the delay between the time at which the heating beam is switched on and the time at which the first $\lambda/4$ fringe movement is detected.

Knowing the thermal expansion coefficient, ΔT_c is given by

$$\Delta T_c \approx \Delta T_h - 2\Delta l_s/\bar{\alpha}l, \quad (5)$$

where $\bar{\alpha}$ is the thermal expansion coefficient. ΔT_h is the rise temperature due to the heating of the CO₂ laser and can be written as

$$\Delta T_h = \frac{2P(1-R)}{cp\chi\pi} \ln\left(\frac{4\chi t_s}{w_0^2} + 1\right) \quad (6)$$

for the case of a gaussian heating source on a semi-infinite slab where w_0 is the laser spot, P is the laser power, R is the surface reflectivity, ρ is the density and c is the specific heat of the sample.

In the case where $4\chi t_s/w_0^2 \ll 1$, eq. (6) becomes

$$\Delta T_h = \frac{2P(1-R)}{cp\pi} \frac{4t_s}{w_0^2}. \quad (7)$$

From eq. (4), knowing ΔT_c , ΔT_h and t_s , we can estimate the thermal diffusivity value:

$$\chi = l^2/4t_s \ln(\Delta T_h/\Delta T_c). \quad (8)$$

5. Results and conclusions

As an example of the application of the method, the results obtained for different samples are shown

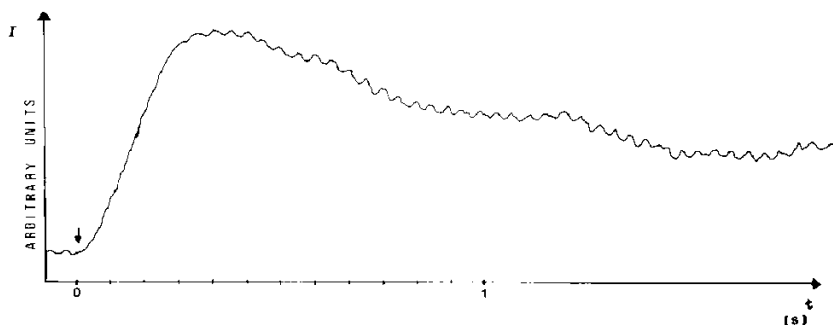


Fig. 3. Fringe movement (sample G6063-Be1). The arrow represents the time at which the heating starts.

in table 1, where l is the thickness of the sample, R is the reflectivity at the surface, P is the laser power on the sample and χ is deduced by eqs. (7) and (8) measuring the time t_s . The time t_s was obtained from the registration of the fringe movements, as shown in figs. 3 and 4, where the arrow shows the time at which the laser beam is switched on and heating starts. The results agree well with those obtained previously and presented in ref. [4], where a slightly different method was used.

A rough estimation of the order of magnitude of the error places it to be 20% on χ , essentially due to the lossless calculation of the temperature increases ΔT_h and ΔT_c .

From table 1, samples S2210 and G1 showed higher thermal diffusivities. The addition of a low concentration of Be in sample G6063 (G6063-Be05) decreases the thermal diffusivity (see fig. 5). However, increasing the Be concentration the χ value tends to increase. This is probably due to the insertion of Be atoms (which is non-stoichiometric) in interstitial positions for low concentrations, which causes an increase of the disorder, whilst for higher

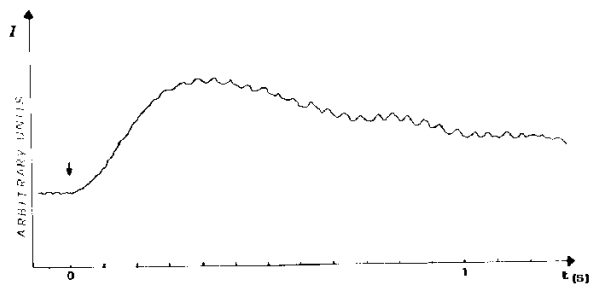


Fig. 4. Fringe movement (sample G6063-Be1.5). The arrow represents the time at which the heating starts.

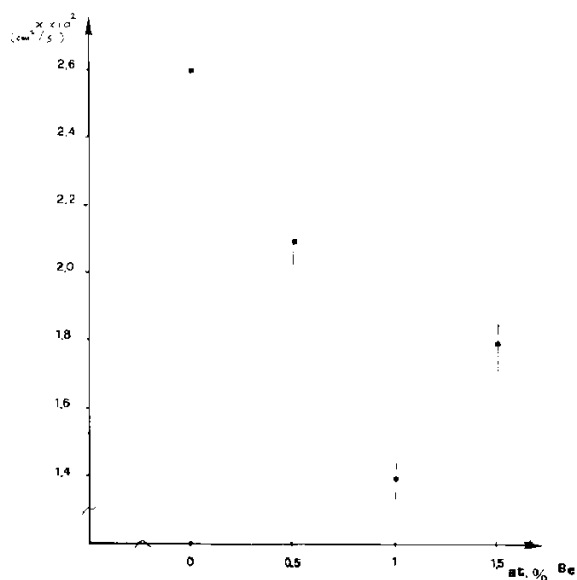


Fig. 5. Thermal diffusivity as a function of the atomic percent of Be for samples G6063.

concentrations a substitution of Fe^{2+} in tetrahedral sites probably occurs. We are testing this hypothesis with a more accurate structural analysis.

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