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Photoacoustics and laser-ultrasonics applied to the characterization of a ZnO ceramic sample

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Abstract: we present results of photoacoustics and laser-ultrasonics experiments that were performed on a ZnO ceramic sample and that led to optical, thermal, thermo-mechanical and mechanical characterizations of this material.

1. INTRODUCTION

Due to their mechanical, thermal or electrical properties, ceramic materials are increasingly being used in industrial applications. The development of characterization and non-destructive evaluation techniques adapted to these materials has now become a research subject of great importance.

In this paper, we report results of photoacoustics and laser-ultrasonics experiments applied to a ZnO ceramic sample. The combination of these two techniques permits to perform optical (optical absorption coefficient), thermal (thermal diffusivity), thermo-mechanical (thermal expansion coefficient) and mechanical (rigidity tensor) characterizations of the material inspected.

2. THE PHOTOACOUSTICS EXPERIMENT

2.1 Principle of the experiment

In this experiment, the sample is placed in a closed cell, surrounded by an inert gas, and its top surface is impinged by an intensity-modulated monochromatic light. The absorption of optical energy results in modulated temperature variations in the sample near its top surface. These local temperature variations have two effects: (i) the gas near the sample's top surface is periodically heated by thermal conduction, giving rise to a modulated pressure variation in the cell, and (ii) the sample itself undergoes a modulated thermal expansion, which also results in a modulated pressure variation in the cell. The experimental signals recorded are the amplitude and phase of the pressure modulation in the cell.

McDonald [1] derived the following expression for the complex pressure modulation, assuming a thermally and optically thick sample surrounded by a thermally thick gas layer, and neglecting the thermal expansion contribution:

$$q = -i\frac{K}{\omega}\frac{\beta\mu}{1+\beta\mu+i}e^{i\omega t} \qquad (1)$$

In this formula, K is a constant related to the physical properties of the sample and the gas, ω the angular frequency, β the optical absorption coefficient of the sample at the excitation wavelength, and μ the thermal diffusion length in the sample defined by $\mu = (2a/\omega)^{1/2}$ (a being the thermal diffusivity of the sample).

If one performs a series of experiments for different modulation frequencies, first on the sample to study, then on a reference blackbody sample (for which β_{ref} is very large), the ratio of the two complex pressure modulations q and q_{ref} and the amplitude A of this ratio are given by:

$$\frac{q}{q_{ref}} = \frac{K}{K_{ref}}\frac{\beta\mu}{1+\beta\mu+i} \qquad (2) \qquad A = \frac{K}{K_{ref}}\frac{\beta\mu}{\sqrt{(1+\beta\mu)^2+1}} \qquad (3)$$

Since $\beta\mu = \beta\sqrt{a/\pi f}$, a numerical fit of the experimental curve $A_{exp}(1/\sqrt{f})$ permits to determine $\beta\sqrt{a}$.

2.2 Experimental apparatus

It is shown on figure 1. The ceramic sample was 2.6 mm thick, and the reference was a graphite sample about 4 mm in thickness. The gas used in the photoacoustic cell was helium at atmospheric pressure. The bandpass of the microphone (recording the pressure signal) limited our experiments to modulation frequencies between 10 Hz and a few kHz.

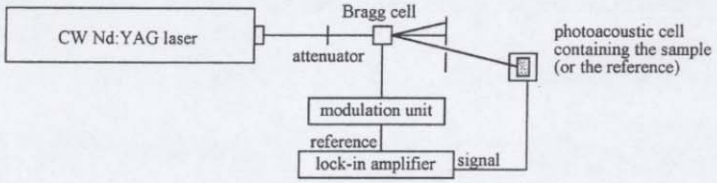

Figure 1: scheme of the photoacoustics experiment

2.3 Experimental results - determination of $\beta\sqrt{a}$

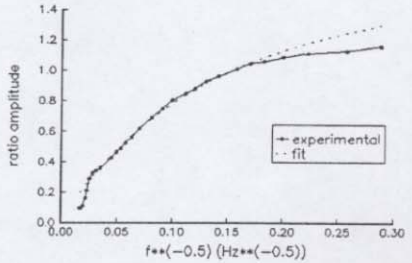

Figure 2: comparison of the experimental and fitted ratio amplitude curves

We made measurements on the ceramic and reference samples over the frequency range from 12 Hz to 3.51 kHz. Then we plotted the experimental ratio amplitude curve and fitted it with a theoretical curve having an equation given by formula (3). This way, we obtained $K/K_{ref} = 1.85$ and $\beta\sqrt{a} = 16.3\ Hz^{1/2}$.

Figure 2 shows the good agreement between the experiment and the model over the frequency range from 35 Hz to 1510 Hz, which ensures a reliable experimental evaluation of the product $\beta\sqrt{a}$.

We are now going to see how, using the laser-ultrasonics technique, we can evaluate β separately, and then deduce the value of a.

3. THE LASER-ULTRASONICS EXPERIMENTS

3.1 Experimental apparatus

It is shown on figure 3. The pulsed Nd:YAG laser has a variable pulse duration (from 12 to ≈ 100 ns), is monomode, and can deliver energies up to 100 mJ. The optical detection probe is an *Ultra-Optec* OP35-I/O heterodyne interferometer, that can perform quantitative measurements of both normal and in-plane (in a given direction) displacements.

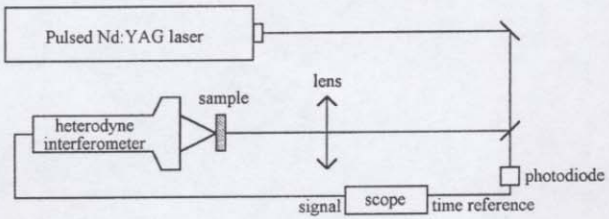

Figure 3: scheme of the laser-ultrasonics experiment

3.2 First experiment - determination of the rigidity tensor

This experiment was performed with a focused beam: its Gaussian radius at 1/e was 250 μm on the sample. The interest of having a small generation source compared to the sample's thickness (2 mm) was to produce a clearly defined shear wave arrival (see figure 4). The counterpoint was the low pulse energy of 1.9 mJ and the long pulse duration of 87 ns that we used in order not to reach the ablation regime. The normal displacements are thus very weak and the large value of the pulse duration makes the accuracy of the measurements quite error-prone. We measured for the times of flight of the longitudinal and shear waves through the sample the values of 345 and 750 ns. We then obtained the longitudinal (V_l) and shear (V_s) velocities, from which we calculated the components C_{11} and C_{44} of the rigidity tensor (the density ρ of the material being known: 5420 kg/m^3). We obtained the following values: C_{11} = 18.2 10^{10} N/m^2 and C_{44} = 3.85 10^{10} N/m^2. We deduced the third component C_{12} of the rigidity tensor from these two parameters and found 10.5 10^{10} N/m^2.

3.3 Second experiment - determination of the optical absorption and thermal expansion coefficients

In this experiment, the laser spot impinging the sample was completely defocused: its Gaussian radius at 1/e was 1.6 mm, whereas the sample was still 2 mm thick. In this configuration, the irradiation of the sample could be considered as uniform, and thus all the phenomena involved in the laser thermoelastic generation of ultrasound (optical absorption, thermal diffusion, thermal expansion, propagation of ultrasonic waves) could in turn be considered as monodimensional in the direction of the sample's thickness. We have developed an analytical 1-D model that predicts the normal displacement of the rear surface as a function of time, the ultrasound generation being assumed to result only from the optical penetration and not from the thermal diffusion. This model led to a very simple expression relating the Full Width at Half Maximum (FWHM) of the precursor to the optical absorption coefficient β and the longitudinal velocity V_l:

$$FWHM = \frac{\ln(4)}{\beta V_l} \qquad (4)$$

The experimental displacement recorded on the rear surface of the sample is shown on figure 5. In comparison to the result of figure 4, one can see that this time, no shear wave arrival is apparent. The measurement of the experimental FWHM (46 ns) plus the knowledge of the value of V_l (determined in the last section: 5800 m/s) permit to evaluate the optical absorption coefficient: β = 5200 m^{-1}.

For the evaluation of the thermal expansion coefficient α, we used a semi-analytical 3-D model [2] that solves the heat and acoustic wave equations using temporal Laplace and spatial 2-D Fourier transformations. The use of these integral transforms permits to consider an orthotropic sample and any time or surface profiles of the irradiation. From the Christoffel equations, it appears that the group $\alpha E/\rho C_p$ is a multiplicative factor of the mechanical displacement field **u**(**r**,t), where E is the energy absorbed by the sample and C_p its specific heat. C_p was given by the manufacturer (506 J/kg.K). Concerning E, we measured the pulse energy of the experiment (22.4 mJ) and the reflection coefficient of the sample at the YAG wavelength (18%); E was then 0.82 x 22.4 = 18.4 mJ. We ran the model with all these values plus the ones of β and [C] found above, and we determined α by fitting the amplitude of the calculated normal displacement to the experimental result. One can see on figure 5 that for α = 3.8 10^{-6} K^{-1}, the agreement between the model and the experiment is excellent. Moreover, the value of α that we have determined is quite consistent with the one proposed in the literature [3] for the bulk ZnO material (4.3 10^{-6} K^{-1}).

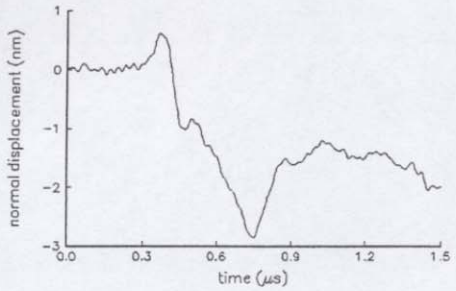

Figure 4
Normal displacement vs time
for a small spot size

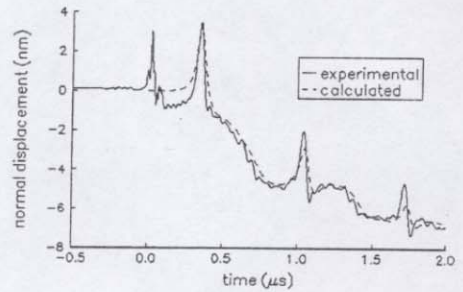

Figure 5
Experimental and calculated normal displacements
vs time for a large spot size

4. DETERMINATION OF a - CONCLUSION

Now that $\beta\sqrt{a}$ and β have been evaluated, it is easy to determine the value of the thermal diffusivity a: we find a = 9.8 10^{-6} m^2/s, which is in very good accordance with the value obtained from a "flash" technique measurement (9.0 10^{-6} m^2/s).

The combination of the photoacoustics and laser-ultrasonics techniques have allowed us to measure optical, thermal, thermo-mechanical and mechanical parameters with the need of only two experimental apparati. This represents a new non-destructive technique for the investigation of material properties.

References:

[1] McDonald F.A., *J. Opt. Soc. Am.* **70**(5) (1980) 555-557.
[2] Dubois M., Enguehard F., Bertrand L., Choquet M. and Monchalin J.P., *Appl. Phys. Lett.* **64**(5) (1994), to be published.
[3] Touloukian Y.S., Kirby R.K., Taylor R.E. and Lee T.Y.R., Thermophysical Properties of Matter - Volume 13 - Thermal Expansion - Nonmetallic Solids (IFI/Plenum, New York, 1977) p. 444.