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Measurement of internal friction in polycrystalline materials using laser-generated ultrasound

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Abstract

A new technique is presented allowing one to measure ultrasonic absorption or internal friction over a wide temperature range in polycrystalline materials by generating reverberation fields using short laser pulses and by observing their decay using optical interferometers for detection.

1. Introduction

Ultrasonic methods have been successfully applied for many years in non-destructive testing [1], physical acoustics [2], solid state physics [3] and materials science [4–6]. The ultrasonic attenuation contains mainly two different types of losses namely elastic losses which conserve the mechanical energy in the sample volume and inelastic losses or internal friction where the mechanical energy is dissipated, *i.e.* transferred into heat. In polycrystalline materials an important elastic loss occurs when the ultrasound is scattered by grain boundaries due to the elastic anisotropy. Ultrasonic scattering has been studied intensively [7], and if the wavelength is much larger than the grain size, the attenuation coefficient α_S due to scattering can be described by Rayleigh's law

$$\alpha_S = Sd^3f^4 \tag{1}$$

where S is the scattering parameter, d is the grain size and f is the ultrasonic frequency. The total attenuation coefficient α can then be expressed as

$$\alpha = \alpha_G + \alpha_A + \alpha_S \tag{2}$$

where α_G stands for the attenuation coefficient representing geometric losses and α_A stands for the attenuation coefficient for inelastic losses. By using ultrasonic backscattering measurements and exploiting eqns. (1) and (2), one can determine the grain size non-destructively and perform characterization of the microstructure [7]. Such measurements are based on the evaluation of α_S and its dependence on the microstructure. However, it is not possible to derive from these measurements either the absolute value α_A or its frequency dependence with sufficient accuracy. This is due to the fact that α_A is often much smaller than α_S. In steels the ratio α_A/α_S extends from 0.05 to 0.3 depending on the material and the frequency used [7].

Recently several methods for absorption measurements in scattering media have been suggested by observing the diffusion of scattered ultrasound [8], the IR radiation caused by absorption [9] and the damping of the resonance of small cylindrical samples [10]. Another way for direct measurements of absorption is the reverberation technique. In this technique the property that an incident ultrasonic beam loses its coherence by scattering is exploited [11]. After a certain time the elastic energy of the beam will be distributed over the whole volume of the sample and the temporal decay of this "homogeneous diffuse field" will only be determined by absorption. The distribution of ultrasound and its decay corresponds to the reverberation of sound in rooms. The energy distribution of the various wave types in a diffuse field has been calculated showing that the shear modes are dominant [12]. This distribution of energy in the various modes, *i.e.* longitudinal, shear and surface waves, depends on the sound velocities of the material under consideration. The ratio of the energy content of the transverse and longitudinal waves in the reverberation field is constant and does not depend on the volume of the sample, whereas the energy content of the surface waves varies with the sample volume and with frequency. Because all these modes contribute to the reverberation field, measurements will yield a weighted absorption coefficient α_A which can only be given in units per time and not per distance and the corresponding logarithmic decrement δ is then $= \alpha_A/f$. An example might elucidate this. The reverberation field in a cube of Al of 1 cm side length would contain at 5 MHz, 86% of the energy in the shear modes, 5% in the longitudinal modes and 9% in the surface waves. Hence measuring α_A by observing

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the decay of a reverberation field will not give a value which can be assigned to one mode only, because high absorption of one mode may dominate the decay of the reverberation field. In view of the fact that the inaccuracies in determining α_A by normal back-wall echo or backscattering measurements are only accurate within one order of magnitude, this can be tolerated.

In this paper we report on a non-contact technique allowing measurements of α_A by observing the decay of the reverberation field. Ultrasonic waves are generated by short laser pulses and detected by an interferometer. It is our intention to show the capability of laser-generated ultrasound (also called laser ultrasonics [13]) to obtain reliable data on the absorption coefficient α_A (albeit with the restriction mentioned above) in technical materials. We want to use α_A as a parameter for material characterization. We measured α_A of fine-grained aluminium, of ceramic silicon carbide, of white cast iron exhibiting a phase transition near 480 K and of several other materials, even monocrystalline copper.

2. Generation of ultrasound by laser pulses and its interferometric detection

When a solid is irradiated by short laser pulses, broad band ultrasonic pulses are launched [13] allowing the remote excitation of ultrasound. Depending on the optical power of the laser pulses, one can distinguish two different generation mechanisms. In the thermoelastic regime where the laser power density is below the material ablation threshold, pressure, shear and surface waves are generated simultaneously by the stresses induced by thermal expansion of the heated volume. At higher power densities, material ablation changes the mechanism. In this case a plasma is formed close to the sample surface, resulting in an increased pressure wave amplitude owing to the momentum transfer due to the plasma pressure. As a result the generated waveform depends on the laser intensity [14]. In conjunction with interferometric detection, laser ultrasonics is suitable for application at very high temperatures. Laser interferometers are less sensitive than piezoelectric, capacitive or electromagnetic transducers but are suitable for non-contact and remote detection. There are different types of interferometers with typical sensitivities of 10^{-5} nm $Hz^{-1/2}$ at 1 mW collected light power. A review of interferometric measurement techniques can be found in ref. 15.

3. Experimental arrangement

Figure 1 shows the experimental set-up used. The laser pulse for ultrasonic generation is directed through a window of the furnace and focused on to the sample. The beam of the interferometer impinges on to the opposite and polished surface of the sample through another window of the front door. The ultrasonic signals are detected by the interferometer, demodulated, stored in a digital transient recorder and then processed by a computer. For generation of the ultrasonic signals an Nd–YAG laser at a wavelength of 1064 nm with a maximal energy of 450 mJ and about 10 ns pulse duration was used. The beam diameter of 7 mm was focused to 3 mm on the sample surface. For detection we used a heterodyne Michelson interferometer which is stabilized against vibrations [16].

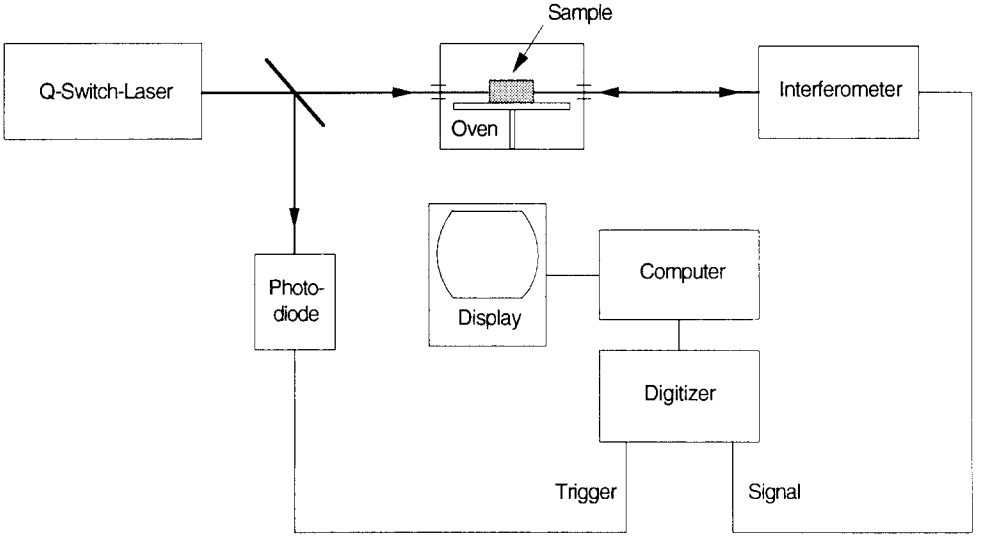

Fig. 1. Principle of experimental arrangement. A laser pulse impinging on the surface of the sample generates broad band ultrasonic pulses. After travelling through the sample, they are detected on the other side of the sample by an interferometer of suitable design and bandwidth. For high temperature measurements (presently up to 1000 °C) the sample is placed in an oven.

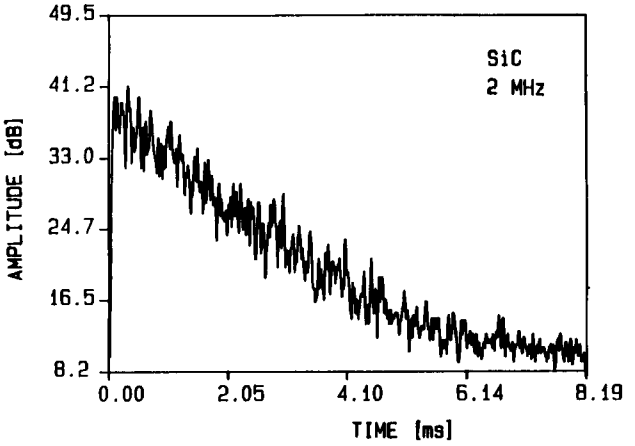

Fig. 2. Decay of a reverberation field obtained in SiC at 2 MHz at room temperature. As can be seen, the decay time is approximately 4 ms. Assuming that transverse waves are dominant in the reverberation field (see text), this corresponds to a path-length of 30 m.

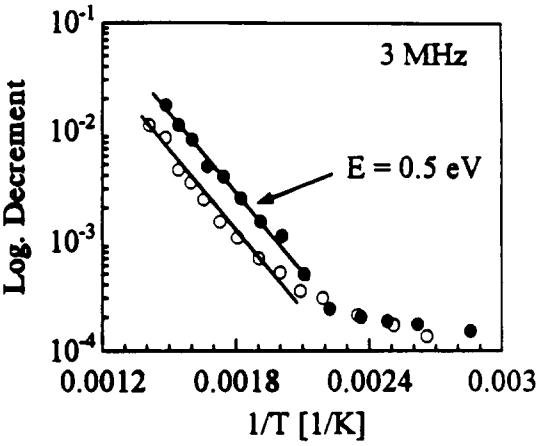

Fig. 3. Logarithmic decrement at 3 MHz in the two different aluminium materials (○) AlCuPb and (●) $AlMg_3$ as a function of inverse temperature. Assuming a Debye relaxation, an activation energy of 0.5 eV is obtained.

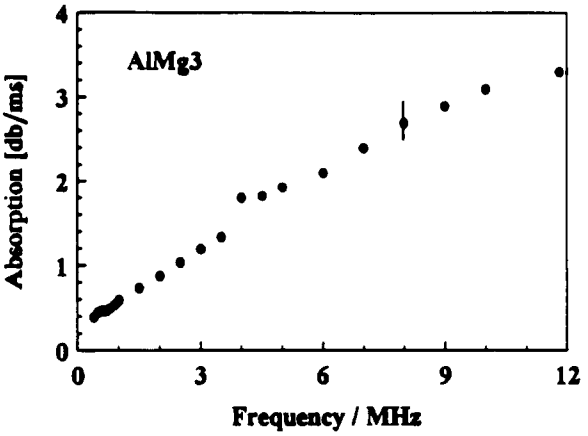

Fig. 4. Frequency dependence of the ultrasonic absorption α_A in the aluminium alloy $AlMg_3$ at room temperature. A linear dependence is obtained which is also observed in many other technical materials.

The absorption measurements were carried at power densities from 400 to 600 MW cm^{-2}, generating a reverberation field of sufficiently large amplitude. For measurements at a set frequency the demodulated signal was passed through a variable narrow band logarithmic amplifier (Fig. 2). Depending on the signal-to-noise ratio of the reverberation field in a given sample, absorption data could be taken from 200 kHz to about 15 MHz. In order to improve the signal-to-noise ratio, the signals could be averaged several times.

Samples of different materials, size and shape have been chosen for measurements: (i) a disc of the aluminium alloy AlCuPb 5 cm in diameter and 1 cm thick with a grain size of about 20 μm and containing 4.7 wt.% Cu inclusions mainly as $AlCu_2$ precipitates, and an $AlMg_3$ sample; (ii) a ceramic SiC plate of about 5 μm grain size with the dimensions $4.7\times4.8\times0.5$ cm^3; (iii) a piece of white cast iron containing 57.6 wt.% cementite with the dimensions $2.0\times1.4\times0.5$ cm^3; (iv) monocrystalline copper 99.999% pure. The samples were supported in the furnace on ceramic rings. The maximal temperature of the furnace was 1400 K. The temperature was measured with a thermocouple near the sample. During a given absorption measurement temperature variations of ±2 K were possible. For the absorption measurements signals excited at various locations in one sample (usually along a line) were averaged. Additionally, digital filtering of the signals was sometimes used. These procedures resulted in a decay pattern whose slope can be more easily evaluated by linear regression.

4. Results and discussion

Figure 3 shows a logarithmic plot of δ *vs.* inverse temperature $1/T$ for the two aluminium samples for temperatures up to 830 K at 3 MHz. For small values of $1/T$ an Arrhenius behavior of δ can be observed.

We have also measured the frequency dependence of the absorption coefficient α_A in the aluminium alloys at room temperature. Figure 4 shows a typical result for $AlMg_3$. The frequency dependence is found to be almost linear. This linear frequency dependence can be found in many polycrystalline metals, *i.e.* there is no distinct evidence of a relaxational type of damping in the usual way, because in this case α_A would vary as ω^2 for $\omega\tau \ll 1$ (where τ is the relaxation time). Some time ago a hysteretic type of absorption was invoked in order to explain the linear frequency dependence [17, 18]. It would be interesting to test this idea for polycrystalline materials in order to gain insight to the relaxing species. If one measures the absorption in a pure monocrystalline sample, one can observe, however, the usual relaxational behaviour due to dislocation

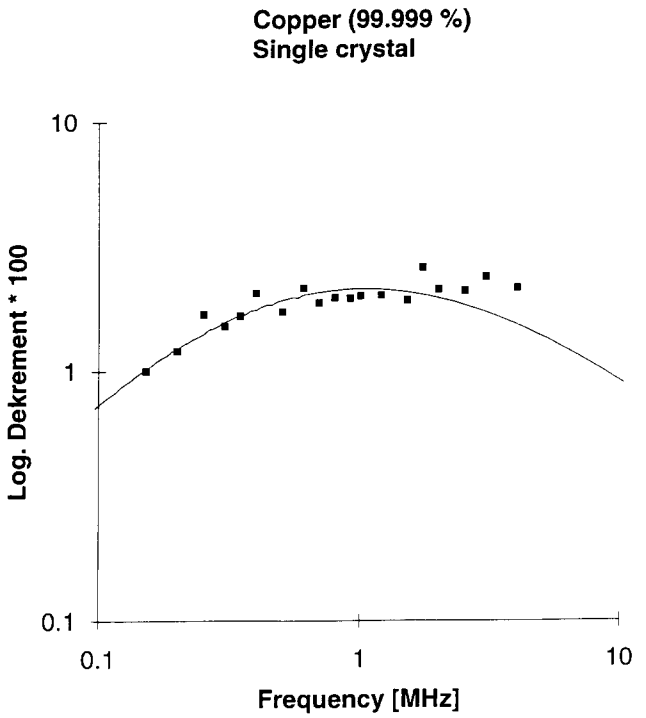

Fig. 5. Logarithmic decrement of pure copper as a function of frequency from 0.15 to 5 MHz at room temperature. A clear maximum is discernible due to the movement of dislocations which can be described by the Granato–Lücke model (solid curve [20]). The dislocation density is approximately 10^7 cm^{-2}.

damping as described by the Granato–Lücke model [19] (Fig. 5). Reverberation measurements in monocrystalline samples are possible provided that the sample is sufficiently small so that the reverberation field is built up by reflections and the ensuing mode conversions at the side walls [20]. This process is facilitated by the large angular spectrum contained in an ultrasonic source generated by a pulsed laser. Furthermore, in silicon carbide measurements of the absorption were performed up to a temperature of 1370 K. The logarithmic decrement first decreases with increasing temperature and exhibits a minimum at about 580 K; δ then increases again and passes through a maximum. Finally we measured the ultrasonic absorption at 5 MHz in white cast iron containing 57.6 wt.% cementite. A sharp maximum is obtained at 480 K indicating the occurrence of a phase transition, which is also reflected in the data for the elastic moduli as a function of temperature. Further details can be found elsewhere [21, 22].

5. Conclusions

We have shown that laser-generated ultrasound combined with its interferometric detection offers the possibility to obtain ultrasonic absorption data in polycrystalline samples even at high temperatures by observing the decay of reverberation fields. Shear modes contribute predominantly to the logarithmic decrement measured in this way. It appears to us that the standard model describing absorption by dislocation movement does not apply to polycrystalline material. It is our aim to elucidate this question by further experiments.

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