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#### ACOUSTIC MICROSCOPY OF ULTRASONIC ATTENUATION

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<u>Résumé</u> L'atténuation ultrasonique peutêtre imagée à l'aide d'un microscope acoustique à balayage. La zone de plastique située au niveau d'une craquelure sous contrainte a été imagée par transmission, le contraste provenant d'un amortissement de dislocation. Il est aussi montré que le contraste en mode réflexion est sensible à latténuation ce qui permet de détecter et d'imager l'attenuation avec une résolution spatiale d'environ 2µm.

<u>Abstract</u> The scanning acoustic microscope can image ultrasonic attenuation. The plastic zone at a stressed notch has been imaged in transmission with the contrast arising from dislocation damping. Contrast in reflection is also shown to be sensitive to attenuation, enabling attenuation to be detected and imaged with spatial resolution of  $\nu 2\mu m$ .

#### TRANSMISSION ACOUSTIC MICROSCOPY

The scanning acoustic microscope has now been developed to the point where it can be used routinely to image elastic properties of solids with high spatial resolution<sup>1,2</sup>. The most obvious way to image variations in ultrasonic attenuation is to use the microscope in transmission; in this mode two lenses are arranged confocally, with one acting as transmitter and the other as receiver, and the specimen is placed between them and scanned mechanically to build up an image. Regions of high attenuation would appear dark in an image made in this way.

At the interface between the coupling fluid and a metal strong refraction occurs. Resolution of properties inside the solid is therefore optimised by reducing the aperture of the lens<sup>3</sup>, in order to minimise the convolution of the geometrical aberations and diffraction effects. The optimum numerical aperture is found to be

$$NA = n \sin[\tan^{-1}(\frac{.704\lambda}{n(1-n)^{2}s})^{\frac{1}{4}}]$$

where n is the refractive index of the solid,  $\lambda$  is the wavelength in the liquid and optimum focus is required at a depth s below the surface. Thus, for example, in our transmission microscope operating at 140MHz imaging inside an aluminium specimen 2mm thick, the optimum NA is about 0.1, giving a resolution better than 0.05mm.

This acoustic microscope has been used to image the plastic zone at a stressed notch. The material was polycrystaline aluminium of 99.999% purity. Its acoustic behaviour was first studied during homogeneous deformation. A specimen was glued into grips in a tensile testing machine, and the attenuation of longitudinal waves propagating along a 30mm gauge length was measured. It was confirmed that the onset of plastic deformation caused a dramatic increase in attenuation, and that when deformation was stopped relaxation occured permitting the attenuation to decrease eventually to a low level. These effects were attributed to dislocation damping<sup>4</sup>, and provided encouragement to use this contrast mechanism to image a plastic zone.

Specimens of the same material were therefore prepared in the form of discs of 6mm diameter and 1mm thickness, with flat and parallel faces. A fine radial slot 2mm long was spark machined into the disc, which was then annealed just below its melting



0.2mm

(b)

ig.1: Transmission acoustic micrographs at 140MHz of the region around the tip of a park cut slot in a polycrystalline aluminium disc. (a) is a photograph before stressing; (b) was taken after loading the disk by driving a wedge into the upper end of the slot.

point. A special specimen stage had been prepared with a small wedge which could be driven into the slot, and the specimen was glued into this holder. Before applying any force to the wedge the specimen was imaged while it was still unstressed; this is shown in Figure 1a. The wedge was then driven into the crack so as to apply a tensile stress at the notch tip and thereby cause a small amount of plastic deformation, and new images were made as seen in Figure 1b.

The preliminary nature of these results must be emphasised. A particular concern was to ensure that the change in contrast was not due to geometrical effects such as surface deformation. To check this the specimen was left for  $\frac{1}{2}$  hour and imaged again; it was found that the attenuation had reduced and images similar to Figure 1a were obtained, thus confirming that the dark contrast in Figure 1b might indeed be due to dislocations. Unfortunately the area imaged in Figure 1 is not large enough to show clearly the shape and size of the plastic zone; nevertheless, it shows that the zone can be revealed. Modifications are now being made to increase the usefulness of such pictures taken in transmission.

#### REFLECTION ACOUSTIC MICROSCOPY

(a)

Higher resolution may be obtained by imaging a surface in reflection. This is because higher frequencies may be used, the wavelength in the liquid is shorter than in the solid, geometrical aberrations are negligible and large numerical apertures are used. The experimental results presented here were obtained at 730MHz, giving a resolution of about  $2\mu m$ . Ultrasonic attenuation can be observed in the form of attenuation of leaky Rayleigh waves propagating along the surface of the specimen; this effect may be understood in terms of the imaging theory of the acoustic microscope.

The acoustic wave reflected by a specimen at focus in an acoustic microscope gives rise to a signal

$$V(o) = A_{O} \int_{0}^{\frac{1}{2}\pi} P^{2}(\theta) R(\theta) \sin\theta \cos\theta d\theta$$

where A is a normalising constant,  $\theta$  is the angle to the optic axis,  $P(\theta)$  is the pupil function of the lens (including aberrations) and  $R(\theta)$  is the reflection function of the specimen surface. If the specimen is now defocussed by an amount z then a phase shift 2k.z is introduced (where k is the wave vector in the coupling fluid), which is a function of  $\cos\theta$ , giving

$$V(z) = A_0 \int_0^{\frac{1}{2}\pi} P^2(\theta) R(\theta) \exp(i2kz\cos\theta) \sin\theta\cos\theta d\theta.$$

For a given lens,  $P(\theta)$  is fixed, so that the function V(z) depends only on  $R(\theta)$ . By a change of variable,  $t = 2\cos\theta$ , and adjustment of the limits, this may be expressed as a Fourier transform

$$V(kz) = \frac{A}{4} F[P^{2}(t)R(t).t]$$

which may be inverted to give

$$R(t) = \frac{4}{A_{0} \cdot P^{2}(t) \cdot t} F^{-1}[V(kz)]$$

sc that from an experimental measurement of V(kz), R(t) may be deduced over the range of t for which P(t) is non zero.

The impedance mismatch between water (the most commonly used coupling fluid in the acoustic microscope) and metals/rather large, so that little contrast is seen from specimens at focus. However, as the specimen is defocused the variation of the signal is determined by the reflection function, so that regions of a specimen with different reflectance functions will give rise to differing contrast. The reflectance function is a complex quantity; in Figure 2 the reflectance function for isotropic aluminium is shown in terms of its amplitude and phase. The most prominant feature is a change of phase by approximately  $2\pi$  at an angle just greater than the shear wave critical angle. This corresponds to excitation of a leaky Rayleigh wave on the solid surface, and is centred around  $\theta_R$ , the angle of incidence for excitation of such waves. The presence of this strong feature might be expected to give a periodicity in V(kz) of spacing  $\frac{\pi}{\cos(\theta) - \cos(\theta)}$ , and this is indeed found to be

If the solid is lossy, so that the leaky Rayleigh wave is attenuated by damping mechanisms as it propagates, this affects the features of the reflectance function around  $\theta_R$ , as illustrated in Figures 2b and c. As the attenuation increases a dip in the model of the reflectance function at the Rayleigh angle appears, which first



Fig.2: The reflectance function for waves incident on isotropic aluminium from water. The curves show 1, the amplitude (left ordinate) and 2, the phase (right ordinate) of the reflection coefficient as a function of angle of incidence for the following bulk attenuations: a, zero; b,  $0.85dB/\lambda$ ; c,  $4.5dB/\lambda$ .





Fig.3: V(z) for an isotropic aluminium specimen with varying attenuation as in Figure 2. The parameters used in the computation are appropriate for 730MHz. The parameter z indicates the amount of defocus, negative z corresponding to movement of the specimen towards the lens.

grows and then diminishes and broadens. This is accompanied by variation in the associated phase change. The form of V(z) may be calculated for each of the reflectance functions in Figure 2, and this is shown in Figure 3. For the loss-free case (a) the periodic nulls are clearly seen for  $z \leq 0$ . For a small amount of attenuation (b), the mean level of the signal initially becomes lower; then as the attenuation increases further (c) the depth of the nulls decreases. Thus, variations in the damping in a material manifest themselves as variation in  $V(z)^{\frac{6}{7}}$ .

It should be noted that Figure 2 has been calculated for an ideal pupil function for a lens of NA = 0.87. The actual pupil function of the lens used to obtain the experimental results below is rather more complicated, and therefore caution should be excercised in comparing Figure 4 with the theoretically generated curves.

The effect of dislocation damping in pure aluminium has been observed by in situ deformation in a scanning acoustic microscope operating at 730MHz. Another polycrystalline specimen of 99.999% pure aluminium was polished to ordinary metallographic standards and again annealed just below its melting point. It was then mounted in a compression straining stage on the microscope, and levelled. For this experiment, the imaging scan of the microscope was then turned off, and the specimen was scanned in the z direction (i.e. along the lens axis) in order to measure V(z) for a single spot on the specimen. The results are shown in Figure 4. Fig.4a shows V(z) for the specimen as annealed, it should therefore correspond to damping due to a very low dislocation density, together with other attenuation mechanisms. Figure 4b was measured within three minutes of a compressive deformation. This was sufficiently small not to cause significant geometrical changes in the surface, but large enough to generate a fairly high dislocation density. The effect of the dislocation damping on V(z) can be seen on this curve, in particular, the intensity at  $z = -5\mu m$ increases considerably. In order to confirm that the change was not due to topographical effects, relaxation was observed without touching the microscope or the specimen. Figure 4c was taken after 10 minutes, and shows partial relaxation. Figure 4d was taken after 30 minutes, and comparison with Figure 4a indicates that the specimen has relaxed almost to its original level of damping or even less, noting however, that the two are not directly comparable.

The resolution of the reflection microscope at this frequency is approximately  $2\mu m$ , depending on the defocus. These measurements demonstrate the possibility of measuring attenuation over a region of this size. It is therefore possible to image the extent of a plastic zone with high resolution by holding z fixed and scanning the lens in a plane parallel to the surface of the specimen. Figure 4 shows that a suitable defocus to choose would be z = 14um because at this value the signal V is strongly dependent on the amount of damping.



Fig.4: Experimental V(z) curves at 730MHz for polycrystalline aluminium @  $40^{\circ}$ C. (a) unstrained; (b) 2mins after compressive straining; (c) relaxed for 10 mins at constant strain; (d) relaxed for 30 mins. The total z scan in all cases was  $31\mu$ m.

The results of an early experiment to image plastic deformation are shown in Fig.5. The sample, a 30mm x 6mm x 1mm bar, was prepared as in the V(z) experiment, and in addition, a 2mm deep spark cut was made before annealing. The specimen was then imaged at a defocus of  $z = -14\mu$ m (Fig.5(a)). The slot tip is just out of the picture on the right hand side; the patterning on the surface is topographical in origin and arises from the polishing process.

The specimen was then subjected to a tensile strain of approximately 0.2%, after which the average signal was observed to be much reduced (cf. Fig4o). The gain of the amplifier was then increased to give a similar average signal to that used in the first picture and another scan made (Fig.5(b)).

As the specimen was allowed to relax, at constant strain, pinning of dislocations resulted in a reduction of attenuation, and hence an increase in signal (cf. Figs. 4(c), 4(d)). This led to a zone of increased signal which swept across the picture from left to right, ie. from regions of low plastic deformation to regions of high plastic deformation. (Figs. 5(c)-5(e)). These were made using exactly the same settings as Fig 5(b), at times of 10, 14 and 30 minutes after straining. As a final check that this was not a topological effect due to bending of the

specimen, a scan was made at  $z=0\mu$ , at which defocus the microscope is insensitive to attenuation, but is still as sensitive to topography as was the scan at  $z=-4\mu$ m (Fig 5(f)). This is seen to be fairly uniformly bright, corresponding to a flat specimen.

#### CONCLUSION

The unique advantages of the acoustic microscope lie in its ability to image through opaque solids and to reveal the elastic properties of materials. If the elastic properties are expressed as complex numbers, then the imaginary part corresponds to losses due to damping. In the transmission microscope this may be measured directly, in reflection it may be deduced from its effect on the reflectance function. In both cases the possibility exists of imaging variations in attenuation. This has been illustrated by the case of plastic deformation; it is hoped that the technique may also be of value in other cases where local variation of attenuation is of importance.

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Fig.5(a). Unstrained. z=-14jum



Fig.5(b). Strained c.0.2% & relaxed for 1 min.



Fig.5(c). Relaxed for 10 mins.



Fig.5(d). Relaxed for 14 mins.



Fig.5(e). Relaxed for 30 mins.



Fig.5(f). z=0, m.

Fig.5: Reflection acoustic micrographs at 730MHz of the region to the left of the tip of a spark cut slot. Specimen defocus was z=-14µm. (See text)

The plastic zone plays a vital role in fatigue and fracture. Various techniques have been devised to measure its size and shape, including etching, microhardness, selected area channelling pattern degradation and (most recently) positron anihilation. Acoustic microscopy offers the possibility of measuring plastic zones more rapidly (picture aquisition time typically 20s) and with good resolution, using the mechanism of dislocation damping.

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