JOURNAL OF MATERIALS SCIENCE 23 (1988) 1748–1751

Acoustic scanning microscopy of grain structure in isotropic solids: pure aluminium and Al–2.5% Mg alloy

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A scanning acoustic microscope (SAM) has been built, and its main features are described. Acoustical imaging of the grain structures in elastically isotropic 99.995% pure aluminium, Al–2.5% Mg alloy, and of a composite structure of Al + SiC particles has been performed using this instrument. We obtain surprisingly good contrast and therefore good imaging in both pure aluminium and Al–2.5% Mg. The fact that we obtain good contrast even in elastically isotropic materials like aluminium and Al–2.5% Mg shows that SAM imaging is very sensitive to mechanical near-surface properties.

1. Introduction

The scanning acoustic microscope (SAM) was developed into a scientific instrument by Quate and co-workers at Stanford during the 1970s and early 1980s. Towards the end of that period, when the power of the instrument became obvious, a large number of groups around the world joined in the task. The instrument has now reached a degree of sophistication where it has become a natural supplement to traditional optical, electron and other microscopy in any materials characterization laboratory. Essential features like resolution and contrast are reasonably well understood. The lens theory is well developed. A resolution range from 500 μm to 20 nm is available. These developments have been described in a series of reviews [1–9].

Today much work is devoted to the use of the instrument. The range of demonstrated applicability is rapidly expanding, particularly in materials characterization and in medicine.

It is essential to obtain a precise understanding of the imaging principle and how it differs from the more traditional imaging techniques. One immediate answer is that since we are dealing with acoustic waves, we are dealing with a totally different contrast principle. A second answer is that the instrument is capable of viewing through structures that are either opaque to, or too thick for, optical and electron microscopes. A third feature is that, beyond being an instrument for structural characterization, it offers a quantitative method for the characterization of mechanical near-surface properties over microscopic regions of the sample [10–12]. This is obviously a very important fact since surface properties are so important for the understanding and use of materials. In both pure aluminium and Al–2.5 wt % Mg samples, apart from observing contrast reversal in the grains, contrast reversal in the grain boundaries is also observed.

The SAM has been used by several groups to study the grain structure in different materials. The origin of contrast and the acoustical material signature have been discussed in a series of papers [13–17]. The effect of anisotropy has been utilized to explain the contrast from grain to grain [18, 19]. It has been stated [18] that an elastically isotropic material like aluminium cannot show good contrast from grain to grain. We have therefore undertaken a study of auminium: 99.995% pure elemental, Al–2.5% Mg alloy, and a composite of Al + SiC. We were able to demonstrate that the previous assertions about the difficulty of imaging aluminium are far too pessimistic. We do in fact obtain nearly full contrast on a black and white scale simply due to the difference in grain orientation, a great surprise in view of what has been said [18]. These findings again point to the extremely high sensitivity to the factors influencing the elastic properties near the surface. Our observations have convinced us that the instrument will continue to find new and important applications in surface characterization, and in the ongoing efforts to tailor material surface properties to technological requirements.

2. The SAM system: design and properties

The scanning acoustic microscope [20] is an integrated system for r.f. acoustic wave excitation, propagation and detection, mechanical line-scanning of the lens over the sample area, timing of scanning and r.f. pulse generation and detection, video display, data storage and system control. To make it easily applicable it should run and collect the image at the touch of a button. In principle all such systems will be similar in

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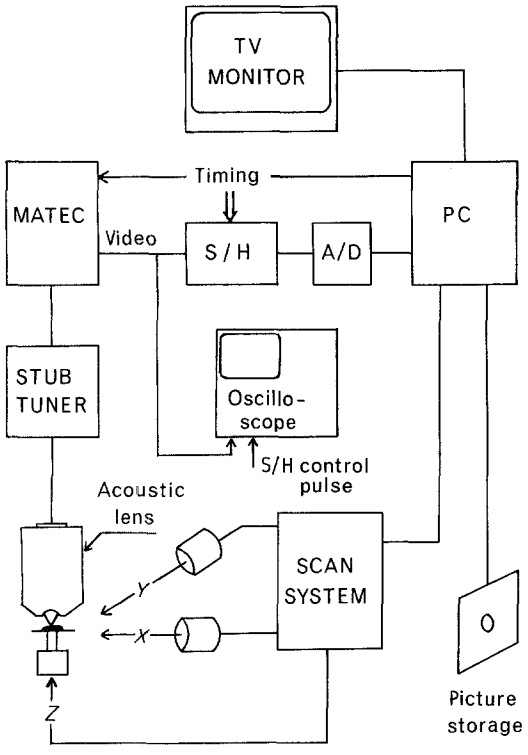

Figure 1 Block diagram of the scanning acoustic microscope, showing schematically how the different parts of the system are connected.

most respects, although the practical implementation of the various parts may be very different from one system to the next. Our system is to a large extent home-made, as regards the control system, timing circuits, software, lenses and mechanical parts. The r.f. system, personal computer (PC), vibrators, vibration detectors and z-scan unit are all standard commercial items.

A schematic diagram of the whole system is shown in Fig. 1. In Fig. 2 we show a close-up of the mechanical scan system. We refer to the figure captions for details.

The lens used in the present work is a single-crystal sapphire lens whose c axis is along the direction of propagation of acoustic waves. The length is 16.2 mm, the aperture 2.5 mm, the effective opening angle is 50°, and the focal length 1.73 mm.

With the system operated at 90 MHz it has a two-point resolution of 13 μm, while at 150 MHz the resolution is 8 μm. The two-point resolution is calculated according to the expression

$$h = 1.13 \frac{\lambda f}{D} \quad (1)$$

Here λ is the wavelength in water, f is the focal length and D is the aperture diameter.

As is well known, the sapphire–water combination gives a diffraction-limited resolution with insignificant spherical aberration.

The radiofrequency (r.f.) transmitter/receiver system we used is a Matec 6600 (Matec Inc., Warwick, RI, USA) unit. The output from the transmitter is sent to the lens via a stub tuner for impedance matching. The receiver has a video output giving the positive envelope of the received pulse. This output is passed to two sample-and-hold (S/H) circuits mounted in succession, and also to the oscilloscope for viewing. The first S/H circuit is a high speed (25 nsec), high accuracy (0.1%) S/H circuit, and the second one is a slower one to ensure that the signal is held long enough to be digitized and stored without drop in signal level. The output from the second S/H circuit is passed to an 8-bit analogue–digital (A/D) converter where the digital output is read and stored by the PC via a parallel port.

In order to obtain a full image, the lens must be scanned over the sample in a raster pattern. This is done by two vibrators, one moving the lens fast along the x axis, the other moving the sample one step along the y axis per line scan along the x-axis. The vibrators consist of a permanent magnet and a moving coil, and can move a total of 5 mm peak to peak. The signals controlling the vibrators is generated by a single-board computer called Bitten, made by ELAB, Norwegian Institute of Technology, via parallel ports and D/A converters. The Bitten computer is linked to the PC by a general-purpose interface bus (GPIB) to receive information regarding the size of the scan, frequency, etc., before starting the exposure of the image. The vibrators are also connected to two linear voltage differential transducers (LVDTs) which measure the actual displacement. This measurement is used as a feedback to a regulator to make the positioning of the lens as accurate as possible.

The lens can also be moved along the z axis using a stepping motor. This motor can be either manually controlled or software-controlled from the PC via the GPIB bus. It has a resolution of 1/8 μm per step.

An important feature is the timing of the mechanical scan and of the r.f. pulse detection. This is achieved by different trigger and strobe pulses generated from a specially designed circuit. The PC is provided with a programmable interrupt timer which is used as a rate generator to make the main trigger pulse. This pulse triggers the generation of r.f. pulses in the Matec unit, and is also used by another circuit to produce three additional pulses delayed in time relative to the r.f. pulse generation. Two of these are control pulses to the S/H circuits, and the time delay of these can be shifted to select the right reflection pulse for imaging. The third is a strobe pulse to the Bitten computer which uses it to control the motion of the lens and the A/D conversion of the video pulse. The synchronization is obtained by having the PC read the A/D

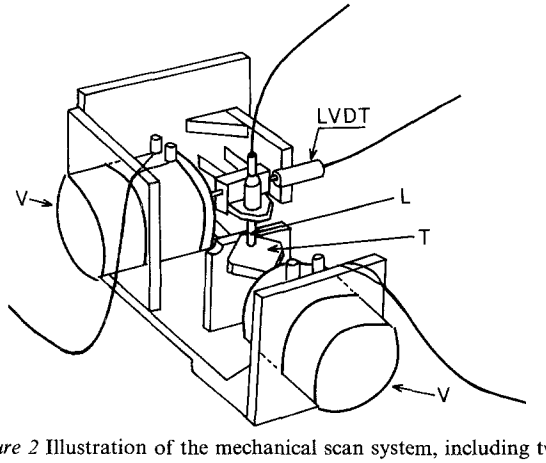

Figure 2 Illustration of the mechanical scan system, including two vibrators (V), two LVDT transducers (LVDT), a table (T) for mounting the sample and an acoustic lens (L).

converter when receiving an interrupt signal from the Bitten computer, and returning a handshake signal when ready for a new input.

The software running in the PC consists of several modules written in Basic and assembly language. This main control program is command-driven to make the use of the acoustic microscope as easy as possible for the operator. The main features are:

(a) To start the exposure of an acoustic image by sending data about the scan size, etc. and a starting directive to the Bitten computer.

(b) To alter a set of parameters determining the scan size, scan position, scan frequency and the r.f. pulse repetition rate.

(c) To receive the image from a video camera mounted on an optical microscope for comparison.

(d) To store and recover micrographs on magnetic discs.

(e) To control the stepping motor for moving the lens in the z-direction for controlled defocusing.

In addition there are programs for measuring received voltage as a function of defocus along the z axis, so-called $V(z)$ curves, and obtaining line scans, features not yet incorporated in the main control program.

3. Results and discussion

In our investigations all the samples were mechanically polished down to 1 μm and imaged without etching. As reported in other studies, almost no contrast was found at focus. Fig. 3 shows the image at a defocus of $z = -40\,\mu$m for pure aluminium, in a cold-rolled and recrystallized sample. In view of the well-known elastic isotropy of aluminium (anisotropy factor $\eta = 1.22$) compared to copper ($\eta = 3.20$) and nickel ($\eta = 2.38$), we get surprisingly good contrast between the grains. We observe a few grains which are very dark while there is less contrast among other grains. This, we suggest, is mainly because the majority of the grains are oriented in the same direction. It has been observed [21] by electron microscopy that cold-rolled and recrystallized aluminium can have a high degree of single-axis texture. From the maximum change in velocity of sound along the principal crystallographic axes, in aluminium we find that a difference less than 3% in acoustic impedance can give quite good contrast.

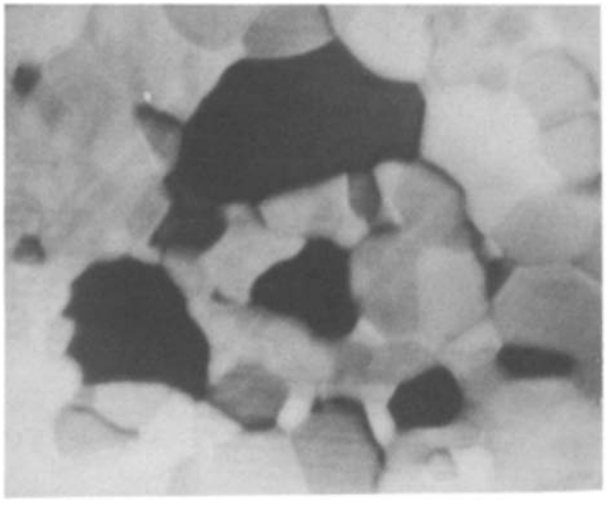

Figure 3 Acoustic micrograph of mechanically polished, unetched polycrystalline sample of 99.995% pure aluminium taken at a defocus of $z = -40\,\mu$m, scan area 4.5 mm × 4.5 mm, frequency 90 MHz.

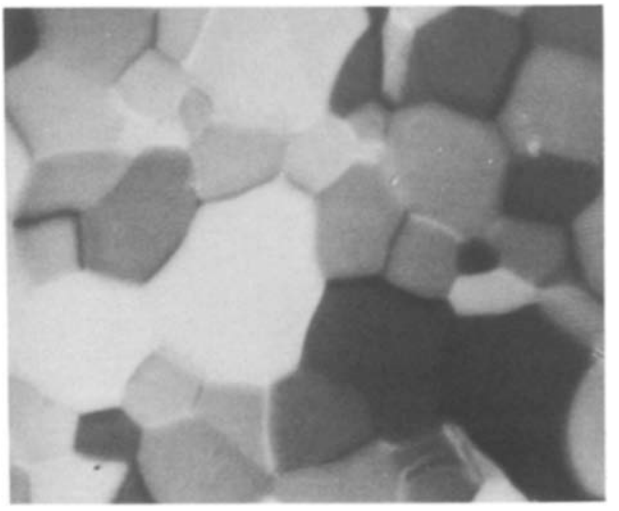

Figure 4 Acoustic micrograph of mechanically polished, unetched polycrystalline alloy of Al–2.5% Mg, taken at a defocus of $z = -33\,\mu$m, scan area 4.5 mm × 4.5 mm, frequency 90 MHz.

The Al–2.5% Mg sample is a recrystallized sample which has an average grain size of $\tilde{D} = 268\,\mu$m. An acoustic image obtained in this sample at $z = -33\,\mu$m is shown in Fig. 4. To demonstrate the contrast reversal in a few grains, an image taken at $z = -73\,\mu$m is shown in Fig. 5. Addition of 2.5% Mg would substitute some of the aluminium atoms in the structure; the distribution being random, the elastic anisotropy will not be changed much. In addition magnesium atoms settle on the grain boundaries. Although the material is elastically isotropic we get nearly full contrast in this material.

In order to illustrate the power of the instrument we show also in Fig. 6 the structure of Al + SiC composite taken at a defocus of $z = -40\,\mu$m. The composite has 25% by weight of finely distributed silicon

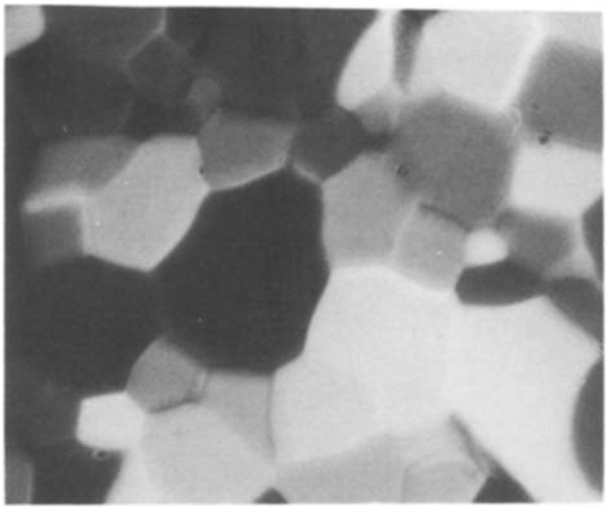

Figure 5 Acoustic micrograph of mechanically polished, unetched polycrystalline alloy of Al–2.5% Mg, taken at a defocus of $z = -73\,\mu$m, scan area 4.5 mm × 4.5 mm, frequency 90 MHz. This figure demonstrates contrast reversal in some grains compared to Fig. 4.

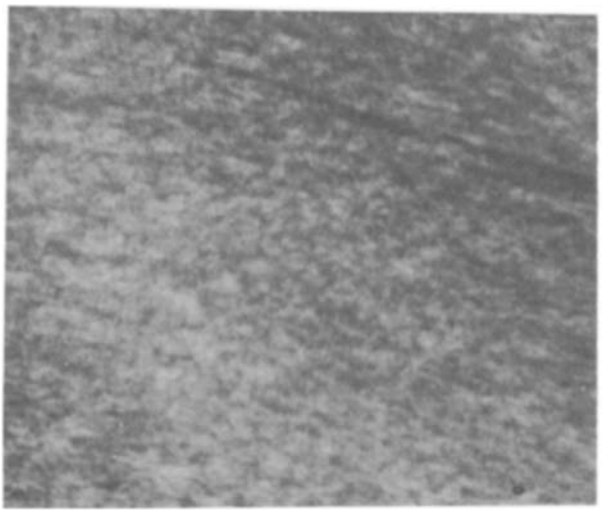

Figure 6 Acoustic micrograph of mechanically polished, unetched Al + SiC composite, taken at a defocus of $z = -45\,\mu m$, scan area 4.5 mm × 4.5 mm, frequency 90 MHz. Particle size before aggregation is 3 to 5 μm.

carbide particles of size between 3 and 5 μm. The dark spots are the silicon carbide particles or aggregates of these, in the aluminium matrix.

In conclusion, we have demonstrated that the SAM instrument is very sensitive to small changes in elastic properties near the surface of materials (here within 10 μm of the surface). It will therefore be suitable for studying the influence of various kinds of surface treatment, an area of great technological importance, which will be our next objective.

Acknowledgements

The authors are grateful to T. Landmark, B. Brattheim and V. Grener for major contributions to hardware and software implementations, and to B. Førre, T. H. Løvaas, K. Ramsøskar, A. Bjølstad and K. Rokhaug for their skilled technical assistance. We thank Professor C. Quate and students at Stanford University, and Dr Hollis and Dr Hammer of IBM, Yorktown Heights for helpful discussions and encouragement. We also thank H. Westengen for supplying us with Al–2.5% Mg samples.

Our work on acoustic microscopy is supported by The Norwegian Council for Science and Humanities (NAVF), The Norwegian Council for Technical and Industrial Research (NTNF), The Norwegian Institute of Technology (NTH), The Norwegian Institute of Technology Fund and Forretningsbanken A/S.

One of the authors (S.S.) would like to thank NTNF for a postdoctoral fellowship and the Indian Institute of Science, Bangalore, for providing leave.

References

1. R. A. LEMONS and C. F. QUATE, in "Acoustic Microscopy in Physical Acoustics", edited by W. P. Mason and R. N. Thurston (Academic, New York, 1979) p. 1.
2. C. F. QUATE, A. ATALAR and H. K. WICKRAMASINGHE, *Proc. IEEE* **67** (1979) 1092.
3. H. K. WICKRAMASINGHE, *J. Microsc.* **129** (1982) 63.
4. J. E. HEISERMAN and C. F. QUATE, in "Frontiers in Physical Acoustics", edited by D. Sette (North-Holland, Amsterdam, 1986) p. 343.
5. M. NIKOONAHAD, *Contemp. Phys.* **25** (1984) 129.
6. M. HOOPE and A. THAER, in "Topics in Current Physics", edited by U. Gonser (Springer, Heidelberg, 1986) p. 7.
7. J. S. FOSTER and D. RUGAR, *IEEE Trans. Son. Ultrason.* **SU-32** (1985) 139.
8. A. BRIGGS, "An Introduction to Scanning Acoustic Microscopy" (Oxford University Press, Oxford, 1985) p. 67.
9. *IEEE Trans. Son. Ultrason.* **SU-32** (1985).
10. J. M. R. WEAVER, M. G. SOMEKH, A. D. BRIGGS, S. D. PECK and C. ILETT, *ibid.* **SU-32** (1985) 225.
11. J. KUSHIBIKI and N. CHUBACHI, *ibid.* **SU-32** (1985) 189.
12. R. D. WEGLEIN, *ibid.* **SU-32** (1985) 225.
13. A. ATLAR, *J. Appl. Phys.* **49** (1978) 5130.
14. *Idem, ibid.* **50** (1979) 8237.
15. H. K. WICKRAMASINGHE, *El. Lett.* **14** (1978) 305.
16. R. D. WEGLEIN, *Appl. Phys. Lett.* **34** (1979) 179.
17. W. PARMON and H. L. BERTONI, *El. Lett.* **15** (1979) 684.
18. M. G. SOMEKH, G. A. D. BRIGGS and C. ILETT, *Phil. Mag.* **49** (1984) 204.
19. C. ILETT, M. G. SOMEKH and G. A. D. BRIGGS, *Proc. R. Soc. L* **A393** (1984) 171.
20. V. B. JIPSON, doctoral dissertation (PhD), Stanford University (1979).
21. J. HJELEN, E. NES and R. HØYER, in Proceedings of International Conference on Aluminium Alloys: Their Physical and Mechanical Properties, Charlottesville, June 1986, Vol. 1, edited by E. A. Starke Jr, T. H. Sanders Jr, G. E. Spangler, P. V. Mara and R. A. Bonewitz (Engineering Materials Advisory Services Ltd, Warley, 1986) p. 471.

Received 6 July
and accepted 12 October 1987