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Rapid Imaging of Microstructure using Spatially Resolved Acoustic Spectroscopy

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Abstract
Microstructure can have profound effects on the bulk mechanical properties of a material, such as its strength and susceptibility to failure under stress. Several well known methods for imaging microstructure exist, including essentially destructive techniques such as etching and electron back-scattered diffraction, and contact techniques such as the scanning acoustic microscope. SRAS – spatially resolved acoustic spectroscopy – is a relatively new laser ultrasound technique that uses surface acoustic wave velocity as its contrast mechanism. Unlike the techniques above it is non-contact, non-destructive, can be used to image large samples, and provides quantitative velocity information for acoustic waves propagating in one or more directions. The technique uses the relative efficiency with which waves of either a fixed frequency or *k*-vector are excited by a grating to obtain the velocity, unlike most laser ultrasound techniques which rely on direct phase or time of flight measurements. This makes SRAS very tolerant to acoustic aberrations and poor signal to noise ratios, and hence is a robust technique. Lateral resolutions of the order of 25μm, and velocity resolutions better than 1m/s are illustrated by striking images of microstructure from industrially relevant materials.
Keywords: Laser ultrasound, material characterization, microstructure, acoustic imaging, surface acoustic waves (SAWs), acoustic velocity, crystal orientation, orientation imaging.

1. Introduction

Material characterization techniques are important because engineering properties such as yield strength, fracture toughness and thermal conductivity are highly structure-sensitive. The ability to image microstructure can provide valuable insights into the suitability of a material – or a specific sample – to perform adequately in its intended role, as well as providing feedback about the success or otherwise of alloy refinement techniques (ageing, cold rolling and so on). For these reasons, a number of techniques have been developed to image microstructure and to interrogate surface material properties. Amongst these techniques are: chemical etching to reveal the grain structure; orientation imaging microscopy, usually a scanning electron microscope using electron back-scatter diffraction (EBSD); and acoustic techniques such as the scanning acoustic microscope (SAM). Acoustic techniques tend to use surface acoustic wave (SAW) relative phase or velocity as the contrast mechanism, as this varies with crystallographic orientation. This can be measured in a number of ways, and for many years the SAM has been used to produce excellent qualitative images of material microstructure [1]. Quantitative images of SAW velocity are difficult to achieve on the SAM, as the *V(z)* method would be too time consuming for imaging, although techniques using the relative arrival times of the direct reflected signal and the surface wave for two values of *z* have been published in recent years [2].

Spatially resolved acoustic spectroscopy (SRAS) is a technique that has been developed by the authors to directly and quantitatively image the local SAW velocity of a material's surface, using lasers to excite and detect the ultrasound. Rather than rely on attempting to directly measure the SAW velocity by time-of-flight or relative phase measurement, the technique determines the acoustic phase velocity by measuring the efficiency with which SAWs are excited by a grating. This makes the technique particularly robust, being tolerant to texture effects (acoustic aberration, scattering). It is non-contact, non-destructive, and rapid.

This paper reviews the development of the technique to date, and describes the instrumentation used to acquire the results presented in this paper. The results demonstrate some of the capabilities of the technique in general and the instrument in its present form.

2. Spatially Resolved Acoustic Spectroscopy

Although the SRAS technique has been described previously [3, 4], it is worth repeating the most salient details here. The technique itself will be described, and a description of the instrument itself follows in the next section.

The spatially resolved acoustic spectroscopy technique has its roots in the optical spectroscopy technique, whereby analysing a spectrum (of light) we can discover some of its properties (dominant wavelengths). In the acoustic sense we are analysing the spectrum of ultrasound emitted from a grating. Either (1) the frequency is fixed, and the grating period is swept, or (2) the grating period is fixed, and the frequency is swept. For the results shown in this paper, the first of these two methods is used.

To acquire the acoustic spectrum for one “point” on the sample, the SAWs are excited by a fixed frequency source – in our case a pulsed laser – using a grating of regularly spaced lines. The SAWs are detected close to – but not overlapping – the grating. The efficiency with which the SAWs are excited depends on how well the grating period matches the wavelength (λ) of the SAWs at that frequency (f). If the frequency is fixed, then the SAW wavelength depends on the SAW phase velocity: $v = f\lambda$. The velocity can be determined by finding the grating period that excites the largest SAWs.

This is how we calculate the velocity for one “point,” where a point corresponds to the area under the grating. To build up a velocity map, the grating is raster-scanned over the material surface. It is important to note that the calculated velocity corresponds to the area under the grating, not the point at which the SAWs are detected.

3. The O-SAM Instrument

The SRAS results in this paper were acquired using our O-SAM instrument (optical scanning acoustic microscope). This laser ultrasound system has been described previously [5] and so only the details relevant to the SRAS technique and the images shown will be described here. The system, configured to perform SRAS measurements, is shown schematically in figure 1.

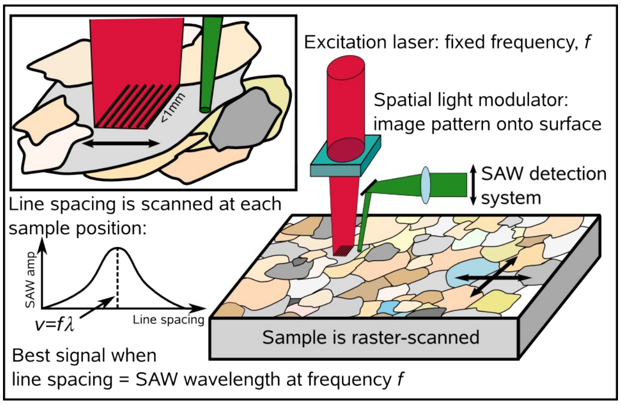

Figure 1. Schematic of the experimental system

The laser used to excite the SAWs is a lamp pumped Q-switched mode locked Nd:YAG laser. The Q-switcher determines the repetition rate, and this is set to 1kHz. Every millisecond, around 30 pulses are emitted as a tone burst, each pulse is separated from its neighbour by 12.1ns; this separation time is determined by the mode lock frequency (which is itself determined by the cavity length of the laser). This gives the laser a fundamental frequency of 82MHz. As the pulses each have a width of approximately 200ps, the harmonics – at multiples of 82MHz – extend into the GHz region.

A spatial light modulator (SLM) is used to image the light emitted from the excitation laser onto the sample. The SLM is a 512x512 pixel device, and we use this to image a grating of an arbitrary line spacing onto the sample surface. The SLM can cycle through a number of different images (gratings of different line spacings) at up to 1kHz, and so successive tone bursts from the laser could produce a different grating if required. The SLM image is 384x384µm in size on the material surface when all the pixels are used, giving a spatial resolution for SRAS velocity measurements of approximately half that. If fewer pixels are used for the grating – for instance just the central 128x128 pixels – then the lateral velocity resolution can be improved, without the need to change the imaging optics. Mean power onto the sample when all the SLM area is used is 75mW; the energy in each of the 200ps-long pulses in each 82MHz tone burst is of the order of 2.5µJ, giving a peak optical power of approximately 12.5kW. When fewer pixels are used, the power is reduced accordingly; for example when 128x128 pixels are used, SAWs are excited using less than 15mW of optical power.

The SAWs are detected at a point just outside the footprint of the grating. This is done by a continuous wave frequency-doubled Nd:YAG laser, with a mean optical power of 50mW focused onto the sample. Optical beam deflection ("knife edge") is used to detect the SAWs; although this technique requires a reasonable surface finish (it is not a "rough surface" technique), it should be noted that this is not in any way a limitation of the SRAS technique in general, rather the O-SAM instrument itself. The high frequency detected signal – a tone burst of 82MHz or one of its harmonics – is mixed down in quadrature with a coherent reference, and the baseband signals are acquired using a very modest digital acquisition card into a PC. As there is no digital processing involved up to this point, the amplitude and phase of high frequency SAWs (for example at 328MHz) can be acquired at rates which are limited only by the Q-switch rate (in this case, 1kHz). The same PC controls the mechanical stages used to raster-scan the sample with respect to the grating and the detection point (the detection point can also be moved if desired), and the PC also controls the images on the SLM. The control and acquisition software runs under Linux® and is open source.

It is possible to perform in excess of 100 velocity measurements per second, even taking into account the fact that several amplitude measurements are required for each acoustic spectrum, although 10-20 measurements per second is a more realistic rate for good quality data. It is also important to note that the SRAS technique is very tolerant to acoustic aberrations and scattering, which can have a profoundly deleterious effect on other techniques used to measure SAW velocity, such as time of flight or relative phase measurement. Acoustic aberration affects the propagation of SAWs from excitation region to detection point when propagating through random microstructure [5], and this can completely swamp conventional measurements. With SRAS, the overall detected amplitude (and phase) may vary significantly as the sample is scanned from point to point, as the waves propagate through different microstructure; but crucially, the propagation between the excitation region and the detection point is not affected by a change in fringe spacing. It is true that if the grating is on a region consisting of more than one grain (of more than one velocity) then as the fringe spacing is adjusted the relative efficiency within each grain will change. This does affect the properties of the propagating SAWs, but is a second order effect, and generally manifests itself as indicating the limit of spatial resolution for a given grating size.

4. Results

The images in figure 2 are all of the same sample, Ti-685. This large-grained sample has cracked, this is due to a load controlled low cycle fatigue fracture, and the failure process is described in detail in [6]. Figure 2a shows the optical image of the etched sample, figure 2b shows the EBSD orientation image (for which the sample had to be polished to a very high standard). The next two images are SRAS velocity maps, where the 82MHz SAWs are propagated in orthogonal directions. By combining velocity information in more than one direction (figure 2e), some degree of orientation information can be obtained: for instance blue areas in the velocity vector image indicate regions where the velocity is "fast" in both directions. This in turn indicates that the basal plane is parallel to the surface. The images are striking in their similarity.

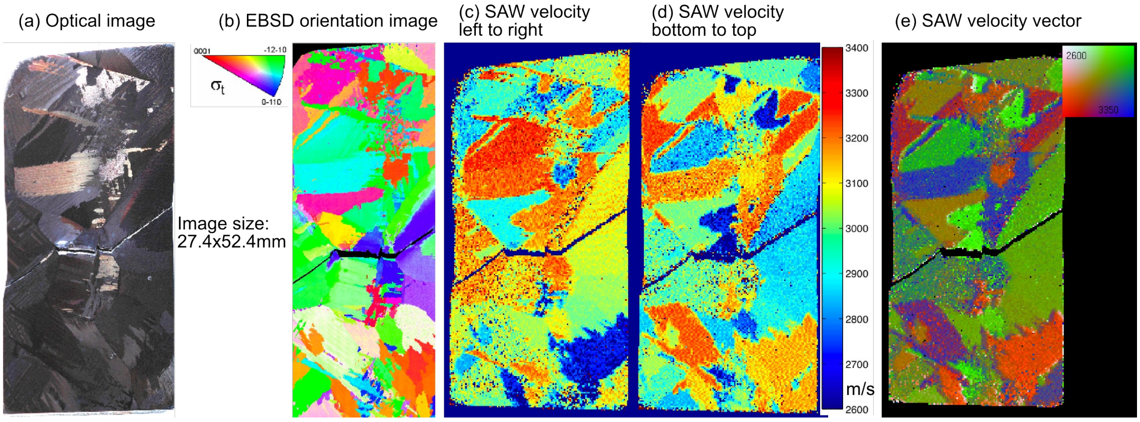

Figure 2. Images of a Ti-685 sample, 27.4x52.4mm in size. (a) optical image, after chemical etching; (b) EBSD orientation image; (c) SRAS velocity map, waves propagating left-right; (d) SRAS velocity map, waves propagating bottom-top; (e) SRAS velocity vector map.

Figure 3 illustrates the effect of changing the excitation grating size, from 384µm on the left, down to 48µm on the right. Here, lateral resolutions of approximately 25µm have been achieved. 328MHz SAWs were used to acquire these images. By increasing the SAW frequency, the excitation grating can be made smaller for the same number of fringes. The number of fringes used determines the velocity resolution, as the grating period must match the SAW wavelength more accurately for constructive interference to take place when there are more fringes. This reduces the width of the curve in the spectrum, which means the location of the peak can be more accurately determined. In [4], a velocity resolution corresponding to better than 0.03% (<1m/s) was experimentally achieved for a grating consisting of 44 fringes.

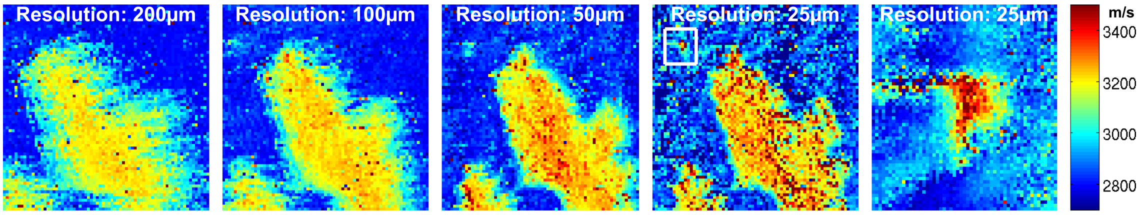

Figure 3. SRAS velocity maps of a Ti-685 sample, acquired using an excitation frequency of 328MHz. The first four images are 700x700µm, each pixel is 10µm square. They differ by the number of pixels used in the SLM image of the grating, which in turn determines the lateral resolution. The image on the right is 108x120µm, and is a subset of the fourth image, acquired using a smaller step size (2µm).

Figure 4 illustrates how SRAS can be used to image large samples, generally beyond the means of other techniques used to image grain structures. The Ti-6-4 sample shown is 153x106mm in size, and the pixel size is 250µm square. The SAW frequency used was 164MHz.

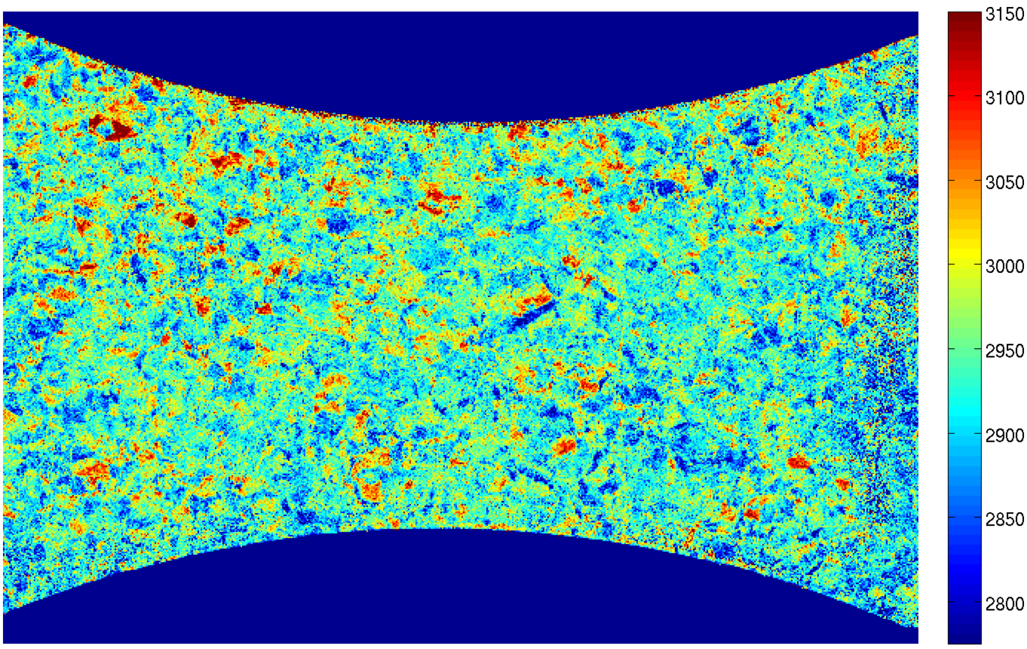

Figure 4. SRAS velocity map of a Ti-6-4 sample, using an excitation frequency of 164MHz. The area of the scan is 154x106mm, each pixel is 250µm square. The lateral resolution is approximately 100µm square. The image took around 6 hours to acquire, corresponding to a scan rate of approximately 12 points per second.

5. Conclusions

Spatially resolved acoustic spectroscopy is a powerful and robust laser ultrasound technique that uses local SAW phase velocity as its contrast mechanism. The practical lateral resolution achievable on the O-SAM instrument is currently of the order of 25µm; at the other end of the scale, the sample size is limited by how far the mechanical stages can travel. Although only titanium alloys have been shown in this paper, the technique can be (and has been) used on a wide range of materials, such as aluminium, silicon nitride and steel. Acquiring the SAW velocity in multiple directions can be used to ascertain information about crystallographic orientation of the microstructure, although further work needs to be done in this area. The absolute SAW velocity can also be used to acquire information other than the microstructure, for instance it can be used to measure coating thickness of thin films or residual surface stress.

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