Orientation imaging using spatially resolved acoustic spectroscopy

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Abstract. The term ‘orientation imaging microscopy’ describes generic techniques for imaging the orientation of crystalline structures of heterogeneous media. Typically, scanning electron microscope techniques are used, such as electron backscatter diffraction or transmission electron microscopy. We have developed an acoustic technique that can perform equivalent measurements. The key to enabling a practical acoustic method is spatially resolved acoustic microscopy (SRAS), a robust laser ultrasonic technique for quantitatively determining the surface acoustic wave velocity at a high spatial resolution. Here we present quantitative determination of crystallographic orientation of large nickel grains by comparing the measured SAW velocity in multiple propagation directions, with a data base of calculated velocity surfaces for all orientations. As well as discussing the method of determining the crystallographic orientation details are also presented of the recent advances in the capabilities of the latest generation of instrumentation, including significant increase in the data acquisitions rate and the reduction in size and complexity of the SRAS instrument itself.

1. Introduction - Grain orientation and SRAS
Measuring the crystallographic orientation of grains of different materials is generally performed with electron microscope techniques, such as transmission electron microscopy [1] and electron backscatter diffraction microscopy [2]. These techniques provide the crystallographic orientation with exceptional spatial resolution across the sample of interest. The main drawbacks to these electron based techniques are the requirements on samples size and preparation. The samples have to be small in size to fit inside the electron microscope so typical samples sizes are of the order of 10x10mm. The electron microscopy techniques are very sensitive to the surface finish and so special care must be given to ensuring the finish of the sample is sufficient, this typically requires polishing the sample to a mirror like finish. We have developed an acoustic technique that can perform equivalent measurements, on samples of any size and varying surface finish, although with a lower spatial resolution than that obtained by electron techniques (10’s microns).

Although the SRAS technique has been described previously [3, 4], it is worth repeating the salient details here as the new instrument uses the fixed grating method. The SRAS technique itself will be described, and a description of the new instrumentation will follow in the next section. We will then describe the process from going from velocity contrast maps to true orientation, before finally providing examples of experimental measurements on single crystal nickel.

2. Spatially Resolved Acoustic Spectroscopy for velocity imaging
The spatially resolved acoustic spectroscopy technique is analogous to optical spectroscopy techniques where analysis of the optical spectrum and the wavelengths that are most readily transmitted or absorbed tells us some properties of the sample being examined. In the acoustic sense we monitor the spectrum of sound waves emitted from a grating source. The technique can be performed two ways, the first is to use a fixed frequency source and sweep the grating spacing or the grating period can be fixed and the frequency is swept. The previous versions of the SRAS instrument used the first...
approach, the new instrument introduced here uses the second approach and this will be used to explain how the technique works.

![Figure 1. Cartoon of a SRAS system.](image)

To measure the acoustic spectrum at each point on the sample a fixed grating is illuminated with a short laser pulse (typically ~1ns). As the laser pulse contains a broad range of frequencies, only the frequencies which match the grating spacing and acoustic velocity of that sample point will be generated. We measure the waves that have been generated close to the excitation region (see figure 1). We can then calculate the frequencies generated and with the knowledge of the grating spacing we can obtain the acoustic velocity using $v = f \lambda$. The advantage this approach has over the fixed frequency laser approach is that all the information we require to calculate the velocity is obtained in a single measurement, whereas previously the grating spacing needed to be changed several times per measurement point leading to a lower data acquisition rate. To build up a velocity map the sample is raster scanned and the measurement is repeated for each point.

Using this approach the data rate is greatly increased, single shot measurements on well prepared samples can be carried out at 6000 points/second. The overall data rate obtained including data read out / download and flyback of the stages is around 1200 points / second. An example image of TiLG685 obtained with the new system is shown in figure 2. This 3.2 megapixel image was obtained in ~50 minutes. The new instrument is also considerably smaller than previous instruments. The main optics of the system fit in a head of around 350x200x150 mm with an umbilical to the electronics rack. When fully complete this new instrument will also have the ability to detect waves on optically rough surfaces and propagate the sound in any chosen direction without the need to rotate the sample itself.
Figure 2. 3.2 Mega pixel velocity map of TiLG685 sample, waves propagating up/down direction.
3. From contrast to orientation measurements

To provide orientation measurements we need to convert from velocity contrast maps to true orientation information. This process will now be described before presenting experimental results on large nickel grains.

The acoustic velocity of a known material in a known orientation can be calculated analytically. We use the method outlined by Farnell [5]. This method is used to calculate SAW and pseudo-SAW velocities on different crystallographic planes by using iterative search procedures and the known materials' elastic constants. The method requires the iteration through different SAW velocities in a specified range to find out which velocities satisfy the boundary conditions. In the case of pseudo-SAW a similar method is used, with the additional assumption that the wave is attenuated along the propagation direction. Using this method we can calculate the velocity surface for any crystallographic orientation and a selection of surfaces are shown in figure 3.

We plot the residuals of the boundary conditions and the minimas of this curve suggest the possible propagation of different wave modes e.g. surface waves, pseudo surface waves and leaky modes. The detector is sensitive only to the out of plane motion of the waves, so we calculate the out of plane motion for all of the modes found during the search and choose the dominant mode. This then gives us an indication of the expected velocity surface for that crystallographic orientation that will be measured with our experiment.

![Figure 3. velocity surface calculations for different orientations.](image)

The Inverse problem is difficult to solve, so to provide orientation measurements we use the measured velocity surface and fit it to the model data by searching a database of all known orientation angles to get the orientation of the measured point. As mentioned above the velocity surface maps we have calculated have been modified to take into account the workings of the detector so we used these adjusted maps for the fitting process. The fitting is achieved using cross correlation; we record the strength of the correlation of the experimental data with each plane and rotation in the calculated
velocity values database. Once all planes and rotation angles have been compared we look for the largest correlation value as this corresponds to the best fit orientation.

This process is shown in figure 4 below. It shows the correlation strength for different planes and rotational angles and we can see various peaks in the image showing partial matches. The figure 5 shows the modelled velocities for the chosen plane superimposed on the experimental data. In this case we have only overlaid the modelled values for every 5 degrees of rotation for clarity. We see the fit is extremely good and we can be confident that the crystallographic plane has been recovered by the fitting algorithm.

![Figure 4](image_url)  
**Figure 4.** The correlation factors obtained during the fitting process for different rotations and plane orientations.

![Figure 5](image_url)  
**Figure 5.** Unknown plane overlaid with best fit chosen velocity values.
4. Experimental Results – Single crystal nickel.
A velocity map was produced for a nominally single crystal nickel sample. This map (see figure 6 below) shows two regions of differing velocity showing that this sample is not a single crystal but is made of two grains with differing orientations. A velocity surface was then taken for each grain by measuring the surface wave velocity around 360 degrees of rotation with 5 degree increments.

![Velocity map of single crystal nickel sample.](image)

**Figure 6.** Velocity map of single crystal nickel sample.

The velocity surface is shown in figure 7 for the two regions. The white stars are the modelled velocity values for the output orientation obtained via the fitting process. We see excellent agreement between the experimental velocity surface and the chosen best fit plane. The chosen plane and angle are shown in the inserts of figure 7. Figure 8 shows the velocity map with a cube representing the orientation of the grain for comparison. The height of the sides of the cube show the slight change in the plane orientation and the top face shows the large rotational change.

![Velocity surface for region A and B with modelled grain orientation superimposed.](image)

**Figure 7.** Velocity surface for region A and B with modelled grain orientation superimposed.
5. Conclusions
We have presented the determination of grain orientation using spatially resolved acoustic spectroscopy. The SRAS instrumentation has been improved to allow faster data acquisition rates of 1200 points/second and a much smaller total footprint making the system easily transportable. Obtaining the grain orientation is achieved by fitting the experimentally recorded velocity surface to a database of calculated velocity surfaces. The database is modified to take into account the response of the detector to different wave modes.

To date we can do this for any cubic material where the elastic constants are known and we are in the process of expanding this to other crystal types and optimising the fitting algorithm without comprising its robustness.

6. References.
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Figure 8. Velocity map with the grain orientation represented by a cube