Crystallographic Orientation Determination of Hexagonal Structure Crystals by Laser Ultrasonic Technique

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Abstract. Spatially resolved acoustic spectroscopy (SRAS) is a laser ultrasonic technique that shows qualitative contrast between grains of different orientation, illustrating the sensitivity of acoustic waves to the material structure. The technique has been improved significantly on determining the full orientation of multigrain cubic metals, by comparing the measured surface acoustic wave (SAW) velocity to a pre-calculated model.

In this paper we demonstrate the ability of this technique to determine the orientation of hexagonal structure crystals, such as magnesium and titanium based alloys. Because of the isotropy of the SAW velocity on the basal plane (0001) of hexagonal crystals, the slowness surface is shown as a circle. As the plane moves from (0001) towards (11̅20) or towards (10̅10), the slowness surface gradually turns into an oval. These acoustic properties increase the difficulty in orientation determination.

The orientation results of a grade 1 commercially pure titanium by SRAS is presented, with comparison with electron backscattered diffraction (EBSD) results. Due to the nature of SAWs on hexagonal structure crystals, only the results of Euler angles 1 and 2 are discussed. The error between SRAS and EBSD is also investigated.

1. Introduction
Spatially resolved acoustic spectroscopy (SRAS) is a laser ultrasonic technique for crystallographic orientation determination. It non-destructively measures the surface acoustic wave (SAW) velocity on the material’s surface. The microstructure is not only revealed by the velocity contrast, but also the orientation results can be illustrated by the velocities in multiple directions. Although it is complicated to solve the SAW velocity at an arbitrary direction analytically, numerical solutions [1–3], based on the materials elastic constants and the mass density can be determined. We have modified the approach described by Farnell [1] in order to perform the current calculations. The orientation of measured grains can be determined by a brute-force search on this pre-calculated SAWs velocity model as described previously [4].

SRAS has been used for examining cubic crystal structure materials such as nickel-based alloys, aluminium and stainless steel; the SRAS orientation results have been compared to the electron backscattered diffraction (EBSD) technique [5]. The present paper extends previous
work to show for the first time how it may be used to obtain quantitative crystallographic orientation for hexagonal structure materials.

In terms of SAWs, the difficulty for hexagonal crystals is that the basal plane (0001) is isotropic; hence, the slowness surface of primary planes (11\(\bar{2}0\)) and (10\(\bar{1}0\)) are identical. The slowness surface of all planes has both inversion and reflection symmetry. In other words, the hexagonal prism behaves like a cylinder acoustically. Because of this character, on certain planes, where the Euler angle 2 of both planes are the same and both plane normals are in the same plane with axis \(z\) as shown in Figure 1, Euler angle 1 can be narrowed down to either 0\(^{\circ}\) or 180\(^{\circ}\) by SRAS. Although only Euler angles 1 and 2 can be determined by SRAS technique, it gives sufficient information for certain applications, for which knowledge of the \(c\)-axis direction is of interest.

![Figure 1: SRAS ambiguity: Euler 1 could be narrowed down to 0\(^{\circ}\) or 180\(^{\circ}\) when axes \(z\), \(z'\) and \(z''\) are in the same plane and at the meantime \(\angle z''oz = \angle zoz'\).](image)

2. SAW Model of Hexagonal Crystals

The SAW velocity model is calculated based on the material’s elastic constants and mass density [4]. An iterative search is performed until the assumed velocity value satisfies the wave equation and the boundary condition of the SAW simultaneously at the known plane and direction. Repeating the calculation on all planes and directions, a numerical model is generated.

Typical planes of titanium \(\alpha\) model is shown in Figure 2. The slowness surface is a perfect circle on the basal plane (0001); when the plane gradually moves towards plane (11\(\bar{2}0\)) (or plane (10\(\bar{1}0\)) as they are acoustically identical), it elongates to an ellipse shape. On some planes, only one wave mode would be detected. Two wave modes could be seen on the planes which are in between the plane (0001) and (11\(\bar{2}0\)) (or plane (10\(\bar{1}0\))); it is confirmed by the experimental data in later sections.

3. SRAS Instrument

The SRAS technique can be used to obtain SAW velocity information on the local area where the waves are generated. A SAW excitation pattern is generated by projecting a grating pattern of laser light on sample surface. We vary the frequency by using a broadband Nd:YAG laser in combination with a fixed grating [8], allowing the local velocity \(v\) can be calculated, through \(v = f\lambda\) where \(f\) is the frequency and \(\lambda\) is the grating period. By repeating the scan while changing the fringe pattern’s direction, a velocity surface spectrum (which can be seen as a 3D slowness surface) of each point on the sample surface is obtained. The current system is capable of scanning at 1200 points/second; the spatial resolution is 100\(\mu\)m, this can be pushed to 80\(\mu\)m for smaller grain size samples. Its operation does not require a vacuum, hence the tested sample size is only limited by the scanning stages’ range.
4. Orientation Comparison of EBSD and SRAS

A brute-force search, termed the overlap function, was introduced [4] to match the velocity surface spectrum of each point to the pre-calculated SAW model, from which the orientation results are obtained. The inverse pole figures (IPF) on cubic materials were verified by comparison with EBSD technique [5]. This section shows the orientation results of a commercial purity (CP) grade 1 titanium sample.

4.1. Results by the Overlap Function

Figure 3 shown the crystallographic orientation of a CP titanium sample by both EBSD and SRAS. Figure 3a is the IPF measured by EBSD; it is the most common method of presenting materials’ texture. A IPF shows the combination of Euler angles 2 and 3, however, due to the limitation of SAWs sensitivity, only Euler angles 1 and 2 can been achieved by SRAS. In order to compare the two techniques visually, a new interpretation for illustrating crystallographic orientation of hexagonal structure materials is introduced, which is as seen in Figure 3b. This method shows the combination of Euler angles 1 and 2, while using three dimensional space of the combination of c-axis pointing direction projected to a colour map. Therefore Figure 3b and Figure 3c are illustrating the same information deduced by EBSD and SRAS respectively. The figure demonstrates the two techniques have a high level of agreement on grains orientation; the few mistakes which occur have been identified for further discussion in the next section.

4.2. Error Analysis

In order to have a better understanding why the overlap function occasionally picked the orientation which is slightly different from EBSD results, experimental data of the grains are shown in Figure 4b and Figure 4d. In addition, the velocity surface spectra are simulated according to the given orientation of EBSD and illustrated in Figure 4a and Figure 4c; they are what SRAS would measure if the orientation is accurate as stated by EBSD results.
Figure 3: Titanium orientation results by EBSD (left and middle) and SRAS (right).

| Grains | Euler angle 1 (°) | Euler angle 2 (°) |
|--------|------------------|------------------|
|        | EBSD | SRAS | EBSD | SRAS |
| A      | 80.19 | 79.90 | 49.63 | 63.96 |
| B      | 2.71  | 2.01  | 76.76 | 87.91 |
| C      | 49.01 | 48.02 | 68.67 | 67.98 |
|        | 50.00 | 85.99 |        |        |

Table 1: Euler angles 1 and 2 of the three grains measured by EBSD and SRAS.

Figure 4: Simulated (from EBSD data) and experimental (measured by SRAS) velocity surface spectra of grains A and B on the CP titanium sample (see Figure 3).

It can be seen in Figure 4, SRAS measured data on some grains are different from what EBSD results show. On grain A, two wave modes should be observed according to EBSD where only one wave mode is measured. On the contrary, two wave modes were detected by SRAS on grain B rather than one, which is indicated by EBSD. Therefore, SRAS and EBSD have minor disagreement on orientation of those grains.

Figure 5 shown the $c$-axis maps zoom in on grain C by EBSD and SRAS respectively. Figure 5a is the EBSD results which contain Euler angles 1 and 2 shown uniform colour on
Figure 5: The EBSD and SRAS c-axis maps zoom in on grain C. The EBSD results pointed out the alpha-phase lath boundaries on Euler angle 3, hence in this figure an uniform colour is shown on this grain while it appeared on the SRAS results.

grain C; the primary alpha laths on this grain can only be seen in Euler angle 3, which should not be observed by SRAS. However, it appeared on the SRAS c-axis map in Figure 5b. In Table 1, two sets of data for SRAS as it gives two possible orientations. Euler angle 1 of the two techniques are very close with error less than 1°; one of the SRAS Euler angle 2 is very close to EBSD when the other one is approximately 17° away.

Figure 6a shown the simulated velocity surface spectra from the EBSD data and the measured velocity surface spectra by SRAS (the middle two), of two different areas in yellow and green pixels on SRAS c-axis map grain C. The latter are not very easy to distinguish until plotted as slowness surfaces in Figure 6c. The colour of the slowness surfaces match the area on the grain. They are very close in value however one of them has slightly faster velocity in the fastest direction, hence SRAS decides grain C has two different orientations. The disagreement in the results was due to deformation twins being present in the sample, which would mean that there was a second orientation in the grain. The deformation caused different effect on the twinning planes; accordingly, evidence of the third Euler angle is presented. In conclusion, although SRAS has disagreement with EBSD on the orientations of a few grains, it does truly display the elastic properties of the grain’s stress/strain conditions.

5. Conclusion
SRAS is a non-destructive evaluation technique to measure the SAW velocity by laser ultrasound. It has proved to be a successful tool for measuring crystallographic orientation quantitatively for cubic structure materials. In this paper CP grade 1 titanium is studied, to show the technique can be extended to the hexagonal structure materials. The calculated SAW velocity model of titanium has shown that its slowness surface is isotropic on the basal plane (0001); when it gradually rotates to plane (1120) or (1010), it elongates into an ellipse shape. The overlap function has been performed to examine the samples’ microstructure and orientation of the grains. Although SRAS can only presents two of the Euler angles out of three – physics limits the information that elastic waves can extract of the orientation – the comparison between the c-axis maps by EBSD and SRAS confirm it is a robust technique not only for cubic crystals but also hexagonal ones. The mismatches of the two techniques are discussed in the last section. It is believed that the presence of deformation twinning caused the mismatch by SRAS, which indicates this technique is truly representing the elastic properties.
Figure 6: The left three figures are the theoretical (simulated from EBSD data) and experimental (measured by SRAS) velocity surface spectra of grains C. The right figure shows the slowness surface of the yellow and green area on grain C by SRAS; the colour of the curves correspond to the pixels’ colour.

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