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Assessing the precision of strain measurements using electron backscatter diffraction – Part 2: Experimental demonstration

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ABSTRACT

The residual impression after performing a microhardness indent in silicon has been mapped with high resolution EBSD to reveal residual elastic strain and lattice rotation fields. Mapping of the same area has been performed with variable pattern binning and exposure times to reveal the qualitative and quantitative differences resulting from reducing the pattern size and exposure time. Two dimension 'image' plots of these fields indicate that qualitative assessment of the shape and size of the fields can be performed with as much as 4×4 binning. However, quantitative assessment using line scans reveals that the smoothest profile can be obtained using minimal pattern binning and long exposure times. To compare and contrast with these experimental maps, finite element analysis has been performed using a continuum damage-plasticity material law which has been independently calibrated to Si [9]. The constitutive law incorporates isotropic hardening in compression, and isotropic hardening and damage in tension. To accurately capture the localised damage which develops during indentation via the nucleation and propagation of cracks around the indentation site cohesive elements were assigned along the interfaces between the planes which experience the maximum traction. The residual strain state around the indenter and the size of the cracks agree very well with the experimentally measured value.

1. Introduction

Residual stresses and defect populations in engineering materials dominate performance and failure. Quantitative assessment of these fields on a local scale is driving forward the development and understanding of new materials and helping to close the structure-properties loop. High resolution electron backscatter diffraction (HR-EBSD) is one such tool employed to measure elastic strains, stresses, lattice rotations and stored dislocation content with a spatial resolution of $\sim$20 nm.

The strain resolution of the technique is enhanced in comparison to conventional (Hough-based) EBSD as a reference and test pattern are directly compared using image correlation to measure the change in strain state and misorientation. This technique is based upon the work of Troost et al. [1] and Wilkinson et al. [2], and the modern implementations are based upon the work of Wilkinson et al. [3,4]. Recent developments have focussed upon extending this route to deal with significant lattice rotations, which are commonly found in metals [5–7]. Fundamental to the technique is precise measurement of pattern shifts between sub-regions of test and reference patterns, using image correlation. These pattern shifts are then linked through simple geometry to components of the displacement gradient tensor. Precise recovery of the pattern shifts requires diffraction patterns with a high signal to noise ratio. Therefore the accuracy of the high resolution technique is linked to the quality of the diffraction patterns, which is determined significantly by the physical detector used, exposure time and pattern binning employed.

Part 1 of this study [8] has indicated that strain accuracy is limited by pattern size, where less binning results in pixels that subtend a smaller angle of the diffraction pattern and therefore smaller changes in strain state can be measured. Equally, the ultimate accuracy of the technique is limited by the signal to noise within the diffraction pattern, which can be adjusted by changing the exposure time or probe current.

In Part 2 of this study, the effect of different pattern capture settings is demonstrated by exploring the strain field around a micro-hardness indent in silicon. To emphasise the utility of the technique to recover data, rather than simple micrographs, these strain fields have been used to validate a continuum finite element model including continuum damage-plasticity and localised damage through cohesive zones. This is required to model the behaviour of silicon during an indentation test which produces a non-trivial loading history and stress state involving compression and tension, plasticity and damage. In order to independently confirm that the

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Table 1
Comparison of binning, image size, intensity ranges and electron budgets (2 s.f.) for silicon diffraction patterns captured with hardware binning.

Hardware binning	1×1	2×2	4×4	8×8
Phosphor diameter (pixels)	904	452	226	113
Acquisition time (s)	1.04	0.24	0.05	0.02
Maximum intensity (arbitrary units)	3550	3450	3400	3550
Minimum intensity (arbitrary units)	1750	1800	1800	1750
Approximate electron budget (electrons per super-pixel)	3900	3600	3000	4700

measured strain fields are sensible, we did not perform any calibration of the model parameters, instead we used values published in [9]. Cohesive elements were assigned along the principal planes to accurately capture the crack nucleation and propagation there but the same damage law as for the bulk was used.

Characterising deformation around indents with HR-EBSD and validation using other techniques has previously been reported in both silicon and titanium. In silicon, validation of HR-EBSD results has been performed with both Raman spectroscopy [10] and atomic force microscopy (AFM) [11]. In these two studies, Vaudin et al. focussed on a wedge indent and studied the strain fields perpendicular to the length of the wedge. This provided a relatively simple strain profile which could be directly compared between techniques, using peak shifts with Raman to infer the presence of elastic stress, and integrating the displacement gradients measured with HR-EBSD to compare directly with the surface profiles measured with AFM. In titanium, deformation is significantly more plastic and therefore the modelling is necessarily more involved. Britton et al. [12] used a crystal-plasticity finite element model and demonstrated good agreement for indents made into different crystals of different orientations.

Similarly, simulation of deformation fields around indents in silicon has also been performed. Wan et al. [9] used a continuum damage model to compare experimental load displacement curves and crack lengths of Vickers, cube corner and Berkovich indentations made with a nanoindenter. Their focus was on matching the load-displacement curves and subsequently evaluating crack types and sizes between experiment and model. Calibration of their model was performed by fitting the compressive yield stress and compressive hardening ratio such that the experimental and modelling load-displacement curves matched well. The tensile damage behaviour in their model was extracted from prior work based upon three point bend specimens [13]. Following from this calibration, they reasonably reproduced different cracking behaviours using their model, yet the crystallography of the cracking geometry was not included due to the reduced symmetry imposed to decrease computational times.

Modelling the precise nature of cracking using finite elements is difficult. While damage based models can account broadly for the features seen, they can fail to capture the local nature of the strain fields. In cases where the plane of the cracking is known then cohesive elements can be introduced which allow localised damage to be captured accurately, as they explicitly include fracture mechanics components to predict crack propagation [14].

2. Experimental method

To demonstrate the value of understanding the resolution limits of the technique a residual impression from a 50 gf Vickers microhardness indent in Si was mapped by EBSD.

A semiconductor grade silicon single crystal wafer sample was examined in a JEOL JSM-6500F at an accelerating voltage of 20 kV, and a nominal probe current of $\sim$10 nA. The sample was tilted to 70° and it was aligned such that the sample x-axis and the phosphor x-axis were coincident along the tilt axis.

Before insertion into the chamber, the sample was dipped in 40% HF and cleaned with acetone to reduce contamination for the duration of the experiment. EBSD patterns were captured using a TSL-OIM DC v5.3 and a Digiview II camera which uses both a variable aperture f0.95 lens (opened fully) and a 1024×1280 pixel chip. Each pattern was cropped and centred on the circular phosphor screen. Flat fielding was performed using background subtraction with a background obtained using a mechanically ground aluminium stub at a different sample height, set to overlap the differing backscatter emission centres for Si and Al.

Patterns were captured with different hardware binning and therefore acquisition times, given in Table 1. Approximate electron budgets, B, which indicates the number of electrons hitting the phosphor per (binned) pixel in the image were estimated using Eq. 1) and summarised in Table 1.

$$B = \frac{4It\zeta h}{e\pi D^2} \tag{1}$$

Where I is the nominal probe current according to the factory specification (10 nA); t is the exposure time in seconds; ζ is the backscatter coefficient for silicon ($\sim$0.5 at 70° tilt); h is the area fraction of the diffraction sphere covered by the detector, approximating it to a spherical cap ($\sim$0.08) and D is the diameter of the phosphor in pixels, which varies with the binning used.

The single crystal was oriented such that the [001] direction was aligned out of the plane and the two perpendicular $<110>$ fracture directions aligned along the X and Y axes. The same indent impression was mapped with the different levels of hardware binning, and hence exposure times, listed in Table 1. The four maps: 1×1, 2×2, 4×4, and 8×8 binning; took $\sim$2 h, $\sim$15 min, $\sim$3 min and $\sim$2 min respectively. As the lattice rotations are relatively small (typically $< 2°$/0.035 rad), analysis was performed without use of image remapping [6]. Data was filtered to remove points with a mean peak height [5] less than 0.3 and a mean angular error [5] greater than 1×10^{-3}. A point far from the indent in the top left of the map was selected as the zero strain reference point.

3. Experimental results

Maps showing the residual deformation around the indent after unloading are shown in Fig. 1. These include the full lattice rotation tensor and elastic strain tensor. Qualitatively there is relatively little difference between the maps for 1×1 and 2×2 binning, whereas maps for 4×4 and 8×8 binning show similar spatial features and senses for each component of the strain and lattice rotation tensors but with visibly increased noise, as expected.

In addition to these maps, line scans were performed just ahead of the crack tip, $\sim$15 μm above the centre of the microhardness indent. The direction of the scan was perpendicular to the crack length (i.e. along the X direction). For each binning level, five line scans were taken in quick succession as before [8] and analysis of the line scans using both one and five averaged patterns per point are presented in Fig. 2. Strain free reference points for these scans were the first 'image' for each line.