High angular resolution electron backscatter diffraction: measurement of strain in functional and structural materials

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AN OVERVIEW OF STRAIN MEASUREMENT

Human history has been forged by progress in the extraction and manufacture of materials, particularly crystalline metals, ceramics and semiconductors. Technological innovations require their continuous adaptation to accommodate new engineering designs and operation in new and restrictive environments. This can only be delivered through deep understanding of microstructure and properties. An engineer can use this knowledge as a limit on design and tolerances and a materials scientist can use it to improve the material properties through innovative materials processing and microstructural design. The cornerstone of this process is the characterization of microstructure.

One of the newest tools in the box is high angular resolution electron backscatter diffraction (HR-EBSD) which can map elastic strain, stress and evidence of plastic strain. This development realizes the full potential of the scanning electron microscope and enables us to close the loop on microstructure property relationships.

Conventional electron backscatter diffraction (EBSD) is a commonly available tool in which a focused electron probe is scanned across a crystalline material and at each point an electron diffraction pattern is generated, recorded and interrogated.

HR-EBSD augments this by enabling very small changes in the geometry of the crystal to be measured through interrogation of the diffraction pattern. The sensitivity is sufficient to determine residual elastic strains within the material and local crystal misorientation at levels of \(10^{-4}\) and \(10^{-4}\) radians (0.006°) respectively [1, 2]. At these sensitivities it is possible to track the elastic stresses and strains, for example ahead of deformation bands [3].

HR-EBSD does not require new apparatus or experimental procedure, using patterns captured from conventional EBSD [4]. It competes with other strain measurement techniques available both in the laboratory and at larger diffraction facilities, such as at ESRF and the Diamond Light Source. In the field of diffraction, X-rays are commonly used for strain measurement but typically this method has limited spatial resolution (~few μm) and may not resolve the full strain tensor. At the large synchrotron facilities the X-ray beam can be collimated to give a spatial resolution of 1 μm and a strain resolution comparable to HR-EBSD [5]. For improved spatial resolution (~1 nm) beyond that achievable in the SEM, samples can be examined in the TEM but samples must be electron transparent and therefore routine analysis can be difficult. Strain is most commonly measured using convergent beam methods through which complete strain tensor data can be extracted.

Many non-diffraction-based strain measurement techniques function by determining a change of another material property such as the observations of fluorescence peaks in Raman spectroscopy [6, 7]. More indirect still are techniques which measure strain through surface displacements and the most recent variations of this involve patterning the surface of the sample with a contrast medium and following the surface movements with high-resolution optical microscopy or scanning electron microscopy [8, 9]. Computer-assisted digital image correlation (DIC) is carried out between two or more micrographs captured before and during a test. DIC delivers sub-pixel shift resolution providing a combined measure local plastic and elastic strain at a sensitivity typically ~10^{-3} and at a spatial resolution dependent upon the patterning method.

THE HIGH RESOLUTION ELECTRON BACKSCATTER DIFFRACTION METHOD

EBSD involves the formation of a diffraction pattern in a scanning electron microscope. A schematic of this process is shown in Figure 1A. Briefly a small focused probe of (~20 kV) electrons is scanned over a highly tilted sample (~70°). At
each point, electrons enter the sample and some are backscattered towards the phosphor screen and contribute to the diffraction pattern. These patterns contain many Kikuchi bands, which consist of the intersection of a pair of diffracting cones from one diffracting lattice plane, separated by the Bragg angle, projected onto a flat phosphor screen.

The diffracted electrons which escape the surface are those excited within the top ~20 nm and from an area equal to the footprint of the electron beam as it enters the crystal. The lateral resolution depends on the SEM emitter, with a FEG emitter resolving ~5 nm parallel to the tilt axis and ~15 nm along the inclined plane. The patterns are imaged with a CCD camera from where they are transferred to a computer for image enhancement and analysis. The collection of a diffraction pattern is very fast (up to ~800 Hz) and so a map containing many millions of patterns can be routinely captured in a standard SEM session.

All EBSD systems facilitate this process by synchronizing control of the electron beam with the pattern capture process. The patterns are analysed automatically using a Hough procedure \([4]\) in real time to generate basic microstructural maps depicting crystal phase, orientation and misorientation as well as such derivatives as overall crystal orientation texture, average misorientation within each crystal, and grain size and grain shape.

High-resolution EBSD adds to this functionality by directly comparing the patterns. As the analysis is mathematically intense it is performed offline using saved patterns. Patterns should be captured at the full resolution of the CCD in order to obtain the highest strain sensitivity.

The most recent approaches to HR-EBSD are derived from the cross-correlation approach developed by Wilkinson et al. \([1, 2]\). A schematic of the process is shown in Figure 1B. A sub-window (266x266 pixels) is extracted at the same location from both test and reference diffraction patterns and cross-correlated. Cross-correlation measures the average shift required to achieve the best overlay of the windowed image from the test image with that from the reference image by searching for a peak in the cross-correlation function. Interpolation functions can be used to up-sample the cross-correlation peak and estimate the peak shift required with sub-pixel precision, which is vital. Elastic strain in most materials at yield or failure is limited to at most a few tenths percent and therefore interrogating elastic strain variations requires measurement of shifts of less than one pixel in 1000.

Shifts in features within the diffraction pattern can be related to distortions in the crystal through basic geometry as the diffraction pattern can be imagined as a simple gnomonic projection of all the diffracting lattice planes within the crystal. Rotation of the crystal results in a coherent motion of features across the diffraction pattern. Elastic strains that result in changes in the angle between lattice planes are observed as symmetric shifts of features across the diffraction pattern. Elastic strains and lattice rotations cause independent changes in the diffraction which can be separated with mathematics.

There are eight degrees of freedom (3 rotations and 5 strains) that contribute towards pattern distortions. Arbitrary deformation has a ninth that describes a volumetric strain, which changes the band width and is not readily measured with cross-correlation. As the diffraction pattern is formed in the top 20 nm of the sample, we can assume that the out-of-plane normal stress \(\sigma_{zz}\) is therefore equal to zero. Hooke’s law is used to define the change in zone axes positions must be measured in the horizontal and vertical directions at a minimum of four locations in the pattern. Wilkinson et al. initially noted that use of twenty regions of interest (ROIs) with some simple image filtering resulted in precise measurement of lattice rotations with a sensitivity of \(-1\times10^{-4}\) \([2]\). More recent advances include use of the redundancy of employing many more ROIs to generate a statistically robust solution for the strain state \([10]\) and pattern remapping to measure small elastic strains \((-1\times10^{-4})\) in the presence of significant lattice rotations \((0.02-0.2\) rads / 1 \(\times\) 10\(^{-4}\)) \([11, 12]\). Note that there have been many other advances by other groups in this area testing accuracy and calibration \([13-17]\).

Although this procedure may seem complex, once implemented it can be used repeatedly on many patterns in turn to generate high fidelity maps of elastic strain variation and lattice rotation.

Similar to most other strain measurement approaches, obtaining a reference strain state can be problematic \([16]\). In many instances an unstrained area of the crystal may be readily found, e.g. far field from an indent impression, or the strain gradients are of interest, e.g. studying dislocation patterning. In other cases, e.g. in plastically deformed material or with strain heterogeneity associated with a specific microstructural feature, it can cause significant ambiguity and overcoming the problem is very interesting in its own right. Nevertheless the approach described thus far has been successfully employed to study elastic strains in brittle materials (e.g. silicon) \([6, 7, 13, 18, 19]\), strains in lightly deformed metals (e.g. indents \([19-21]\)) and lattice rotation gradients in metals to ascertain a lower bound estimate of the stored dislocation content which is related to plastic strain \([20-22]\).

To demonstrate the potency of this technique, four examples are presented: (1) measurement of displacements associated with stepping the
SEM electron beam systematically across the surface of an unstrained sample; (2) measurement of the strain state ahead of a microcrack in germanium; (3) measurement of the strain state around a microhardness indent in silicon; and (4) evaluation of the residual plastic strain in rolled titanium.

Data used in examples 1-3 were collected using a JEOL JSM 6500F with a TSL Digiview II camera operating with a 1×1 binning (~1 k x 1 k pixels) at the University of Oxford. A probe current of ~10 nA was used with an accelerating voltage of 20 kV. Each pattern was captured in ~2 s. Data used in example 4 were collected using a Zeiss Auriga CrossBeam FIB-SEM, with a Gemini SEM column and a HKL Nordlys S camera operating with a 1×1 binning (cropped to ~1 k x 1 k pixels) at Imperial College London. High current mode was used with a large aperture and an accelerating voltage of 20 kV. Each pattern was captured in ~0.3 s.

All the strain maps shown here were produced using commercial computer software Crosscourt 3, a description of which can be found on the website www.blgvantage.com.

EXAMPLE 1: MEASURING THE PRECISION OF THE METHOD
The precision of the method relates to the precision with which pattern shifts can be measured. As the EBSD pattern is a simple projection onto the phosphor screen in the SEM, a 1 μm movement of the SEM electron beam across a sample surface will move the entire pattern 1 μm across the phosphor screen. Therefore precision can be measured by capturing a series of patterns as the beam was moved across the surface of an unstrained silicon crystal. Pattern displacements measured with cross-correlation are shown in Figure 2. A linear trend is observed, with a constant gradient related to the pixel size. The precision, measured as the standard deviation of 20 ROIs, is better than 0.05 pixels. This is better than 1 part in 10,000 for a one megapixel CCD.

EXAMPLE 2: DEMONSTRATING THE ACCURACY OF THE METHOD
Testing the accuracy of any method is more difficult than testing precision. We needed a sample in which the strain distribution was known at a spatial resolution equal to that possible with EBSD and for which the complete strain tensor information was available. Corroboration with other techniques is helpful, but not entirely satisfactory in this case, as no other experimental method is capable of providing comparable data. We turned therefore to testing the experimentally measured distortions against a sample where we could impose a known strain gradient. We used a germanium crystal and introduced a sharp lenticular crack by loading and unloading the surface with an indenter. The calculated strain field that remains locked into the crystal is a classic formulation.

Figure 3 shows the experimental data analysed by Angus Wilkinson and reported by Dingley et al. [25] plotted together with two sets of calculated values. The result shows the shifts of the [114] zone axis on the pattern from the germanium sample as a function of distance ahead of the crack tip. The precision values are calculated from 5 repeat measurements. As the exact position of the crack tip could not be observed experimentally, theoretical curves and experimental results were normalized at a point 2.3 μm from the crack tip. The experimental values fall within the theoretical curves and match within 2 parts in 10,000 over the full extent of the measurements.

EXAMPLE 3: STRAIN MEASUREMENTS CLOSE TO A MICROHARDNESS INDENTATION IN SILICON
The next example shows the potency of the technique using a simple indentation impression in silicon. A diamond microhardness indenter was pressed into its surface so that the diagonals of the indenter lay along <110> in the exposed (001) surface and unloaded. The impression measured 12 μm across the diagonal and microcracks extended a short distance from each apex, shown in the ‘Image Quality’ map in Figure 4A (derived from the Hough analysis of conventional EBSD).

A host of information can be derived from the HR-EBSD technique as shown in Figure 4. This includes data quality, and all components of the stress, elastic strain and lattice rotation tensors.

For large maps it is impossible to visually check the quality of each pattern so we have developed two simple metrics: peak height describes the quality of the cross correlation result, which is normalized to 1 for autocorrelation and decreases towards zero for poor correlation; and mean angular error describes the difference between the best fit solution, from using more than four ROIs, with the measurement of the pattern shift at each ROI. A low value of peak height, (typically <0.3), or a high value of mean angular error (greater than the measured strain or lattice rotation component), indicates an improper measurement and should be discarded. In this case, patterns taken from the indent crater, with much shadowing and very poor patterns, have been automatically filtered using a peak height filter (PH >0.3).

The elastic stresses (Figure 4B) are calculated using Hooke’s law from the elastic strains and our simple boundary condition (σzz = 0). The remaining two out-of-plane shear stresses (σxz and σyz) are found to be close to zero from the analysis, as expected. This leaves the three remaining in-plane stresses to consider.

Indentation into this material can broadly be considered as a point load into an infinite half space, and so as we expect, the σxz and σyz fields are rotated 90° with respect to each other. The shear stress field is also consistent with this approximation, as demonstrated when the Von Mises stress field is calculated. There are notable exceptions to this simple point force analogy,
especially around the crack tips along the (110) directions. Here elastic strain has been relaxed by the formation of a new free surface, i.e. the crack. The magnitude of these stresses is entirely reasonable, as the failure stress of silicon is ~7 GPa [24].

The elastic strains are similar in form to the stresses, as they are related directly through Hooke’s law. Note that out-of-plane strains are not necessarily zero as a stress-free boundary condition does not imply a strain-free boundary condition.

Lattice rotations reflect the rigid body rotation of the crystal lattice. In this example, the lattice rotations are caused by the upheaval of material to accommodate the free space generated by the formation of the indentation crater. Material is uplifted to form a hilllock around the crater and when this is represented as rotations about the X and Y axes there is clearly a neutral line running through the indenter center and along the rotation axis. Measurements of this form can be related directly to surface displacements, e.g. AFM surface traces [7]. The in-plane lattice rotations are difficult to obtain by other means and in this example they show the presence of the discontinuity introduced by the formation of cracks.

**EXAMPLE 4: EVIDENCE OF PLASTIC STRAIN IN POLYCRYSTALLINE MATERIALS**

Plastic strain is typically accommodated by the nucleation and propagation of dislocations. Dislocation motion and storage is a significant focus of materials science as we strive to understand local failure processes. In complex alloys, such as titanium, these processes may involve multiple phases and interfaces and will vary significantly through a component even when it is subjected macroscopically to a uniform deformation state. As EBSD measures the local lattice orientation, there is an opportunity to study the effect of stored dislocations.

Initial efforts focused on interrogating local deformation gradients within the electron interaction volume which cause pattern blurring [25]. However with the rise of automated methods, effort was refocused towards linking local misorientation gradients to plastic strain [26, 27]. We must be careful here; single slip in a uniaxial test will result in the gradual rotation of the crystal form, but there will be no local rotation gradients. Rotation gradients form when slip is impeded and dislocations pile up to form structure with net lattice curvature. This is the basis of work hardening and strength in engineering alloys. A direct link between applied plastic strain and lattice rotation gradients is tricky, but in some cases this can be addressed through the careful use of calibration samples [28]. Precise measurement of measurement lattice rotation gradients, a strength of HR-EBSD, can be utilized to understand local dislocation structure, e.g. likely failure points and dislocation-microstructure interactions. The key benefit is that the resolution of dislocation structure is related to the accuracy of the technique, and the two orders of magnitude improvement using HR-EBSD affords a resolution of dislocations in lightly deformed materials [14, 20].

Our example is a cold-rolled sample of Ti-6Al-2Sn-4Zr-6Mo reduced by ~17% in one pass. Maps from the EBSD analysis are shown in Figure 5. Conventional EBSD maps show local crystallography of this complex alloy. This titanium alloy undergoes a solid-state phase transformation during thermomechanical processing. At elevated temperatures, the BCC beta phase forms large grains, which on cooling transform to HCP alpha phase in the solid state. This preserves an orientation relationship between the two crystals, and therefore colonies of alpha grains form from the same prior-beta grain. In this sample, alpha first nucleates and coats the prior beta grain boundaries. Subsequently, alpha nucleates elsewhere within the grains and forms a series of similarly oriented alpha laths. Finally the remaining beta transforms around these laths. These maps show the intersection of two prior beta grains, indicated by the grain boundary alpha laths. This complex series of reactions and the orientation relationship creates a tangle.

**Figure 5**

Analysis of a cold rolled Ti-6246 sample, reduced by 17% in one pass with conventional and high-resolution EBSD. Crystal orientation maps reveal an area which contains the intersection of two prior beta grains, grain boundary alpha and numerous alpha lath colonies. Image quality maps reveal morphology and the presence of many slip bands. Conventional EBSD-based KAM measurements reveal significant orientation noise using Hough-based analysis. The GND map derived from HR-EBSD measurements reveals lots of dislocation substructure. The complementary nature of backscatter imaging and GND maps is indicated with the inserts.
of microstructural units which interact during deformation and cause local variations in plastic strain gradients, which we can observe using EBSD. Engineering microstructures like this is at the heart of modern engineering alloy design. Image quality metrics from conventional EBSD show surface features including slip bands but this map is not quantitative and separation of dislocation structure from other artifacts is difficult. Orientation maps may be used to discriminate grains and sharp changes in their colouring can be an indication of significant plastic strain. Assessing these changes can be tricky, as the human visual system is not equally perceptive to the entire visible spectrum. Overlaying low-angle grain boundaries on these maps provides some insight, provided that polygonisation occurs, but in this case it is not helpful.

Thus dislocation structure is best determined by direct measurement of local misorientations. These can be calculated from conventional EBSD data, as shown here with the kernel average misorientation (KAM). This measures the local average orientation gradient surrounding each pixel. This averaging process may smooth out orientation noise but the low resolution of conventional KAM maps reduces their usefulness. In addition, there is no physical merit to the KAM, as it ignores length, gives no measure of dislocation density and cannot be linked to any mechanical process directly.

The higher angular precision of the pattern correlation procedure described above, however, can be utilised to provide the link between stored dislocation content and plastic strain though analysis based on the pioneering work of Nye [29]. An example map is shown in Figure 5, where the analysis has been performed to map the necessary geometric dislocation density structure using the method described elsewhere [21]. Comparing the two inserts shows the complementary nature of the pseudo-backscatter quality map and the GND distributions. For example, one slip band (marked with an arrow) has not punched fully through the surface and results in a significant stored dislocation density.

Linking this map with the Hough-based data provides a wealth of high fidelity, statistically significant multimodal information ripe for statistical interrogation. Insights from these data can be utilised to provide the link between stored dislocation density and cannot be linked to any mechanical process directly.

Looking to the Future

The HR-EBSD technique has emerged as a powerful laboratory-based technique for strain measurement. Some of its potential has been described with these four simple examples. The analysis procedure is now widely employed in laboratories worldwide. Application to the study of basic questions such as the patterning of deformation in polycrystalline materials [14, 22, 30], the effect of inclusions [31], the strength of individual grain boundaries [3], and stress fields measured in silicon have been undertaken and reported extensively [6, 7, 13, 18].

Direct comparison of measurements obtained via AFM and Raman spectroscopy have been positive [6, 7, 18]. Development of the technique continues, most notably to solve the ‘reference pattern problem’ [16, 32]. This stems from the need to know the geometry of the experimental configuration with a precision equal to or greater than the desired strain resolution (i.e. better than 1 in 10,000) so that we can remove the effect of beam shift more precisely, or simulate a high quality diffraction pattern using dynamical diffraction theory [33] and use that as the reference pattern.

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BIOGRAPHY

Ben Britton has a DPhil on the deformation behaviour of titanium alloys for aerospace applications from Oxford University (2010). After working at the Department of Materials at the University of Oxford researching materials for fission and fusion power, in 2012 he joined the Department of Materials at Imperial College London as a Nuclear Metallurgy Fellow. His research interests include techniques such as electron backscatter diffraction and digital image correlation, as well as the mechanical performance and behaviour of metals.

ABSTRACT

The performance of crystalline materials is dominated by the nature of elastic strains and defects. The interaction of stress fields and defects, such as dislocations, and microstructure at the meso-scale is of significant importance. We discuss and explore applications of the high angular resolution electron backscatter (HR-EBSD) technique, which is capable of resolving elastic strains and defect populations with high fidelity and high spatial resolution, which enables us to probe the behaviour of materials and link structure to properties.

Our examples span the realms of functional and structural materials, including silicon, germanium and titanium.

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