Mapping the full lattice strain tensor of a single dislocation by high angular resolution transmission Kikuchi diffraction (HR-TKD)

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Abstract

The full lattice strain tensor and lattice rotations induced by a dislocation in pure tungsten were mapped using high-resolution transmission Kikuchi diffraction (HR-TKD) in a SEM. The HR-TKD measurement agrees very well with a forward calculation using an elastically isotropic model of the dislocation and its Burgers vector. Our results demonstrate that the spatial and angular resolution of HR-TKD in SEM is sufficiently high to resolve the details of lattice distortions near individual dislocations. This capability opens a number of new interesting opportunities, for example determining the Burgers vector of an unknown dislocation in a fast and straightforward way.

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Electron backscatter diffraction (EBSD) in a scanning electron microscope (SEM) is widely applied in material characterisation at the meso-scale. By rastering a focused electron beam across a grid of points on the sample surface and analyzing EBSD patterns, the crystallographic orientation of each point is obtained [1,2]. Based on the point-by-point orientation, information, such as grain structure [3], phase identification [3], intragranular misorientations [1,4,5], micro-texture [4,6], grain boundaries [4–7] and orientation relationships between phases [1], can be retrieved. The angular resolution of EBSD is ~1° [8]. The cross-correlation based EBSD analysis approach introduced by Wilkinson (HR-EBSD) improves the angular resolution to 0.005° by measuring small shifts of features in the EBSD patterns compared to a reference EBSD pattern [8–11]. These small shifts can be interpreted in terms of lattice rotations and lattice distortions [1,8–11].

HR-EBSD has been widely adopted to characterise geometrically necessary dislocation (GND) density and residual lattice strains in crystalline materials [1,12–17]. A comparative study showed good agreement between HR-EBSD and X-ray measurements of GND density [13]. Importantly, HR-EBSD can access the spatial distribution of GND density and lattice strain at the nano-scale near interesting features, such as grain boundaries [18–20], indents [21,22], second phases [20,23] or slip bands [24]. The spatial resolution of HR-EBSD is governed by the electron interaction volume with estimates of the probed volume ranging from several tens to hundreds of nanometers in bulk material [25]. This, and experimental issues with drift, have prevented the study of the strain fields associated with individual dislocations using HR-EBSD, though statistical analysis by Wilkinson et al. [26] indicates that sufficient spatial resolution should be available to probe the lattice strains near dislocations.

Dislocations are among the most important lattice defects in crystalline materials. Thus far, dislocation characterisation has mostly relied on TEM for determination of dislocation type, Burgers vector and associated strain fields. The two most common methods for measuring lattice strain at the atomic scale are geometric phase algorithms (GPA) [27,28] and nano-beam diffraction in TEM [29–31]. Both offer a strain sensitivity of ~10^-3 and a spatial resolution of 2 to 3 nm [32]. However, only the 2D in-plane strain tensor can be measured using these techniques, and the measurement must be performed on a certain zone axis, placing stringent requirements on sample preparation.

By using transmission Kikuchi diffraction [25] (TKD) in a SEM, i.e. detecting the Kikuchi pattern from the bottom surface of a thin foil, the absolute spatial resolution can be improved to ~10 nm [25], while the effective spatial resolution falls to 2–4 nm [33]. Here we show that by combining TKD with HR-EBSD approaches in the SEM, it becomes possible to measure the full deviatoric strain tensor associated with an individual dislocation. These measurements are compared to predicted strain fields, calculated using an isotropic elasticity model. Our results show that HR-TKD provides a convenient and reliable way of probing nano-scale strain fields, with sufficient sensitivity to study strains associated with specific dislocations.

High purity tungsten foil (99.99% purity and 120 μm thickness) was punched into 3 mm diameter discs. The samples were thinned to electron transparency by twin-jet electropolishing (0.5 wt% NaOH aqueous...
solution, 0 °C, 14 V). \( \mathbf{g} \cdot \mathbf{b} \) analysis was performed on a JEOL 2100 TEM. TEM bright field images under 8 independent \( \mathbf{g} \) vectors from 4 zone axes were acquired. The thickness of the region of interest is 40 ± 10 nm, measured using electron energy loss spectroscopy on a JEOL ARM200F TEM. A Zeiss Merlin SEM with a Bruker eFlash detector was then used to carry out HR-TKD measurements (20 kV, 3 nA). The TKD setup is shown in Fig. 1(a). The sample was tilted −45° to the electron beam, a TKD pattern size of 800 × 600 selected and a scanning step size of 4 nm used. The cross-correlation analysis of the Kikuchi patterns was done using the XEBSD matlab code described by Britton & Wilkinson [34,35].

The anticipated spatial variation of the deviatoric lattice strain and lattice rotations in the vicinity of a dislocation was calculated using isotropic elasticity. This is reasonable since tungsten is almost perfectly elastically isotropic [36]. Since a thin foil was used for the measurement, surface relaxation was taken into account. For simplicity, we assume that the dislocation is straight with line direction normal to the foil surface, i.e. along the -\( \mathbf{z} \) direction (see supplementary Fig. S1). For a dislocation with arbitrary Burgers vector \( \mathbf{b} \) that meets the free surface at (0, 0, 0), the displacement field at (\( x, y, z \)) can be found by decomposing \( \mathbf{b} \) into components along \( x, y, z \) directions:

\[
b = \sum_{i=1}^{3} b_i \mathbf{n}_i,
\]

where \( \mathbf{n}_i \) are unit vectors along \( x, y, \) and \( z \) directions, \( b_i \) are the corresponding coefficients.

The displacement field caused by the dislocation is then the linear superposition of the displacements caused by a screw dislocation with Burgers vector \( b_3 \mathbf{n}_3 \) and two edge dislocations with Burgers vectors \( b_1 \mathbf{n}_1 \) and \( b_2 \mathbf{n}_2 \). The displacement field can be expressed as:

\[
u_{ki} = \sum_{j=1}^{3} u_{ki}^j \quad (j = 1, 2, 3),
\]

where \( u_{ki} \) is total displacement along the \( k \) direction (\( k \) refers to \( x, y \) or \( z \)), \( u_{ki}^j \) is the displacement along \( j \) induced by dislocation with Burgers vector \( b_j \mathbf{n}_j \).

The displacement field of an edge or screw dislocation meeting a free surface can be obtained by superposing the displacement of the dislocation in an infinite body and the displacement induced by image forces due to the traction free surface condition,

\[
u_{ki}^\text{image} = u_{ki}^\text{image} + u_{ki}^\text{image} \quad (i, j = 1, 2, 3).
\]

Here we use the solution for \( u_{ki}^\text{image} \) and \( u_{ki}^\text{image} \) given by Anderson et al. [37] and Yoffe [38] (see supplementary material). The components of the 3D strain tensor of the dislocation at any position (\( x, y, z \)) are obtained by differentiating:

\[
\varepsilon_{ij} = \frac{1}{2} \left( \frac{\partial u_{xi}}{\partial x_j} + \frac{\partial u_{xj}}{\partial x_i} \right) \quad (i, j = 1, 2, 3).
\]

The lattice rotation can be obtained as

\[
\alpha_{ij} = \frac{1}{2} \left( \frac{\partial u_{xi}}{\partial x_j} - \frac{\partial u_{xj}}{\partial x_i} \right) \quad (i, j = 1, 2, 3).
\]

As the TKD patterns are dominated by a tens of nanometers thick surface layer, the reported elastic strain and lattice rotations are an average over a depth of half of the foil thickness (20 nm).

In SEM, dislocations can be imaged with high-energy primary electrons (PE) without needing to set up specific diffraction conditions. Fig. 1(b) shows a PE image of the grain under investigation. Several dislocations with line direction normal to the surface can be seen with black–white contrast. The crystal orientation of the grain, determined by TKD, is (227.8°, 23.2°, 322.3°) in Euler angles and the corresponding coordinate frames for the image and the crystal are shown inset in Fig. 1(b). A dislocation close to a grain boundary (2–3° misorientation) was selected for TEM \( \mathbf{g} \cdot \mathbf{b} \) analysis and HR-TKD (red rectangle in Fig. 1(b)) as the grain boundary provides a convenient reference for judging drift during the HR-TKD measurement.

Fig. 2(a) shows the same grain imaged by TEM bright field. The dislocations imaged by the two different techniques agree very well. \( \mathbf{g} \cdot \mathbf{b} \) analysis for the dislocation of interest is shown in Fig. 2(b). The \( \mathbf{g} \) vectors were determined consistent with the crystal orientation found by EBSD. The dislocation shows contrast except under \( \mathbf{g} = 110 \) and \( \mathbf{g} = 21T \) conditions. The Burgers vector of the dislocation can thus be determined as ±[111]/2 according to the \( \mathbf{g} \cdot \mathbf{b} \) table (Table 1).

Strain maps for the 6 components of the 3D strain tensor (upper triangle and diagonal in the matrix map) and 3 lattice rotations (lower triangle in the matrix map) near the dislocation, measured by HR-TKD, are shown in Fig. 3(a). The strains and rotations are plotted in the microscope coordinate frame shown in the upper left corner, which is the same as used in Figs. 1 and 2. The deviatoric, rather than full, lattice strain tensor is measured, as HR-TKD is not sensitive to lattice dilation [8]. However, the volumetric strain can be calculated by assuming stress along the out of plane direction to be zero (see supplementary Fig. S2). From the normal strain components, the contrast formed by compressive and tensile strains on either side of the dislocation is clearly visible. The core of the dislocation can be determined as the mid-point between peaks of compressive and tensile strain in the \( \varepsilon_{xz} \) strain map, which is the clearest of the measured lattice strain components. The core is marked by two intersecting arrows superposed on the \( \varepsilon_{xz} \) strain map.

![Fig. 1](image_url) (a) The setup for HR-TKD measurement. (b) Transmitted primary electron image showing the contrast from dislocations. The inset cube shows the orientation of the grain. \( x_m-y_m \) is the coordinate frame of the electron image and the EBSD map. \( y-z \) is the crystal coordinate frame.
The strain maps for shear components of $\varepsilon_{12}$ and $\varepsilon_{13}$ are a little noisy, while the map for $\varepsilon_{23}$ shows obvious contrast between positive and negative shear strains. Lattice rotations around axes $z_m (\omega_{12})$ and $x_m (\omega_{23})$ display clear negative and positive contrast between the two sides of the dislocation, while the lattice rotations around axis $y_m (\omega_{13})$ are small.

It is interesting to note that the separation between the tensile and compressive strain peak in the $\varepsilon_{22}$ strain map is ~25 nm. The isotropic elasticity model, on the other hand, predicts no separation between these maxima (see Supplementary Fig. S3). The elasticity model has a singularity at the dislocation core, however, at $>10 \, b$ (~2.7 nm) away from the core [39], elasticity is valid. This suggests that the large separation between extreme $\varepsilon_{22}$ values we observe is due to the finite size of the electron interaction volume. To enable a better comparison between the measured and predicted strain fields, the forward calculated lattice strain and lattice rotation maps were convolved with a 2D Gaussian function ($\sigma = 5$ nm). This estimate of the probe resolution (~2.4 $\sigma = 12$ nm) is consistent with previous reports of TKD spatial resolution [25]. Fig. 3(b) shows the forward calculation of the variation of deviatoric strain and lattice rotations near the dislocation after convolution with the probe function. Importantly only the strain fields for $b = \frac{1}{2} \left[ 111 \right]$ correctly capture the spatial variation of strains and rotations. The alternative $b = \left( \frac{1}{2} 111 \right)$, which is also consistent with the $g \cdot b$ analysis, yields the opposite, incorrect, sign of all strain and rotation components.

Good agreement between the measurement and the calculation can be found in all the components except $\varepsilon_{13}$, $\varepsilon_{23}$, and $\omega_{23}$. Particularly we note that key features, such as the line separating positive and negative lobes, as well as the direction pointing from the negative to the positive lobes match remarkably well (see the maps for $\varepsilon_{11}$, $\varepsilon_{22}$, $\varepsilon_{33}$, $\varepsilon_{12}$, and $\varepsilon_{13}$).

The agreement can be further quantified by considering the angular variation of the lattice strain and lattice rotation fields along a circular path around the dislocation 20 nm from the core (see path drawn on $\varepsilon_{12}$ maps in Fig. 3). The resulting profiles for all strain components are shown in Fig. 4. For the direct strain components ($\varepsilon_{11}$, $\varepsilon_{22}$, $\varepsilon_{33}$), the $\varepsilon_{12}$ shear component and $\omega_{12}$ and $\omega_{13}$ lattice rotations the measurement agree rather closely with the expected angular variation. In particular it is very interesting that the locations of key features, such as peaks and troughs, are correctly captured. Several of the profiles appear to

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**Table 1**

The $g \cdot b$ table for visibility (v) and invisibility (i) of dislocations in bcc crystal.

| $\mathbf{b}$  | $\mathbf{g}$        | $(\mathbf{200})$ | $(\mathbf{100})$ | $(\mathbf{020})$ | $(\mathbf{110})$ | $(\mathbf{21T})$ | $(\mathbf{12T})$ | $(\mathbf{22T})$ | $(\mathbf{111})$ |
|---------------|---------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| $\pm[100]$    | v                   | v                | i                | v                | v                | v                | v                | v                | v                |
| $\pm[010]$    | i                   | v                | v                | v                | v                | v                | v                | v                | v                |
| $\pm[001]$    | i                   | i                | i                | i                | v                | v                | v                | i                | i                |
| $\pm[111]$    | i                   | v                | v                | v                | v                | v                | v                | i                | i                |
| $\pm[1\bar{1}T]$ | v           | v                | v                | v                | v                | v                | v                | v                | v                |
| $\pm[1\bar{1}T]$ | v           | v                | v                | v                | i                | v                | v                | i                | i                |
| $\pm[1T1]$    | v                   | v                | v                | i                | i                | v                | v                | v                | v                |
| $\pm[1T1]$    | v                   | v                | v                | v                | i                | v                | v                | v                | v                |
have a small vertical offset. This may be explained as the lattice rotations and strains in HR-TKD are computed with respect to a reference pattern assumed to come from a nominally strain-free region of the sample.

Surprisingly poor quantitative agreement is found in $\varepsilon_{23}$ and $\omega_{23}$ as obvious positive and negative contrast is observed in the measurement, while only small variations are expected from calculations. Qualitatively, the directions from negative to positive lobes match well in measurements and calculations for both $\varepsilon_{23}$ and $\omega_{23}$ (better visible in Supplementary Fig. S3). Considering Eqs. (4) & (5), summation of $\varepsilon_{23}$ and $\omega_{23}$ provides a map of $\partial u_y / \partial z$, which is small, as expected from calculations. The large signal in the experimental maps of $\varepsilon_{23}$ and $\omega_{23}$ is due to $\partial u_y / \partial y$, which is obtained by subtracting $\omega_{23}$ from $\varepsilon_{23}$ (see supplementary Fig. S4). The unexpectedly large magnitude of $\partial u_y / \partial y$ may be due to larger experimental uncertainty associated with $\partial u_y / \partial y$ and $\partial u_x / \partial x$ components in the present geometry [10]. The variation in $\varepsilon_{13}$ is dominated by strains associated with the grain boundary that are superimposed on the dislocation strain field.

The good agreement of the strain tensor and lattice rotation between measurement and calculation shows that the spatial and angular resolution of HR-TKD is sufficient to study in detail the lattice distortions caused by individual dislocations. Importantly this characterisation can be done in the SEM, allowing the determination of dislocation Burgers vector, which was previously only possible by TEM. In TEM, Burgers vector determination commonly relies on $g \cdot b$ analysis and black-white contrast. However, $g \cdot b$ analysis becomes ambiguous when applied to small irradiation induced dislocation loops and dislocations normal to the thin foil. Here, because of surface relaxation, dislocations may still show contrast even when $g \cdot b = 0$ [40–42]. Black-white contrast relies on computation of the dynamical diffraction images of dislocations recorded at different $g$ vectors [42]. Instead, direct comparison of lattice strain and rotations (which actually cause the intensity contrast in TEM) from just one HR-TKD measurement with predicted strain maps provides a straightforward approach to unambiguously determining Burgers vector, sign and magnitude of unknown dislocations. To illustrate this point, the strain tensors for all other possible Burgers vectors in tungsten are calculated. No agreement can be found between those maps and the measured maps (see supplementary Figs. S5–10). Only the correct Burgers vector, [T1T]/2, provides a good match to the measured strain profiles. We note that the model used in this paper only applies to dislocations with line direction near-normal to the surface and assumes an elastically isotropic material. For inclined dislocations the direction of the dislocation line and an incline dislocation model, e.g. [38,43], are required.

In summary, we have demonstrated that, using tungsten as a case study, the full deviatoric lattice strain tensor and rotation field due to an individual dislocation can be quantitatively mapped using HR-TKD. The experimentally measured lattice distortions are in remarkably

\[ Fig. 3. (a) Maps for 3D lattice strain tensor and lattice rotations around the dislocation measured by HR-TKD. (b) The forward calculation of the distribution of strain tensor and lattice rotation around a [T1T]/2 dislocation using an isotropic elastic forward model convolved with a 2D Gaussian probe function. Red arrows show the direction of Burgers vector projection. The cross shown in the $\varepsilon_{12}$ map is the place where reference pattern was taken. \]
good agreement with those expected from a forward calculation using an isotropic elasticity model of the dislocation. Our results suggest that the combination of strain field simulation and HR-TKD may offer a straightforward approach to determining Burgers vector magnitude, direction and sign in SEM.

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Appendix A. Supplementary information

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scriptamat.2018.12.039.

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