The Analysis of Plastic and Elastic Deformation from Indentation

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Abstract. A technique is described using convergent beam electron diffraction to measure the magnitude of the elastic and plastic strains in the material around an indentation. Cross-sections through indentations are extracted and thinned for transmission electron microscopy using focussed ion beam milling. The technique has been demonstrated on 500 nm deep indentations in single crystals of Cu and MgO.

1. Introduction
When a sample is indented, the material from the final impression must be extruded to the surface [1], giving pile-up, or accommodated elastically in the bulk of the crystal [2]. The former is generally thought to occur in materials whose yield stress, \(Y\), is a relatively low compared to its Young modulus, \(E\), whilst the latter occurs in materials whose \(Y/E\) ratio is higher. Thus far, quantitative small-scale study of the deformation underneath indentations has been hampered by the very high dislocation densities.

A solution to this challenge is to measure the rotations of the crystal lattice. According to strain gradient plasticity theory, these rotations arise from geometrically necessary dislocations in materials with a low \(Y/E\) ratio, such as Cu [3]. However, in materials with a higher \(Y/E\) ratio such as MgO or spinel, a greater fraction of the rotations might be associated with elastic deformation. Yang et al. [4] were able to measure rotations from volumes of about 1 \(\mu\)m\(^3\) in Cu, but other work has shown that variations in orientations can occur on a smaller scale [5]. We are developing a method that will allow both the elastic and plastic contributions to the overall deformation to be measured in much smaller volumes than has been possible so far.

2. Experimental Method
Single crystals of [001]-oriented Cu and MgO were indented using a Berkovich tip (Nanotest 600, Micromaterials) to a depth of about 500 nm. Electron-transparent cross-sections were made parallel to the [010] axes using the “trench” technique in Cu and the “lift-out” technique in MgO by focussed ion beam milling [6,7]. No bending of the foil was observed in the last stages of milling in either material and the final thickness was about 200 nm. Low camera length convergent beam electron diffraction (CBED) patterns were obtained by transmission electron microscopy (TEM) (Philips CM30), operating at 300 keV. Rotations about the [100] axis were measured by comparing the orientation of the [002] Kikuchi bands in the deformed region to those in the un-deformed region, as seen in figure 1. A clockwise rotation is taken as positive, and the rotations are measured to an accuracy of 1º.

Tilts about [001] and [010] axes are calculated from CBED patterns using the method described by Wang and Starink [8]. Figure 1 (b) shows how these tilts are measured in the new system of axes created by the rotation of the crystal around the [100] axis. This method has an accuracy of 0.1º.
although it is less accurate for angles greater than 10° [8]. Tilts within 0.2° of the [100] axis were not measured due to diffraction from the zone axis. It should be noted that the tilts around the [001] and [010] axes are opposite when the foil is opposite side-up in the TEM.

Figure 1. CBED pattern from Cu on the [100] axis. Plates are taken at several exposures and nested so detail from different intensities can be seen. (a) Central beam is parallel to the [100] axis. The dark area at the bottom of the pattern is due to overhang from the trench. (b) CBED pattern of crystal which has rotated clockwise by 12°, and then tilted by -2.1° around the new [001] axis and -5.1° around the new [010] axis. The tilts are measured in relation to the rotated [020] and [002] Kikuchi bands.

3. Results and Discussion

Figure 2 shows the rotations around the indentation in Cu and figure 3 shows those in MgO. Both bright field (BF) images are taken from the [100] orientation, and both show “lobes” of deformation on either side of the indentation. The in-plane rotations around the [100] axes follow similar patterns in both materials, with the exception of the one of the sides of the MgO sample.

Table 1 shows that the magnitude of the rotations in the Cu and MgO are about the same, and are similar to those reported elsewhere in spinel [9]. This similarity occurs despite differences in slip systems and differences in the predominant obstacles to dislocation motion. To determine the contribution of elastic strains to the rotations, measurements of the lattice parameters from the HOLZ lines in the CBED patterns are underway. Preliminary measurements in the MgO have shown that at point “X” in figure 3, there are no elastic strains in the [001] or [010] directions.

In the MgO, the transition between the deformed and undeformed regions takes place suddenly, which is different from Cu. This can be seen in points “F” and “G” of figure 3, which are 200 nm apart but show a rotation of 5.0° to 0.6° respectively. The more gradual transition from rotated to unrotated lattice in the Cu is possibly due to the lower flow stress and the ease with which dislocations can move through the crystal lattice, or because deformation of MgO requires the operation of both the softer {110} \langle 1 \bar{T} 0 \rangle and the harder {100} \langle 01 \bar{T} \rangle slip systems [6].
The quality of the CBED patterns varied greatly between the two samples. In Cu, clear CBED patterns could be obtained in the deformed zone provided the probe-size was smaller than about 30 nm. In MgO, the Kikuchi lines were blurred in the deformed region, even with a 10 nm probe size; however, outside the deformed region, the CBED patterns obtained from MgO were much sharper than those from Cu.
Table I. Numerical results for Cu and MgO. Note that the spots are associated with the points shown in figure 2(b) and figure 3(b) and are not equivalent locations.

| Spot No. | Cu | MgO |
|----------|----|-----|
|          | 100 (°) | 010 (°) | 001 (°) | 100 (°) | 010 (°) | 001 (°) |
| A        | -1  | 3.4 | -2.1 | 4      | -4.6 | -0.8 |
| B        | -2  | 0.6 | -2.2 | 3      | 4.2  | 0.8  |
| C        | 1   | 1.4 | -2.1 | 5      | -3.2 | -0.2 |
| D        | 7   | 4.0 | -2.1 | 0      | 0    | 0    |
| E        | -1  | 0   | 0    | 0      | 5.5  |
| F        | 0   | 0   | 0    | 0      | -4.8 | 1.7  |
| G        | 4   | 7.7 | 0.2  | 0      | -0.6 | 0.1  |
| H        | 1   | 0.3 | -0.3 | 0      | 2.7  | -1.2 |
| I        | -5  | 4.0 | 0.5  |
| J        | -3  | 3.0 | 1.0  |
| K        | -7  | 3.3 | 0.1  |
| L        | -1  | 0.0 | 0.2  |
| M        | 2   | -0.3 | -0.3 |
| N        | -5  | 2.6 | -0.1 |
| O        | 1   | 1.6 | 1.4  |
| P        | -2  | 1.8 | 1.3  |

4. Conclusions
The magnitudes of the rotations of the crystal lattice around indentations approximately 500 nm deep were similar in both Cu and MgO when measured with CBED. However, the transition from rotated to unrotated material was much more gradual in and the quality of the CBED patterns was more consistent in the Cu. Further work is underway to investigate the contribution of the elastic strain to the rotations using the HOLZ lines from the CBED patterns.

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