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Systematic study of structural changes in the vicinity of indentation marks with HR-EBSD

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Abstract. In crystalline materials a detailed understanding of the effects of external deformation is essential to develop new materials. The mechanical properties are highly influenced by the collective motion of lattice defects (mainly dislocations). The primary task of the research is to gain more experimental knowledge on the stress and strain distribution evolving in the crystal due to external deformation. In the reported investigation an indenter was pressed into the sample to a depth of a few micrometers. After the removal of the head high resolution electron backscatter diffraction (HR-EBSD) measurements were carried out in the vicinity of the mark, giving the 3D depth distribution of stress values.

1. Introduction

Plastic deformation of crystalline materials typically occurs by the collective motion of dislocation ensembles. In a macroscopic sample the large number of grains and moving dislocations generally produce smooth and reproducible stress–strain response allowing highly accurate predictions of the material's properties. In contrast, on the scale of a single grain, the inhomogeneities in the dislocation structure can be in the order of the grain size, leading to a stochastic activation of dislocation avalanches producing discontinuous response. The effect of dislocations is localized to an extremely small volume, so most of the time one can only investigate the cumulative influence of numerous defects.

The effect of crystal orientation on the indentation response is widely studied on single crystal titanium [1], nickel [2], copper [3] and even on perovskite oxide ceramic materials [4]. Synchrotron X-ray microdiffraction measurements show that indentation hardness increases with decreasing indentation depth on copper single crystal [5]. In these measurements Berkovich or conical indenters were used. For the better understanding of the indentation process it is important to get a detailed knowledge about how the dislocation cell structure forms, how it evolves due to external deformation, and inspect the stress state developing around the mark. As a model material we used a previously heat treated copper (Cu) single crystal, that has been studied extensively because of its unique properties like cubic symmetry, relatively low Young's modulus and ease of preparation.

Traditional 3D electron backscatter diffraction (EBSD) measurements, using conical indenter, were published in Ref. [3], showing size effect at small indents due to the geometrically necessary dislocations generated during the indentation. In the present work we aim to investigate the 3D distribution of strain developing under a Vickers indenter. In order to achieve this, around the indentation mark, a series of thin layers perpendicular to the sample surface were removed by focused ion beam (FIB). High resolution electron backscatter diffraction (HR-EBSD) measurements were carried out in each layer giving the maps of the ε_{ij} strain and σ_{ij} stress tensors.

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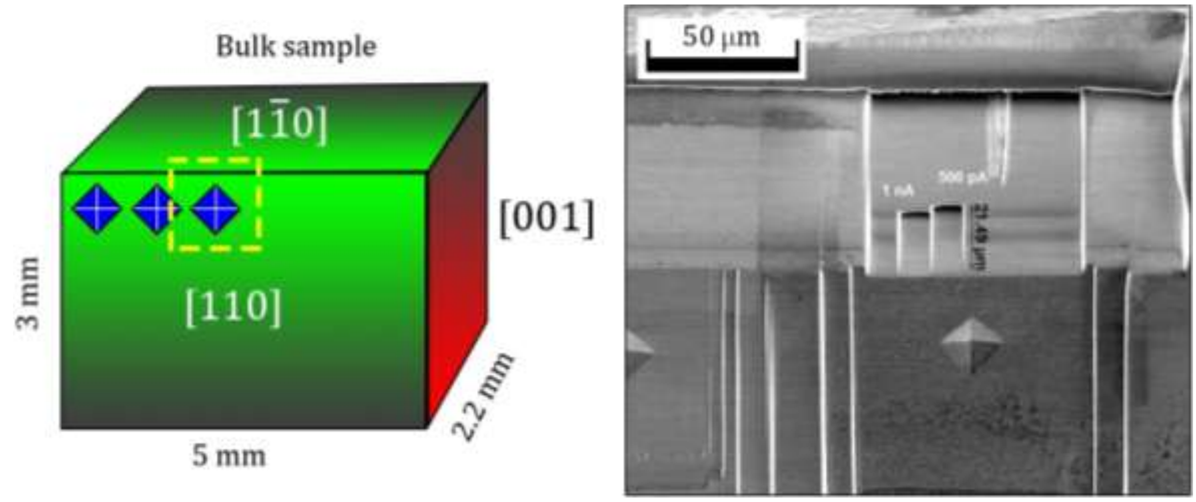
High resolution EBSD is a cross-correlation based evaluation technique. It is a widely accepted methodology for strain calculation from the relative deformation of the EBSD patterns obtained at different points. The development of the HR-EBSD technique is mainly related to Wilkinson and his co-workers [6]. The evaluation, which was executed by a program developed by one of the presenting authors [7], enables us to calculate the relative distortion of the lattice and investigate the local stress variations. The distortion and stress values can be plotted as a function of the position on the sample surface. Combining these 2D maps leads to a 3D map of the structural changes occurred due to plastic deformation. This so called "slice and view" method is a novel approach in HR-EBSD measurements.

2. Methods

Using FIB technique thin layers can be removed from the surface of the material in a well-controlled and precise way. With the application of preliminary determined ion current it is possible to produce smooth and EBSD-ready surfaces. By repeating the slicing and EBSD mapping a relatively high volume (in the order of tens of micrometers) can be explored.

The measurements were performed on a previously heat treated and undeformed copper single crystal. The sample was cut from an oriented ingot by electrical discharge machining. The orientations and the dimensions of the specimen are indicated in Fig 1a). After cutting, the samples were electropolished, and heat treated in a vacuum furnace to remove any excess distortions that may have accumulated during the cutting. After the heat treatment the sample was electropolished again with Struers D2 formula.



a) **b)**

Figure 1. a) The schematics of the specimen shown with the crystal orientations. On the sides the dimensions of the bulk sample is noted, and the coloring of the surfaces indicates the orientation. The three blue squares represent the position of the indentation marks. The yellow dashed area indicates the photographed area in b). An indentation mark with an estimated diagonal of 20 μm shows the initial arrangement before the slicing. This mark was only used for testing the FIB currents to check the quality of the diffraction patterns. The middle mark was used for the actual measurement.

2.1. Preparation of the indentation mark

In order to create a perfectly leveled and flat surface for the indentation marks, focused ion beam was used to form a plateau (with a normal vector parallel to the [110] crystal direction) near the sample's edge. The side of the sample was also cut by FIB to create a surface perpendicular to the plateau. As the quality of the surface is crucial for the accuracy of the evaluation, this newly formed surface was the one measured later by EBSD.

Since focused ion beam milling creates an amorphous layer on the surface that disturbs or even totally hides the diffraction patterns, to minimize this effect, during the FIB processing the ion current was decreased as much as it was necessary to be able to collect good quality diffraction patterns from the ion-treated surface. The biggest challenge of the preparation was to create perfect conditions for the EBSD measurements.

Three indentation marks were placed on the sample (100 μm from each other) by a *Zwick/Roell ZHμ Indentec* Vickers hardness testing device. The smallest available load (HV 0.01) was chosen while the Vickers head was pressed into the sample for 10 seconds. The marks were approximately 20 μm wide (see in Fig 1 b)).

2.2. EBSD measurements

To be able to make both the FIB slicing and the EBSD measurements we mounted the sample on a 45° sample holder. This allowed us to rotate the specimen between the ion slicing and the EBSD mapping positions. For the manipulation and measurements an FEI Quanta 3D FEG dual-beam SEM equipped with an EDAX Hikari EBSD camera was used. Parallel slices of the material were cut with a 30 kV / 15 nA ion beam. Due to the relatively big size of the indentation mark an area of 30 μm × 30 μm was chosen to be scanned by EBSD at 20 kV. Fig 2. shows the surface view of the indentation mark, indicating how the slices perpendicular to the surface were made. Each slice was 1 μm thick, and 30 slices were measured (therefore the reconstructed volume was 30 μm × 30 μm × 30 μm in size) by EBSD with a step size of 200 nm on a square grid.

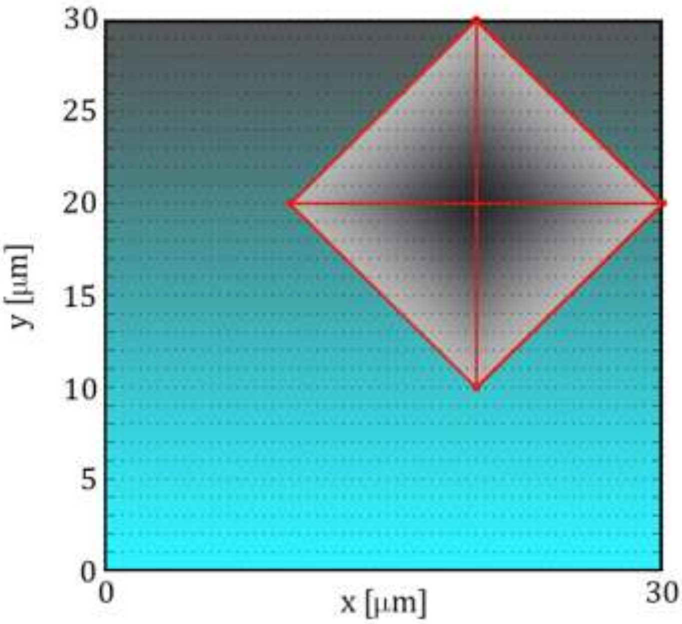


Figure 2. The schematics of the FIB slicing. A volume of 30 μm × 30 μm × 30 μm was mapped, including the indentation mark on the surface. The slicing was performed along the *z* axis, and the cuts are indicated by the dotted lines. The EBSD mapping was performed on the *x* - *z* surface.

The total number of registered EBSD patterns is close to 7×10^5 in a volume of about $3 \times 10^4\ \mu m^3$. Both the cutting of a slice and the creating an EBSD map took half an hour to perform. The whole measurement required 4 days to finish.

The EBSD maps were then evaluated by the *Strain_calc* program developed at the Eötvös Loránd University by one of the authors of this paper. The program is capable of calculating the tensor elements of the distortion, strain and stress matrices. During the cross-correlation-based evaluation the diffraction patterns from the scanned area are compared to a reference patterns (ideally measured at a stress-free point). As the effect of the indentation mark is only confined to a small volume, therefore the reference was recorded on the same surface but further away so it represents the initial (pre-deformed) stress state. All of the 30 slices were evaluated the same way, using the same reference.

3. **Results**

To investigate the residual stress distribution that formed after the indenter was removed from the surface, the $\sigma_{ij}(x,z)$ stress maps were generated. In Fig 3. only the σ_{12} stress component is plotted as a function of the position (x,z). The maps generated from the other components show similar behavior, therefore they are not shown here.

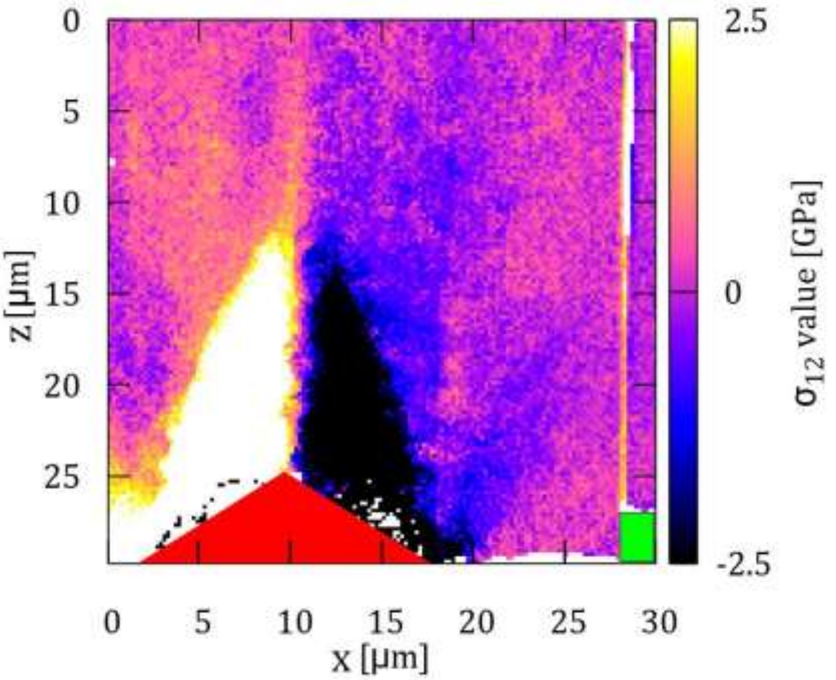
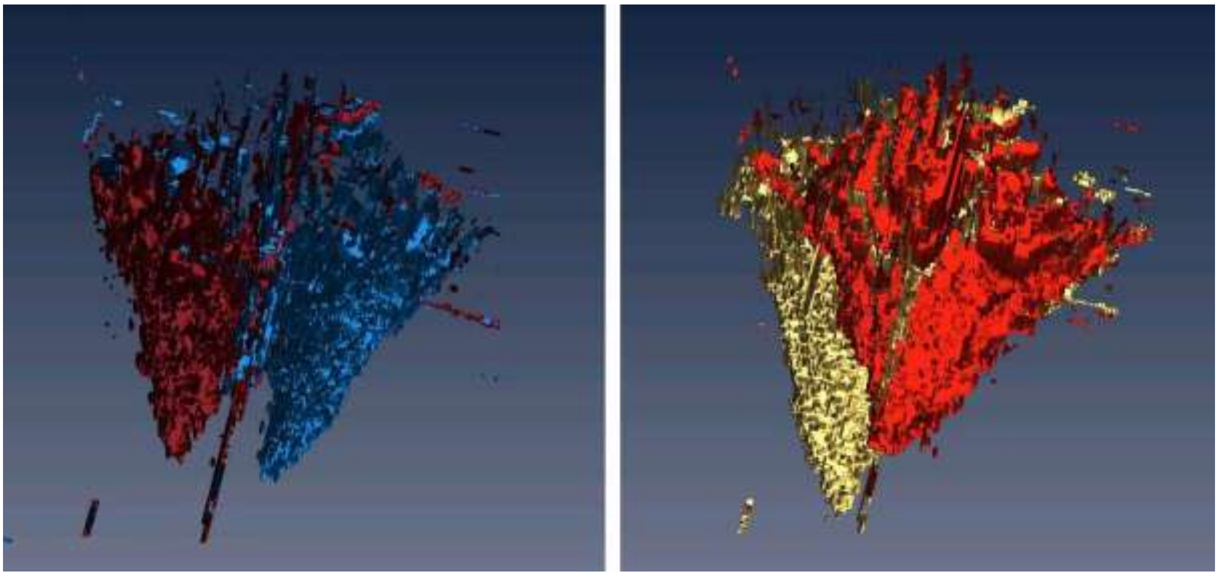


Figure 3. The σ_{12} stress component map measured on the 21st slice.
The red triangle indicates the cross-section of the indentation mark. The green rectangle indicates the FIB-milled ditch used for 3D alignment.

After comparing the results given by the σ_{ij}components some global statements can be made. The volume that was mostly affected by the deformation lies beneath the indentation mark. The σ_{11}, σ_{12} and σ_{13} stress maps show opposite signs of stress levels in these regions, while the σ_{22} and σ_{23} component maps indicate stresses of the same sign. Similar behavior was observed in the earlier GND measurements of *Reuber et al.* [8] along the cross-section of an indentation mark, and also in the strain distribution measured on the surface by *Britton et al.* [9,10].

For the 3D reconstruction of the indentation mark the slices were loaded into the *Amira 3D* software provided by FEI. The 3D model was created by the assembling of the aligned stress maps. The alignment was carried out with the help of a previously FIB-milled ditch on the surface (which can be seen in Fig 3. bottom right corner, green marker). The shapes of the surface corresponding to constant stress levels are seen in Fig 4.



a) **b)**

Figure 4. Constant stress levels at 90% of their maximum values:
(a) σ_{11} component – purple; σ_{12} component – blue; (b) σ_3 component – yellow; σ_{23} component– red.

4. Summary

The "slice and view" method combines the ability of the FIB slicing technique to manipulate the material on a micron scale, and the HR-EBSD method that can calculate the local stress distribution. With this combined method the structural changes in the vicinity of an indentation mark were investigated. A 3D map of the σ_{ij} stress tensor was determined. We found good agreement with previously obtained results, namely that σ_{11}, σ_{12} and σ_{13} stress maps show antisymmetric stress levels in these regions, while σ_{22} and σ_{23} component maps indicate stresses of the same sign. The combined method proposed is a new approach to investigate the deformation and stress distribution in small volumes. It opens the possibility to investigate the microdeformation properties of different problems, like microbending, micropillar compression, etc.

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