

Visualization and Quantification of Plastic Strain Induced by Indentation in Polycrystalline Nickel

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A plastic strain field causes lattice distortion through introduction of dislocations into the crystal [1]. In a scanning electron microscope (SEM), near surface plastic strain fields can be studied using electron channeling contrast imaging (ECCI) and electron backscattered diffraction (EBSD) techniques. In ECCI, plastic strain fields can be visualized through channeling contrast variations due to crystallographic misorientations [2]. In EBSD, plastic strain fields are quantified since presence of plastic strain degrades the electron backscattered diffraction pattern quality [1]. In this study, propagation of indentation-induced plastic zone underneath the surface and in-situ dislocation density measurements were studied using ECCI and EBSD techniques. Ion milling surface preparation technique was used to remove material from the surface and expose the microstructure at maximum depth of the indent.

A Vickers microindentation test was performed on a polished polycrystalline bulk Ni specimen surface using the Clark Microhardness Tester CM-1000AT. To study the microstructure below the indent, ion milling was applied on indentation area, using a Hitachi IM-3000 flat ion milling system. A Hitachi SU-8000 FE-SEM was used to perform ECCI and EBSD on indentation area. Results of ECCI and EBSD studies before and after ion milling are shown in Figure 1. ECCI micrographs showed channeling contrast variations in grains located inside and outside the indent (Fig. 1a and 1c). Inverse pole figure coloring maps showed that regions with channeling contrast also exhibited crystallographic misorientations relative to the underlying grains (Fig. 1b and 1d). In addition, irregular bright and dark bands of channeling contrast were detected in grains subjected to intense deformation inside the indentation area (Fig. 1c). During indentation, a regular change in orientation occurs across grains due to even bending of lattice planes. Systematic crystallographic misorientations in lattice planes result in complex shape and contrast of bend contours. The shape and contrast of bands varies from grain to grain due to changes in grain orientation [3]. Using equations 1 and 2 [3], dislocation density ρ was calculated based on the angle of curvature θ and the length l for a bend contour observed at indentation area after ion milling as shown in Figure 2. For a θ of 17° , l of $3.5 \mu\text{m}$ and lattice parameter a of 0.352 nm , a dislocation density of approximately 10^{11} cm^{-2} was calculated. Considering typical dislocation density of 10^6 cm^{-2} for an annealed specimen prior to indentation test [3], the calculated dislocation density from ECCI micrograph indicates the severity of local lattice bending under microindentation.

$$\rho = \frac{\theta}{lb} \quad (1)$$

$$|b| = \frac{a}{2} |\langle 110 \rangle| = \frac{a}{\sqrt{2}} \quad (2)$$

Results showed that indentation-induced plastic zone around and underneath the indent can be mapped using ECCI and EBSD in FE-SEM. The complicated bend contour pattern observed after ion milling directly maps out the region of severe deformation inside the indent.

References:

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 [3] D. C. Joy, D. E. Newbury, & P. M. Hazzledine, *Proceedings of the 5th annual scanning electron microscope symposium part I and part II, workshop on biological specimen preparation for scanning electron microscopy*, (1972), pp. 97-104.

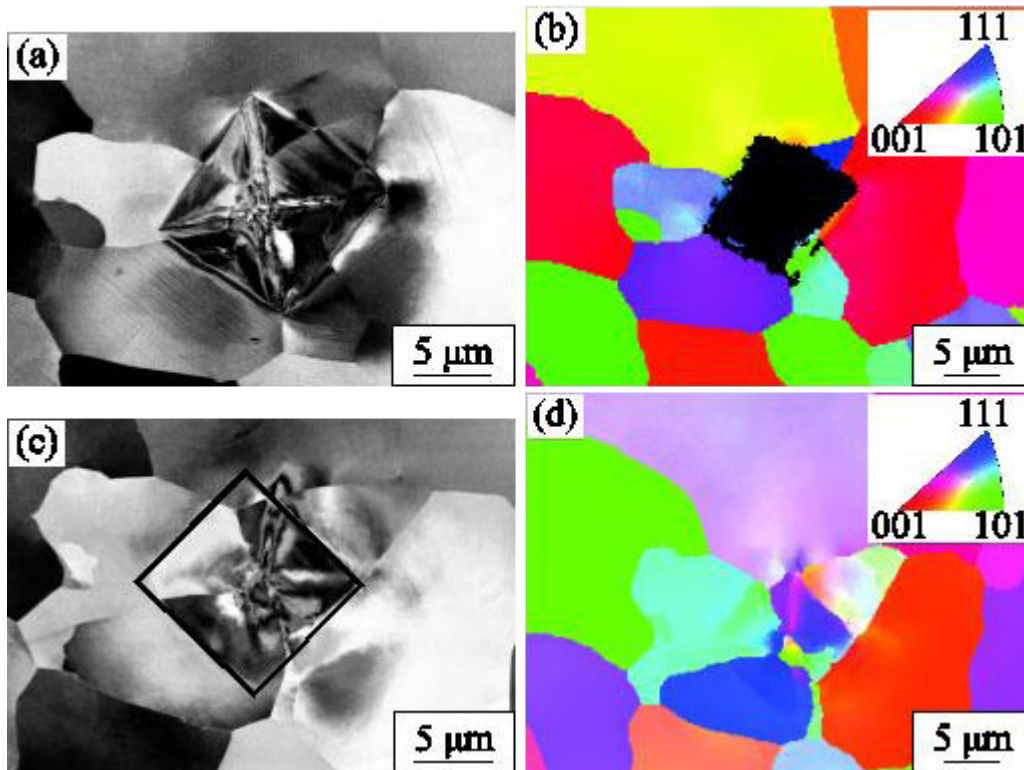


Figure 1. Polycrystalline bulk Ni microstructure after ion milling. (a,c) Backscattered electron micrograph and (c,d) inverse pole figure coloring map of the indentation area. The approximate location of the indent before ion milling is indicated with a $10\ \mu\text{m} \times 10\ \mu\text{m}$ square. Regions of channeling contrast also exhibited crystal misorientations inside and outside the indent.

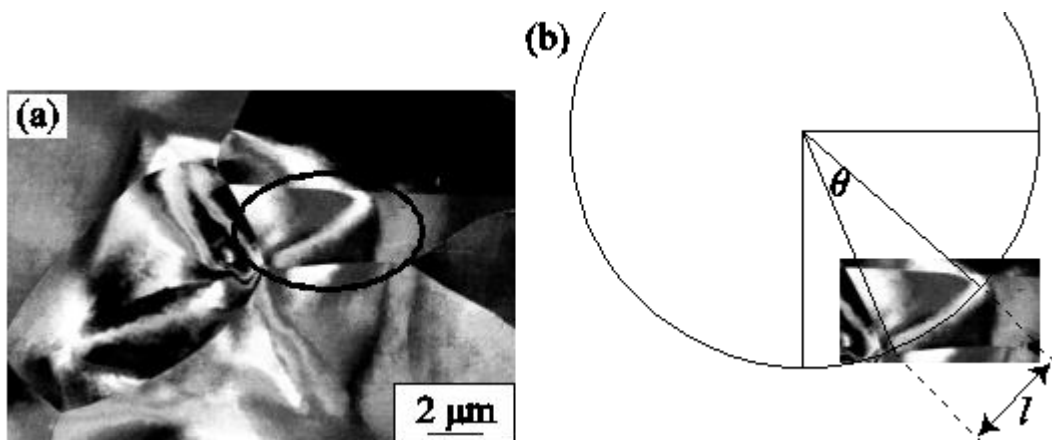


Figure 2. Dislocation density calculation for a bend contour observed in indentation area after ion milling. (a) Backscattered electron micrograph and (b) θ and l measurements for a band indicated with a circle in (a).