INDENTATION SIZE EFFECT AND 3D DISLOCATION STRUCTURE EVOLUTION IN (001) ORIENTED $SrTiO_3$: HR-EBSD AND ETCH-PIT ANALYSIS

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Most crystalline materials exhibit an indentation size effect (ISE), i.e., an intrinsic increase in hardness with decreasing penetration depth. During indentation testing, the material underneath the indenter is heavily deformed, introducing strain gradients in the materials, causing high local dislocation densities. In the present work, the three-dimensional (3D) dislocation structure evolution and ISE in (001) oriented Strontium Titanate (STO) have been studied by direct observation of dislocations using chemical etching and high-resolution electron backscattered diffraction (HR-EBSD) analysis. The sequential polishing, etching and imaging technique was used to reveal the 3D dislocation etch-pit structure at various sub-surface depths using confocal laser and scanning electron microscopy (Fig. 1). The 3D dislocation etch-pit analysis of spherical indentations confirm that, at the early stage of plastic deformation, the dislocation pile-ups were aligned in <100> directions, lying on $\{110\}_{45}$ planes, inclined at 45° to the (001) surface. At higher mean contact pressure and larger indentation depth, however, dislocation pile-ups along <110> directions appeared, lying on $\{110\}_{90}$ planes, perpendicular to the (001) surface. These observations were qualitatively confirmed by corresponding direct Molecular Dynamics Simulations.

The dislocation etch-pits below the Berkovich indentations were digitized for quantification of local dislocation densities. The Geometrically Necessary Dislocation (GNDs) density below Berkovich indentations were calculated using HR-EBSD analysis. Both techniques (etch-pit and HR-EBSD analysis) show higher dislocation densities at lower indentation loads, which provide a direct experimental evidence for the physical basis of the ISE. Moreover, the etch pit analysis does also shed light on the extraordinary combination of high indentation hardness and low yield strength in STO, which can be attributed to the high dislocation density that is observed underneath the indentation.

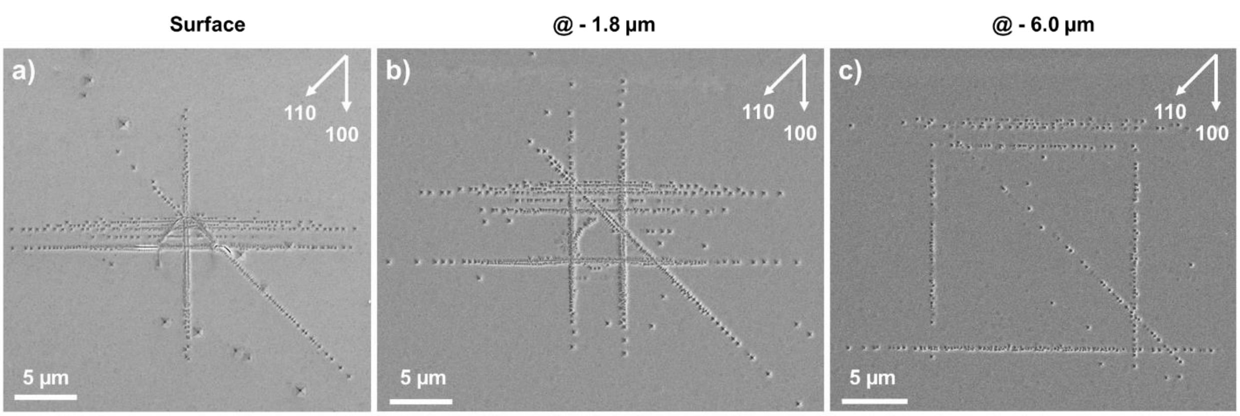

Figure 1 – SEM images of 25 µm tip radius spherical indentation (a) at the surface, (b) and (c) after removing 1.8 µm and 6.0 µm of material from the surface of the specimen, respectively.