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Characterization of Nanoindentations in Silicon by Cross-sectional TEM

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Diamond-anvil tests have shown that normal diamond-cubic Si transforms to the metallic β-tin structure (Si-II) at 11 GPa hydrostatic pressure or at lower pressures when aided by shear stresses. Upon decompression, the β-tin structure transforms to amorphous α-Si if the pressure release rate is high, or to a variety of metastable crystalline forms if the rate is low [1]. The hardness of Si exceeds the pressure needed to induce phase transformations; thus, metastable phases may be observed in and around hardness impressions [2]. The deformation mechanisms and resultant microstructures of indentations are relevant to a basic understanding of micromachining of Si. In this study, the structure of undoped (100) silicon near nanoindentations was examined by TEM of cross-sections prepared by dual-beam focused ion beam (FIB) milling. The indentations were made at room temperature with triangular pyramidal indenters with centerline-to-face angles of 35°, 45°, 55° and 65° and a load of 10 mN (Figure 1).

Near the indents there is a high degree of residual stress with significant rotation of the Si lattice, as evidenced by the strong bend contour patterns (Figure 2). With increasing indenter-angle, TEM reveals an increasing volume of amorphous material, whereas, as expected, the depth of the residual indentation decreases. The other main microstructural features present are cracks, dislocations, microtwins, and material extruded from the indent. These features were characterized by conventional TEM methods, including diffraction contrast and high-angle tilting experiments, on an FEI Tecnai 20. The cracks are more prevalent when indenters with low angles are used, may propagate deeper than 1 μm, change habit plane from nearly normal to nearly parallel to the surface at the greatest depth, and are "filled" with α-Si where they intersect the main amorphous pocket. Microtwins were detected by selected area diffraction (SAD) combined with dark-field (DF) imaging and by high-resolution electron microscopy (HREM) performed on a Philips CM200FEG. Microtwinning is most extensive under the apex of the amorphous pocket and at the amorphous-matrix interface (Figure 3a). Since the FIB-prepared TEM specimens are thicker than optimum for HREM, and the large residual stresses are mostly relieved by lattice rotations when the specimen is thinned, the details of the contrast cannot be easily interpreted. However, diffractograms clearly reveal local maxima at the one-third positions along 111 directions, characteristic of twins. Grains of crystalline Si with dimensions typically between 5 and 20 nm are observed by SAD/DF-TEM and HREM at the amorphous-matrix interface and within the amorphous material. During HREM observations, the α-Si crystallized into heavily twinned grains (Figure 3b), the transformation proceeding by the slow propagation of the crystalline-amorphous interface. This observation generates concerns that the nanocrystalline grains within the α-Si are just artifacts, possibly induced during the FIB preparation by displacement or beam heating effects similar to those that occur with the electron beam. The material extruded from the indent is sometimes α-Si containing nanocrystalline Si, or surprisingly, sometimes it is crystalline Si with almost the same orientation as the matrix. The deformation mechanisms involved in the formation of the microstructures and, in particular, evidence for the presence of metastable crystalline forms of Si are among the goals of continuing research [3].

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[2] G. M. Pharr, J. Mater. Res. 6 (1991) 1129.
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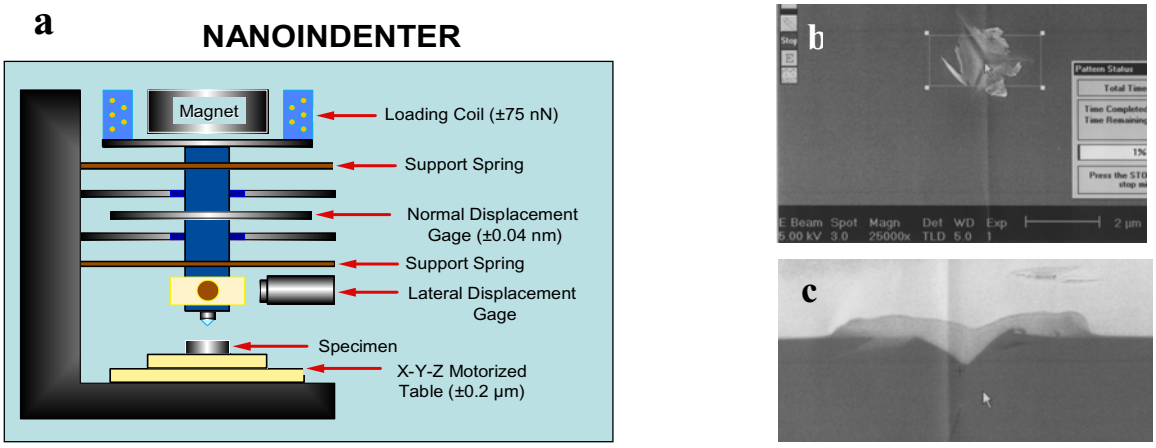

FIG. 1. Cross-section TEM specimen preparation of nanoindents. (a) Nanoindenter schematic, (b) Plan view of an indent selected for FIBing, (c) Cross-sectional view of the indent selected for FIBing

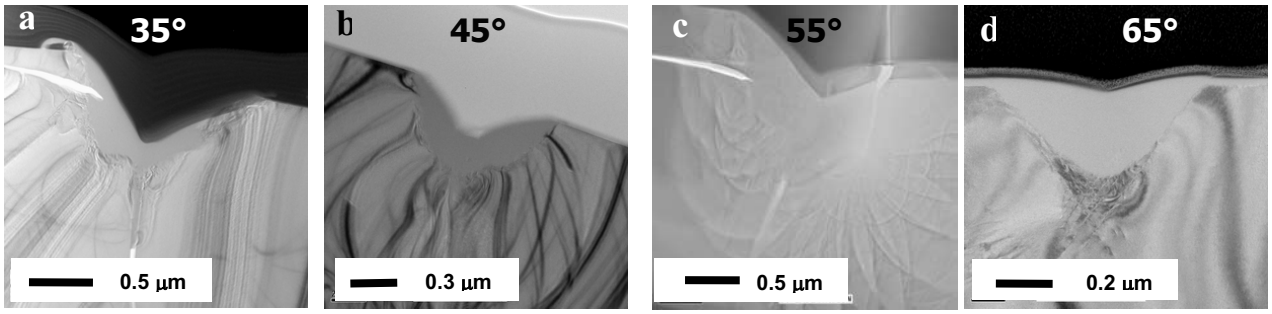

FIG. 2. TEM images of cross-sections of indents from (a) 35°, (b) 45°, (c) 55°, and (d) 65° indenters.

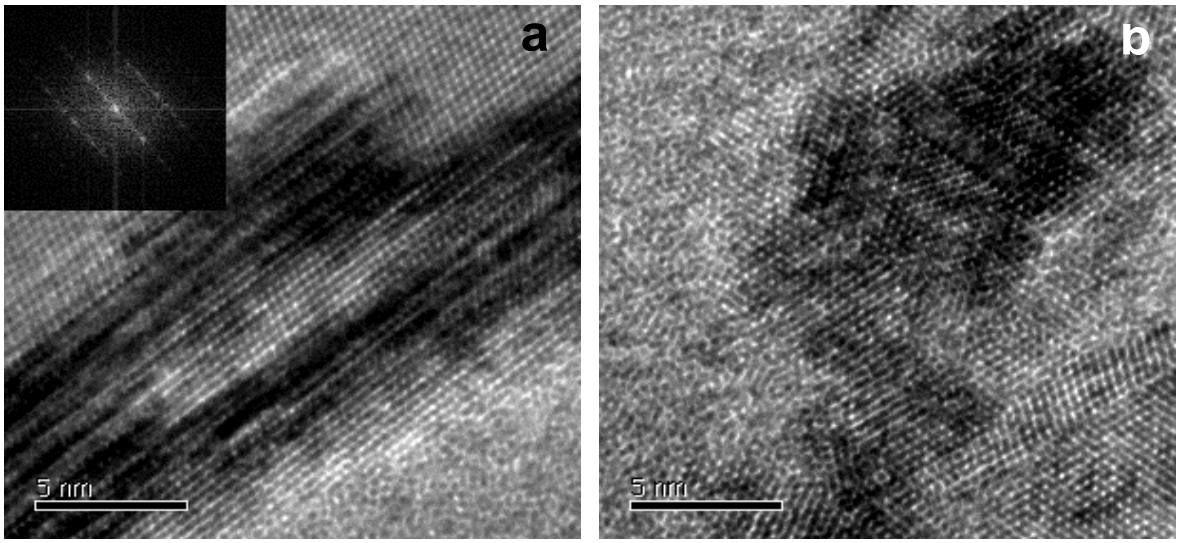

FIG. 3. HREM images from an indent made with a 65° indenter showing (a) 5 to 8nm-wide band of microtwins at the interface between α-Si (bottom-right) and the <110> oriented matrix, with Fourier Transform (diffractogram) inset, and (b) the interface between α-Si (left) and the crystallized Si growing under the action of the electron beam.