

Transmission Electron Microscopy of Indented and Scratched Titanium-Alumina Layers on Silicon

L.C. Whitmore¹, T. Koch², S. Abermann³, K. Whitmore¹ and A. Steiger-Thirsfeld¹

1. Service-Einrichtung für Transmissions-Elektronenmikroskopie (USTEM), Technische Universität Wien, Karlsplatz 8-10, A-1040 Wien
2. Institute of Materials Science and Technology, Technische Universität Wien, Favoritenstrasse 9-11, 1040 Wien
3. Institute of Solid State Electronics, Technische Universität Wien, Floragasse 7, 1040 Wien

whitmore@ustem.tuwien.ac.at

Keywords: indentation, cross-section, amorphous silicon, multi-layers

A 50nm alumina layer has been grown on boron(p)-doped (001) silicon substrates using Atomic Layer Deposition with a Savannah 100 ALD reactor from Cambridge NanoTech Inc. The precursors for the deposition were trimethylaluminum (TMA) and water. Substrate temperatures during ALD growth were set to 200 °C and purging times were adjusted to achieve layer by layer growth (for more details see [1]). A 50 nm titanium layer has been sputter deposited upon that. Real-life contact effects are simulated using nano-indentation and scratch tests. A Hysitron Tribo Indenter has been used to make indentations of between 100 nm and 200 nm depth and increasing-load scratch tests. Scanning electron microscopy (SEM) is used to study the surface effects of the tests, and transmission electron microscopy (TEM) is used to study the subsurface deformation in cross-section.

We are interested in the mechanisms of delamination of the surface layers from the substrate, and of deformation within the layer structure, induced by the indentation process. This knowledge is important for understanding and improving the processes of micro-machining of brittle materials as well as understanding chip failure in semiconductor device manufacture. Earlier work observed the production of amorphous silicon underneath indentations in silicon crystals [eg. 2, 3], as well as underneath the sliding diamond tool of single-point diamond turning and in multi-point grinding of silicon crystals [4].

A typical indentation is shown in cross-section in Figure 1. The image shows the deformed surface and the subsurface changes in material nature. The pale grey region is amorphous silicon as confirmed using EDX and electron diffraction. This contains small regions of crystalline silicon. Directly surrounding the amorphous zone are dense arrays of dislocations. Pile-up from the plastically deformed substrate around the indentation is found to initiate delamination of the surface layers. The interface between the amorphous zone and the silicon crystal is studied in detail to reveal the natures of transformation.

High resolution reveals more detail of the crystal-amorphous transition and the initiation of delamination of the surface layers. This is exemplified in Figure 2a. The diffraction analysis shows amorphous rings from the amorphous zone, clean (011) diffraction spots from the substrate, and elongated spots composed of many small diffraction spots from just beyond the amorphous zone, Figure 2b. This indicates the presence of poly-crystalline silicon within the dislocation region where the structure is disturbed but not amorphous.

Plastic deformation within the surface layers is found directly underneath the indenter. Phase changes from diamond silicon to bcc silicon and amorphous silicon are examined. Residual stress within the layers is investigated with micro-diffraction and high resolution.

1. O. Bethge, S. Abermann, C. Henkel, and E. Bertagnolli, *Thin Solid Films*, 10 (2009)
2. L.C. Whitmore, K.E. Puttick, *Cambridge International Indentation Workshop* 1996

3. I. Zarudi and L.C. Zhang, Tribology International 32 12 (1999) pp701-712
4. K.E. Puttick, L.C. Whitmore et al., Phil. Mag. 69 1 (1993) pp91-103

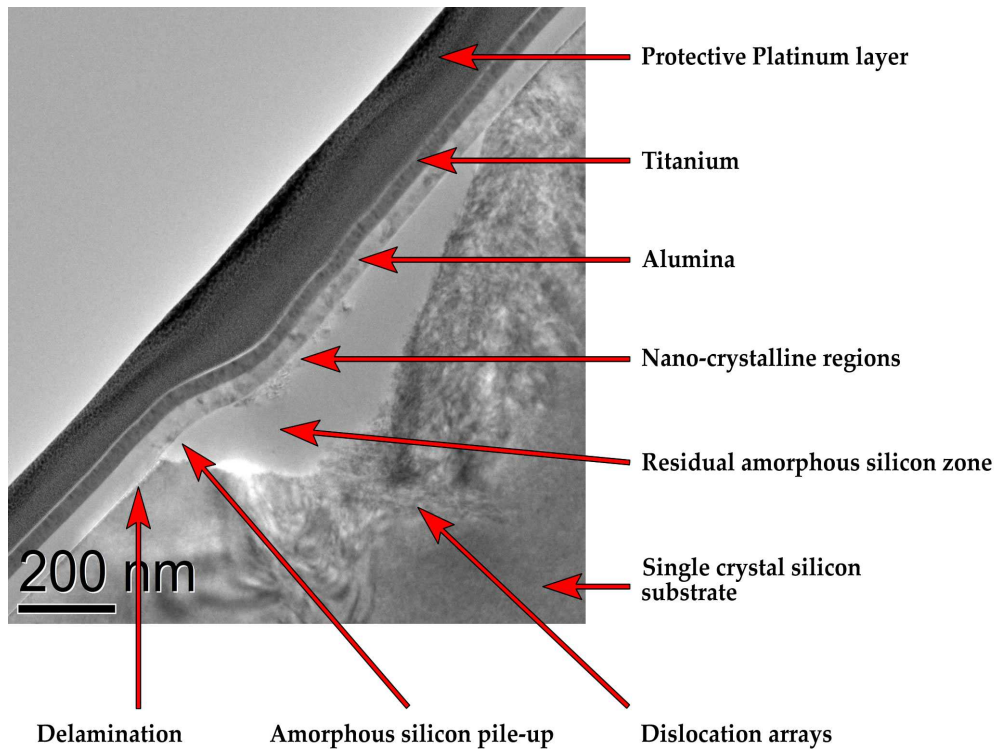


Figure 1. Cross-section image of an indentation shows amorphous silicon directly beneath the indenter impression, deformation of the surface layers and dislocations within the substrate. Initial delamination caused by pile-up is visible on the left side. The platinum layer is deposited to protect the surface during specimen preparation.

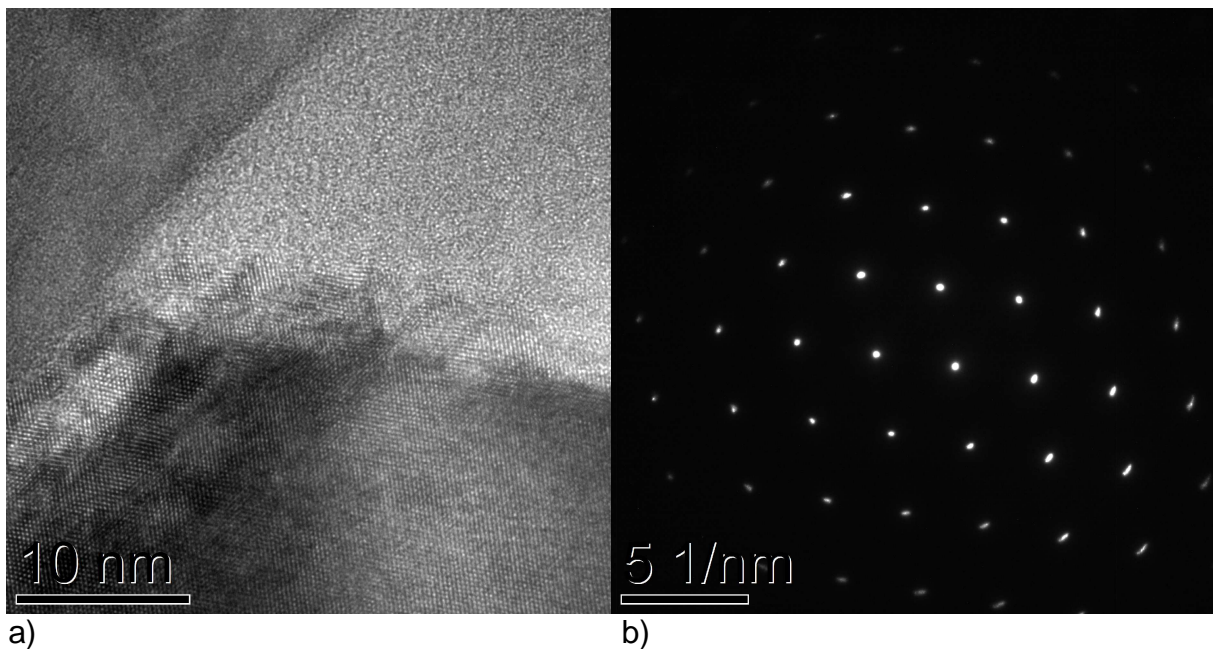


Figure 2. Shows a) high resolution image reveals the transition between crystal and amorphous silicon from top left of Figure 1, and b) diffraction pattern from the region of dense dislocations in Figure 1, showing break-up of the crystallinity in higher order spots.