Application of in situ SEM and TEM mechanical testing in development and optimization of hard coatings

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Hard coatings are now regularly used in various commercial applications but there is a constant pressure for further improvement of their properties. Although hard coatings exhibit incredible strength they commonly suffer from brittleness. This limitation is mainly caused by a lack of plasticity and low cohesive strength of grain boundaries, which favors grain boundary cracking. As a result the fracture toughness is low and cracking often results in catastrophic failure. In the case of bulk materials, fracture toughness is usually studied on notched macroscopic samples. The development of focused ion beam (FIB) technique allowed for the transfer of this concept to the micrometer and nanometer scale and its successful application to testing of coatings.

In this study we present various approaches for fracture toughness enhancement in hard coatings. *In situ* scanning electron microscopy (SEM) and transmission electron microscopy (TEM) indentation of FIB machined micro-cantilever beams was used for a comparison and visualization of differences in fracture behavior. Firstly, in case of grain-boundary designed zig-zag TiN we demonstrate a possibility to increase fracture toughness of ceramic nanostructured materials of more than 150% without loss of hardness by a dedicated grain boundary orientation design with respect to the direction of the expected crack path. Secondly, mechanical tests performed on microcantilever beam specimens of multilayered TiN/SiO_x thin coatings showed that the fracture toughness of this hierarchical, microstructurally and mechanically heterogeneous material can be enhanced up to 60% with respect to either of its single-layered constituents, which is attributed to a large difference in their elastic modulus. Thirdly, self-organized multilayered TiAlN coatings displayed complex fracture behavior based on their hierarchical 3D arrangement which results in greatly enhanced fracture toughness. In this case *in situ* SEM and TEM techniques were combined to obtain an overall picture of fracture behavior.

Our work demonstrates a practical way for obtaining mechanical properties of non-bulk materials using a combination of FIB sample preparation, *in situ* electron microscopy and mechanical testing. The three examples suggest a general approaches for fracture toughness enhancement of hard coatings.

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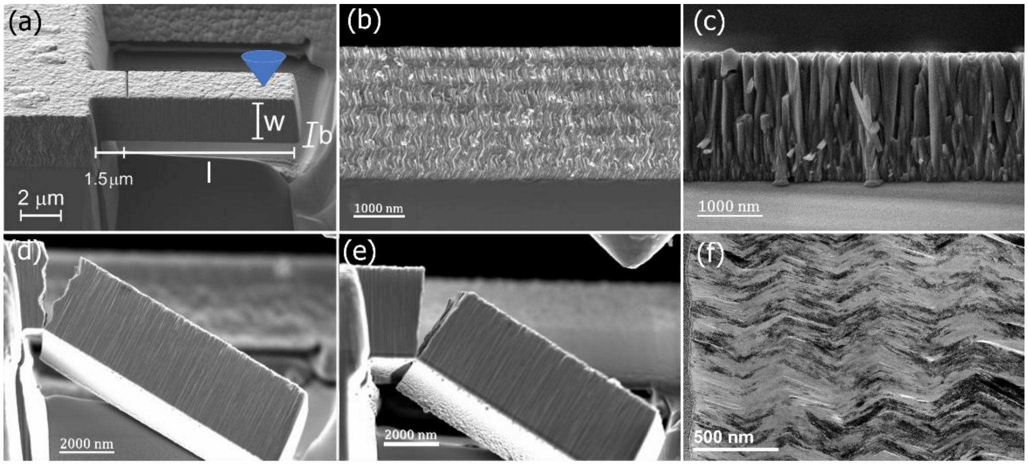

Fig. 1 (a) SEM micrograph of a representative microcantilever beam specimen used for micro-bending testing. Important dimensions are highlighted. (b) Cross-sectional micrograph of a TiN coating with chevron-like microstructure consisting of 12 layers. (c) Cross-sectional micrograph of a reference TiN coating with columnar microstructure. (d) A tested microcantilever of TiN coating with chevron-like microstructure with inclined crack path. (e) A tested microcantilever of reference TiN coating with straith crack path. (f) BF TEM micrograph of the chevron-like microstructure.

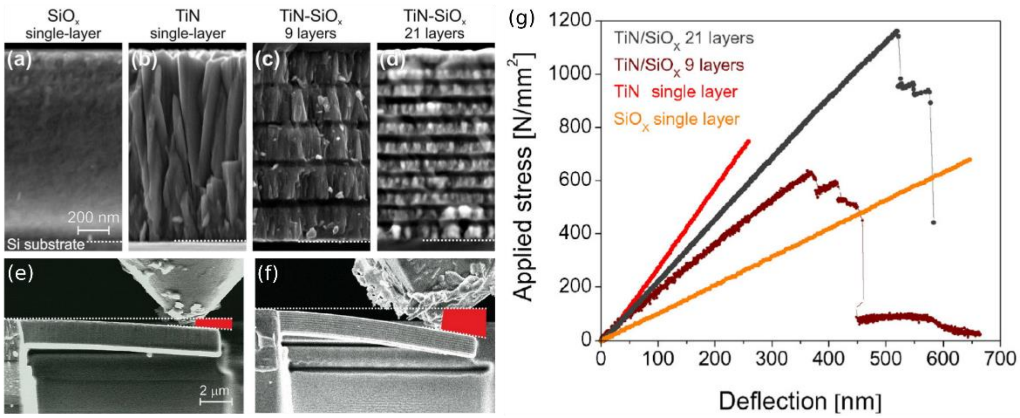

Fig. 2 (a) Cross-sectional SEM micrograph of compared coating systems. (a) SiOx single layer, (b) TiN single layer, (c) TiN/SiOx multilayers (9 layers), (d) TiN/SiOx multilayers (21 layers). (e) The maximum TiN single layer and (f) TiN/SiOx multilayers (21 layers) microcantilever deflection prior fracture. (g) Representative stress-deflection curves of individual samples.

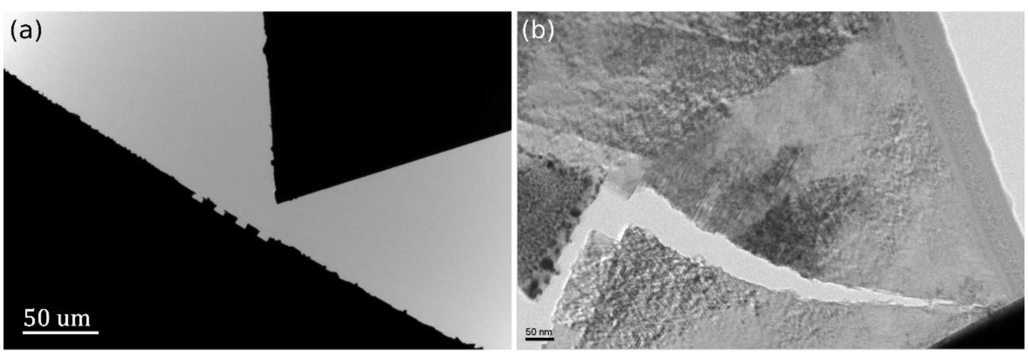

Fig. 3 (a) Low-mag TEM image of a wedge indentor close to sample positions. (b) BF TEM micrograph of a tested sample.