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Correlation of the nanoindentation induced pop-in effects with SEM and TEM observation of nanocomposite nc-TiC/a-C:H coatings

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In the present work a set of nc-TiC/a-C:H coatings was prepared using magnetron sputtering of a titanium target in an acetylene-containing atmosphere [1,2]. The acetylene flow was varied to obtain films with different chemical compositions with carbon content ranging from 30 at.% C to 68 at.% C. The thicknesses of the prepared films were in the range from 5 to 7 μm.

Nanoindentation tests were used to study the mechanical properties of the film/substrate system from near surface up to film-substrate interface. Indentation loads ranging from 20 to 1000 mN were applied to each sample to obtain the hardness and elastic modulus as a function of indentation depth. The highest hardness and Young's modulus of (46±2) GPa and (415±5) GPa respectively was reached for films with the Ti/C ratio of approximately 3/4.

In case of films with complex structures (different phases, micro- or nanocrystallites, columnar structure, differences in grain orientation) measurements at the low depths are giving a large scatter because the indentation response corresponds with the material structure. The resulting load-penetration curves contain a lot of information about the tested material. We can visualize them using the so called differential hardness curves. The differential hardness H_{dif} ($H_{dif} = k\partial L/\partial(h^2)$, where L is the load, k is geometric constant and h is the indentation depth) represents the ratio of the small load increase and the corresponding change in the square of the indentation depth (i.e. change in contact area of the indenter and the tested material). This value is very sensitive to any changes occurring during indentation test. The H_{dif} dependence on the indentation depth may be used to visualize the increase of the substrate influence, moreover it can be used to determine the depth of indentation induced crack creation or film delamination. The extended evaluation of H_{dif} curves can give us image about the grain boundaries, layered structures, pressure induced phase transformations etc. In order to study the indentation induced deformation and cracking indentation tests made with maximum load of 1N. In Figure 1 an example of SEM image of an indent performed using a load of 1N on film with the highest hardness is shown in both secondary electrons and backscattered electrons signals. The corresponding loading-unloading curves and the differential hardness H_{dif} on the indentation depth are shown in Figure 2. The crack lines inside the indentation print correlate with the pop-ins on the loading curves. Using the differential hardness curves it is possible to determine the depths of the crack creations. The first crack appeared at indentation depth around 850 nm. The crack creation started when the titanium interlayer influence became dominating. The single crystal titanium hardness (H_{Ti}=1 GPa) and elastic modulus (Y_{Ti}=129 GPa) are substantially lower than those of both the coating and the stainless steel substrate. The fracture did not spread further, it was observed only inside indentation prints. TEM results obtained from the lamella taken from the surroundings of the indentation print did not show any cracks in the material structure. The diffraction studies revealed that the deformation was proceeded by rearrangement of crystallites within the a-C:H matrix.

Details of microstructure in the vicinity of the indentation print were studied using a Philips CM12 STEM transmission electron microscope on thin lamellas prepared using focused ion beam (FIB) technique in a LYRA 3 XMU FEG/SEM×FIB by Tescan.

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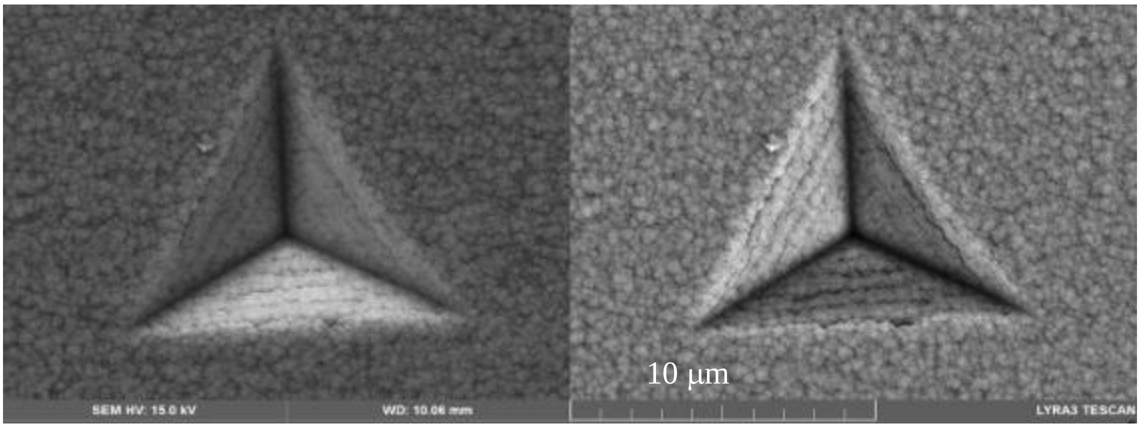

Figure 1. Indentation print made with 1N on tested coating (signal of SE on the left, BSE on the right.)

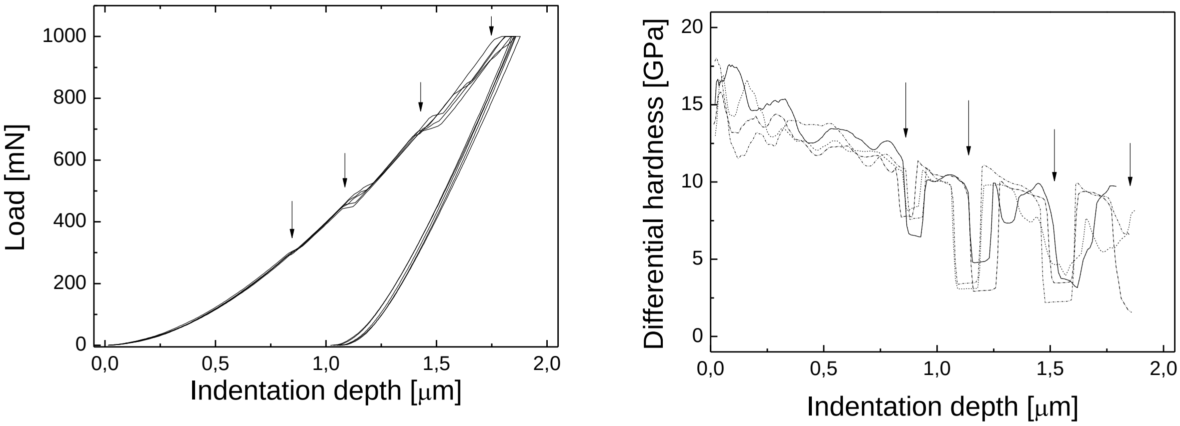

Figure 2. Loading and unloading dependences (on the left) on the indentation depth made with maximum load of 1N and the corresponding differential hardness dependences on the indentation depth (on the right). The arrows indicate the crack creation in the inner part of the indentation print.