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TEM investigation of droplets in TiAlN based coatings

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Wear resistant coatings are widely used to improve the performance and lifetime of cutting tools. Often, these coatings are grown by cathodic arc evaporation, where a target is evaporated by a cathodic arc, thus creating a vapor phase which condenses together with reactive process gases on the substrates to be coated. An inherent drawback of this process is the formation of droplets, which are emitted from the target and incorporated into the coating, representing defects. These droplets affect the cutting performance as they act as diffusion paths, negatively influencing the oxidation resistance, as nuclei for cracks, and they increase the surface roughness of the coating.

The aim of the present work was to determine structure and chemical composition of droplets within a ~3 µm thick TiAlN based coating and a ~300 nm thick TiN base layer deposited onto cemented carbide substrates. Possible crack and oxidation triggering effects of these droplets have been determined for coatings loaded by dry sliding ball-on-disk tests performed at room temperature and at 700°C in ambient air. Coatings were investigated by transmission electron microscopy (TEM), where cross-sectional samples have been prepared within the wear tracks formed. Lamellas for TEM were prepared by means of an Orsay Physics Cobra Z-05 focused ion beam instrument (FIB). The TEM investigations were conducted using a monochromated TF20 TEM, Schottky cathode, operated at 200 kV, equipped for energy-dispersive X-ray spectroscopy (EDX) with a SiLi detector and an ultrathin window. For electron energy-loss spectroscopy (EELS) and energy-filtering (EFTEM), the TEM is equipped with a high-resolution Gatan imaging filter (GIF). All EELS measurements were recorded in scanning mode of the TEM using a probe diameter of about 1 nm.

Figure 1(a) shows a bright-field image of a part of a droplet in the room temperature sample. The EFTEM O K jump ratio image in (b) indicates oxygen-enriched areas in the under-dense regions surrounding the droplet, which originated from shadowing effects during film growth. EFTEM reveals further that the droplet is rich in titanium, within regions which contain more nitrogen (c) and less titanium (d) than the surroundings. In the RGB image, an oxidized area is seen (top right in (e)), which indicates oxidized wear debris formed in the dry sliding test. All EFTEM results have been confirmed by means of EDX and EELS (not shown). An SAED pattern (f) was taken from the nitrogen-rich region marked in the bright-field image (a), which could be clearly interpreted as the -titanium phase (ICSD 43416) in [001] orientation.

The bright-field image for the 700°C sample is shown in Figure 2(a). In contrast to Figure 1, there is more oxygen detected in the surroundings of the droplet (see (b) and (e)). Titanium and nitrogen are homogenously distributed (c, d). The SAED pattern acquired from the marked region in the bright-field image (a) agrees well with $Ti_4N_{2.33}$ (ICSD 40951) in orientation.

In summary, the performed investigations revealed droplets consisting of a metallic Ti-rich core including nitrided areas originating from the nitrogen within the reactive deposition atmosphere. Cavities between droplets and genuine coating material were identified as oxidation sites and are more pronounced for the 700°C dry sliding test. Further, material accumulations were identified as worn coating material, which has also been oxidized within the tribological contact.

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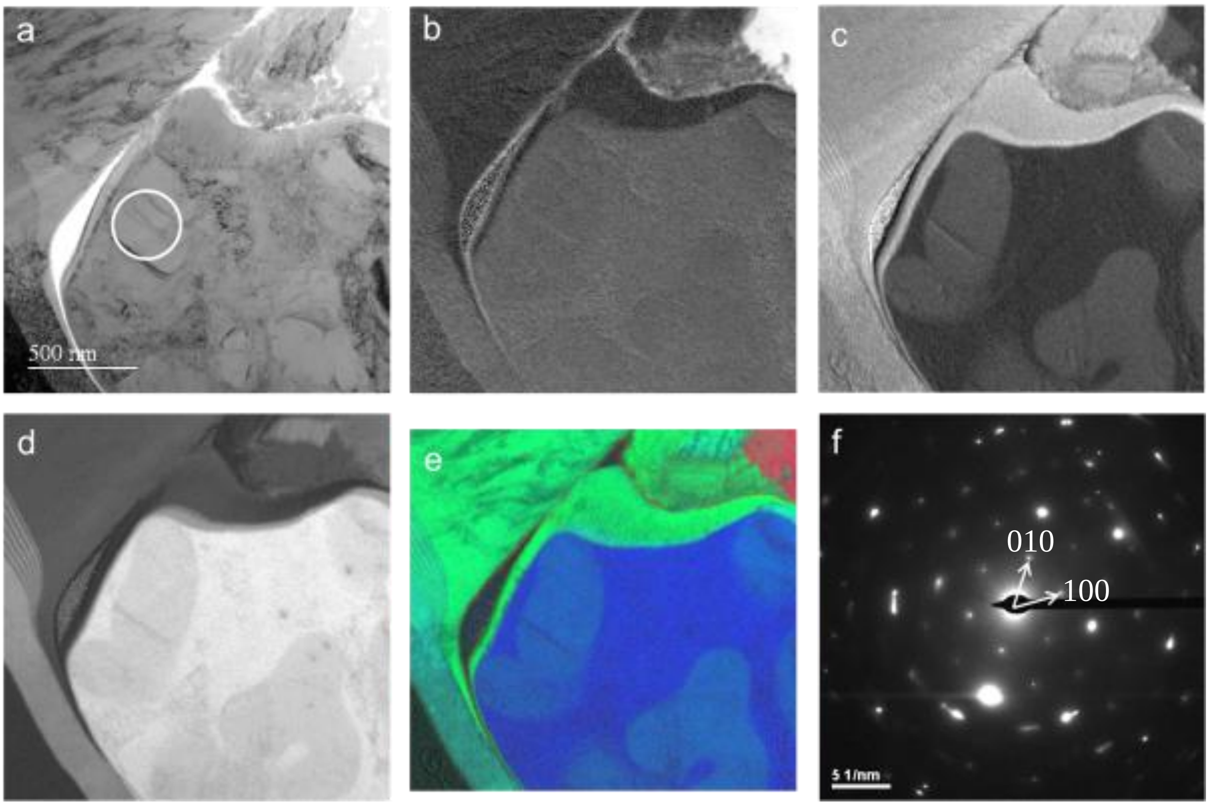

Figure 1. TEM micrographs of room temperature sample. (a) bright-field image, (b) O K jump ratio image, (c) N K jump ratio image, (d) Ti $L_{2,3}$ jump ratio image, (e) RGB image: red=O, green=N, blue=Ti, (f) SAED of marked region in (a).

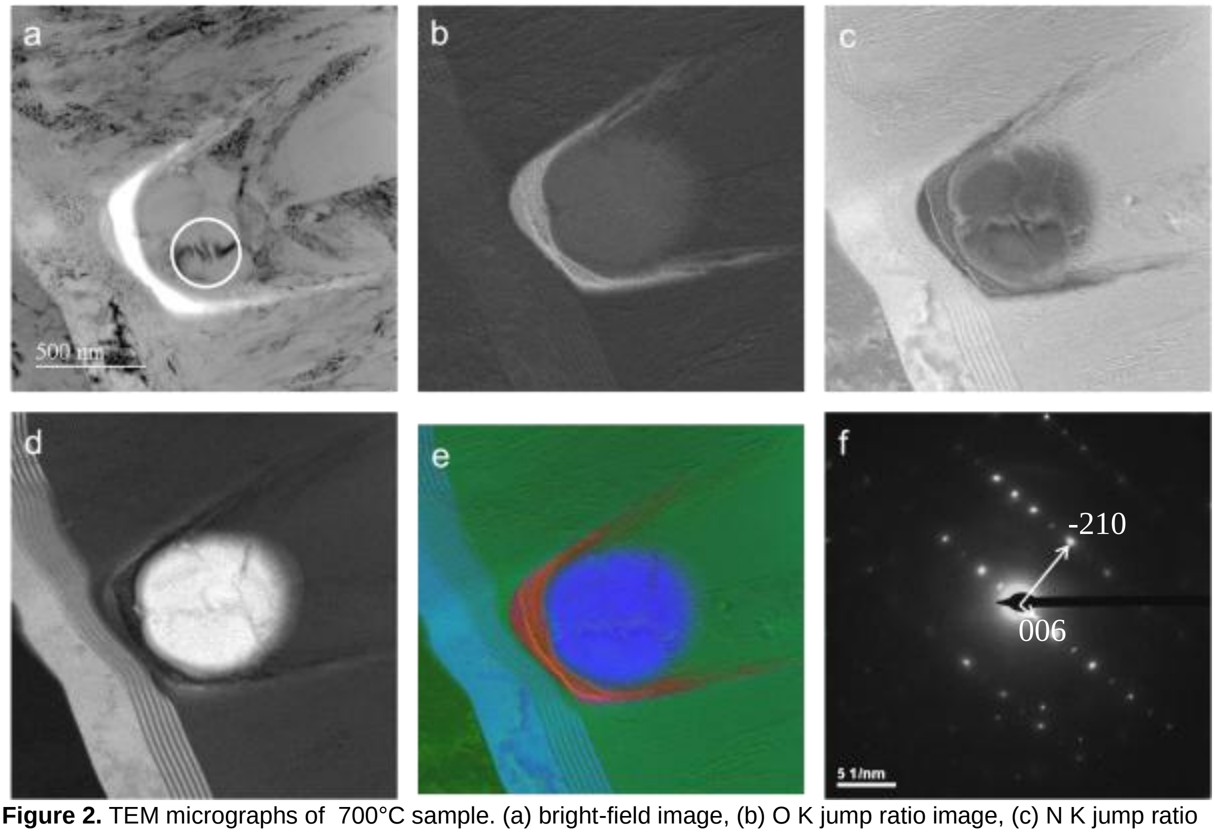

Figure 2. TEM micrographs of 700°C sample. (a) bright-field image, (b) O K jump ratio image, (c) N K jump ratio image, (d) Ti $L_{2,3}$ jump ratio image, (e) RGB image: red=O, green=N, blue=Ti, (f) SAED of marked region in (a)