Disordered alloyed alumina thin films as wear-resistant coatings – a TEM and FIB study.

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Many different alumina phases and ceramic nanostructures, e.g. nano-layers or nano-composites have been successfully synthesized to improve the thermal and mechanical properties of wear-resistant coatings. In this work chromium alloyed alumina thin films were prepared by dual magnetron sputtering deposited on tungsten carbide (WC) substrates. Thin films were investigated by TEM in plan view and cross-section to show a strongly disordered nanostructure, which has not been described before. Micro-indenting of the films was done with subsequent FIB and SEM analysis to demonstrate the improved mechanical properties as a consequence of the nanostructure.

Figure 1a-c shows typical bright and dark-field images of the alumina thin film in plan view, Figure 1d is the corresponding selected-area diffraction pattern. Lattice spacings were measured from the diffraction pattern and are listed in table 1. Note that only d_{hkl}-spacings smaller than 0.2 nm appear and that for smaller scattering angles no reflections are observed rather an intensity distribution typical for amorphous structures is found. Also diffracted intensities are heavily damped for larger scattering angles and the observations do not match with any of the known alumina phases. Both observations indicate a poor long-range order and, therefore, we call this structure disordered. The largest d_{hkl}-value of about 0.2nm suggests a smaller unit cell than alpha or gamma alumina. It was shown by the dark-field image fig. 1c that the whole area contributes to the diffuse part of the diffraction pattern, however, individual grains contribute to the reflections forming a ring like structure (Figure 1b) and the grain size was identified as 50 nm. EELS was used to investigate the disordered structure by acquiring spectra with the objective aperture centered over the transmitted beam and centered in the diffuse part of the diffraction pattern (Figure 2). The goal was to identify atoms that contribute preferentially to the disorder and a more quantitative evaluation of this study will be presented.

Quantitative micro-indenting was used to analyze the local mechanical properties of the alumina films. It was applied to alloyed alumina thin films deposited with different stoichiometries, thicknesses and deposition technologies. Detailed results will be presented elsewhere. Here we show FIB-prepared cross-sections of micro indents.

The SEM FIB cross section (Figure. 3) shows the large plastic deformation of the 1µm thick alumina film. No cracks directly below the tip of the indentation are visible and the circumferential cracks have a maximum length of just 200nm normal to the surface. The disordered film seems to have a reduced brittleness and reduced crack propagation as compared to alumina films with a crystalline structure not showing the observed disorder.

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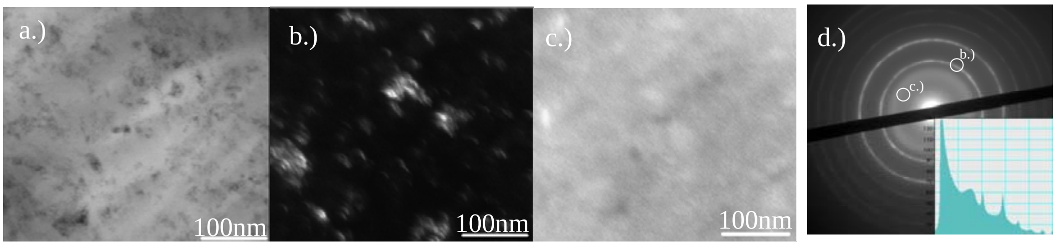

Figure 1. TEM images of an $(AlCr)_2O_3$ PVD deposited alumina coating in plan view: a.) bright-field image, b.) centered dark-field image with the objective aperture positioned at the first diffraction ring, c.) centered dark-field image with the objective aperture centered in the amorphous part of diffraction pattern. d) Selected-area diffraction pattern showing the position of the apertures in dark-field images.

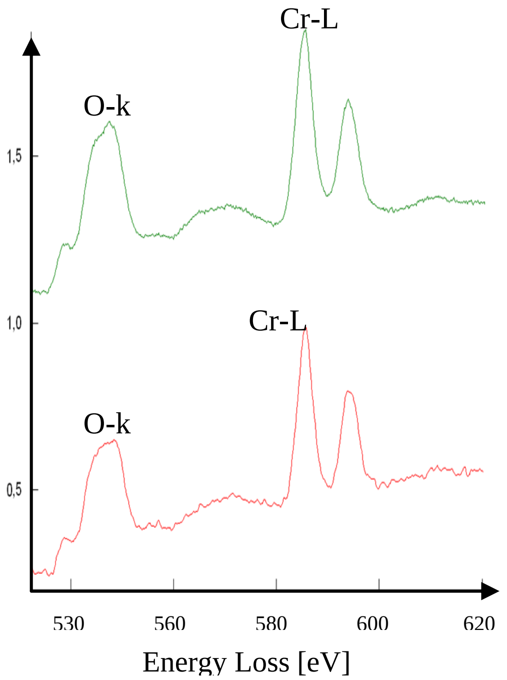

Figure 2. Electron energy-loss spectra of the $(AlCr)_2O_3$ PVD coating. The upper spectrum is acquired with the objective aperture centered over the transmitted beam. In the lower spectrum the objective aperture was placed on position c, Figure 1d)

No.	1	2	3	4	5	6
d [nm]	0.2	0.14	0.12	0.10	0.09	0.08

Table 1. d-values from the electron diffraction pattern (Figure 1d)

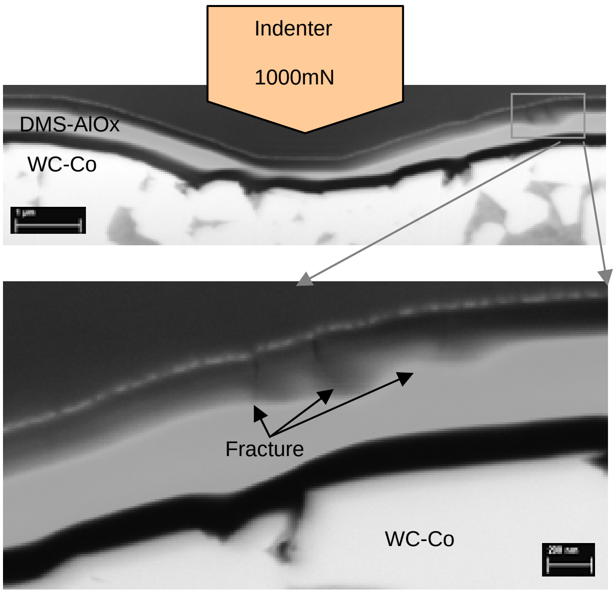

Figure 3. FIB Cross section image of a micro indent shows cracks at the edge of the indent.

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