X-ray characterization of a TiAlN-coating on a mixed ceramic substrate with a resolution of a few microns

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Mixed ceramic cutting tools for finishing of hardened steels are widely-used due to high wear and temperature resistance. Recent developments have shown that surface modification or coating of mixed ceramic inserts by physical vapor deposition (PVD) leads to an enhanced cutting performance [1,2,3]. In order to improve coating adhesion and wear resistance, characterization of coating properties is inevitable in terms of 'residual-stress-engineering' [4]. At the Hamburg University of Technology mixed ceramic cutting tools of Al_2O_3-Ti(O,C)-ZrO_2 are fabricated via aluminothermic reaction sintering followed by an annealing process to form an in-situ TiC-layer on the surface [5]. This TiC-layer serves as an adhesion promoter for an additional TiAlN-coating applied through magnetron sputtering. Thicknesses of the TiC-interlayer and the TiAlN-coating are about 1 - 2 microns each. In order to characterize these coating layers by x-ray diffraction, first experiments were carried out at PETRA III. Results showed that it was possible to separate the diffraction spectra of the coating layers from the spectra of the bulk ceramic material.

Diffraction experiments were performed at the HEMS beamline P07 of PETRA III at HASYLAB using monochromatic synchrotron radiation with an energy of 91 keV, corresponding to a wavelength of 0.1363 A. Diffraction patterns were recorded using a 11 megapixel VHR CCD camera (Photonic Science Ltd., UK) with an input pixel size of 31.18 x 31.18 µm and a sample-to-detector distance of 173 mm. Additionally, a VORTEX-EM® fluorescence detector (Seico Instruments Inc., USA) was installed to collect backscattering fluorescence spectra of the sample.

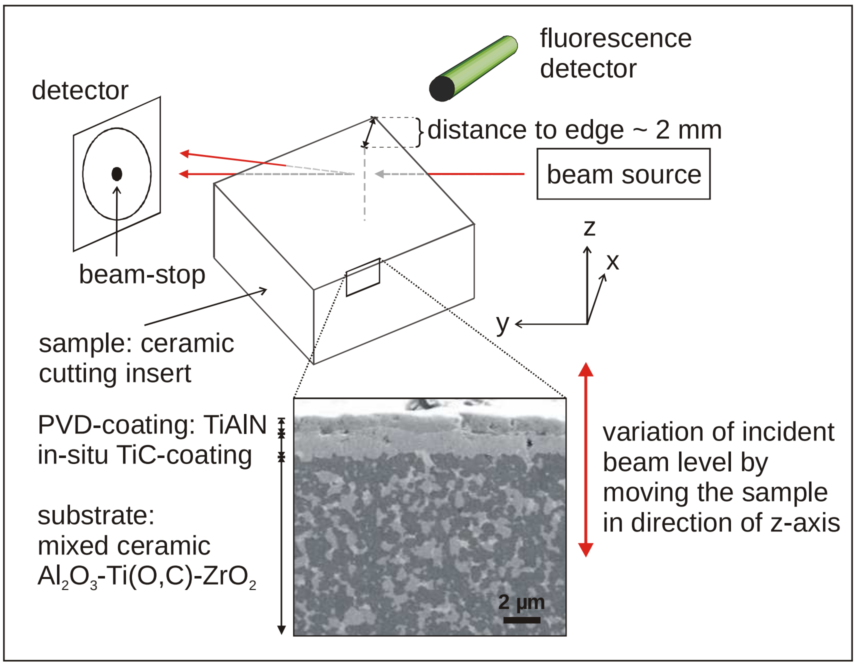

Figure 1: Schematic display of the experimental set-up.

The beam size of the synchrotron radiation was defined to 10 x 10 µm^2 using an optical lens system. The sample used in the experiments was a mixed ceramic cutting tool with an edge length of ~ 12 mm and height of ~ 7 mm according to standard geometry (SNGN 120708 T010020). For alignment in the incident beam the sample was mounted on a stage which could be moved in x-, y-, and z-axis (see Fig. 1). The beam passed the sample at a distance of ~ 2 mm in direction of the x-axis relative to the sample edge. The sample was then shifted in z-direction until the top coating layer of the sample was solely exposed to the beam. It was possible to distinguish the coating from the bulk because of vanishing zirconium peaks from the bulk ceramic and increasing titanium peaks from the TiAlN- and TiC-coating in the fluorescence spectra (see Fig. 2a). In Figure 2b diffraction spectra also show that in the surface layer of the material no zirconia and only small amounts of alumina were detected in comparison to the bulk material, thus indicating the exposition of only the surface layers to the beam.

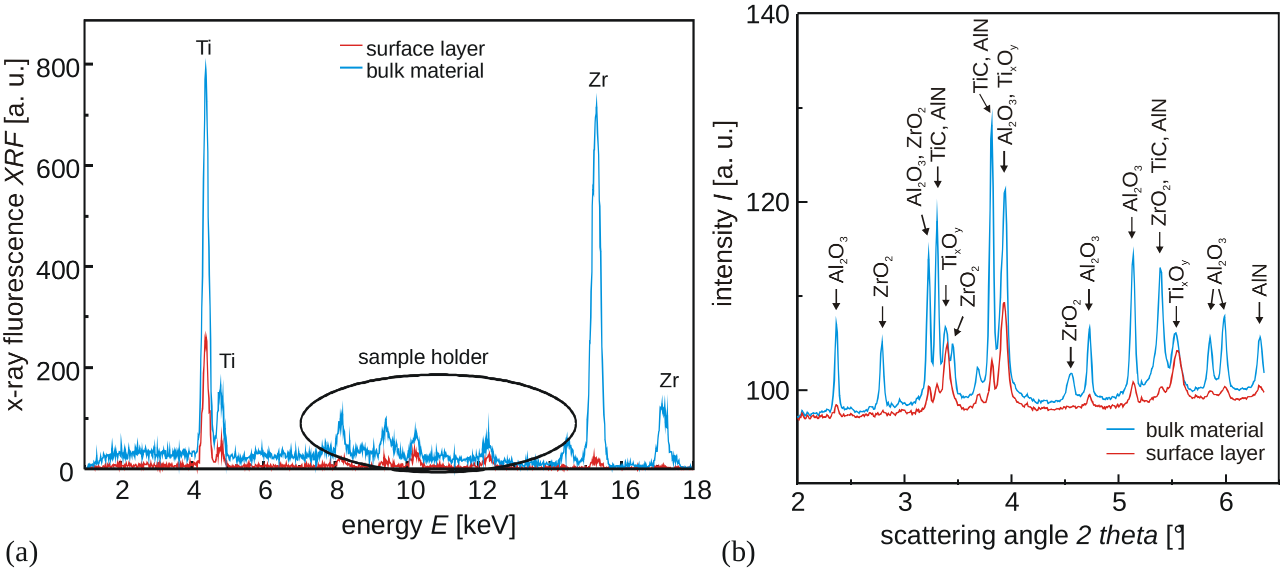

Figure 2: Fluorescence spectra (a) and diffraction patterns (b) of the bulk and the coating of a mixed ceramic cutting tool material.

These results show that with the experimental set-up described above even coatings with only a few microns in thickness can be characterized separately from the bulk material. For further characterization of the coating layers, properties like lattice matching to the substrate and internal stress distribution will be investigated.

References

[1] A. K. Bhattacharya, K. Zimmermann, G. A. Schneider, and W. Hintze, J. Am. Ceram. Soc. **91**, 2982 (2008).

[2] J. Kundrak, K. Schmidt and J. Prilukova, Spanen und Werkzeuge in technologischen Systemen. **76**, 85 (2009).

[3] B. Hüttermann: Anwendung des gepulsten Magnetronsputterns zur Beschichtung keramischer Hochleistungswerkstoffe, Shaker Verlag, Aachen (2005).

[4] M. Klaus: Röntgendiffraktometrische Ermittlung tiefenabhängiger Eigenspannungsverteilungen in Dünnschichtsystemen mit komplexem Aufbau, Universitätsverlag der TU Berlin, Berlin (2009).

[5] K. Zimmermann, G. Schneider, A. Bhattacharya and W. Hintze, J. Am. Ceram. Soc., **90**, 3773 (2007).