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Investigation of hard gradient PVD (Ti,Al,Si)N coating

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Materials

ABSTRACT

Purpose: Investigation of gradient coating of (Ti,Al,Si)N deposited on the Al_2O_3+$SiC_{(w)}$ oxide ceramics substrate by cathodic arc evaporation CAE-PVD method.
Design/methodology/approach: Structure of substrate and coating was investigated with use of scanning electron microscopy (SEM). The X-Ray Photoelectron Spectrometry (XPS) examination was carried out for proving the gradient character of the (Ti,Al,Si)N coating. The investigation includes also microhardness and roughness tests of the deposited coating and used substrate; The R_a surface roughness parameter measurements were made on confocal microscope.
Findings: Gradient structure and main properties of the investigated materials were introduced. It has been stated, that properties of the oxide tool ceramic with gradient (Ti,Al,Si)N coating increase in comparison with uncoated material.
Practical implications: Depositing the wear resistant gradient coating onto the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic results in a significant increase of the surface layer microhardness, contributing most probably in this way in machining to the decrease of the wear intensity of cutting tools' flanks made from the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic.
Originality/value: Functionally gradient coating form is a new class of structures in which the microstructure and properties vary gradually from the surface to the interior of the material.
Keywords: Gradient coatings; XPS; PVD; Oxide ceramics; Whiskers

1. Introduction

Ceramic materials with high hardness and high strength in the broad range of working temperatures and with low abrasion wear, with the Al_2O_3 based ones among them, are used more and more often for cutting tools. Ceramic tools containing the SiC whiskers have the life exceeding other tool materials, allowing at the same time very high cutting speeds. One of the disadvantages of the reinforced ceramics is decay of whiskers in case of machining alloys containing iron, which significantly limits its use [1-3, 7, 8, 14, 15,].

Functionally gradient coating form is a new class of structures in which the microstructure and properties vary gradually from the surface to the interior of the material. Gradient coatings can be deposited onto cutting tools made as well from the high speed steels, cemented carbides, cermets, and also from ceramic materials. PVD processes are used for deposition of the thin wear resistant coatings. In the development of new, contemporary materials the functionality is often improved by combining materials of different properties into composites. Coating composites are designed to specifically get better properties such as tribological, electrical, optical, electronic, chemical and magnetic [4-6, 9-11].

(Ti,Al)N coatings are characteristic of high hardness, good wear-resistance and excellent high-temperature properties. Therefore, (Ti,Al)N coatings have become popular as hard coatings for tools in recent years [11-13].

In this paper the gradient coating of (Ti,Al,Si)N deposited on Al_2O_3+$SiC_{(w)}$ oxide ceramics is investigated.

2. Experimental procedure

Experiments were carried out on Al_2O_3+$SiC_{(w)}$ oxide ceramics using the PVD method of deposition from the gaseous phase in the cathodic arc evaporation process with gradient coating (Ti,Al,Si)N. Specifications of the investigated materials are presented in Table 1.

The R_a surface roughness parameter measurements of the developed coatings were made on LSM 5 PASCAL confocal microscope.

The Vickers microhardness was measured on the Hanemann tester. The tests were made with the load of 0.9 N, making it possible to eliminate to the greatest extent the influence of the substrate material on the measurement results.

Structure and surface of the developed coatings was examined on the transverse fractures with use of SUPRA 25 scanning electron microscope (SEM). To obtain the fracture images the Secondary Electrons (SE) detection method and In-Lens detection one were used with the accelerating voltage in the range of 15-20 kV and maximum magnification 100 000x.

The X-Ray Photoelectron Spectrometry (XPS) examination was carried out for proving the gradient character of the (Ti,Al,Si)N coating deposited onto Al_2O_3+SiC substrate. The XPS technique has been chosen because of highly surface specific achieved due to the short range of the photoelectrons that are excited from the solid. The energy of the photoelectrons leaving the sample are determined using a two metal hemispheres (CHA) and this gives a spectrum with a series of photoelectron peaks. The binding energy of the peaks are characteristic of each element. The peak areas can be used (with appropriate sensitivity factors) to determine the composition of the materials surface. The shape of each peak and the binding energy can be slightly altered by the chemical state of the emitting atom. The schematic diagram of the X-ray photoelectron spectrometry has been shown on Fig. 1. The investigations has been carried out with use of the PHI 5700/660 PHILIPS spectrometer.

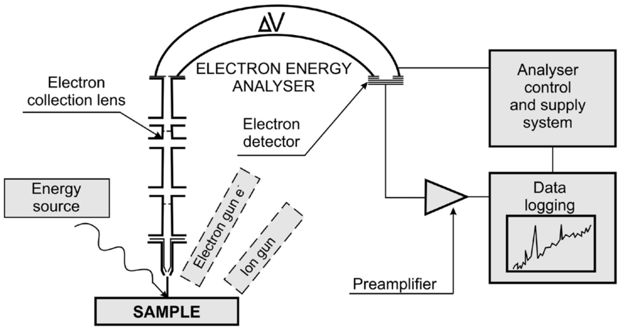

Fig. 1. Schematic diagram of X-Ray Photoelectron Spectroscopy (XPS)

3. Results

Ceramics with whiskers are obtained mostly by the hot pressure isostatic sintering and by the uniaxial hot pressing. The increase of the whiskers portion to 15-20% causes increase of the ceramics strength. Further increase of the SiC whiskers portion deteriorates the mechanical properties because of the increased probability of the development of whiskers agglomerates, featuring the cracking propagation source.

Occurrence of whiskers with the diameters of d=0.5-0.8 µm in the Al_2O_3+$SiC_{(w)}$ tool oxide ceramics sinter was revealed during examinations on the electron scanning microscope, reinforcing the structure and thus ensuring brittle cracking resistance due to the crack bridging process (Figs. 2, 3).

Table 1.
Specifications of the investigated materials

Coating	Thickness, µm	Roughness, µm	Microhardness, HV
uncoated	-	0.07	1890
gradient (Ti,Al,Si)N	2.6	0.18	2650

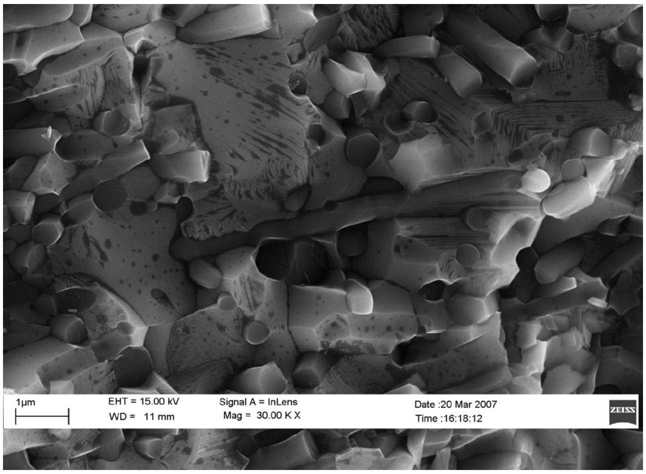

Fig. 2. Structure of the Al_2O_3 oxide tool ceramic reinforced with SiC whiskers

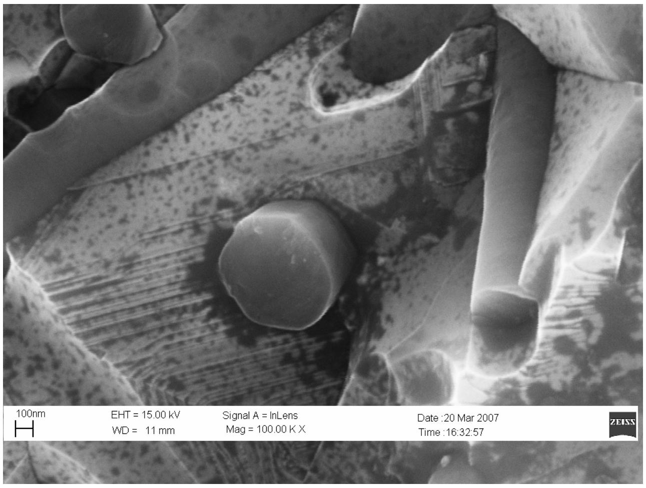

Fig. 3. Structure of the Al_2O_3 oxide tool ceramic reinforced with SiC whiskers

The fractographic examinations that have been carried out give on ground to state that the coating was put down uniformly onto the investigated substrate material and that coating adheres tightly to the substrate (Fig. 4).

Roughness of the substrate defined by R_a parameter is 0.07 µm. Deposition of the gradient (Ti,Al,Si)N coating onto the examined substrate causes increase of the roughness parameter to R_a=0.18 µm (Table 1). Increase of the roughness parameter is connected with occurrence of the drop shaped micro-particles on the surface of coating. The occurrence of the droplets is most probably connected with titanium micro-particles dropping out immediately after completion of the coating deposition process (Fig. 5).

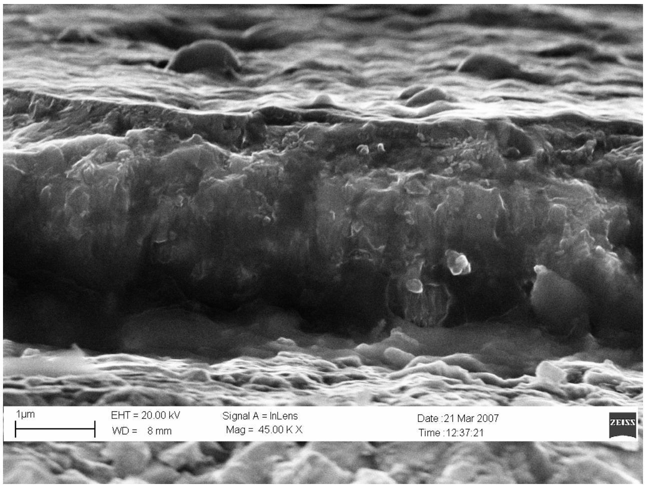

Fig. 4. Fracture surface of the gradient (Ti,Al,Si)N coating deposited onto the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic

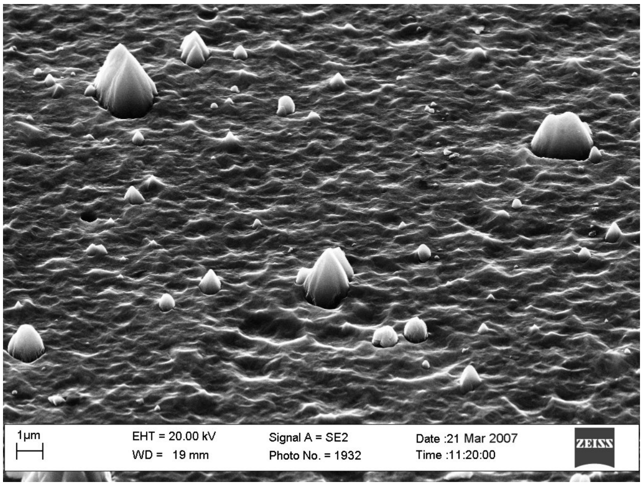

Fig. 5. Topography of the gradient (Ti,Al,Si)N coating surface, deposited on the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic

Depositon of the wear resistant gradient coating onto the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic results in a significant increase of the surface layer microhardness, contributing most probably in this way to the decrease of the wear intensity of cutting tools' flanks made from the Al_2O_3+$SiC_{(w)}$ oxide tool ceramic (Table 1).

During the the X-Ray Photoelectron Spectroscopy (XPS) investigations $Al_{K\alpha}$ monochromatic beam of X-ray radiation was used. Depth profile analysis was obtained after 1000 min of sputtering by Ar+ ions at the accelerating voltage of 2 V (Fig. 6). In one-one-minute cycles of sputtering ion gun scanned the area of square. Side of a square was 2 mm. After each of the cycles the measurement of characteristic peaks of nitride, titanium, aluminium, silicon, carbon and oxygen connected with atomic chemical composition analysis was made in area of the 0.4 mm circle in the centre of scanned square. Two surface spectra in different areas (0.4 mm circle and 2 mm x 0.8 mm rectangle) were made after depth profile analysis (Figs. 7, 8). In spite of the meaningful difference of analysed areas of the surface spectra there is no significant difference of the chemical composition results. It speaks volumes for high homogeneity of the surface pickled with Ar+ ions and precise setting of the sputtering ion gun towards the analysed area.

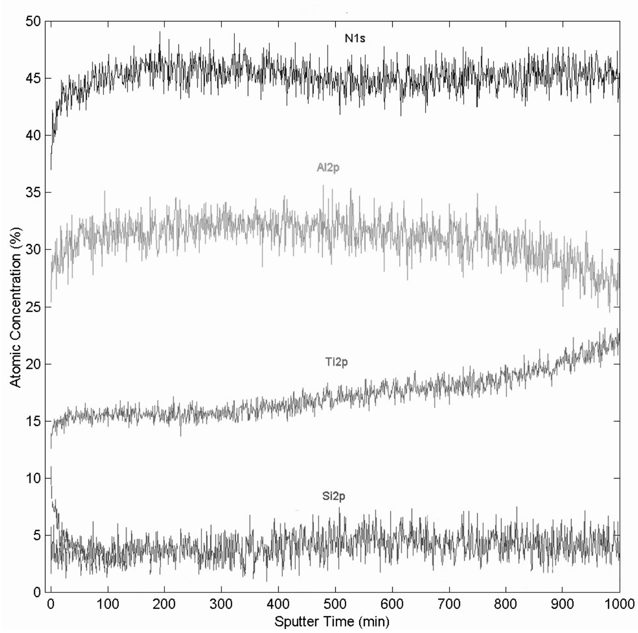

Fig. 6. Depth profile of the chemical composition obtained with XPS method on the (Ti,Al,Si)N coating

Expect of the strong peaks characteristic for nitride, titanium, aluminium and weaker peaks characteristic for silicon, the photoelectron peak O1s of oxygen has identified. This indicate, that the intermediate area between the coating and Al_2O_3+$SiC_{(w)}$ oxide ceramic substrate was reached.

It was found out as a result of depth profile chemical composition analysis (Fig. 6), that investigated (Ti,Al,Si)N coating is characteristic of the gradient structure changing significantly concentration of aluminium and titanium in direction from the surface of coating to the substrate.

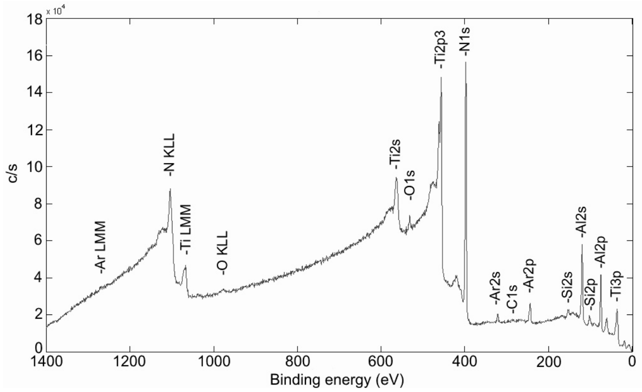

Fig. 7. Surface spectrum of (Ti,Al,Si)N gradient coating deposited on the Al_2O_3 oxide tool ceramic reinforced with SiC whiskers obtained with XPS method (area of analysis: 0.4 mm circle)

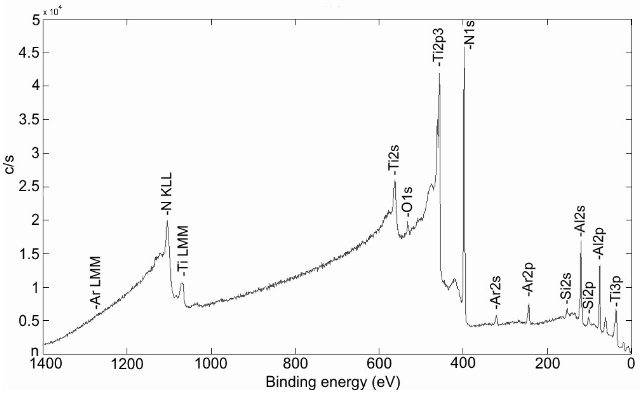

Fig. 8. Surface spectrum of (Ti,Al,Si)N gradient coating deposited on the Al_2O_3 oxide tool ceramic reinforced with SiC whiskers obtained with XPS method (area of analysis: 2 mm x 0.8 mm rectangle)

4. Conclusions

The results of the investigations of a gradient (Ti,Al,Si)N coating deposited onto Al_2O_3+$SiC_{(w)}$ oxide tool ceramic with use of the cathodic arc evaporation CAE-PVD method are given in the paper. Gradient PVD coating deposited on Al_2O_3 oxide tool ceramics reinforced with SiC whiskers has a dense, compact structure and its fracture surface topography attests a high brittleness, characteristic especially for the oxide ceramic materials. The coating was put down uniformly onto the investigated substrate materials and has a fine-graded structure.

The results of roughness and microhardness tests confirm the advantages of the gradient (Ti,Al,Si)N coating. As results of the examination of coating microhardness it has been found out that gradient coating onto investigated materials increase of microhardness value.

It was found out as a result of depth profile chemical composition analysis made with use of X-Ray Photoelectron Spectroscopy (XPS), that investigated (Ti,Al,Si)N coating is characteristic of the gradient structure changing significantly concentration of aluminium and titanium in direction from the surface of coating to the substrate.

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