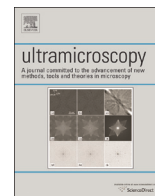




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Atom probe tomography of a Ti–Si–Al–C–N coating grown on a cemented carbide substrate

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ABSTRACT

The elemental distribution within a Ti–Si–Al–C–N coating grown by physical vapour deposition on a Cr-doped WC–Co cemented carbide substrate has been investigated by atom probe tomography. Special attention was paid to the coating/substrate interface region. The results indicated a diffusion of substrate binder phase elements into the Ti–N adhesion layer. The composition of this layer, and the Ti–Al–N interlayer present between the adhesion layer and the main Ti–Si–Al–C–N layer, appeared to be sub-stoichiometric. The analysis of the interlayer showed the presence of internal surfaces, possibly grain boundaries, depleted in Al. The composition of the main Ti–Al–Si–C–N layer varied periodically in the growth direction; layers enriched in Ti appeared with a periodicity of around 30 nm. Laser pulsing resulted in a good mass resolution that made it possible to distinguish between N^+ and Si^{2+} at 14 Da.

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1. Introduction

Titanium nitride based ceramics are widely used as hard and wear resistant coatings on cemented carbide cutting tools. Coating materials based on Ti–Al–N and Ti–Si–N alloys fabricated by physical vapour deposition (PVD) have attracted considerable attention for some time [1–5]. The incorporation of aluminium into the Ti–N lattice reduces the lattice parameter and, as a consequence, the hardness of the compound increases [2]. The addition of silicon to the Ti–N or Ti–Al–N coating may result in a decreased grain size and thereby an increased coating hardness [3,4]. Different technologies for the fabrication of Ti–Si–N films, with or without the incorporation of aluminium, have been presented in the literature [1–5]. It has been noted that the use of trimethylsilane ($(CH_3)_3SiH$; TMS) gas as the silicon precursor may result in a simultaneous incorporation of carbon into the film, i.e. the growth of a Ti–Si–C–N film [5].

The understanding of the relationships between the different parameters in the PVD process and the cutting tool performance in an application requires a thorough knowledge of the structure and chemical composition of the PVD coating. Atom probe tomography (APT) has previously been used in the investigation of Ti–Si–N coatings [6], and in a detailed characterisation of the spinodal

decomposition that occurs during annealing of some Ti–Al–N films [7]. The APT investigation in the present paper is concerned with the elemental distribution within a Ti–Si–Al–C–N coating and, in particular, in the coating/substrate interface region. The structure and elemental distribution in this region is believed to be important to the tool properties and also to the development of the main Ti–Si–Al–C–N layer.

2. Experimental procedures

2.1. Film growth

The Ti–Al–Si–C–N coating was grown on a WC–Co cemented carbide substrate containing 10 wt% Co and 0.4 wt% Cr. The deposition was done by arc evaporation in an Oerlikon Balzers Rapid Coating System (RCS) under industrial production conditions at 600 °C. A Ti–N adhesion layer was deposited on to the substrate surface from titanium targets in a nitrogen atmosphere. This was followed by the deposition of a Ti–Al–N interlayer in a gas mixture of nitrogen and argon using targets with a titanium to aluminium weight ratio of 60:40. Each of these two layers was grown to a thickness of around 100 nm, see Fig. 1. The subsequent deposition of the Ti–Al–Si–C–N layer was done using the Ti:Al targets in a gas mixture of TMS, nitrogen and argon. The final thickness of the coating was 4 μm. X-ray diffraction showed that the coating had the B1 NaCl structure with a lattice parameter of 0.407 nm.

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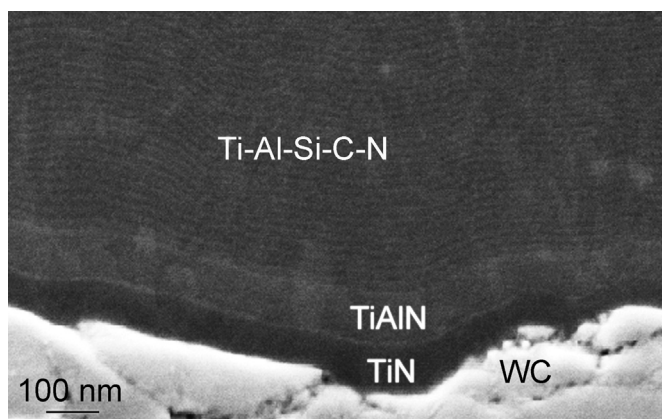


Fig. 1. The three layers of the coating (SEM).

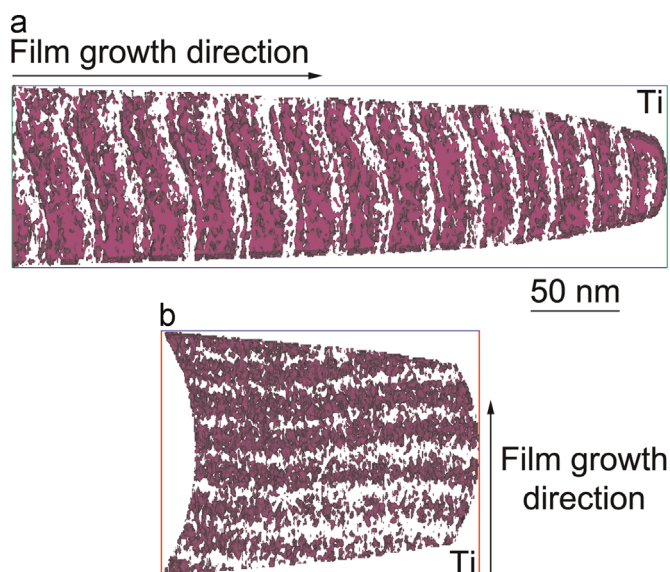


Fig. 2. Iso-concentration surfaces showing the periodically varying Ti content of the Ti–Al–Si–C–N layer (APT). Regions with dark contrast contain 32 at%, or more, Ti. Needle shaped specimens taken (a) parallel to, and (b) perpendicular to, the growth direction of the layer.

2.2. Atom probe tomography

Needle shaped specimens (tip radius < 50 nm) for atom probe analysis were prepared from the Ti–Al–Si–C–N layer parallel and perpendicular to the film growth direction, and from the interface region between the substrate and the Ti–N adhesion layer at right angles to the film growth direction. The specimen preparation was done in a focused ion beam/scanning electron microscope instrument (FIB/SEM; Fei DualBeam Strata 235 equipped with an Omniprobe™ micromanipulator) using the lift-out technique [8]. A strip of platinum was deposited onto a polished section through the PVD coating. Material was removed on both sides of the strip so that a triangular prism, with a trench on each side, was formed. Slices of the prismatic bar were then mounted, *in-situ*, on to pre-fabricated silicon posts with the aid of the Omniprobe™. The mounted slices were subsequently milled into the appropriate shape from the top using an annular milling pattern. The final milling was carried out with low energy ions (5 keV) in order to limit the gallium implantation.

Atom probe analysis was carried out in an Imago LEAP™ 3000X HR local electrode atom probe instrument. The analyses were performed in laser pulse mode (wavelength 532 nm) with a pulse energy of 0.5 nJ and a pulse rate of 200 kHz. The specimen

base temperature was 80 K and the instrument automatically regulated the voltage to keep a constant evaporation rate of 0.5% during analysis.

3. Experimental results

3.1. The Ti–Al–Si–C–N layer

The composition of the Ti–Al–Si–C–N layer varied periodically in the growth direction, see Figs. 1 and 2. This was clearly shown by the distribution of Ti in the reconstructions of analyses made parallel to (Fig. 2a) and perpendicular to (Fig. 2b) the growth direction. The images in Fig. 2 show iso-concentration surfaces at 32 at% Ti. These reconstructions indicate a Ti concentration that varied with a periodicity typically around 30 nm in the growth direction. In addition, the specimen taken parallel to the film growth direction indicated the presence of alternating thin and thicker Ti-rich layers (Fig. 2a).

The mass resolution in the spectra taken from the Ti–Al–Si–C–N layer made it possible to distinguish the N^+ peak from the Si^{2+} peak at 14 Da, see Fig. 3. It was also noticed that the resolution in the spectrum was dependent on the collection point on the detector. A spectrum containing ions from the entire detector is shown in Fig. 3b. A higher mass resolution was obtained when the spectrum only contained ions detected in the right hand part of the detector image, see Fig. 3c. The mass resolving power at full width half maximum (FWHM) was 960 in the spectrum from the entire detector (Fig. 3b) and 1290 in the spectrum from the right hand part of the detector (Fig. 3c), as measured on the nearby Al^{2+} peak at 13.5 Da. This behaviour has also been observed by Liu and Andr en when using the present atom probe instrument in laser pulse mode [9]. They suggested that the observed variation in resolving power with position on the detector was an effect of the asymmetry of the reflectron [9].

Laser pulsing resulted, however, also in a pronounced tailing of the peaks (Fig. 3). As a consequence, a substantial amount of Si^{2+} would still be present in a window set for the N^+ peak. A reconstruction of the Si and N distributions in the layer would, hence, not be able to show representative distributions of these elements. A reconstruction of the N containing areas would, inevitably, also show a certain amount of Si, and a significant part of the Si would be excluded in a Si reconstruction. The tails would probably be somewhat reduced if the analysis was carried out with a lower laser energy and/or at a lower temperature. Both measures would, however, result in an increased risk of specimen fracture.

3.2. The coating/substrate interface region

A reconstructed volume containing a fragment of a substrate surface WC grain, the Ti–N adhesion layer and the lower part of the Ti–Al–N interlayer, is shown in Fig. 4. The chemical composition of the different parts of these two layers was determined as the mean value of the compositions of sub-volumes containing up to 12,500 atoms. The mean composition of the Ti–N adhesion layer was in this way determined to 53.9 at% Ti and 46.1 at% N, both with a standard deviation of 0.4 at%.

A sub-stoichiometric composition was observed also in the Ti–Al–N interlayer. A mean N content of 45.9 at%, with a standard deviation of 0.8 at%, and a mean total Ti+Al content of 53.4 at%, with a standard deviation of 0.7 at%, were determined. The Al content (18.1 ± 0.9 at%) was lower than the Ti content (35.3 ± 1.0 at%), and the Al/(Ti+Al) ratio was determined to 0.34 ± 0.02 .

The Al and Ti distributions in the reconstruction of the Ti–Al–N

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