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Microstructural characterization of the tool-chip interface enabled by focused ion beam and analytical electron microscopy

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Abstract

A method based on focused ion beam milling and analytical electron microscopy to investigate the nature of the tool-chip interface is presented. It is employed to study tool-chip interfaces of the rake face of a $(Ti_{0.83}Si_{0.17})N$ coated sintered c-BN insert after turning operation of case-hardened steel. Analytical electron microscopy shows the presence of a smeared adhered layer on the coating, which consists of steel elements from the work-piece, oxygen, and Si and N, most likely originating from the coating.

Introduction

Understanding the tool-chip contact conditions in chip forming operations in terms of tribo-chemical interactions and prevailing wear mechanisms is important for the development of new coatings for cuttings tool applications. The metallurgy of the system is complex as it contains reaction products in the form of metallic and non-metallic phases of the work material. The quantitative investigations of tool-chip interfaces are a difficult task due to the small contact area available for analysis (order of mm^2) and also because the layers that need to be analyzed are very thin (10-1000 nm).

Several attempts have been made to analyze tool-chip adhesion and layer formation on the micro scale using various methods including Raman spectroscopy [1], secondary ion mass spectrometry (SIMS) [2], and also a set of complementary techniques for characterization in more quantitative terms. An example of the latter can be found in a recent study [3] combining the use of 3D white light interferometry for quantifying adhesion through the layer of reaction products, scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDS), and wavelength dispersive x-ray spectroscopy (WDS) to describe the metallurgical state. Laser mass spectrometry (LA-TOFMS) has been used to identify the chemical species. Combining these techniques also renders information about the contact friction, temperature distribution (IR-CCD), and stress levels.

However, for understanding the wear behavior of thin film or coatings on metal cutting tools at submicron scale it is desirable to have access to methods which can resolve the microstructure and identify the chemical properties of the layers. Analytical transmission

electron microscopy (TEM) is an excellent tool for investigation of thin films and coatings because of its combination of TEM and scanning TEM (STEM) for structural information of superior spatial resolution with associated electron diffraction and fast Fourier transforms for phase analysis, as well as EDS and electron energy loss spectroscopy (EELS) for chemical information and phase analysis. In order to study the tool-chip interface, a technique which can produce high quality electron transparent specimens of the highly localized cutting zone is required. Traditional cross-sectional TEM sample preparation techniques, e.g., mechanical polishing followed by ion-milling of coatings as described in [4], are not well suited. There are a few studies published where the authors have used a refined traditional preparation technique [5,6]. But, also this technique is limited by the precision of choosing a specific area to prepare. Instead, we prepared the TEM specimen by using focused ion beam (FIB) [7], which has the advantage of being able to produce TEM specimens from a specifically chosen area. For instance, FIB has recently been used successfully to prepare cross-section TEM specimens through nanoindents in order to study deformation mechanisms [8,9]. Moreover, multiple TEM specimens can be produced from various locations of the same sample in order to thoroughly study the wear mechanism in the whole area of interest. FIB also offers the possibility to section very hard material, such as c-BN and diamond.

Here, we present a method to study the microstructure and chemical properties of a tool-chip interface by preparing TEM specimens using FIB. The specimens originate from the cutting zone on the rake face of a machined $(Ti_{0.83}Si_{0.17})N$ coated sintered c-BN (CBN) turning insert. We present SEM and TEM/STEM micrographs in combination with EDS and EELS elemental maps as well as energy loss near edge spectroscopy (ELNES) for the

determination of chemical bonding from the coating and adhering material of the work-piece.

Experimental

The films were deposited onto cemented carbide WC-Co(6 wt.%) substrates with 2 mm CBN050C (c-BN-50 vol.% and Ti(C,N) binder) brazed tips at the cutting edge by arc evaporation of an alloyed $Ti_{0.80}Si_{0.20}$ targets in a 99.995% pure reactive N_2 atmosphere using a commercial Metaplas MZR323 deposition system. Prior to deposition, the inserts were cleaned in ultrasonic baths of an alkali solution and alcohol. The system was evacuated to a pressure of less than $2.0x10^{-3}$ Pa, after which the substrates were sputter cleaned with Ar ions. Substrates with a bias of -50 V were kept at a temperature of ~500 °C.

Ti-Si-N coated CBN050C inserts with a TNGN110308 S geometry were tested by hard turning of in a case hardened steel type 16CrMn5 58-62 HRC at cutting speed v_c=170 m/min, cutting depth a_p=0.3 mm, and feed rate f=0.05 mm/rev. The turning time was limited to 4 min.

The as-deposited coating composition was determined by EDS using a LEO 1550 FEG-SEM operating at 15 kV equipped with a Noran Si-detector. Industrial standards and ZAF correction were used for the quantitative analysis. After the turning test, the tool rake face was investigated by SEM and EDS elemental mapping.

Cross-sectional TEM specimens of the cutting zone were prepared using a Zeiss 1540 EsB CrossBeam FIB by the so-called lift-out technique described in Ref. [10]. The

cutting zone includes the coating and any adhered layer from the work material. The area of interest was chosen ~20 µm as shown in from the cutting edge of the rake face (see Fig. 1(a) and the arrow in Fig. 2(a)), which corresponds to the area where the temperature is expected to be highest during machining, hereafter referred to as the "hot-zone". First, a ~1.5 µm thick Pt layer (2x20 μm^2) was deposited in the direction of the chip-flow in order to protect the TEM specimen from ion beam damage. The specimen was then cut out by a Ga ion-beam and transferred via a micro manipulator needle to a copper grid where the final milling and polishing (with an ion current of 20-50 pA) to electron transparency was carried out.

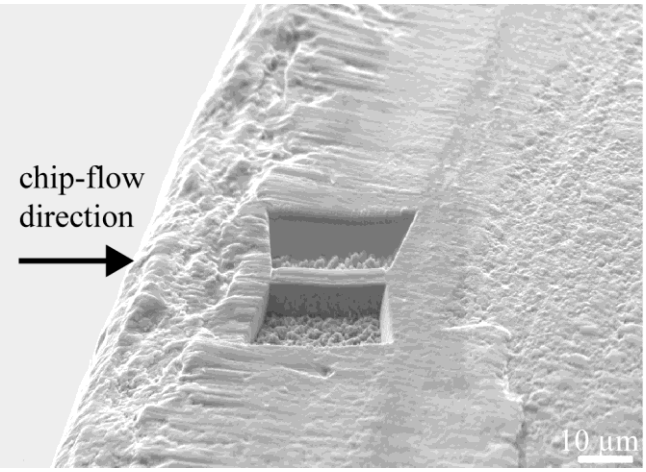

Fig. 1. a) Scanning electron micrograph showing the location and the initial milling of the TEM specimen on the rake face of the $Ti_{0.83}Si_{0.17}N$ film on CBN insert.

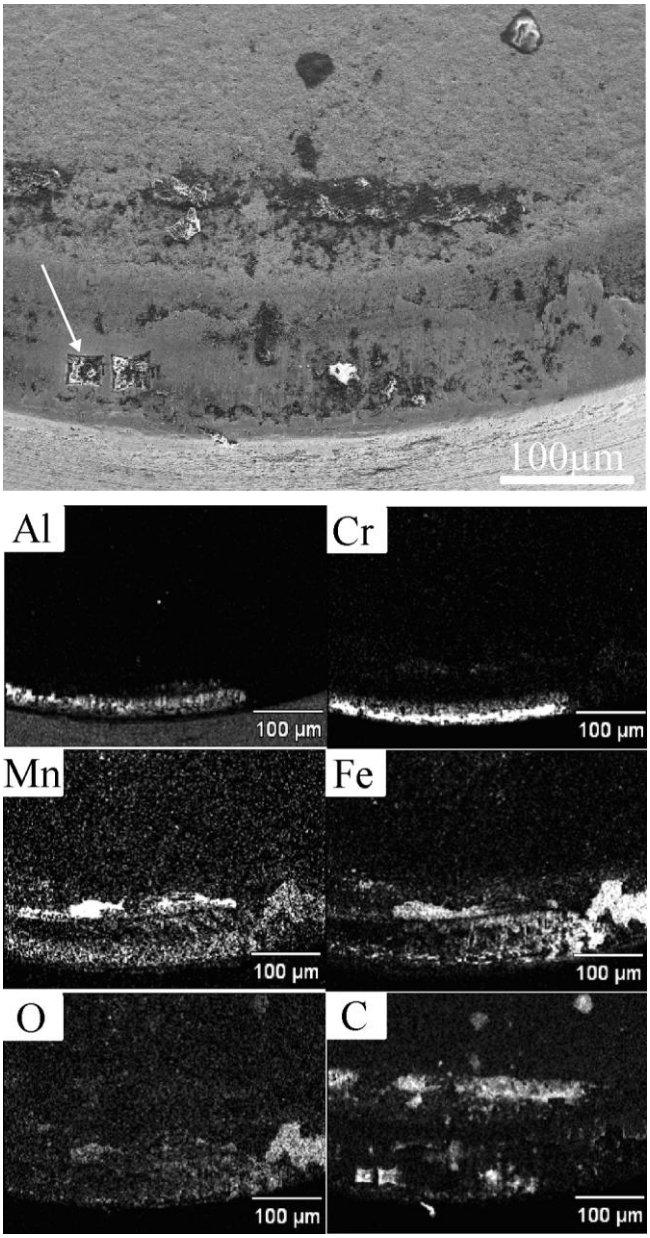

Fig. 2. Scanning electron micrograph and EDX elemental maps from the rake face of the $Ti_{0.83}Si_{0.17}N$ film on CBN insert used in 4 min turning test. The arrow in the SEM image indicates where the TEM specimen was prepared.

The microstructure and chemistry of the tool rake face was further analyzed by TEM and STEM using a FEI Tecnai G^2 TF 20 UT operated at 200 kV. STEM micrographs were obtained by a high angle annular dark field detector (HAADF) and a camera length of 220 mm. Elemental mapping was performed using the Tecnai TIA software. EDS maps

were obtained using an EDAX detector with a pixel size of 10x10 nm^2 and dwell time of 1 s/step across and area of 200x300 nm^2, while mapping by EELS was carried out with a Gatan Enfina spectrometer and a pixel size of 3.33x3.33 nm^2 and dwell time of 1s/step. The ELNES was similarly acquired by adding the EELS spectra in a map, correcting for energy drift during acquisition, background subtraction and a final deconvolution of plural scattering.

Results and Discussion

The relative content of Ti and Si in the as-deposited coatings was quantified to 83 at.% and 17 at.%, respectively, by EDS. The N content was not analyzed in this study, but is expected to be close to 50 at.%, as previously reported for arc evaporated $(Ti_{0.83}Si_{0.17})N_{1.09}$ coatings on c-BN substrates, grown under similar conditions and measured by elastic recoil detection analysis (ERDA) in Ref. [11]. This coating consists of NaCl structure TiN:Si nanocrystallites segregated by a semicoherent SiN_x tissue phase. It has a hardness of ~40 GPa and thermal stability up to ~1100 °C when annealed in Ar atmosphere.

Fig. 2(a) displays an SEM image and Fig. 2(b-g) corresponding EDS elemental maps from the used cutting edge of the insert after turning. The elemental maps show the presence of Al, Mn, Cr, Fe, C, O, Ti, Si, and N. As evident in the SEM image, part of the work-piece material has smeared the surface of the coated insert, in particular close to the hot-zone of the cutting edge. The adhering materials were identified as Al, Mn, Cr, and Fe, which agree with the expected alloying elements in the work-piece material. Closest to the edge were Cr-rich areas, followed by an Al-enriched zone where the temperature is

expected to be highest, ~800-900 °C [12]. The thickest adhesion layer was mainly composed of Fe and Mn and was located where the release between tool and chip occurs. The presence of oxygen in this region also suggests the formation of oxides on the rake surface. Carbon stems mainly from the binder phase of Ti(C,N) from the substrate. Since many elements overlap in these maps, cross-sectional investigation is required in order to determine the composition of the adhered layer.

Fig. 3 shows cross-sectional TEM micrographs of a $(Ti_{0.83}Si_{0.17})N$ film on CBN insert obtained from the hot-zone after turning operation of 4 min (the exact position indicated by the arrow in Fig. 2(a)). As discussed above, the top surface of the coating contains the adhered layer from the work-piece with a thickness up to 150 nm. In the $(Ti_{0.83}Si_{0.17})N$ coating, below the adhered layer, ~100-150 nm broad deformation bands with an angle of ~25° to the growth direction has formed, indicative of intense shear deformation at the tool-chip interface during metal cutting. Moreover, the layer thickness was reduced from ~2 μm in the as-deposited state to ~1 μm due to tool wear during turning.

Fig. 4 shows a cross-section STEM micrograph and EDS elemental maps from the specimen investigated in TEM. The maps show that the adhering material from the work-piece is layered. The contrast from the Si and N maps, reveal their presence in the adhering layer. The likely source for these elements is the tool coating. In fact, earlier

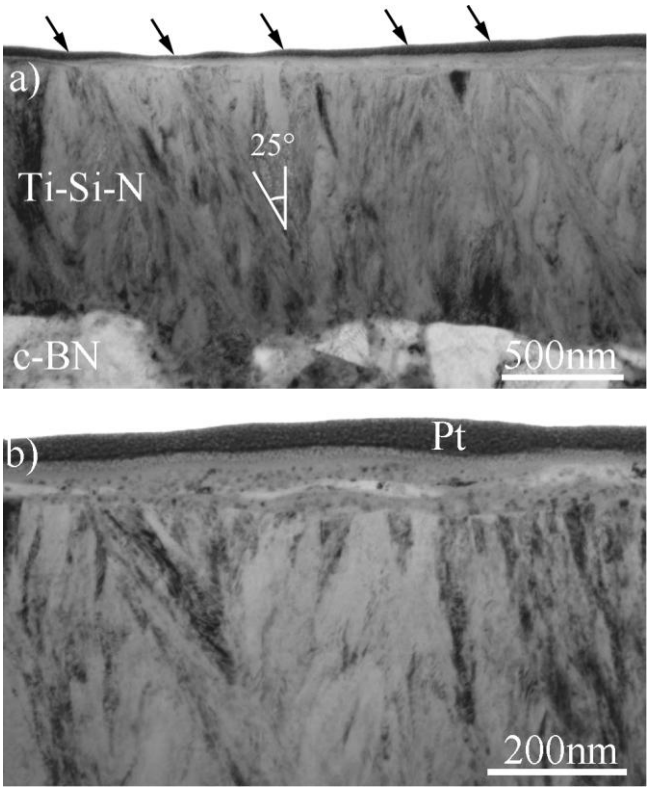

Fig. 3. Cross-sectional TEM micrographs from the rake face of the used $Ti_{0.83}Si_{0.17}N$ film on CBN insert after 4 min turning operation. (a) shows an overview including both substrate, film, and adhered material from the work-piece at the film surface. The arrows indicate shear bands. (b) displays the film and the layered adhering material at higher magnification. The chip-flow-direction is from right to left. The specimen was prepared by FIB.

studies revealed Si and N out-diffusion after annealing at between 900-1100 °C in arc evaporated $(Ti_{1-x}Si_x)N_y$ films for x>0.1 on different substrates [11]. For films deposited on c-BN substrates, in particular, local Si out-diffusion and segregation begin between 1000-1100 °C, i.e., higher than the temperature of 800-900 °C expected at the tool-chip contact in this cutting application. However, the effect of local thermal and pressure spikes during the turning operation may enable the mobility of Si and N. The transfer of some Si from the work-material, which contains up to 0.4 wt.% of Si, into the adhering material is also possible. Correspondingly, N from the ambient may react with the adhered layer.

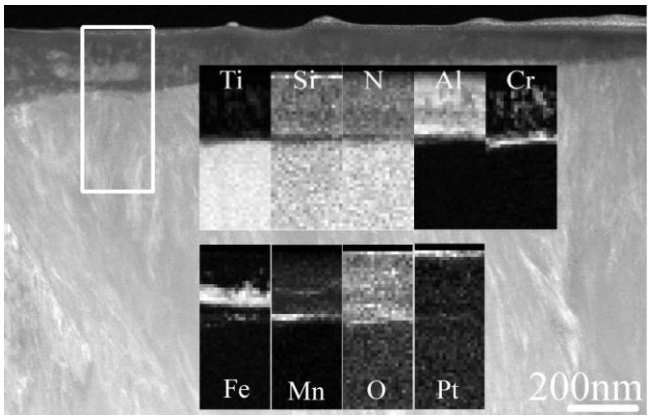

Fig. 4. Cross-sectional STEM micrograph and EDX elemental maps from the rake face of the ($Ti_{0.83}Si_{0.17}$)N on CBN insert after 4 min turning test. The chip-flow-direction is from right to left. The specimen was prepared by FIB.

The here described method for sample preparation also enables EELS fine structural studies. Fig. 5(a) shows a STEM image and EELS elemental maps from Ti and N from another area of the tool-chip interface. The maps display that N is present in the adhering layer while Ti is bound to the coating. The fine structure from the ELNES spectra of the N-K and Ti-L_{23} edges taken from the Ti-Si-N coating are shown in Fig. 5(b). From the peak positions and relative intensities the nature of the chemical bonds can be revealed. Here, N-K spectra resemble both the experimental and theoretical ELNES profiles from NaCl-structure TiN in [13]. Similarly, the fine structure of the Ti-L_{23} edge corresponds to the bond structure of TiN.

Conclusions

We report a method to investigate the microstructure and chemical properties of the tool-chip interface after metal cutting. This method makes use of FIB and analytical TEM to locally address and investigate the nature of tool wear at the tool-chip interface. We demonstrate the method by investigating a ($Ti_{0.83}Si_{0.17}$)N coated sintered c-BN insert after turning of case hardened steel. The coating microstructure was found to form ~100-150

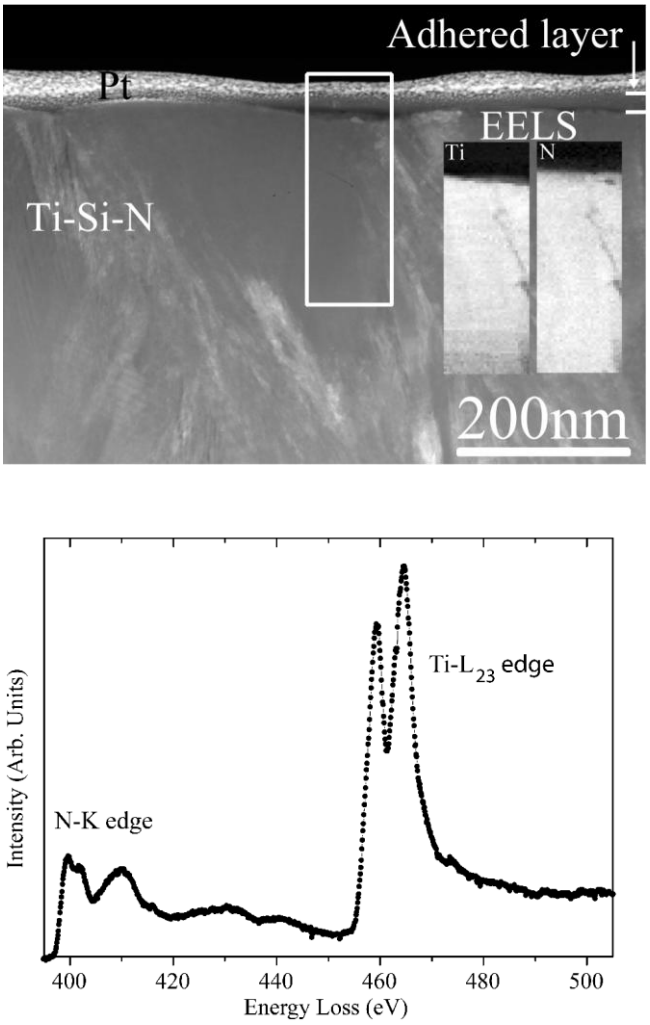

Fig. 5(a) Cross-sectional STEM micrograph and EELS elemental maps of Ti and N from the rake face of the ($Ti_{0.83}Si_{0.17}$)N on CBN insert after 4 min turning test. The chip-flow-direction is from right to left. (b) Show the ELNES fine structure from the N-K and Ti-L_{23} edges. The specimen was prepared by FIB.

nm broad deformation bands, which indicates intense shear deformation. A layer of adhered material originating from the machined work-piece with thickness up to ~150 nm in the hot-zone is present on the top surface of the coating. The distribution of individual elements was resolved by a combination of EDX and EELS in STEM and SEM, respectively, while ELNES enabled determination of the chemical bonds. Finally, our

reported method highlights the potential for characterizing the nature of tool wear mechanisms in metal cutting.

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