Characterisation of worn WC tool using STEM-EDS aided by principal component analysis

Isac Lazar1,2 | Rebecka Lindvall2 | Filip Lenrick2 | Volodymyr Bushlya2 | Martin Ek1

1 Centre for Analysis and Synthesis, Lund University, Lund, Sweden
2 Division of Production and Materials Engineering, Lund University, Lund, Sweden

Correspondence
Martin Ek, Centre for Analysis and Synthesis, Lund University, Box 124, Lund 22100, Sweden.
Email: martin.ek@chem.lu.se

Abstract
Interdiffusion and chemical reactions contribute to tool wear in metal machining. Increased understanding of these processes, through characterisation of worn tools, can facilitate design of more resilient materials through chemical and diffusional passivation. However, the unknown reaction conditions, the large number of elements, and the formation of interspersed phases makes for a complex analysis. Here, we demonstrate the use of scanning transmission electron microscopy and energy dispersive X-ray spectroscopy for characterising the interaction layer between a titanium alloy and a cemented carbide tool. Principal component analysis is used to find chemical correlations and help separate signals from embedded phases. Crucially, we evaluate the required X-ray count statistics from simulated spectrum images and theory prior to the experiment. We find no indications of intermediate phases between the original WC and the metallic W interaction layer. Furthermore, we find enrichment of minor constituents in the titanium alloy closest to the tool which alter the solubility of out-diffusing species, suggesting strong interrelations between the diffusion processes.

KEYWORDS
EDS, metal cutting, PCA, spectrum simulation, titanium, wear

INTRODUCTION
Titanium alloys are used in a wide range of demanding applications due to having properties such as high strength-to-weight ratio, hot hardness, fatigue resistance, and low thermal conductivity.1–3 However, the very same properties makes titanium alloys difficult to cut in machining operations; high stresses and temperatures are generated and concentrated locally at the contact between the cutting tool and workpiece, which can lead to rapid tool wear.4 While mechanical mechanisms (e.g. abrasion and adhesion) play a role, the wear processes are increasingly being connected to interdiffusion and chemical reactions between the tool and the workpiece,5–8 between components in the tool,9 and between the tool and the surrounding atmosphere.10 The diffusional mechanisms have been studied particularly in the context of crater formation5–7,11 – considered the most important wear type in titanium machining – but they will occur in all areas of contact between tool and workpiece, for example, also the flank region. Increased understanding of these phenomena will open up new avenues for improving tool performance.
via chemical and diffusional passivation, for example, through surface coatings deposited ex situ or formed in situ in the cutting process. Consequently, structural and compositional analyses of reaction products formed at the tool–workpiece interface are required. However, the large number of elements involved, the unknown reaction conditions, the highly deformed structure, and the formation of interspersed phases and inclusions makes for a very complex system.

Electron microscopy combined with energy dispersive X-ray spectroscopy (EDS) is an ideal tool for providing the needed nanoscale compositional maps. For scanning electron microscopy (SEM), there are several reports of its application on analysing the wear zone either directly on the surface or in cross section after cleaving and polishing worn tools. The use of scanning transmission electron microscopy (STEM) is more recent, but the higher spatial resolution this technique offers can be crucial; the interaction layer and adjacent material in the workpiece and tool have been shown to contain variations on the nanometre scale after both emulsion-cooled and cryogenic machining. Interpretation of both SEM- and STEM-EDS spectrum images can however be complex due to, for example, physically overlapping phases and inclusions in the sample, and overlapping X-ray peaks in the spectra. Multivariate statistical methods such as principal component analysis (PCA) can greatly aid in the analysis by denoising the spectra and finding chemical correlations, as well as by helping separate signals from embedded materials. However, the use of PCA in the characterisation of worn tools has not been reported previously.

Here, we analyse the interaction layer formed during turning of a Ti6Al4V titanium alloy with an uncoated cemented carbide tool (the most common titanium alloy and tool-type, respectively) using STEM-EDS spectrum imaging aided by PCA. Specifically, we investigate the presence of different carbon-deficient W-phases in the interaction layer. Dissolution of carbon from the tool into Ti6Al4V has long been considered an important wear mechanism. However, important details remain unexplored: for example, the presence or absence of W$_2$C in the transition from WC to W$_7$C could provide important clues on the local conditions during machining. Crucially, prior to recording spectra, we evaluate X-ray count statistics based on simulated spectra to ensure that chemically relevant information is not lost among the PCA noise components. We use the framework adapted by Potapov for this purpose, but reformulated to quantities more easily used during practical spectrum acquisition. Factor rotation is subsequently applied to the principal components to enhance interpretability. In addition to strong indications of a lack of W$_2$C – consistent with expectations from previous temperature estimates at the cutting zone – we find gettering of minor constituents in the Ti6Al4V alloy close to the interface, which could play a hitherto little explored role in the wear process.

MATERIAL AND METHODS

Simulated spectra and principal component analysis

Model X-ray spectra (e.g. of WC and W$_2$C) were generated using Monte–Carlo (MC) simulations in DTSA-II. For each phase, a characteristic spectrum was simulated for 300 keV primary electrons using a 100 nm thick and 1000 μm wide block of the corresponding material in vacuum. To detect X-rays generated in the MC-simulation, a SDD detector with a 10 mm$^2$ detector area, 2048 energy channels, and 6 eV/channel was used. The total electron dose was set to 0.1 nAs and all available X-ray generation modes were included. The generated spectra were then linearly combined to construct noise-free spectrum images corresponding to different distributions of the phases. The spectrum images were subsequently scaled in intensity to represent, for example, a 10$^7$ total X-ray count; this constitutes the base truth which PCA will later attempt to reconstruct. Noise-containing versions of the spectrum images were then generated using a Poisson distribution with an expectation value corresponding to the noise-free, intensity scaled spectrum image. This workflow provides a facile route for generating a large number of model spectrum images with a variety of sampling conditions, total counts, and phase distributions.

Principal component decomposition was performed using SciPy Python routines in the same way for the noise-free and noise-containing, as well as the experimental STEM-EDS, spectrum images. The data was scaled to account for Poisson noise prior to the decomposition. The number of principal components to include in the further analyses was determined using a ‘knee’-finding algorithm. Factor rotation was performed using the varimax method on the spatial domain to aid interpretation of the abstract principal components. Although we used the direct SciPy (and in the case of varimax, NumPy) implementation of these functions to facilitate batch processing, we note that they are also conveniently available through HyperSpy.

Sample preparation and electron microscopy

The $\alpha + \beta$ alloy Ti6Al4V, supplied in mill annealed state with a Rockwell hardness of 31 (HRC), was used as a workpiece material. In accordance with the alloy specification (ASTM B348-13), the material contained mainly the
alloy elements – 6 wt% Al and 4 wt% V – with the main residual being 0.2 wt% Fe. The initial microstructure of the alloy is shown in Figure 1(A). The cutting tool material was a commercial uncoated straight cemented carbide grade provided by Seco Tools AB, consisting of fine-grained WC and 6 wt% Co binder. The initial microstructure of the tool is shown in Figure 1(B). Longitudinal turning of the workpiece material was performed with CNC-lathe SMT Sajo 500 Swedturn, with cutting conditions in the high-speed machining range: constant cutting depth $a_p = 0.3$ mm, feed $f = 0.1$ mm/rev, and cutting speed $v_c = 200$ m/min. High-pressure directed cooling was used with a 8% oil–water emulsion at 90 bar pressure. The cutting tool reached a maximum temperature of approximately 870°C based on IR-CCD images during operation. The turning operation was stopped after 216 seconds to limit the flank wear to 300 μm.

The worn tool was cut, mounted in conductive resin and polished to reveal the wear region in cross section, as shown in Figure 1(C). A lamellae was extracted from the flank region with a focused ion beam (FIB) lift-out procedure using a FEI Nova NanoLab 600 FIB-SEM. The lamellae was subsequently thinned to electron transparency (approximately 100 nm) and characterised using a JEOL 3000F transmission electron microscope (field emission gun, 300 kV, 1 nm FWHM electron probe diameter according to manufacturer-supplied calibration) equipped with an Oxford XMax 80 mm² SDD EDS detector. An EDS spectrum image (10⁷ total counts, 512×448 pixels, 0.4 nm pixel size) was collected from the region shown in Figure 1(D), covering both adhered Ti6Al4V workpiece material, interaction layer and a WC grain from the tool; the three regions can be distinguished based on their contrast in the high-angle annular darkfield (HAADF) image. Note that due to the extreme deformation from the machining process, the adhered Ti6Al4V material no longer shows any ordered microstructure in either the SEM or STEM-HAADF images. Stress-induced dislocations formed during the cutting operation are visible as bright lines in the WC grain. The flank region was chosen for the herein reported characterisation due to the long contact between workpiece and tool materials, and due its comparatively smooth and uniform appearance as evident from Figures 1(C), (D) (as opposed to the crater region where more tool-material has been lost, and which contains more cracks and dislodged grains). Quantification was performed using model-fitting to extract peak intensities, in combination with Cliff–Lorimer ratio routines in HyperSpy. K-factors and X-ray absorption correction factors were used as supplied by the spectrometer software.
RESULTS AND DISCUSSION

Evaluation of PCA using simulated spectrum images

The high heat, pressure, and dynamical contact between the workpiece and tool during machining opens the possibility of forming a great number of different intermetallic compounds and carbides at the interface. Of these, WC and W$_2$C will likely be the most difficult to distinguish by EDS as the variance between their spectra is small: while they differ in carbon content, they have similar density and arrangement of W cations (primitive hexagonal and hexagonal close packing, respectively, differing by a displacement of every other cation layer), which provide the main spectral features. The lower carbide W$_2$C can form at high temperatures in a comproportionation reaction between WC and W$^+$ – in turn formed by carbon out-diffusion from the tool. Potentially, such a W$_2$C layer could be detrimental for tool performance due to having reduced hardness compared to WC.

The required count statistics for distinguishing WC and W$_2$C can be evaluated through comparisons of simulated spectrum images with and without added Poisson noise: the latter provides the ground truth while the former accounts for the discrete and limited number of counts in a real spectrum. After performing PCA on both types of spectrum images, the dot product of the extracted principal component vectors provides a measure of the ability of PCA to describe the true signal variance in the simulation. For a linear transition of WC to W$_2$C (representing either overlapping phases or a non-stoichiometric compositional gradient), the quality of the extracted principal component as a function of the number of pixels in the spectrum image (for a fixed total X-ray count) is shown in Figure 2(A). The improved correspondence between the principal components with fewer pixels is due to the improved count statistics per spectrum in the image, not due to any advantage of having fewer pixels in and of itself. The simulations show a clear threshold effect where the difference in (mainly) C–K intensity between the two phases, reflected by the second principal component as shown in Figures 2(B), (C), cannot be distinguished below about 2000 counts per spectrum.

Theoretical analysis of the principal component decomposition provides additional insight into the factors determining PCA quality and allows us to generalise the behaviour to different distributions of phases. Following the analysis by Nadler as adapted for EDS by Potapov, we express the quality of the extracted principal component (in terms of similarity to the underlying true component) in quantities more familiar to microscopists. First we note that the signal ($\lambda$) and noise ($\sigma^2$) variances in the original formulations are directly related to the mean number of counts per spectrum ($\bar{c}$), which in turn can be expressed in terms of the total number of counts ($C$, often prominently displayed in the EDS software) and pixels ($m$). Their ratio can thus be expressed according to Equation 1:

$$\frac{\lambda}{\sigma^2} = \frac{k\bar{c}^2}{k_{c}\bar{c}} = k \frac{C}{m}.$$  

The signal and noise variances will have different constants of proportionality to the mean counts, the ratio of which can be combined to a single constant ($k$). For a certain number of energy channels ($n$), the correspondence of the extracted component to the underlying true component ($R^2$) can now be expressed according to Equation 2.
In order to make predictions of the principal component quality, values for $k$ can be extracted from the simulated noise-free ($\lambda$) and noisy ($\sigma^2$, as per Potapov) spectrum images. Note that $k$ will depend both on which phases we are trying to distinguish (i.e. how much their respective spectra differ) and their distribution in the spectrum image. The latter effect can easily be tested without simulating new spectrum images by calculating the relative change in variance for different distributions. For instance, the signal variance for a minor discrete inclusion of W$_2$C in WC (representing 2% of the sampled area) is 25% smaller than for the linear transition used in the simulations, and will require slightly higher number of counts per spectrum to be detected by PCA. The predicted principal component quality for this particular example is also included in Figure 2(A) and will be of relevance for the analysis of the experimental spectrum image.

Analysis of tool–workpiece interaction region

Based on the evaluation of the simulated spectra, we expect to be able to extract principal components describing the phases present in the experimental STEM-EDS spectrum image (from the region shown in Figure 1D) at between $\bar{c} = 10^3$–$10^4$ counts per spectrum. To further test this criterion, we perform a series of principal component decompositions while gradually binning the data to increase $\bar{c}$ from the initial (insufficient) 44 counts per spectrum. The resulting variances of the 15 first principal components are shown in Figure 3 as a function of the number of samples, controlled by gradually binning the original STEM-EDS data set. Signal-containing principal components are expected to have a variance distinct from subsequent components, whereas noise containing components are grouped together and continue to decrease in variance as the binning increases.

As expected, PCA initially only finds two significant principal components (i.e. workpiece and tool materials). However, binning reveals two additional principal components (i.e. two additional distinct phases) that break away from the pure noise components at around $\bar{c} = 4000$. As a compromise between having adequate signal for PCA and retaining spatial resolution, binning to $m = 1554$ (37x42 pixels, $\bar{e} \approx 6000$) is used in the further analysis.

The resulting PCA decomposition highlights several important features in the spectrum image, as shown by the extracted vectors and weightings in Figure 4. The first principal component (PC1) represents the mean spectrum and is the weighted strongest to the right, following the strong signal from W. In addition to the elements present in the workpiece and tool, it shows signals from Cu (background from the Cu-grid), Ga (residues from the FIB sample preparation) and O (due to surface oxidation of the lamella). The second principal component (PC2) shows an anticorrelation of Ti and Al to W, suggesting that this component represents the Ti6Al4V alloy. The third principal component (PC3) mainly shows an anticorrelation between Ti and V in the alloy, suggesting a V enrichment at the expense of Ti. As the Ti–K$\beta$ peak overlaps the V–K$\alpha$ peak, and Ti is the major constituent of the alloy, such variations would be difficult to identify unaided by PCA. Correlating with V in PC3 is also Fe, while C shows an anticorrelation. Looking at the weighting, PC3 is located in distinct band in the adhered alloy along the tool interface. The fourth principal component (PC4) shows mainly a strong C signal, but also some anticorrelation to Ti (and to a lesser extent W). While PC4 is present over most parts of the spectrum image due to the presence of carbon contaminants on the specimen surfaces (particularly in the bottom half due to an earlier, aborted SI acquisition), its greatest impact can be seen to the right in the image: the weighting is positive in the interior of the tool, where it is negative along the interaction layer seen as a bright band in the topmost HAADF image. This is consistent with the presence of metallic W (or C-deficient WC) forming in the interaction region.

Varimax factor rotation transforms the principal components into new components, shown in Figure 5, that can be more easily interpreted as regular EDS spectra. The first component vector (C1, representing the interaction layer) closely resembles W with small additions of the alloy elements. C1 does contain a small feature at the C–K energy, but it is only barely distinguishable from the uneven
FIGURE 4 (A) Resulting principal component vectors and weightings from the EDS spectrum image recorded from the region (B) indicated by the HAADF image. The descending order of variance means that the scales for each principal component vector and weighting is different.

background in this energy range. The fourth component (C4, representing the interior of the tool) is similar to C1, but has a clear C–K peak. Also barely visible at approximately 7 keV is a small peak related to Co due to the proximity of binder material further into tool. The second and third components (C2 and C3) both represent the Ti6Al4V alloy and are very similar, differing in the amounts of alloying elements (Al, V, Fe) and interdiffused tool elements (W and C). Their weightings show them located in two distinct regions in the sample corresponding to the bulk (C2) and interface towards the tool (C3). Oxygen is present in all components but fitted with similar intensity after taking the nearby Ti and V L-peaks into consideration; this is characteristic of surface oxidation of the lamella and not due to formation of oxides during the machining process. The presence of carbon-containing contaminants on the surface (c.f. PC4 from PCA in Figure 4 and corresponding discussion) is here accounted for by a combination of components C1 and C4; these components differ mainly in the amount of carbon and have small, complementary weightings throughout the spectrum image.

The principal and rotated components as described above provide strong indication that there are only two W–C phases present: the original WC and metallic W formed as a result of carbon out-diffusion. The former is present in the unaffected interior of the tool (i.e. component C4). The identification of the second phase as metallic W is supported by the C1 component vector: here, the carbon content is so low that the C–K peak is difficult to distinguish from the (strongly) varying background below 0.4 keV in the spectrum. A nanobeam electron diffraction pattern recorded from the interaction layer further corroborates the presence of metallic W; as shown in Figures 5(C), (D) this region yields strong diffraction at spacings that cannot be ascribed to the nearby phases in the tool or alloy. The formation of $\alpha$-W has also been reported previously from X-ray and electron diffraction characterisations of similarly worn tools. Based on the simulations presented in Figure 2(A), it should have been possible to distinguish W$_2$C as a separate component from WC and W under the current conditions if it had been present in as little as 2% of the pixels in the spectrum image. To ensure that no
W–C phases in the tool are overlooked in the transition from C1 to C4, inclusion of additional principal components can be used to increase the degrees of freedom for the varimax algorithm. However, the resulting additional components have weightings only on the Ti6Al4V side and describe the compositional grading\textsuperscript{28} in the alloy and the transition into the metallic W interaction layer. The lack of a distinct W\textsubscript{2}C phase for the current machining conditions is consistent with the estimated tool temperature,\textsuperscript{9} given the W–C phase diagram.\textsuperscript{26} For better detection and quantitative analysis of light elements in future studies, electron energy loss (EEL) spectroscopy can be used to complement the EDS spectrum images.\textsuperscript{14} Such EEL spectrum images similarly stand to benefit from PCA and prior analysis using simulated spectra.\textsuperscript{17,28}

The metallic W layer is alloyed with Ti6AlV4 components diffusing into the tool. While Ti is the predominant alloy element in the interaction layer since it comprises the majority of the workpiece, V is enriched going into the tool. This is evident from the quantified weight ratios of Ti:V in the bulk (C2) and interaction layer (C1) of 15:1 and 12:1, respectively. It is also highlighted by PC4 which directly shows a V and Ti anticorrelation in the tool. Apart from alloying with W in the interaction layer, the in-diffusing elements are also able to form carbides at the tool side of the interaction layer. In the present sample, both Ti and V are present in small quantities in the C4 component vector describing the carbide phase at 3.1 wt% and 0.4 wt% respectively (a 7:1 weight ratio of Ti:V). No separate TiC or VC phases are seen in the investigated area, which can have several explanations; they are too small and interspersed to be extracted as a separate component, they exist in a solid solution with the predominant WC,\textsuperscript{31,32} or they are incorporated primarily at (certain) WC grain surfaces and in the Co binder.\textsuperscript{33} Note that where separate TiC-regions have been reported previously,\textsuperscript{14} they have been located in
pockets in and around the interaction layer; from Figure 1(C) such pockets seem to be present in the crater-region and on the rake-face, but not the flank region which appears smooth.

The in-diffusion and enrichment of alloy elements towards the tool interface is connected to the simultaneous out-diffusion of C and W. The build-up of V and Fe at the expense of Ti in the Ti6Al4V alloy closest to the tool is most clearly seen in PC3. Quantification of the corresponding rotated components for the bulk (C2) and interface (C3) regions yield Al/V/Fe weight percentages of 7.1/5.1/0.1 wt% and 6.3/6.6/1.4 wt%, respectively. Also present in the two segments of the adhered Ti6Al4V alloy are W/C at 4.8/5.2 wt% and 18.0/3.7 wt% in the bulk and the interface regions, respectively. The presence of the two tool-components demonstrates that out-diffusion plays a role in the wear process, resulting in a gradual transfer of material into the Ti6Al4V alloy (where it is removed as chips are formed). The W out-diffusion and V/Fe enrichment at the interface are likely synergistic effects as these elements all stabilise the $\beta$-Ti (body centred cubic) polymorph, thereby increasing each other’s solubility. Note that Co out-diffusing from exposed binder regions in the tool, while not seen in the present region, will also stabilise $\beta$-Ti. Further into the bulk of the Ti6Al4V alloy, the $\beta$-stabilising elements are reduced in concentration, coupled with an increased concentration of elements stabilising the $\alpha$-Ti (hexagonal close packing) polymorph – which comprises the majority of the original alloy structure. The presence of $\beta$-Ti around the interface has previously been identified using nano-beam electron diffraction and has also been shown in static diffusion couples. The exchange of $\beta$-stabilisers for $\alpha$-stabilisers is particularly striking for C which diffuses through the $\beta$-Ti layer at the interface to reach the bulk where it has higher solubility. This behaviour hints at competing diffusional effects: the presence of $\beta$-Ti at the interface might enhance loss of W, thereby exposing new WC to the Ti6Al4V alloy, but simultaneously reduces C out-diffusion. Due to the complex interactions between in- and out-diffusing elements around the interaction layer, comparative studies between wear for other titanium alloys, for example, pure $\alpha$- or $\beta$-alloys, will be useful going forward.

While the present study is focused on the interaction of the carbide phase in the tool with the titanium alloy, future studies will need to analyse also areas where the Co binder is exposed. Here, interactions between the titanium alloy and binder could lead to formation of a plethora of inter-metallic compounds. The consequent loss of binder material has been proposed to lead to dislodging of entire WC grains - a wear mechanism that could be particularly relevant in the crater region. Conversely, the formation of stable compounds could hinder further binder diffusion and enhance the tool life.

**CONCLUSIONS**

To distinguish chemically similar phases in EDS spectrum images aided by PCA – such as WC, W$_2$C and W (with surface carbon contaminants) that can form in worn cemented carbide tools – it is crucial to analyse acquisition conditions and X-ray count statistics based on simulations. Too few counts per spectrum not only reduces the quality of the resulting principal components but will also lead to chemically relevant information being entirely lost among the noise components. For the present combination of tool, workpiece material and cutting data, we find only metallic W in the interaction layer formed by the wear processes during machining. The presence of intermediate W$_2$C carbides is ruled out by the number of detected phases, together with a qualitative analysis of the component vectors. Additionally, we find enrichment of $\beta$-stabilising elements at the tool boundary. This forms a link between diffusion of alloy elements into the tool and tool elements out to the alloy, which could play an important role in the wear process.

In conclusion, EDS spectrum imaging aided by PCA can provide a wealth of information on wear processes in cutting tools by finding chemical correlations and automatically extracting individual phases in the sample. The methodology outlined here can provide an excellent complement to microdiffraction techniques in TEM and SEM by suggesting candidate compounds and enabling analysis of the often highly deformed regions closest to the tool–workpiece interface.

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**CONFLICT OF INTEREST**

The authors declare no conflict of interest.

**ORCID**

Martin Ek https://orcid.org/0000-0002-5705-8495
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