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The influence of surface defects on the mechanical and tribological properties of VN-based arc-evaporated coatings

M. Fallqvist *, M. Olsson

Dalarna University, SE-781 88, Borlänge, Sweden

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ABSTRACT

The influence of surface defects, i.e., droplets and craters, on the mechanical and tribological properties of arc-evaporated V_xN coatings deposited on cemented carbide has been investigated in a scratching contact using a diamond stylus and a sliding contact using a stainless steel pin. Post-test characterisation using 3D optical surface profilometry and scanning electron microscopy was performed in order to investigate the mechanical and tribological response of the coatings. The results show that scratch induced coating cracking mainly is restricted to larger droplets showing a low interfacial bonding to the adjacent coating matrix. The influence of coating defects on the cohesive strength, i.e., the tendency to chipping of small coating fragments, was found to be relatively small. In contrast, the presence of defects may have a significant impact on the interfacial adhesive strength, increasing the tendency to spalling. In sliding contact, surface defects such as droplets and craters have a strong impact on the tribological behaviour of the coatings causing abrasive wear of the less hard counter material surface and material transfer to the coating, both mechanisms affecting the friction characteristics of sliding contact tribo systems.

1. Introduction

PVD coatings are today frequently used in order to improve the tribological performance of cutting tools, forming tools and machine components and a large number of papers have been published in this field. In common for most PVD coatings, and especially arc-evaporated coatings, is the introduction of defects such as droplets (macro-particles), pits and cracks in the coatings. Somewhat surprisingly, relatively few papers have paid attention to how these types of defects influence on the mechanical and tribological properties of the coatings as well as their influence on coating adhesion [1–5].

In cathodic arc-evaporation a discharge between an anode and a cathode is used to melt a small spot of the cathode surface producing vapour particles of the cathode material. Compared with other PVD-techniques arc-evaporation shows a very high degree of ionization of the evaporated particles and consequently an applied bias can be used to control the energy of the particles hitting the substrate and thus the microstructure, defect density and properties of the resulting coating. The arc evaporation technique also allows the growth of dense coatings at relatively low temperatures [6]. However, the arc-evaporation process does not only involve the evaporation of ions and atoms from the cathode surface but also significantly larger particles usually referred to as droplets or macro particles which unfortunately will result in a rough coating surface morphology with hard protruding asperities. However, the studies focusing on droplets are limited in the open literature and mainly restricted to the characterisation of density number, size, angular distribution and chemical composition [7–9].

The fact that surface defects strongly influence on the mechanical strength of ceramic materials and the fact that the surface topography to a large extent controls the friction and wear characteristics of a coated tool/component illuminate the importance and necessity of understanding how these defects influence on the mechanical and tribological properties of the coating.

In the present study, arc-evaporated vanadium nitride based coatings were chosen as a model system in order to evaluate the influence of defects (droplets and craters) on the mechanical and tribological characteristics of the coatings. VN-based coatings are known to show a combination of high hardness and toughness and the possibility to form V_2O_5, a low friction Magnéli phase, promoting low friction coefficients in sliding contacts and consequently these coatings are of interest for sliding contact tribosystems [10–12].

The influence of coating defects on the mechanical properties, i.e., cohesive and interfacial adhesive strength, was investigated using single and multi-pass scratch testing using Rockwell C

* Corresponding author. Tel.: +46 70 540 76 25; fax: +46 23 77 80 50.
E-mail address: mfa@du.se (M. Fallqvist).

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diamond styli of different radii. The tribological performance of the coatings, and especially the influence of defects on friction and wear, was investigated in a sliding contact using austenitic stainless steel as counter material. Post-test characterization of the mechanical and tribological loaded surfaces were performed using 3D optical surface profilometry, scanning electron microscopy, energy dispersive X-ray spectroscopy and Auger electron spectroscopy.

2. Experimental

2.1. Materials

Commercial cemented carbide inserts with a composition of 94 wt% WC and 6 wt% Co were used as substrate material in the present study. Prior to coating deposition the substrate surfaces were polished to a surface finish of $R_a \approx 100$ nm. The vanadium nitride based coatings, V_2N and VN, were deposited by arc-evaporation using V-cathodes. The deposition process was performed at 450 °C in N_2 atmosphere (VN-coatings) and $Ar+N_2$ (flow ratio 10:1) atmosphere (V_2N coatings) at a pressure of 4.5 Pa and a substrate potential of −50 V. The coating growth rate was about 2.0 μm/h giving a coating thickness of about 4.5 μm. In order to evaluate the influence of surface defects on the mechanical and tribological properties of the coatings these were tested both in the as-received state and in a post-treated state in which macro particles (droplets) were removed from the surface by polishing using 3 μm diamond particles.

2.2. Coating characterisation

The surface morphology, microstructure and element composition of the as-deposited coatings were investigated using a 3D optical surface profilometer (WYKO NT-9100) and a ZEISS Ultra 55 field emission gun scanning electron microscope (FEG-SEM) equipped with an Oxford INCA Energy Dispersive X-Ray Spectroscopy (EDX) system. The microstructure of the coatings and particularly the presence of defects within the coatings were studied in cross-section. In order to promote a fully brittle fracture all cross sections were prepared by fracturing pre-notched samples at liquid nitrogen temperature. The defect density was evaluated using stereological methods (disector counting rule and point grid method) [13] and only defects larger than 0.4 μm were considered in five representative areas (30×50 μm) in the centre of each sample.

2.3. Mechanical and tribological testing

The Vickers hardness of the coatings and the coating/substrate composites were measured using a Micro Combi Tester (CSM Instruments) using a load of 200 mN and 4000 mN, respectively, where the maximum residual indentation depth for the lower load did not exceeded 10% of the coating thickness. The indents using the lower load were all done in flat areas free from visible defects, resulting in hardness values for the defect free coating material. Loading and unloading were performed for 30 s, respectively with a holding period of 15 s at the maximum load. By using the obtained indentation curves (load vs displacement) the hardness and Young's modulus were calculated according to Oliver and Pharr [14].

Conventional single pass scratch testing, using the CSM Instruments Revetest®, was performed using a 50 μm and a 200 μm radius Rockwell C diamond stylus. A scratch length of 10 mm, a loading rate of 10 N/mm and a normal load range of 0–100 N or 100–200 N were used in the experiments. In the experiments, two critical normal loads ($F_{N,C1}$ and $F_{N,C2}$, respectively) were used as a measure of the coating cohesive strength and interfacial adhesive strength and were taken as the normal load corresponding to the first coating failure ($F_{N,C1}$) and continuous substrate exposure ($F_{N,C2}$), respectively.

The Micro Combi Tester was also used for evaluating the wear/low cycle fatigue behaviour of the coatings under repeated/cyclic stresses. Multi-pass unidirectional scratch testing at constant normal load with a scratch length of 10 mm and a scratching velocity of 10 mm/min using a Rockwell C diamond stylus (50 μm radius) was performed in order to evaluate the influence of surface defects on the coating cohesion strength and interfacial (adhesion) strength during a repeated scratching contact [15–19]. The multi-pass scratch tests were performed at the critical normal load $F_{N,C1}$ (as obtained from the conventional scratch tests) and subsequently at decreasing normal loads using a step length of 0.5 N. The maximum number of cycles was set to 17. After testing all scratches were carefully characterized by the FEG-SEM in order to evaluate the influence of normal load and number of scratching cycles on the prevailing coating failure mechanisms.

The Revetest® was also used in order to evaluate the friction and material pick-up characteristics of the coatings (both in as-deposited and post-polished condition) in sliding contact against AISI 304 austenitic stainless steel (chemical composition; 0.06%C, 18.5%Cr, 1.5%Mn, 9%Ni, 71%Fe, hardness 200 HV_{20}). Multi-pass linear sliding contact experiments were performed at a normal load of 20 N, a velocity of 5 mm/min and a sliding distance of 10 mm using a stainless steel pin with a hemispherical shaped end surface (radius 5 mm). All experiments were performed in ambient air (21–22 °C, 30–32% RH). During testing the friction force and acoustic emission (A.E.) signals were continuously recorded.

In order to evaluate the tendency to material pick-up, tribo oxidation, etc., the element composition of the sliding wear tracks was analysed by EDX and Auger electron spectroscopy (AES). The AES analyses were performed using a PHI 660 Scanning Auger Microprobe (SAM) with an accelerating voltage of 10 kV and a primary beam current of 140 nA. Before analysing the surface was cleaned by sputtering (5 s) using 3.5 keV Ar^+ ion sputtering in the SAM system. The sputter rate was 18 nm/min as measured on Ta_2O_5 reference sample with known thickness (100 Å). Semi-quantitative analyses were made using the peak-to-peak height of the Auger transitions of a specific element together with sensitivity factors provided by PHI.

3. Results

3.1. Coating morphology and microstructure

Figs. 1 and 2 show the surface morphology of the as-deposited coatings investigated. As can be seen, both coatings show a pronounced roughness due to the generation of μm- and sub-μm-sized droplets during the deposition process. Of these, the larger droplets (> 3–6 μm in diameter) typically show an inter-droplet distance in the range of ~8–10 μm, i.e., they constitute a significant area fraction of the coating surface. Also, μm- and sub-μm-sized craters contribute to the observed roughness. When comparing the two coatings, see Table 1, it is observed that the metal rich V_2N coating shows a rougher surface mainly due to a higher number of relatively large droplets and areas of high droplet density. Polishing (using 3 μm diamond in the last step) of the coatings removes the protruding droplets but increases the number of craters which indicates a poor bonding between the droplets and the surrounding coating matrix.

Fig. 3 shows fractured cross sections of the VN coating and illustrates the rough surface morphology and the presence of