



The influence of surrounding atmosphere on tribological properties of hard protective coatings



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ARTICLE INFO

Available online 3 December 2014

Keywords:

Tribology
Atmosphere
Hard coatings
Pin-on-disc

ABSTRACT

In this research paper we compare the tribological properties of conventional hard coatings (TiN, CrN, TiAlN) and more advanced protective coatings ($\text{Cr}_x\text{Al}_{1-x}\text{N}$ and $\text{Cr}_x\text{V}_{1-x}\text{N}$). Numerous factors can influence the coating tribological properties; thus we limit our research to evaluate the tribological behaviour in different surrounding atmospheres. We performed tribological tests in the air (high, medium and low humidity), nitrogen and oxygen environment. Experiments indicate that oxygen-rich environment causes the formation of oxides at lower temperatures while the use of the nitrogen environment can effectively stop the oxidation process which results in a reduction of wear. We encountered different friction and wear behaviour in dependence of the atmosphere type. To fully understand the processes during tribological testing, the wear mechanisms were investigated for each hard coating separately. The wear debris and the wear tracks were analysed by SEM, focused ion beam (FIB), 3D-stylus profilometry and confocal optical microscopy.

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1. Introduction

The development of new PVD hard coatings for protection of cutting tools is stimulated by the requirements to increase machining productivity, by environmental mandates and by the need to machine difficult-to-cut materials. A huge progress has been achieved in the development of PVD hard coatings through the design of composition and microstructure ranging from simple, first-generation binary coatings like TiN [1–4] and CrN [5,6] to multicomponent (TiAlN) [7], multilayer (TiN/TiAlN) [8,9] and nanostructured coatings [10,11]. Recently the emphasis has been on the development of coatings based on oxides and oxynitrides due to their superior hot hardness and chemical inertness [12].

Nowadays, the new industrial techniques impose environmental and production mandates. In cryogenic machining [13,14] conventional cooling and lubricating fluids (CLFs) are replaced by the liquid nitrogen. Although the main efficiency gain comes from lower tool temperature, there is another aspect of nitrogen not so frequently mentioned; when the liquid nitrogen evaporates, it also forms inert atmosphere around the cutting edge-workpiece contact surface. Food and pharmaceutical industries also require high standards for controlling the manufacturing environment. The majority of reports on environmental influences are focused on the diamond-like coatings [15,16]. In many of the new

industrial techniques the conventional hard coatings are used, but reports of their tribological and wear properties in different environments are rare [3,4,17,18]. Wilson et al. [3,4] showed that the wear changes from a high wear regime by TiN oxidation in the air to a mild TiN wear regime in a low-oxygen atmosphere. Gant et al. [17] also compared wear behaviour of several engineering coatings in an ambient air and dry nitrogen.

In this work the influence on friction and wear of the surrounding atmosphere is reported. We used different gases, including the air, nitrogen and oxygen, in standard reciprocal tribological tests. Such experiments show that the oxidation processes are accelerated by an oxygen-rich environment or they can be prevented in a nitrogen atmosphere. Comparing the tribological properties in these environments to those obtained at ambient air conditions provides useful information about the role of oxidation and humidity in such systems. Nevertheless, additional tests were conducted in the high and low air humidity environment. We selected conventional TiN, CrN and TiAlN and more advanced (CrAlN , CrVN) hard coatings for tribological tests. All these coatings were prepared by magnetron sputtering in an industrial deposition system.

2. Experimental

The industrial magnetron sputtering system CemeCon CC800/7 was used for the deposition of conventional TiAlN (Ti:Al = 55%:45%) and CrN coatings using TiAl and Cr targets, while the $\text{Cr}_x\text{V}_{1-x}\text{N}$ and $\text{Cr}_x\text{Al}_{1-x}\text{N}$ coatings were prepared in the CemeCon

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CC800/9 deposition system using triangular targets [19]. Both are DC magnetron sputtering systems with four unbalanced targets. For the deposition of $\text{Cr}_x\text{V}_{1-x}\text{N}$ and $\text{Cr}_x\text{Al}_{1-x}\text{N}$, we used two sets of triangular targets, which were faced diagonally, and two pure Cr targets also facing diagonally. This approach enabled us to prepare a large set of samples of different compositions in a single deposition run therefore enabling us a significant reduction of time and costs of the development of coatings. The XRD spectra of all the coatings used in our experiments are presented in Fig. 1. The crystal structure of all the coatings is f.c.c. We notice the (111) peak shift in the Cr-based coatings due to alloying with aluminium or vanadium (up to 12% of alloying when using Al or V triangular targets). Different deposition rates of Cr versus Al or V were detected in vertically positioned samples. Because of this, a small variation of the peak width, i.e. grain size, can be noticed in Cr-based coatings.

Polished tool steel, alumina and silicon wafer substrates were mounted on a one-, two- and threefold rotating substrate holder. In this way we prepared nanolayered coatings composed of two different layers, i.e. CrN/ $\text{Cr}_x\text{V}_{1-x}\text{N}$ and CrN/ $\text{Cr}_x\text{Al}_{1-x}\text{N}$.

The thickness of the coatings was measured by the ball crater method. The film thickness of CrVN and CrAlN varied from 3.7 to 8.5 μm and from 4.5 to 7 μm , respectively. The thickness of the vanadium and aluminium-rich coatings is significantly lower than of the chromium-rich ones due to the difference in sputtering yield [19,20]. Individual thickness values are presented in Table 1. The tool steel samples were ground and polished by a 3 μm diamond paste. All samples were ultrasonically cleaned and sputter-etched prior to coating deposition.

The friction and wear tests were conducted by using the standard CSM tribometer with a cover and a built-in gas inlet. The tribometer was set to reciprocal sliding mode. A 6 mm radius Al_2O_3 ball was used as a counter body ($\text{HV} \approx 2000$). An Al_2O_3 ball was chosen as the counter body due to its inertness to high oxidising atmosphere. In the literature [7,8,21–23] the Al_2O_3 ball has also been widely used in wear testing of hard coatings. Thus the current results can be better compared to those in the literature. For all the experimental results in this report the normal load was 5 N and the movement frequency was 2 Hz over a 5 mm track. The number of cycles was set to constant 5000 for all the hard coatings with an exception of TiAlN which was set to 3000 due to premature wear off the coating. The cleaning procedure was the same for all coatings. The samples were cleaned with ethanol in the ultrasonic bath for 10 min, dried, wiped with 2-propanol and purged with nitrogen. The sample was purged after each individual test by blowing with the dry nitrogen to remove the loosely attached wear particles.

A constant flow of pure gas was blown through the tribometer chamber. We used nitrogen and oxygen both of 99.9% purity. Before the experiment, we let the constant flow of the gas through the closed

chamber for at least 10 min. For ambient air atmosphere experiments, the gas flow was closed and the tribometer chamber was ventilated for approximately 10 min. The humidity meter (Votcraft HT-200) was placed in the vicinity of the test sample to measure the humidity level. The general ambient laboratory conditions were 24 °C and approximately 40% relative humidity (RH); as in the case of the nitrogen or oxygen atmosphere the humidity level was 0%. Wear tracks were analysed by confocal optical microscopy (COM), scanning electron microscopy (SEM) and 3D stylus profilometry. In order to detect chemical changes on the surface of the wear track images with backscattered electrons (BSE) were recorded. For the evaluation of the chemical composition change the energy dispersive spectroscopy (EDS) was used.

3. Results and discussion

3.1. Friction and wear in ambient air

The tribological tests were first performed in an ambient air atmosphere (Fig. 2). Within the first few cycles we noticed a rapid increase in friction coefficient from 0.2 to a higher value. The highest jump in friction coefficient exhibited the TiAlN coating with the maximum value of 0.9. Other coatings exhibited an initial jump, reaching values around 0.4. The CrVN and CrAlN, on the other hand, showed different friction behaviour; they immediately reached the peak friction coefficient. During the first 100 cycles the coatings behaved differently. The friction coefficient increased from 0.3 to 0.4 and from 0.4 to 0.8 for CrN and TiN. TiAlN coating friction coefficient rose to a stable value of 1.0 after approximately 1000 cycles, not changing significantly throughout the test. CrVN and CrAlN (6% Al or V) coatings both experienced a decrease of the friction coefficient with an exception of the rapid friction coefficient drop for CrAlN coating from 0.3 to 0.2 after 2800 cycles.

The wear volumes (Fig. 2b) of the coatings correlated well with the friction values, being the highest for TiAlN and TiN. Chromium-based coatings exhibit only minor wear, approximately one tenth of that for TiN.

3.2. Friction and wear in nitrogen

The results of tribological tests in pure nitrogen atmosphere are presented in Fig. 3. The pure nitrogen atmosphere changed the tribological system completely. The inert and moisture-free environment during the test caused different friction and wear behaviour in comparison to that in the air. At the early stage of the test the coatings behave similarly with an exception of TiAlN and CrAlN. Both aluminium-based coatings exhibit a friction bump that gradually subsides closer to the value of the other coatings. In the literature such behaviour is explained by formation of hydrated oxides that can form during storage of test samples in the ambient air [17,24]. The top oxide layer is worn down after some initial laps in the pure nitrogen environment, while the oxide layer is continuously growing when testing in the pure oxygen or ambient air environment [25]. Other coatings experience a slower increase, reaching close to steady-state value after a maximum of 1000 cycles. The overall average friction is slightly higher for all Cr-based coatings, while TiN and TiAlN experience a friction reduction in comparison to the ambient air.

The wear rate for all the coatings tested in nitrogen decreased in comparison to those obtained in the air conditions (Fig. 3b). TiN and TiAlN exhibit up to 10 times lower wear rate, while with the chromium-based coatings the wear rate change was smaller.

3.3. Friction and wear in oxygen

In contrast to nitrogen, the pure oxygen stimulates the formation of oxide phases during tribological tests. The tribological system is closer to that of the ambient air, except the oxygen concentration is higher and the moisture is removed from the system.

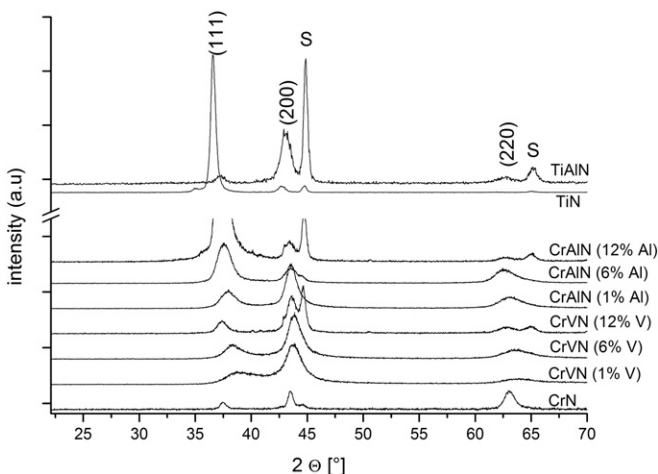


Fig. 1. XRD spectra of all the hard coatings that were used in the experiment.

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