



Mechanical properties of Cr-O-N coatings deposited by cathodic arc evaporation



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ABSTRACT

Ternary chromium based coatings (Cr-O-N) were formed on HS6-5-2 steel substrates using a vacuum arc plasma flux coating system at different relative oxygen concentrations, $O_{2(x)} = O_2/(N_2 + O_2)$. The mechanical and tribological properties of the coatings were characterized using scanning electron microscopy (SEM), atomic force microscopy (AFM), hardness measurements, wear test and adhesion tests (scratch test and Daimler-Benz test). Special attention was paid to coating adhesion and failure modes in the adhesion tests. The coefficient of friction, wear rate and adhesion decreased as the relative oxygen concentration increased. Hardness of the coatings showed the opposite effect, increasing from approximately 19 GPa–30 GPa. The dominant failure mode of Cr-O-N coatings was abrasion. Individual micro fragmentation was also observed for Cr-O-N coating with $O_{2(x)} = 20\%$.

1. Introduction

To improve durability of tools and machine parts, protective coatings characterized by high hardness and adhesion, corrosion and abrasion resistance, high temperature strength and chemical stability are required. One of such groups is the transition metal nitrides. Among these, chromium nitride coatings applied by physical vapor deposition (PVD) fulfill some of the demands [1–4].

Mechanical or chemical properties of two-component CrN coatings are not always sufficient. For example, CrN thin coatings are characterized by rather low hardness compared to other materials, such as titanium nitride or titanium aluminum nitride. This feature limits their applications. By adding a third element to the CrN coating microstructure, various properties can be improved, such as wear resistance and thermal stability. Many publications have reported the results of ternary systems Cr-X-N, where X is a metallic or non-metallic element, such as boron [5,6], carbon [7,8], aluminum [9,10], silicon [11], titanium [12] and vanadium [9]. It should be mentioned that the Cr-O-N system was also investigated, but the main topic of interest was the structure and morphology of the coatings. The mechanical and tribological properties were not a major focus of investigations [2], [13–20].

The findings of Ho et al. [21,22] indicate that Cr₂O₃/CrN coatings

can be applied to aluminum die casting due to good oxidation resistance, excellent thermal stability and high hardness. This ternary (Cr-O-N) system is very interesting as, at increased temperature, partial transformation of CrN → Cr₂O₃ can be observed and a passive Cr₂O₃ layer with interstitial nitrogen is formed. Such a layer, with a fine nanocrystalline zone at the surface [19], prevents oxygen diffusion through the coating at high temperature [13,18].

The Cr-O-N coatings can be widely applied on tools in order to protect them against oxidation [14]. They can also be used in photo-thermal solar energy conversion as solar selective absorber coatings [23] or as decorative coatings due to their distinct colors [24].

Chromium oxynitride thin coatings are formed by different methods, including reactive sputtering [2,23], pulsed laser deposition [17,25], arc ion plating [16] and cathodic arc deposition [13,14,26,27]. All of them enable deposition of a coating with the required chemical composition. For industrial applications, magnetron sputtering and cathodic arc evaporation are mainly used to deposit the coatings. These two PVD methods differ significantly and have advantages and disadvantages. The coatings obtained using cathodic arc evaporation (CAE) show much denser microstructure and better adhesion strength between the coating and the substrate material compared to coatings deposited using magnetron sputtering [28,29]. The other benefits of

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CAE technique are the high degree of particle ionization and high deposition rate. The kinetic energy of the particles reaches tens or even hundreds of electron volts, which is often greater than the energy for atom displacement near the surface [30]. However, the disadvantage of the coatings obtained by cathodic arc evaporation is the large number of micro-droplets from the cathode surface.

The hardness was the most often tested mechanical property. Suzuki et al. [25] indicated that the hardness of Cr-O-N thin films prepared by pulsed laser deposition increased due to oxygen substitution for nitrogen in the CrN structure at amount of oxygen up to 37 at.%. Above this level, hardness decreased. In turn, the results of investigations of CrON coatings prepared by magnetron sputtering, presented by Collard et al. [2], indicated that, with an increase in oxygen content, hardness not monotonically changed. Gautier and Machet [14] found that Cr-O-N coatings obtained by vacuum arc evaporation in a wide range of oxygen concentrations were characterized by almost constant hardness. Urgan et al. [27] found that addition of oxygen to CrN formed using reactive cathodic arc evaporation reduced the hardness of the coating from 30 GPa to 21 GPa. These divergent results led us to attempt to address this issue.

Other mechanical and tribological properties have been rarely presented in the literature. The studies of Urgan et al. [27] on coatings deposited using cathodic arc evaporation indicated that the coefficient of friction ranged from 0.2 to 0.5, dependent on the chemical composition of the coatings. It should be noted that the reciprocating testing system with a load of 2 N, amplitude of 1 mm and 10 Hz frequency was used in these investigations. The adhesion of coatings using scratch test was investigated by Gautier and Machet [14]. They found that the coatings deposited at higher oxygen flow rate present reduced adhesion from about 50 N for almost pure CrN coating to about 10 N for coating deposited at higher oxygen flow rate. They stated that this behavior is probably related to chemical composition and structure of the coatings. Some data on tribological properties, especially using the AFM method, is presented in Ref. [31]. It was found that the coefficient of friction of the coating with respect to diamond probe with radius of 10 nm and applied load 230 nN ranges from 0.1 to 0.3 dependent on its chemical composition. The wear rate ranges from 1×10^{-13} to 2×10^{-14} m³/Nm and is about two orders higher than the wear rate determined in ball-on-disc method.

The structure of the Cr-O-N coatings presented here, including chemical and phase composition, has previously been investigated [20]. It was found that CrON coatings deposited in relative oxygen concentration $O_{2(x)}$ with x amounting to 0, 5 and 20% show a CrN cubic lattice, even to 23 at.% oxygen concentration. The coating obtained with a relative oxygen concentration of 50% had a rhombohedral Cr₂O₃ structure. The crystallite size calculations showed their reduction from approximately 109 nm–23 nm in coatings deposited at 0 and 20% of relative oxygen concentration and half that (approximately 11 nm) for $O_{2(50)}$. The decrease in grain size with increasing oxygen concentration in the coating could be a result of the formation of undetected different phases of low solubility on the growing surface by XRD [13]. The lattice parameter increases from 0.41847 ± 0.00003 nm for coating deposited at $O_{2(0)}$ to 0.4187 ± 0.0001 nm for coating deposited at $O_{2(20)}$.

The coatings have also been investigated by Raman spectroscopy. The tests confirmed that only CrN structure was present in coatings deposited with relative oxygen concentrations of 0–20%, and Cr₂O₃ structure in coatings synthesized with relative oxygen concentration of 50% [20].

The goal of this study was to investigate Cr-O-N coatings deposited using cathodic arc evaporation with different relative oxygen concentration $O_{2(x)} = O_2/(N_2 + O_2)$. This work is a continuation of the research reported in Ref. [31]. The mechanical and tribological properties, such as hardness, adhesion, friction and wear were characterized using scanning electron microscopy (SEM), atomic force microscopy (AFM), nanoindentation, wear test and adhesion tests (scratch test and Daimler-Benz test).

2. Experimental details

2.1. Technology

The deposition process of CrN and CrON coatings was performed by the unfiltered cathodic arc evaporation method using a “Bulat 3T” system. It was equipped with a Cr (99.99%) cathode of 60 mm in diameter. A vacuum arc plasma source with magnetic stabilization of a cathode spot was used [32]. Samples of 32 mm in diameter and 3 mm in thickness were taken from HS6-5-2 steel (EU standard) with the following chemical composition (wt.%): C - 0.87, W - 6.4, Mo - 5.0, V - 1.9, Cr - 4.2, Mn - 0.3, Si - 0.4 and Fe balanced. Samples were quenched and tempered to a hardness of 63 HRC and then polished using abrasive silicon carbide paper to a mean roughness Ra of about 0.02 μm. The samples were chemically degreased, ultrasonically cleaned in a hot alkaline bath for 10 min and then dried in warm air. After that they were placed within a distance of 300 mm from the cathode in the vacuum chamber on a planetary rotating holder with a rotation speed of about 30 rpm.

The chamber was evacuated to a pressure of 2×10^{-3} Pa. Substrates were ion etched with chromium ion bombardment by applying a DC bias of –1300 V for 3 min. The arc current was 90 A. To improve adhesion of CrN and Cr-O-N films, a pure chromium layer (about 0.1 μm thick) was deposited on the substrate at a bias voltage of –100 V for 5 min. Nitrogen at a pressure of 1.8 Pa was used as a reactive gas. The substrate temperature was maintained at about 400 °C. In case of Cr-O-N coatings, a gas mixture (N₂ + O₂) with different relative oxygen concentrations, $O_{2(x)} = O_2/(N_2 + O_2)\%$, was used where x equals 0, 5, 20 and 50%. The deposition process was performed at a substrate bias voltage of –150 V for 45 min in all cases. The thickness of all deposited coatings was about 7 μm.

Taking into account the relative concentration of oxygen, deposited coatings were labeled as follows Cr-O(x)-N. This means that, for example, Cr-O(20)-N coating was obtained at a relative concentration of oxygen of 20%. In addition, the notation Cr-O(0)-N coating has been simplified to CrN.

2.2. Characterization

Hardness and elastic modulus of the coatings were determined by nanoindentation using a Nano Indenter-G200 system (Agilent Technologies, USA) equipped with Berkovich diamond tip. Ten indentations were made on each sample. The depth of indentation was approximately 500 nm.

The adhesion of the coatings to the substrate was characterized using the scratch test (REVETEST). The tester was equipped in an acoustic sound sensor which enabled determination of the loss of coating adhesion. The specimens were scratched with a standard sliding speed of 10 mm/min. Normal force varied linearly from 0 to 100 N or 150 N. The L_{c1} critical load was defined as the minimum load at which damage first appeared in the coating, and the L_{c2} critical load as the load at which the coating was totally detached from the substrate. These loads were evaluated by observations using an optical microscope and expressed as the mean of at least three measurements. In order to investigate the coating failure mechanism, Cr-O-N-substrate systems were indented using a Rockwell C indenter with a load of 1470 N [33].

The wear rate was defined as the wear volume V divided by sliding distance s and normal load L [34]: $k_v = \frac{V}{Ls}$. The wear volume was estimated from the sectional area of wear S (made perpendicular to wear track) in the coating and the perimeter of wear track. The area of five randomly chosen wear profiles were averaged. It was computed based on studies carried out with the ball-on-disk test with a load of 20 N and sliding speed of approximately 0.2 m/s under dry friction conditions at a distance of 1000 m. The radius in wear tests was 12 mm. An alumina ball with a diameter of 10 mm and Ra < 0.03 μm was used as a

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