



Microstructure and wear performance of arc-deposited Ti–N–O coatings on AISI 304 stainless steel

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ABSTRACT

A cathodic arc plasma deposition system (with variation of the O_2/N_2 flow ratio) was used to synthesize different Ti–N–O films on the AISI 304 stainless steel. The coating morphologies and structures were analyzed by using SEM, XRD, and TEM. The coating properties, such as adhesion, hardness, elastic modulus, and wear behavior, were explored to evaluate the impact of the O_2/N_2 flow ratio on the coating microstructure and properties. The results showed that the O_2/N_2 ratio properly controlled at 0.25 could produce an optimal film with a dense crystalline structure consisting of TiN, anatase-TiO₂, and rutile-TiO₂ phases, showing the best adhesion strength using a standardized ball indentation method, highest nano-indentation hardness (22.8 GPa), and highest elastic modulus in comparison to other coatings. AISI 304 stainless steel with the Ti–N–O coating not only reduced the friction coefficient from 0.8 to 0.35, but also remarkably improved the adhesion wear resistance.

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1. Introduction

AISI 304 stainless steel possesses excellent corrosion resistance, oxidation resistance, formability, and weldability. Due to its desirable properties, the stainless steel has been widely adopted not only in common use, such as appliances and architecture, but also in sophisticated technologies, such as cryogenic devices and nuclear reactors [1,2]. For all its advantages, however, the steel with an austenitic matrix in microstructure is unsatisfactory for specific industrial applications that require good performance in wear resistance. Fortunately, the wear resistance of engineering materials can be improved by depositing appropriate hard coatings [3]. For example, various ceramic nitride films, such as TiN, CrN, ZrN, TiAlN, and their combinations, are used to prolong the lifetime of materials exposed to abrasive environments [4–11]. In particular, titanium nitride, with its pleasing golden color and high hardness, is often selected as a coating material to improve the wear resistance of austenitic stainless steels [12–14]. Makino et al. [15] also reported that Ti–N–O ternary coatings could be successfully synthesized using the arc ion plating method, which achieved a dramatic efficiency in characterization. For instance, titanium nitride films improved their electric resistivity through the addition of oxygen. Moreover, a suitable addition of oxygen to TiN was conducive to raising the hardness of TiN film. A previous study [16] used sputtering to coat AISI 304 stainless steel with different hard films (such as CrN, TiCN,

DLC) with the main purpose of enhancing the corrosion resistance without regard for the wear resistance. Though various ceramic hard films have been explored as mentioned above, there is still a lack of information on the wear resistance performance of Ti–N–O films on AISI 304 stainless steel.

This study investigated the microstructure and wear behavior of Ti–N–O films coated on AISI 304 stainless steel. The PVD coatings were deposited on the steel through a cathodic arc plasma deposition (CAPD) system with two titanium targets and O_2 and N_2 reactive gases. Coating characteristics, such as structure, chemical composition, surface roughness, adhesion, hardness and elastic modulus were analyzed. In addition, the wear tests were carried out to evaluate the effects of the O_2/N_2 flow ratio on the friction coefficient and wear rate of the coated specimens.

2. Material and methods

2.1. Material preparation and CAPD treatment

In this study, the substrates were made of commercial AISI 304 stainless steel (18–20%Cr, 8–10.5%Ni, 0.08%C, 1.0%Si, 2.0%Mn, 0.03%S, 0.045%P, and balanced Fe in wt%), and had a circular shape with a diameter of 20 mm and a thickness of 5 mm in size for the wear tests. Prior to the CAPD process, the substrates were mechanically ground and polished to an average surface roughness of 0.04 μm (Ra value) and thoroughly cleansed in an ultrasonic bath. The substrates were then fixed in the vacuum chamber and subjected to an argon ion bombardment (ion etching) at a substrate

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bias of -1000 V for 10 min to ensure a clean surface for good adhesion of the deposited films. The argon pressure was set at 0.1 Pa during ion etching. Two titanium targets were placed on opposite walls of the vacuum chamber to carry out the coating experiment. The N_2 and O_2 reactive gases were fed into the chamber simultaneously to obtain Ti–N–O films. In this study, the $O_2:N_2$ gas ratio were controlled at three levels: 0 (0:85 sccm), 0.25 (17:68 sccm), and 0.5 (28:56 sccm). The distance between substrate and target was about 15 cm. Table 1 lists the details of the processing parameters.

2.2. Analysis of coating characteristics

A field emission scanning electron microscopy (FESEM, LEO 1530) was used at an accelerating voltage of 15 kV to observe the surface morphology and to measure the coating thickness of the specimens. A glancing incidence X-ray diffractometer (XRD, Rigaku-TTRAX III) was employed to identify the coating structure with a Cu-target $K\alpha$ radiation at 40 kV and 30 mA and a glancing incident angle of 2° ; the scanning angular (2θ) ranged from 20° to 80° at $2^\circ/\text{min}$. The structure of the optimal film was further examined using a transmission electron microscope (TEM, Philips TECHNAI G2F20, 200 kV). The chemical composition of the Ti–N–O films was determined by the quantitative electron probe micro-analysis (EPMA, JEOL JXA-8200) with the mode of line scanning for Ti, N, and O elements. A surface roughness analyzer (Mitutoyo SV-400) was applied to measure the average surface roughness (Ra value) for each specimen. Adhesion strength quality (ASQ) of the coatings was evaluated by using Rockwell-C indentation testing with a load of 1471 N [17]. The damage to the coatings was compared with a defined ASQ basis, where HF1–HF4 were acceptable adhesion and HF5–HF6 represented insufficient adhesion (HF is the German short form for adhesion strength). The nano-indentation experiments were performed using a nano-indenter (MTS XP system) with a diamond Berkovich tip. According to the Oliver and McHargue method [18], the values of nano-indentation hardness (nH) and elastic modulus (E) for the coatings were obtained by analyzing load–displacement curves using a load of 15 mN. The averages of 20 indentations for each film were reported as the resultant values.

Table 1
Parameters of the coating process in this study.

Target material (wt%)	Ti (99.9) × 2
Working pressure (Pa)	2.7
Substrate bias (V)	−50
Cathode current (A)	40
O_2/N_2 ratio (flow rate, sccm)	0/85=0, 17/68=0.25, 28/56=0.5
Substrate temperature ($^\circ\text{C}$)	200
Rotation rate (rpm)	4
Deposition time (min)	50
Source to substrate distance (cm)	15

Table 2
Chemical composition and properties of various coatings in this study.

Specimen	Concentration (at%)			Film thickness (μm)	Surface roughness, Ra (μm)	Adhesion strength, ASQ [17]	Film hardness, nH (GPa)	Elastic modulus, E (GPa)	nH/ E ratio
	Ti	N	O						
S1 ($O_2/N_2=0$)	50.3	49.7	–	1.50	0.25	HF2	19.6 ± 1.0	204.8 ± 10.2	0.096
S2 ($O_2/N_2=0.25$)	40.2	21.5	38.3	2.12	0.17	HF1	22.8 ± 1.1	217.5 ± 10.9	0.105
S3 ($O_2/N_2=0.5$)	35.4	10.7	53.9	1.76	0.26	HF4	16.9 ± 0.8	193.6 ± 9.7	0.088

2.3. Wear tests

Friction and wear properties of the coated and uncoated specimens were evaluated in wear tests using a ball-on-disc tribometer (CSM Instruments, Switzerland). The counterface material was a 6 mm diameter WC-6%Co ball, which had a hardness of 1780 HV and a surface roughness of approximately $0.2 \mu\text{m}$ (Ra). As frequently selected conditions for coating tests [19,20], the applied load was kept at 5 N with a linear sliding speed of 0.2 m/s. All the tests were conducted without lubricant at an ambient temperature of 25°C as well as 65% relative humidity (in the laboratory atmosphere). For each coating condition, the wear tests were performed three times. The relationship between the friction coefficient and the wearing time of about 3000 s (total travel distance: 600 m) was continuously recorded during the tests. Furthermore, the wear rate of specimen was determined from weight loss divided by total travel distance. The weight loss of each specimen after the wear test was measured with a micro-balance ($\pm 1 \times 10^{-4}$ g). In addition, the worn surface of each specimen after the wear test was observed using SEM.

3. Results and discussion

3.1. Coating composition and structure

Based on the different O_2/N_2 ratios, the three coated specimens in this paper were marked as S1 (for N_2 only), S2 (for $O_2/N_2=0.25$), and S3 (for $O_2/N_2=0.5$), respectively. Atomic concentration in each film analyzed by the EPMA method was obtained as listed in Table 2.

With only N_2 used, the S1 specimen had almost the same contents in Ti (50.3 at%) and N (49.7 at%) elements in its Ti–N binary film. When oxygen was added during deposition to form a ternary film, as in the case of the S2 specimen ($O_2/N_2=0.25$), the content of oxygen in the Ti–N–O film was higher than that of nitrogen (38.3 vs. 21.5 at%). This raise in the oxygen content was also observed when the O_2/N_2 ratio was at 0.5 (S3); the oxygen content in the film increased (38.3 \rightarrow 53.9 at%) while both the nitrogen content (21.5 \rightarrow 10.7 at%) and the titanium content (40.2 \rightarrow 35.4 at%) decreased substantially. This observation of the effect of oxygen addition on the Ti–N–O film is consistent with related studies [15,21]. One conjectures that the affinity of titanium and oxygen is stronger than that of titanium and nitrogen. We found, as compared with the use of N_2 only (S1), there was an increase in the film thickness of S2 ($O_2/N_2=0.25$) but a decrease in that of S3 ($O_2/N_2=0.5$). One plausible explanation for such a finding may lie in the fact that oxygen addition during film deposition produces power reaction of Ti and O ions, which heightens the deposition rate. Nevertheless, an excess of oxygen during deposition may also have a drastic impact on the interactive ions such that the deposition rate is lowered. The specimens, ranking in terms of the averaged film thicknesses, are as follows: S2 ($2.12 \mu\text{m}$) > S3 ($1.76 \mu\text{m}$) > S1 ($1.50 \mu\text{m}$).

Fig. 1 compares the crystal structure of the coatings in terms of the XRD patterns. The results showed that S1 had peaks mainly on

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