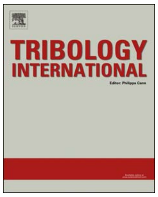
Tribology International 109 (2017) 285–296

Contents lists available at ScienceDirect

Tribology International

journal homepage: www.elsevier.com/locate/triboint



Influence of carbon contents on the structure and tribocorrosion properties of TiSiCN coatings on Ti6Al4V

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ARTICLE INFO

Keywords:
TiSiCN coatings
Arc ion plating
Structure
Tribocorrosion properties

ABSTRACT

The TiSiCN coatings were fabricated on Ti6Al4V. The effect of carbon contents on the structure and tribocorrosion properties were studied. The coatings consist of TiN, TiC and Ti(C,N) nanocrystallites, amorphous Si_3N_4, SiC and little amorphous carbon. The coating (11.9 at% C) exhibits maximum hardness of 39.8 GPa and the best tribocorrosion resistance, which is attributed to the special nanocomposite structure, and the lower friction coefficient of the TiSiCN coating associated with the graphitization effect during sliding in atmosphere. Moreover, the results of tribocorrosion indicate that the volume loss of TiSiCN coatings ascribed to main wear loss and a little corrosion loss, and the synergy between wear and corrosion is a key factor in degenerating the TiSiCN coating.

1. Introduction

Ti6Al4V alloy is widely applied in the fields of navigation, aerospace, engine turbine and ocean exploration due to its excellent combination of low density, high strength-to-weight ratio, high toughness, excellent resistance to corrosion [1,2]. However, the low hardness and poor wear resistance limit its application [3]. In order to improve the wear resistance of the Ti6Al4V alloy, the thermal oxidation, nitriding and coatings have been employed to guarantee its stable running as key components [4–8].

The coating is one of the most common surface treatments, which not only can increase the hardness of the materials, but also improve the wear resistance and thus prolong the life of the components [9,10]. The TiCN coating with complete solid solubility in the B1 cubic phase, which has obvious advantages including low friction coefficient and excellent corrosion resistance, but has low ductility at room temperature, which limits its practical application [11]. While the element Si was incorporated into TiN coating to form special nanometer-size TiN grains in the matrix of amorphous Si_3N_4 structure—TiSiN coating, which exhibits superhardness, improved wear resistance and oxidation resistance properties, however, it has high friction coefficient [12]. The quaternary TiSiCN system, which combines the advantages of TiCN and TiSiN, performs high hardness, excellent wear resistance and self-lubrication, which was prized under heavy load condition and in high humidity or corrosive medium [13–16]. Up to now, many reports have been focused on investigating the effect of the deposition conditions and chemical compositions of the TiSiCN nanocomposite coating. Ma et al. found that the influence of the C contents on mechanical and tribological properties of TiSiCN coating using PECVD technology and revealed the strengthening mechanism of the coating [17]. The influence of the sources of C on microstructure and properties of TiSiCN coating was investigated by Eriksson et al. [13]. Wei et al. investigated the influence of the silicon contents by varying the TMS gas flow rate on microstructure and corrosion properties using magnetron sputter technique [18]. There is a few investigation about the tribocorrosion property of TiSiCN coating in seawater. Besides, coating failure is accelerated resulting from synergistic action of wear and corrosion which was defined as tribocorrosion that conforms the real running environment and evaluated failure mechanism in the corrosive medium.

The TiSiCN coating combines the excellent wear and corrosion resistance, which may exhibit great performance in the tribocorrosion property. In this paper, the TiSiCN coatings are prepared on Ti6Al4V by arc ion plating. The influence of the carbon contents on structure and tribocorrosion behavior is studied and we evaluate the tribocorrosion mechanisms of degradation of TiSiCN coating simultaneously.

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http://dx.doi.org/10.1016/j.triboint.2017.01.002
Received 6 October 2016; Received in revised form 5 December 2016; Accepted 3 January 2017
Available online 04 January 2017
0301-679X/ © 2017 Published by Elsevier Ltd.

2. Experimental details

2.1. Coating deposition

The TiSiCN coatings were deposited on Ti6Al4V alloy with 20 mm×30 mm×3 mm by arc ion plating technique (Hauzer Flexicoat 850). The Ti6Al4V alloy substrates were ground using abrasive papers from 1000# to 7000#, and then polished with 0.5 µm diamond grinding paste. Besides, the Si wafer (p-type (100)) with dimensions 10 mm×10 mm×0.625 mm was fixed on the substrate seat as well to be coated TiSiCN coating synchronously which provided additional samples to analyze the microstructure. Deposition of the TiSiCN coating was carried out in an Ar+C_2H_2+N_2 mixture gases with TiSi targets (90 at% Ti, 10 at% Si; purity 99.99 at%). Prior to deposition, the chamber was heated to 450 ℃ and pumped to 4×10^{-5} mbar, then the specimens were etched by Ti ions for 3 min under the bias voltage of −900 V, −1100 V and −1200 V, respectively. A stress gradient TiN transition layer was first deposited to improve the adhesion between the TiSiCN coatings and substrates. In order to deposite the TiSiCN coatings with different carbon contents, we designed four variates of the proportion of C_2H_2 and N_2, of which the amount of C_2H_2 and N_2 remained constant and the chamber pressure remained stable by argon. The deposition time of all TiSiCN layers was kept fixed 1 h. The deposition parameters are listed in Table 1.

2.2. Characterization

The cross-sectional images of TiSiCN coatings were observed using secondary electron microscopy mode by field emission scanning electron microscope (Hitachi S4800). The phase structure of the coatings was obtained by X-ray diffraction (Bruker D8 X-ray facility) with Cu K_α radiation. The scanning angle ranged from 10° to 90° at a scanning speed of 10°/min with a 0.02° step size. The element chemical states of the coatings were characterized by X-ray photoelectron spectroscopy (AXIS ULTRA^{DLD}, Kratos) with Al Kα X-ray source. The C1s peak at 284.8 eV was used as reference for the energy calibration. All curve-fitting was carried out using a non-linear least squares fitting method employing the Gaussian–Lorentzian function and considering the background as linear and shirley type. The hardness and elastic modulus of the coatings were measured by nanoindentation using continuous stiffness measurements by MTS Nano Indenter G200 instrument with a Berkovich diamond indenter. The hardness and elastic modulus of the coatings were chosen in a depth of around 1/10 of the coating thickness, which was not affected by the substrate. For each sample, 8 indentation tests were performed to obtain the mean value. The structure of the coating was characterized by high resolution transmission electron microscope (FEI Tecnai F20). The chemical compositions of the coatings were identified by energy dispersive X-ray spectroscopy.

The tribological properties of the coatings were evaluated by performing dry friction tests against a ZrO_2 ball (φ=6 mm) in atmosphere at room temperature (25 ± 5 °C) and relative humidity of 80 ± 5%. The tests were performed under reciprocating sliding movement in a ball-on-disk tribometer (Rtec Instrument). The parameters were as follows: load of 5 N, sliding length of 5 mm, the frequency of 2 Hz and sliding time of 3600 s.

Table 2
Chemical composition of artificial seawater.

Compound	Concentration (g/L)
NaCl	24.53
$MgCl_2$	5.20
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.695
$NaHCO_3$	0.201
KBr	0.101
H_3BO_3	0.027
$SrCl_2$	0.025
NaF	0.003

The tribocorrosion tests were performed on the coated Ti6Al4V using a tribometer and a Potentiostat/Galvanostat (Modulab, Solarton Analytical), of which the counterparts were ZrO_2 balls (φ=6 mm) and the detailed assembly of sample and electrodes can be found elsewhere [19]. A platinum electrode, a saturated Ag/AgCl electrode and the TiSiCN coated Ti6Al4V alloy were used as the counter, reference and working electrodes in all the electrochemical tests, respectively. The electrolyte was artificial seawater, which was prepared according to the standard ASTM D1141-98. The chemical composition of the artificial seawater is listed in Table 2 and the PH value of the seawater was adjusted to 8.2 using 0.1 N NaOH solution. Prior to the tribocorrosion tests, all samples were immersed in artificial seawater for 1 h to establish a complete and dense passive film on the TiSiCN coating. For tribocorrosion tests in open circuit potential (OCP) conditions, the potential kept stable for 600 s before sliding, then the load was applied to the samples under the same conditions as the dry friction tests. The potential recording continued for 1200 s after sliding finished. For tribocorrosion in cathodic protection (CP) conditions, the sliding set-up and procedure were also the same as OCP condition and the cathodic protection potential imposed on the samples were listed in Table 3, which is according to the experimental results of the tribocorrosion in OCP conditions. Potentiodynamic polarization measurements were carried out from −550 to +500 mV at a sweep rate of 2 mV s^{-1} using the same set-up under the condition of sliding, when the friction coefficient kept stable. The tests that the ball was placed above (~5 mm) the coating and the ball moved without any contact with the specimen surface under potentiodynamic polarization measurements, which act the comparison testing to evaluate the influence of wear on corrosion current density and corrosion potential.

The morphologies of the wear tracks were analyzed by secondary electron microscopy mode using field emission scanning electron microscope (FEI Quantal FEG 250) equipped with EDS (OXFORD X-Max). And the cross-sectional morphologies of the wear track were investigated by focused ion beam (Auriga). A Pt layer was deposited on top of the area of interest before Ga^+ milling. The Raman spectra of the wear tracks and coatings were excited by 532 nm laser radiation and recorded using a Raman spectroscopic (HR800) measurement with a resolution of 1 cm^{-1}. Based on the depth profiles of the wear track detected by Alpha-Step IQ profilometer, the volume losses of the coatings V (mm^3) can be obtained after the sliding tests. The volume loss rate of the coatings were calculated based on the classical equation,

$$W = V/FS \tag{1}$$

Table 1
Synthesis conditions for TiSiCN layer.

Samples	C_2H_2 flow rate (sccm)	N_2 flow rate (sccm)	Ar flow rate (sccm)	Temperature (℃)	Negative bias (V)	Pressure (Pa)
6.9 at% C	20	300	684	450	20	3
11.9 at% C	40	280	677	450	20	3
16.7 at% C	60	260	672	450	20	3
22.2 at% C	80	240	656	450	20	3

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