



Effects of process parameters on microstructure and wear resistance of TiN coatings deposited on TC11 titanium alloy by electrospark deposition



Xiang HONG, Ke FENG, Ye-fa TAN, Xiao-long WANG, Hua TAN

College of Field Engineering, PLA University of Science and Technology, Nanjing 210007, China

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Abstract: In the present study, the effects of process parameters (output voltage x , nitrogen flux l and specific strengthening time s) on the microstructure and wear resistance properties of TiN coatings prepared by electrospark deposition (ESD) were investigated systematically. The microstructure of the coatings was characterized for thickness (TOC), content of TiN (CON) and porosity (POC). A statistical model was developed to identify the significant factors affecting the microstructure and wear resistance of the coatings. The results show that the output voltage x and nitrogen flux l present significant effects on majority of the evaluation indexes such as TOC, friction coefficient (COF) and wear mass loss (I_d), while the specific strengthening time s has a significant effect on POC and a small effect on the other indexes. The optimal process parameters were obtained as follows: output voltage (x , 60 V), nitrogen flux (l , 15 L/min) and specific strengthening time (s , 3 min/cm²). The variation of wear mass loss (I_d) by the variation of the output voltage (x) and nitrogen flux (l) is attributed to the change of wear mechanisms of TiN coatings. The main wear mechanism of TiN coating prepared under optimal process parameters is micro-cutting wear accompanied by micro-fracture wear.

Key words: electrospark deposition (ESD); TiN; coating; wear resistance; statistical model; process parameters

1 Introduction

Titanium alloys have many advantages such as low density, high specific strength, excellent corrosion resistance and biocompatibility [1–3]. Thus, they have been widely applied to aerospace, chemical engineering, metallurgy, medicine and nuclear industries. However, titanium alloys limit their further application due to their poor tribological properties such as high friction coefficient and prone to adhesive wear, leading to their failure in the early service stage [4]. Surface modification and treatment of titanium alloys by using methods such as physical or chemical vapor deposition (PVD, CVD) [5,6], ion implantation [7], thermal oxidation [8], and magnetron sputtering techniques [9] have achieved good results. However, the applications of these surface technologies are restricted for the generation of some adverse effects on the titanium alloys. For example, due to the high temperature, it is easy to cause the deformation of the substrates by the nitriding, sulfurizing and CVD process. The equipment of PVD is relatively complicated, and it is hard to carry on the processing for the large scale parts because of the

necessary vacuum or gas environment for PVD. The coating deposited by magnetron sputtering method is relatively thin with low efficiency [10]. Therefore, it is urgent to prepare excellent coatings on titanium alloys bonding with substrate metallurgically while minimizing the total heat input into the substrate.

Electrospark deposition (ESD) is a typical surface modification technology with high energy density and low heat input, which has been widely used in the preparation of modified strengthening coatings [11–13]. Utilizing the high energy pulsed discharge between electrode and metallic substrate, the ESD process can deposit the melting electrode materials onto the substrate to accomplish metallurgical reaction with substrate. Currently, several coatings have been deposited on titanium alloys by ESD process. WANG et al [14] deposited WC92–Co8 hard alloy coating on titanium alloys by ESD and studied the microstructure and interface of the coatings. The microhardness of coating reached HV 1192, but the coating was thin (26.3–56.12 μm) and inhomogeneous. TANG et al [15] carried out ESD in the conditions of air, nitrogen gas atmosphere and silicone oil respectively using a graphite electrode on titanium alloy. The results showed that the ESD coating

treated in silicone oil exhibited better biocompatibility and biological activity in comparison with other conditions, but the silicone oil dielectric environment restricted to the application for large mechanical parts by the ESD. HAO et al [16] prepared TiN coating on TC4 titanium alloy by ESD, but the coating was relatively thin (12.1–24.3 μm) and the influence of process parameters on the properties of the coatings had not been studied.

Based on a large number of experiments on developing coatings on titanium alloys by ESD, it is evident that the process parameters have great effects on the properties of coatings. In view of the excellent properties of TiN coating such as high hardness, high strength, low friction coefficient, anti-wear and corrosion resistance, it is a promising surface coating to strengthen the titanium alloy. Thus in this work, the TiN coatings were deposited under different process parameters and the effects of parameters on microstructure, mechanical properties and tribological properties of the coatings were studied systematically to provide the theoretical basis and technical support for the further engineering applications of titanium alloys.

2 Experimental

2.1 Materials and processing

Titanium alloy plate of TC11 with the dimension of 10 mm $\times$ 10 mm $\times$ 3 mm was selected as the substrate material. The chemical composition of TC11 is listed in Table 1. The pure titanium rod of TA2 with diameter of 3 mm was used as the electrode. Prior to ESD, the substrate specimens and the electrode tip were ground with 800-grit SiC paper to remove the oxide films and then rinsed with acetone to remove surface oil. The experiments were carried out by a 3H-ES ESD deposition machine. The schematic diagram of ESD process is shown in Fig. 1. The deposition gun connects with high frequency power supply through the wire. The electrode rod is fixed by the clamp in the deposition gun and rotates with high-speed of 2500 r/min driven by the deposition gun. The deposition gun is hold by hands with a certain angle (30° to 45°) to the substrate surface, moving back and forth in the speed of 0.001 m/s.

Table 1 Chemical composition of TC11 titanium alloys (mass fraction, %)

Al	Mo	Zr	Si	Fe	O	Ti
6.32	3.29	1.79	0.23	0.077	0.096	Bal.

2.2 Factorial experimental design

A factorial experimental method was applied to investigating the factors on the properties of wear resistance of the coatings. In this study, the influence of

three factors (output voltage, nitrogen flux and specific strengthening time labeled as x , l and s respectively) on the microstructure and wear resistance of TiN coatings was been studied systematically. The Taguchi orthogonal array (TOA) was used to design the experiment. Three levels at same intervals of the parameters were selected as experiment factors, and its distribution is shown in Table 2. A standard L_93^4 orthogonal table containing nine test combinations was used to analyze the importance of four independent factors with three levels, the lists indicate the factor levels, and the line has determined the concrete test plan.

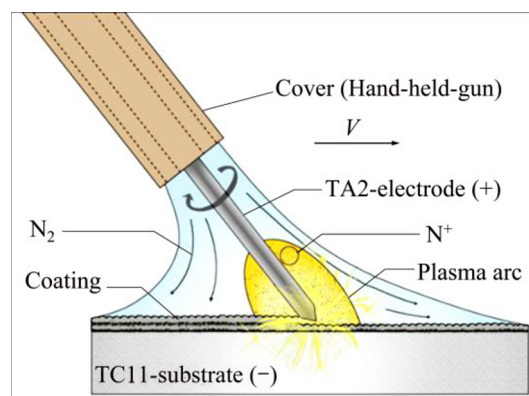


Fig. 1 Schematic diagram of preparing in-situ TiN coatings on TC11 by ESD [17]

Table 2 Factors and their levels for TiN coatings deposition in experimental design

Level	Output voltage, x/V	Nitrogen flux, $l/(L \cdot \text{min}^{-1})$	Specific strengthening time, $s/(\text{min} \cdot \text{cm}^{-2})$
1	40	5	3
2	60	10	4
3	80	15	5

2.3 Surface analysis

The microstructure and the worn surface of ESD coatings were observed by the scanning electron microscope (SEM) of QUANTA 200. In addition, the phase composition was identified by a D/max 2500/PC type X-ray diffractometer with Cu target operated at 40 kV and 200 mA. The microhardness of the coatings was measured using the DHV-1000 microhardness tester under the load of 1.96 N with a dwell time of 15 s. The surface roughness of the coatings was measured by Sufcorder SE300 surface roughometer. Using MATLAB software programming to calculate the porosity of the coating based on the image gray method.

2.4 Tribological tests

Tribological tests of ESD coatings sliding against a GCr15 ball with diameter of 4 mm and surface roughness of 0.05 μm were carried out by an HT-500 ball-on-disk

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