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Characterization and mechanical properties investigation of TiN-Ag films onto Ti-6Al-4V



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ABSTRACT

To investigate their effect on fretting fatigue (FF) resistance of a Ti-6Al-4V alloy, hard solid lubricating composite films of TiN with varying silver contents (TiN-Ag) were deposited on a Ti-6Al-4V alloy using ion-assisted magnetron sputtering. The surface morphology and structure were analyzed by atomic force microscopy, X-ray diffractometry, X-ray photoelectron spectroscopy, and transmission electron microscopy. The hardness, bonding strength, and toughness of films were tested using a micro-hardness tester, scratch tester, and a repeated press–press test system that was manufactured in-house, respectively. The FF resistance of TiN-Ag composite films was studied using self-developed devices. The results show that the FF resistance of a titanium alloy can be improved by TiN-Ag composite films, which were fabricated using hard TiN coating doped with soft Ag. The FF life of Ag0.5, Ag2, Ag5, Ag10 and Ag20 composite films is 2.41, 3.18, 3.20, 2.94 and 2.87 times as great as that of the titanium alloy, respectively. This is because the composite films have the better toughness, friction lubrication, and high bonding strength. When the atomic fraction of Ag changes from 2% to 5%, the FF resistance of the composite films shows the best performance. This is attributed to the surface integrity of the composite film is sufficiently fine to prevent the initiation and early propagation of FF cracks.

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1. Introduction

Titanium alloy is used widely in aviation, aerospace, biomedical, and other industrial fields because of its high specific strength, good thermal stability, excellent corrosion resistance, and other superior characteristics [1,2]. However, titanium alloy also has disadvantages such as low hardness, and poor wear resistance, which lead to fretting fatigue (FF) damage [3,4]. FF is the most common form of failure for key aircraft structures of titanium alloy such as the landing gear, flap track, and engine compressor blade dovetail. TiN films have a high hardness, wear resistance, and good chemical stability, and are used in a wide range of industrial applications. Previous studies show that TiN films improve the wear and fretting wear resistance of metal materials [5,6]. However, our group [7] reported that TiN films produced by chemical vapor deposition or plasma nitriding reduce rather than improve the FF resistance of titanium alloys. Both of the wear resistance and fatigue resistance problems must be solved to improve the FF resistance. Anti-wear and anti-fatigue methods often contradict each other. Ion-assisted

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http://dx.doi.org/10.1016/j.apsusc.2016.01.005 0169-4332/© 2016 Elsevier B.V. All rights reserved. magnetron sputtering deposition (IAMSD) is an organic combination technology combined with ion implantation and physical vapor deposition technology. It produces films with high strength and compactness, which has great advantages in surface modification applications [8,9].

Hard ceramic coatings or surface-hardened nitriding improve anti-wear performance, but often lead to a decrease in fatigue performance because of lower surface toughness, thus the anti-FF problem is difficult to solve [10,11]. Research shows that TiN or CrN composite ceramic films with an appropriate amount of soft elemental Ag reduce the friction coefficient significantly and improve the abrasion resistance of ceramic films [12–15]. Therefore, TiN doped with Ag(TiN-Ag) composite films on Ti-6Al-4V titanium alloy prepared by IAMSD is a promising candidate to improve the FF properties of titanium alloy and anti-wear performance.

2. Materials and methods

2.1. Materials and specimens

FF specimens and fretting pads were machined from a Ti-6Al-4V alloy bar (16 mm diameter, see Fig. 1). The material was



Fig. 1. Schematic drawing of FF specimens and fretting pads.

subjected to solution and aging heat treatment (820 °C for 1 h, quenching in water, 640 °C for 6 h, cooling in air). The Ti-6Al-4V alloy microstructure consists of $\alpha + \beta$ phases. The chemical composition of Ti-6Al-4V alloy consists of 6.7% Al, 4.2% V, 0.1% Fe, 0.03% C, 0.015% N, 0.03% H, and 0.14% O, with the balance being Ti. Mechanical properties were measured at room temperature, and indicate that the alloy has an ultimate strength of 1080 MPa, a yield strength of 1010 MPa, an elongation of 15%, and an area reduction of 41%.

2.2. Film preparation

PIEMAD-03 multifunctional ion-enhanced coating equipment was used to prepare the IAMSD coatings [4]. Two independent Ti and Ag targets with 99.99% purity were distributed symmetrically on both sides of the samples. During the deposition, the sample holder was rotated at a specified velocity. Specimens were treated by grinding using 1200# SiC sandpaper, mechanical polishing, and ultrasonic cleaning in acetone for 15 minutes at room temperature before coating. Before deposition, the specimen surface was cleaned with a slit ion source of 1 keV Ar ion beam for 30 min. To improve the bonding strength between the coating and substrate, a 300-nm-thick pure Ti film was deposited on the surface, and then a $5-\mu$ m-thick TiN-Ag composite coating was deposited on the pure Ti coating. The interfacing Ti layer coating parameters were as follows: The Ti target power was 400 W, the bias voltage was -300 V, and the argon flow rate was 180 sccm. The TiN-Ag composite coating preparation parameters were as follows: The chamber base pressure was 1×10^{-4} Pa, the Ti target power was 400W, the bias voltage was -300V, the argon flow rate was 180 sccm, the nitrogen flow rate was 125 sccm, and the change in control power was 50 W, 75 W, and 100 W of the Ag target to realize TiN-Ag composite films with different Ag content. The working pressure was set at 0.12 Pa during TiN-Ag composite coating deposition. A high-energy assisting ion beam was used to bombard the surface of the coating during deposition. And the deposition rate was calculated by film thickness of cross-section of the monocrystalline silicon wafer and treatment time.

2.3. Characterizations

Atomic force microscopy imaging and surface roughness of the films was performed by using an SPA400-SPI3800N with a silicon nitride tip with a diameter of 20 nm in contact mode (AFM, Seiko Instruments Inc., Japan). The crystalline structure of the coatings was measured by X-ray diffractometry with a Cu-K α source and a D/MAX 2200 PC X-ray diffraction analyzer (XRD, Rigaku Corporation, Japan). XRD working conditions were as follows: the tube voltage was 40 kV, the tube current was 30 mA, the scanning step size was 0.02°, and the scanning range was 20°-90°. X-ray photoelectron spectroscopy measurements (XPS, Thermo Electron Inc., US) and a Tecnai F30 G² transmission electron microscope (TEM, FEI Inc., US) were used to analyze the film structure. The film bonding strength and toughness were characterized using a WS2005 scratch tester and a cyclic in-house press-press device [16], respectively. A micro-hardness instrument was used to measure the Knoop hardness of the films with a range load of 0-0.98 N and a loading time of 20 s.

2.4. Fretting fatigue test

To evaluate the FF performance of surface-modified titanium alloy, a home-made FF device was used in the GPS100 high frequency fatigue test machine, and the fatigue loading was set to pull-pull mode. A schematic drawing of the FF test setup is shown in Fig. 2.

The contact state between the specimen and the pad was flat surface to flat surface with a contact area of 2×6 mm. The contact zone stress was controlled at 85 MPa by the stress ring. The maximum cyclic stress force was 550 MPa in sinusoidal form at 110 Hz and a stress ratio of 0.1 at room temperature. Five specimens were used for each condition during the FF tests, and the average sample fracture life was assessed to determine the FF resistance of the titanium alloy. Friction coefficients of the fretting fatigue process were tested and calculated using devices that we manufactured in-house.



Fig. 2. Devices and sketch diagram of fretting fatigue test.

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