

Effect of SPD surface layer on plasma nitriding of Ti–6Al–4V alloy

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ABSTRACT

A severe plastic deformation (SPD) surface layer was introduced by shot peening to enhance the nitriding kinetics in low-temperature (600 °C) plasma nitriding of Ti–6Al–4V alloy. The effect of this pretreatment on the nitrided microstructures and phase compositions was investigated by analytical microscopy techniques e.g. scanning and transmission electron microscopy (SEM, TEM) and X-ray diffraction (XRD) analysis. Microstructural investigations revealed the formation of a compound layer consisting of a 0.6 μm thick nanocrystalline TiN layer followed by a 0.5 μm thick layer of Ti₂N with a larger grain size (0.1–0.5 μm). The development of TiN nanograins was attributed to accelerated nitriding kinetics due to the increased preferential nucleation sites in the SPD layer. Furthermore, the thickness of nitrogen diffusion zone (DZ) increased by 50% in the pretreated plasma nitrided alloy when compared with that of the untreated one. This is likely promoted by an increase in density of subsurface microstructural defects, such as twins and grain boundaries.

The sliding behaviour and interfacial adhesion of the nitrided surfaces were evaluated by micro-scratch tests within a load range of 1–20 N. Compared with untreated-plasma-nitrided alloy, the pretreated nitrided surfaces exhibited a higher load bearing capacity and better interfacial bonding. They exhibited no chipping or spallation, even after multiple sliding passes at the highest applied load of 20 N in contrary to the untreated plasma nitrided surfaces.

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1. Introduction

Titanium alloys have been attractive candidates for aerospace and automotive applications due to their high strength-to-weight ratio, corrosion/oxidation resistance, stability at elevated temperatures, and fatigue resistance [1,2]. Nonetheless, a major drawback to their extensive application is their susceptibility to scuffing and poor tribological behaviour [3–5]. Over the past few decades, wear resistance of Ti alloys has been improved by coating deposition and thermochemical treatments such as plasma (ion-) nitriding [6–8]. Plasma nitriding of titanium alloys results in the formation of a thin layer composed of TiN and Ti₂N titanium nitrides (e.g. compound layer), an area depleted of β particles (the α-Case), and a nitrogen diffusion zone. Formation of the compound layer, despite its thin morphology, leads to significant improvements in the galling and scuffing behaviour of Ti surfaces. A conventional plasma nitriding treatment is performed at 800–1000 °C for up to 80 h in a nitrogen-rich atmosphere due to the limited rate of nitrogen diffusion in titanium [9–11]. However, the high temperatures and prolonged durations involved in these treatments result in the formation of a thick compound layer and α-Case as well as grain growth and

microstructural transformations in the bulk. The former promotes premature crack initiation from the surface while the latter decreases the substrate resistance to crack propagation. This will lead to the degradation of mechanical properties especially ductility and fatigue strength [11–14]. Reducing the temperature and duration of plasma nitriding, on the other hand, does not provide sufficient nitrogen diffusion depths due to slower nitriding kinetics [9,15–17].

There is a large difference between the mechanical properties of titanium nitrides (TiN ~ 2000 HV, Ti₂N ~ 1400 HV, $E = 250\text{--}640$ GPa [18,19]) and those of Ti substrate (Ti–6Al–4V ~ 400 HV, $E = 110$ GPa [20]). Therefore, the compound layer cannot conform to the extensive plastic deformation of the softer Ti substrate and eventually fails under sliding conditions. An effective measure to improve the sliding resistance in such cases is to minimize the incompatible coating-substrate mechanical responses by the design and fabrication of multilayer coatings, or through diffusion treatments (e.g. the incorporation of interstitial atoms, such as oxygen and nitrogen, to achieve a deep diffusion zone) [7,21–24].

Alteration of surface microstructure by introducing high-diffusivity paths such as grains boundaries, dislocations and atomic level microstructural defects have been employed to accelerate the diffusion of interstitial nitrogen atoms in iron and steels by several researchers [25–28]. Gu et al. [28] also reported that the high stored energy associated with the higher density of these non-equilibrium defects increases chemical reactivity of surfaces and enhances the

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kinetics of surface reactions. A severe plastic deformation surface layer introduced by mechanical attrition, shot peening, etc. resulted in significant grain refinement in the outermost layer and generation of non-equilibrium defects in the subsurface region. During the posterior nitriding treatments, these microstructural changes enhanced the nitrogen diffusion rate and increased the efficiency of nitriding at lower temperatures [25–28]. The thickness of this severely deformed layer varied in the range of 10–80 μm depending on the microstructure and properties of the substrate material; metals of a lower yield strength experienced a more intense plastic deformation that extended deeper into the substrate [29].

In this study, shot peening was employed as a pretreatment step to alter the near-surface microstructure of Ti–6Al–4V alloy and facilitate plasma nitriding kinetics at a low temperature. Comprehensive characterization techniques were performed to study the effect of this pretreatment on the nitrided microstructures and subsequently, on the load bearing capacity and sliding behaviours of the plasma nitrided alloy.

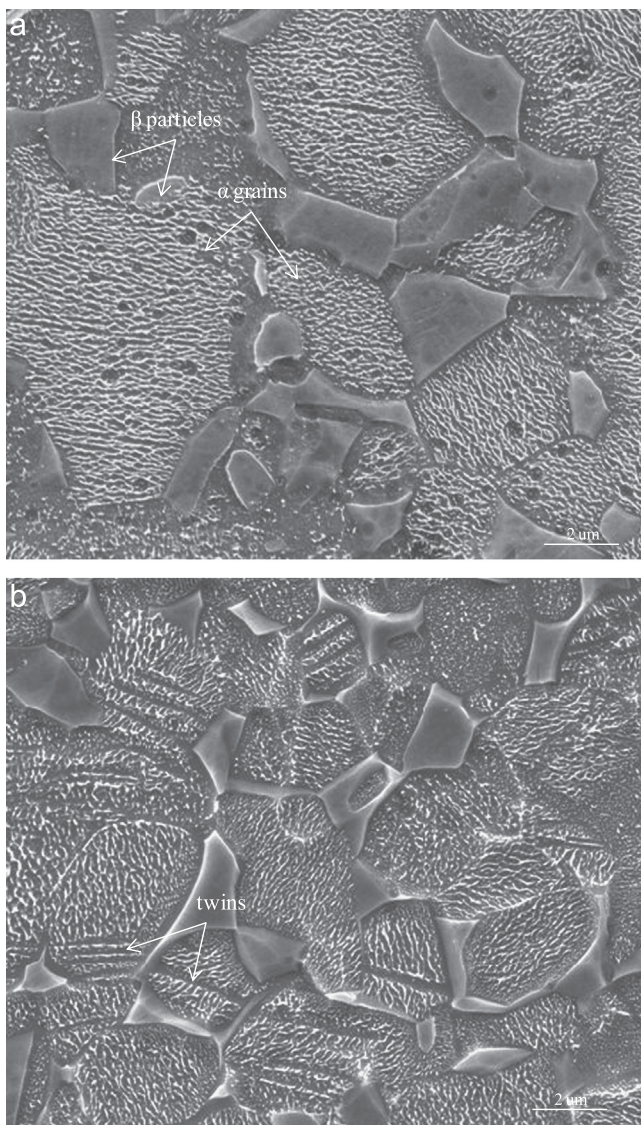


Fig. 1. Secondary electron images (SEI) of Ti–6Al–4V surface (a) before and (b) after shot peening. (a) The as-received microstructure contained equiaxed α grains with fine recrystallized β particles and retained β particles at α grain boundaries (marked by the arrows). The α grains can be differentiated by the different orientations of recrystallized β particles inside each grain. (b) The shot peened surface microstructure revealed the abundance of twins inside the α grains as marked by the arrows. (Etched in glycerol+HF (1:1) solution).

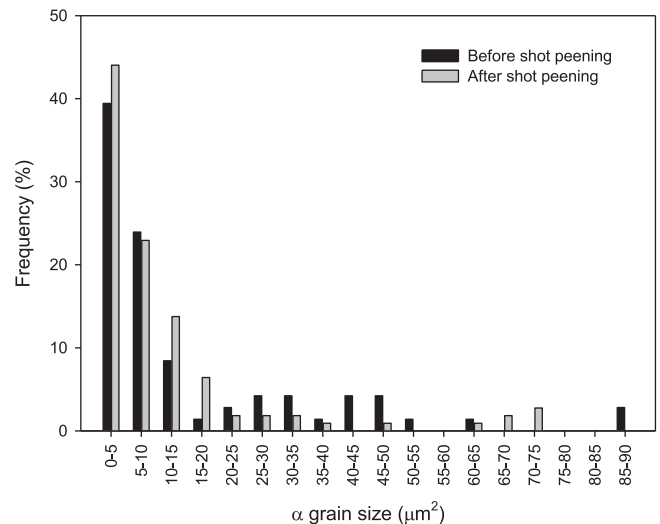


Fig. 2. Distribution of α grain size on the surface of Ti–6Al–4V alloy before and after shot peening. The measurements were made based on SEM images similar to those shown in Fig. 1.

2. Experimental procedure

Shot peening pretreatment was carried out on 2 cm \times 2 cm test coupons cut from a mill-annealed ELI-grade Ti–6Al–4V plate in an industrial shot peening facility. The surfaces were blasted in an air powered system at 90 psi (\sim 621 Pa) for 30 s with S–460 grade steel shots with an average diameter of 1.2 mm. The top surface of the coupons was completely covered with shot peening collision dents—100% coverage. The severe plastic deformation (SPD) imposed on the surfaces by shot peening increased the surface roughness. The average surface roughness (R_a) and peak-to-valley ratio (R_t) of shot peened surfaces as measured by an optical surface profilometer (Veeco, Wyko) were $6.2 \pm 0.02 \mu\text{m}$ and $108.0 \pm 10.5 \mu\text{m}$, respectively.

Subsequently, an approximately 2 μm thick layer was removed from the surface of the shot peened samples by mechanical grinding to eliminate surface oxide scales and other contaminants. The surfaces were then polished with 3 μm diamond suspension and passivated in an aqueous solution prior to plasma nitriding treatment to remove inherent oxide scales from the surface. The samples were placed inside a Ti cathodic cage in the nitriding chamber and plasma nitriding was performed at 600 $^\circ\text{C}$ for 24 h in an atmosphere containing 6% N_2 . The working pressure was approximately 67 Pa and the glow discharge was maintained at a voltage of 600 V. Subsequently, the nitrided samples were cooled down to room temperature under vacuum conditions. A set of untreated Ti–6Al–4V samples were also prepared and nitrided under the same conditions for comparative reasons.

The surface and subsurface microstructural evolutions were studied after shot peening by a field emission gun scanning electron microscope (Quanta 200 FEG–SEM) fitted with an energy dispersive X-ray spectrometer (EDS) at 10 kV. The average roughness, morphology, and elemental composition of the nitrided surfaces were examined using optical profilometry and SEM/EDS, respectively. Phase identifications were performed by a Rigaku DMAX–1200 X-ray diffractometer (XRD) with monochromatic Cu– K_α radiation. The microstructure of the plasma nitrided layers was studied under the SEM and an FEI Titan 80–3000 Cubed transmission electron microscope (TEM) which operated at 300 kV. The TEM investigations were performed at the Canadian Centre for Electron Microscopy (CCEM).¹ The TEM samples were prepared by the focused ion beam (FIB) lift-out

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