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Fatigue improvement in low temperature plasma nitrided Ti–6Al–4V alloy

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ABSTRACT

In this study a low temperature (600 °C) treatment was utilized to improve the fatigue performance of plasma nitrided Ti–6Al–4V alloy by optimization of microstructure. In order to study the fatigue properties, rotation bending tests were conducted, the *S–N* curves were constructed, and the results were compared with those obtained by an elevated temperature treatment (900 °C) as well as conventional gas/plasma nitriding treatments reported in literature. The plasma nitrided alloy at 600 °C showed an endurance limit of 552 MPa which was higher than those achieved by conventional nitriding treatments performed at 750–1100 °C. In contrast, plasma nitriding at 900 °C resulted in the reduction of fatigue life by at least two orders of magnitude compared to the 600 °C treatment, accompanied by a 13% reduction of tensile strength and a 78% reduction of ductility. The deterioration of mechanical properties after the elevated temperature treatment was attributed to the formation of a thick compound layer (~6 μm) on the surface followed by an α-Case (~20 μm) and phase transformation in the bulk microstructure from fully equiaxed to bimodal with coarse grains (~5 times higher average grain size value). The microstructure developed at 600 °C consisted of a thin compound layer (<2 μm) and a deep nitrogen diffusion zone (~45 μm) while the bulk microstructure was maintained with only 40% grain growth. The micromechanisms of fatigue failures were identified by examination of the fracture surfaces under a scanning electron microscope (SEM). It was found that fatigue failure in the plasma nitrided alloy initiated from the surface in the low cycle region ($N \leq 10^5$ cycles) and propagated in a ductile manner leading to the final rupture. No failures were observed in the high cycle region ($N > 10^5$ cycles) and the nitrided alloy endured cyclic loading until the tests were stopped at 10^7 cycles. The thin morphology of the compound layer in this study restricted the extent of premature crack initiation from the surface. Moreover, a deep diffusion zone with a well-bonded interface decreased the likelihood of fatigue initiation at (or below) the compound layer interface. Another notable feature was that the fatigue strength of the nitrided alloy was correlated with the surface roughness and in fact when the nitrided surfaces were polished, a higher number of cycles were dedicated to the formation of fatigue cracks compared to the as-treated condition resulting in an improved fatigue life.

1. Introduction

Titanium alloys are potential candidates for many applications due to their high specific strength, corrosion and fatigue resistance, but their poor tribological behavior is a major drawback [1–4]. Currently, the surface engineering techniques that improve the wear resistance of Ti alloys (e.g., nitriding) are accompanied by an inevitable negative side effect on their mechanical properties such as ductility and fatigue strength [5–9]. The gas and plasma-based nitriding treatments are normally performed at temperatures in the range of 750–1100 °C for several hours and exposure of the alloy to the temperatures results in fatigue deterioration by grain growth and overaging of the bulk microstructure. The nitriding microstructure generally consists of a "compound layer" composed of TiN and Ti_2N on the surface, followed by a nitrogen-stabilized α-titanium layer (α-Case) and a nitrogen diffusion zone [5,10–11]. Investigations by Morita et al. [12] on the fatigue properties of nitrided pure titanium showed that the compound layer and grain growth contributed to the loss of fatigue strength. After eliminating the effect of grain growth by exposing the alloy to the same thermal cycle under vacuum, they found that the brittle nature of the compound layer with a high stiffness difference with the Ti substrate promoted initiation of microcracks from the surface and led to premature failure and the reduction of fatigue strength.

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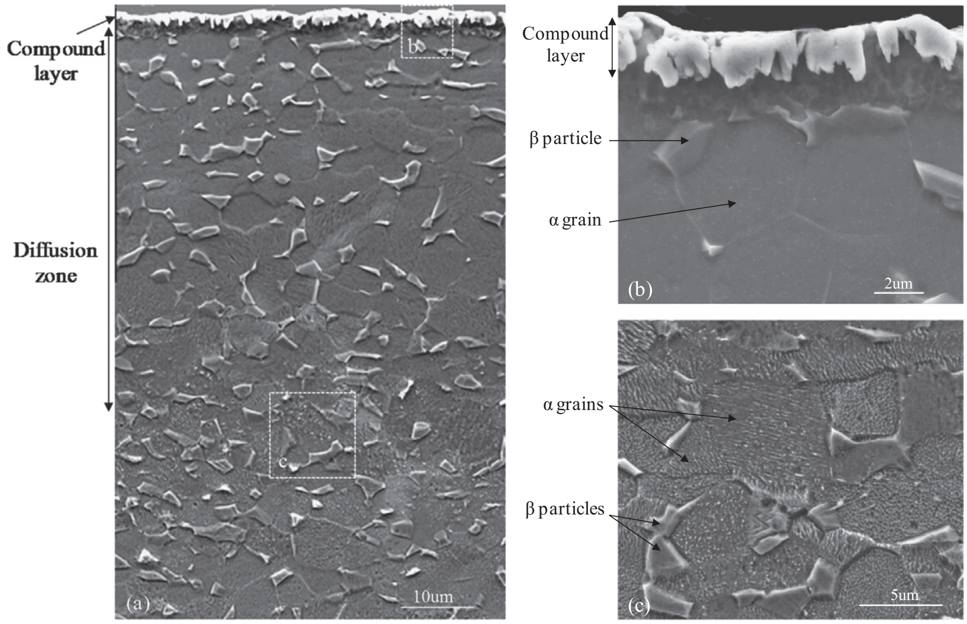

Fig. 1. (a) A typical cross-sectional secondary electron SEM image (SEI) of plasma nitrided Ti–6Al–4V alloy at 600 °C. The enclosed areas marked as "b" and "c" are the locations where higher magnification images in Figs. 1b and 1c were taken. The nitrided microstructure consisted of a thin compound layer (1.9 μm) on top of a deep diffusion zone (~45 μm). The diffusion zone was differentiated by the dissolution of fine recrystallized β particles inside the α grains since nitrogen is an α-stabilizing element. (b) A cross-sectional SEM image showing that the compound layer was well-bonded to the underlying diffusion zone at the interface without any evidence of the formation of an α-Case. (c) A cross-sectional SEM image showing that the bulk microstructure, consisting of equiaxed α grains, grain boundary β particles and fine recrystallized β particles inside the α grains, remained unchanged after the plasma nitriding treatment at 600 °C.

Similarly, Tokaji et al. [13] found deterioration of fatigue strength and endurance limit for Ti–6Al–4V (an $\alpha+\beta$ Ti alloy) and Ti–15Mo–5Zr–3Al (a β-Ti alloy) after gas nitriding at different temperatures (750–850 °C) and durations (4–60 h). They observed improvements in fatigue life by complete removal of the compound layer and partial removal of the diffusion zone and therefore reported that the formation of compound layer and diffusion zone were both responsible for impaired fatigue properties. It is also know that the formation of a brittle α-Case has adverse effects on the fracture toughness and fatigue properties of Ti alloys and according to the studies by Bell et al. [14] on plasma nitrided Ti–6Al–4V alloy at different temperatures (750–1050 °C), the reduction of fatigue limit was correlated with the thickness of the α-Case.

In this study the fatigue behavior of nitrided Ti alloys is improved by microstructural optimization through the formation of a thin compound layer supported by a deep diffusion zone and restricting the bulk microstructural changes. For this purpose, a low temperature plasma nitriding treatment is performed on Ti–6Al–4V alloy and the influence of nitrided microstructure on static and cyclic strength of the alloy is evaluated by uniaxial tensile and rotation bending fatigue tests. Subsequently, SEM observations of the fracture surfaces are utilized to identify the responsible failure mechanisms.

2. Experimental procedure

The as-received Ti–6Al–4V (ELI grade) alloy used in this investigation had a mill-annealed microstructure consisting of equiaxed α grains and retained β particles at α grain boundaries. Fine β precipitates were also observed inside the α grains formed by recrystallization during the final annealing step in the processing route. The average α grain size of the as-received alloy was 3.8 μm, measured using an image analyzing software.

Samples for fatigue tests were machined to an hourglass configuration with tapered length and minimum gauge diameter of 50 mm and 5.6 mm, respectively. Tensile samples having a gauge length and diameter of 24 mm and 6 mm were machined according to the ASTM E8 standard. The surface of reduced sections were ground through 240, 400, 600, 1200 grade SiC paper and subsequently polished using 3 μm and 1 μm diamond suspensions. The polished surfaces were then chemically activated before being placed inside the nitriding chamber. The plasma nitriding treatment was performed at 600 °C for 24 h. In order to study the effect of high temperature on microstructure, tensile and fatigue properties, the plasma nitriding temperature of 900 °C was investigated as well for the purpose of comparison. The details of the plasma nitriding treatment procedure are explained elsewhere [15].

The surface roughness measurements were carried out using a Veeco (Wyko) optical profilometer before and after the plasma nitriding treatment. The average residual stress level at the surface after nitriding measured using the $\sin^2\psi$ X-ray diffraction method (LXRD-Y[1]) was −530 MPa (compressive). Uniaxial tensile tests were conducted at a loading rate of 0.5 mm/min (MTS Criterion-43) and strain values were measured using an axial extensometer clipped to the gauge lengths. The load cell and the extensometer had an accuracy better than ±0.5% of the applied load/displacement. The fatigue properties were investigated by rotation bending fatigue tests (fully reversed cyclic loading, $R=-1$) using an R. R. Moore fatigue tester (Instron) at a frequency of 3000 rpm. The first samples were tested at ~20% of the tensile strength where failures were expected to occur at a relatively short number of cycles. The testing stress was decreased for the rest of the samples until at least three samples did not fail at 10^7 cycles (run-out) and the highest stress at the run-out was taken as the fatigue endurance limit. The tensile and fatigue tests were also performed on untreated Ti–6Al–4V samples under the same conditions for comparison.

The microstructure of the alloy before and after nitriding and the fracture surfaces after tensile and fatigue tests were studied using a field emission gun scanning electron microscope (Quanta 200

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