Low cycle fatigue behavior of Ti6Al4V thermochemically nitrided for its use in hip prostheses

D. RODRÍGUEZ, J. M. MANERO, F. J. GIL, J. A. PLANELL
CREB – Centre de Recerca en Enginyeria Biomèdica, ETSEIB – Departament de Ciència dels Materials i Enginyeria Metal.lúrgica, Av. Diagonal 647; 08028 – Barcelona, Spain

Titanium and its alloys have many attractive properties including high specific strength, low density, and excellent corrosion resistance. Besides, titanium and the Ti6Al4V alloy have long been recognized as materials with high biocompatibility. These properties have led to the use of these materials in biomedical applications. Despite these advantages, the lack of good wear resistance makes difficult the use of titanium and Ti6Al4V in some biomedical applications, like articulating components of prostheses.

Some surface treatments are available in order to correct these problems, like thermal surface treatment by means of nitrogen gaseous diffusion at high temperature. Nitrogen enters into the material by diffusion, creating a surface layer of increased hardness.

Low cycle fatigue behavior in air of Ti6Al4V alloy has been studied. Results show a reduction of low cycle fatigue life up to 10% compared to the not-treated material. Studies suggest it is not related to the titanium nitride surface layer, but to microstructural changes caused by the high temperature treatment.

Introduction

Titanium and its alloys are materials with a high specific strength and fatigue resistance, as well as and excellent corrosion resistance and biocompatibility. However, the lack of good wear resistance prevents the use of titanium and Ti6Al4V in some biomedical applications, like articulating components of prostheses.

One existing method for improving wear resistance properties is surface treatment of the material. Thermochemical modification of titanium alloys involving the introduction of carbon, oxygen, nitrogen, and other interstitial elements in titanium via a gaseous medium have long been studied [1]. This treatment produces a hard case of depths up to 100–200 μm.

This work studies the low cycle fatigue properties of Ti6Al4V treated with nitrogen diffusion hardening. The nitrogen diffuses into the titanium, producing an enriched α-case of 20–200 μm width, with an external layer of nitrides, especially Ti_2N (ε-nitride). Surface hardness and wear properties of the modified surface are greatly improved, with surface microhardness over 1000 $HV_{0.05}$.

Despite these promising results, it is necessary to study the fatigue behavior of thermochemically nitrided Ti6Al4V before its use in biomedical applications. Low cycle fatigue performance of Ti6Al4V shows important differences with microstructure as well as surface finishing [2]. This work studies the low cycle fatigue behavior of nitrided Ti6Al4V in air, at different deformation amplitudes.

Materials and methods

The material used in this study was received as rods of Ti6Al4V alloy, with mill-annealed microstructure and an ASTM E112 grain size of 11.8. Both chemical composition and microstructure complies with ASTM F136 standard for Ti6Al4V alloy application in surgical implants.

The received material was machined following the ASTM standard E606 to obtain normalized test specimens. Due to the significance of surface finishing on fatigue behavior, the test specimens were polished with SiC (up to 1200 grit) and aqueous alumina suspension (up to 0.05 μm) until the measure of the surface roughness parameter R_a were under 0.05 μm.

Once polished, samples were cleaned in an ultrasound bath, first with acetone for 20 min and afterwards with isopropilic alcohol for 10 min. When the test specimens were clean, they were thermochemically nitrided with a procedure already described [3].

A DOE (design of experiments) of 2^2 with temperature and time of treatment as independent variables and low cycle fatigue life as response was planned. The results were also compared to the behavior of non-treated test specimens, as well as specimens enduring the same process of the nitrided ones, but in a high purity inert gas (argon) atmosphere. Temperatures of treatment were 850 °C and 900 °C, and times were 1 and 4 h. Samples were codified with temperature and treatment time (Ti64_800_1 : specimen treated 1 h at 800 °C), besides

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TABLE I Cycles to failure of samples tested to low cycle fatigue

Load [kN]	σ^{*}_{norm}	$\Delta\varepsilon/2$	Cycles $\pm\,\sigma$ ($n=3$)					
			NT	900_a_AR	850_1	850_4	900_1	900_4
11.8	0.926	$\pm\,5\cdot10^{-3}$	69 229 ± 5115	19 204 ± 1989	12 423 ± 2132	8841 ± 1.526	10 702 ± 1374	5383 ± 636
14.9	1.169	$\pm\,6\cdot10^{-3}$	11 128 ± 435	1950 ± 421	3313 ± 549	1511 ± 385	3026 ± 522	1178 ± 257
16.7	1.311	$\pm\,7\cdot10^{-3}$	5033 ± 550	156 ± 47	207 ± 55	138 ± 37	191 ± 57	102 ± 29

* Monotonic yield stress in air: 649 MPa [2].

of control samples (_NT : non-treated) and the ones treated in argon (_AR).

Strain-controlled fatigue tests were made in a servo-hydraulic testing machine Instron 8500 with a 100 KN load cell and a coupled extensometer. Tests were made with deformation amplitudes of $\pm\,5\cdot10^{-3}$, $\pm\,6\cdot10^{-3}$ y $\pm\,7\cdot10^{-3}$, a ratio $R_e = -1$ and a load frequency of 0.5–3 Hz. These values were obtained from previous studies in order to assure low cycle fatigue behavior [2, 4]. Both sample fixations allowed for lateral movement during test preparation allowing for a correct alignment. All the tests were made in air at room temperature (24 °C).

Results

The data obtained from the fatigue tests (Table I) show a low cycle fatigue behavior for tests carried out with deformation amplitudes of $\pm6\cdot10^{-3}$ and $\pm7\cdot10^{-3}$, while tests made with an amplitude control of $\pm5\cdot10^{-3}$ presents a longer fatigue live. Besides, differences between nitrided and non-treated samples are also evident.

Comparison of the tests have also exhibited a softening effect in the specimens tested with strains over $\pm5\cdot10^{-3}$. Fig. 1 shows a comparison of cyclic softening of Ti64_NT and Ti64_900_1 samples tested at different strain values compared to data from [2].

Although the behavior of the nitrided samples is similar to the non-nitrided samples, fractography shows clear differences between them in the external layer. The nitrided layer suffers brittle transgranular fracture propagation, while non-treated samples presents a usual fatigue crack propagation.

Non-nitrided and argon-treated samples exhibit, for all the strain range studied, a fatigue starting point in the border of the samples, usually from a machining surface defect. The nucleated crack grows with load cycling. However, nitrided samples present a different fractography. Transgranular brittle fracture appears in the outer layer of the sample (50 μm), corresponding to the nitrided layer (Fig. 2b). In this place a transition to common fatigue mechanism is observed (Fig. 2a), and the crack progress until ductile fracture appears (Fig. 3).

Discussion

The obtained low cycle fatigue data were compared to existing S–N curves for Ti6Al4V [2, 5] in Fig. 4 and some interesting characteristics appeared. Low cycle fatigue resistance of nitrided Ti6Al4V is reduced when compared to samples of Ti6Al4V that were not treated, but it is similar to the values presented by Ti6Al4V treated in vacuum, and it is even better for fatigue over 10^5 cycles. This change could be due to the micro-structure change from mill-annealed to a Widmanstätten

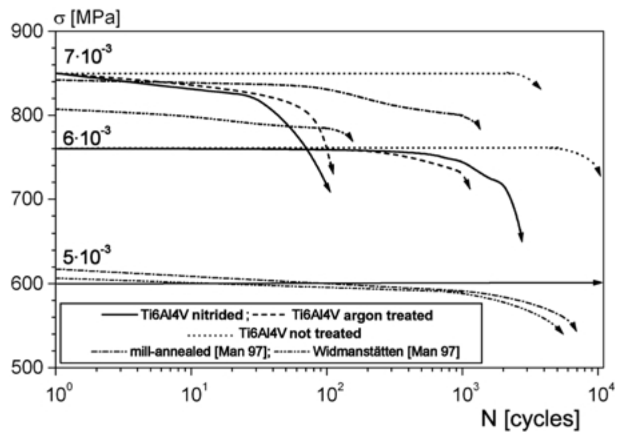

Figure 1 Cyclic softening curves for the sample Ti64_900_1 and samples of Ti6Al4V with mill-annealed and Widmanstätten structures.

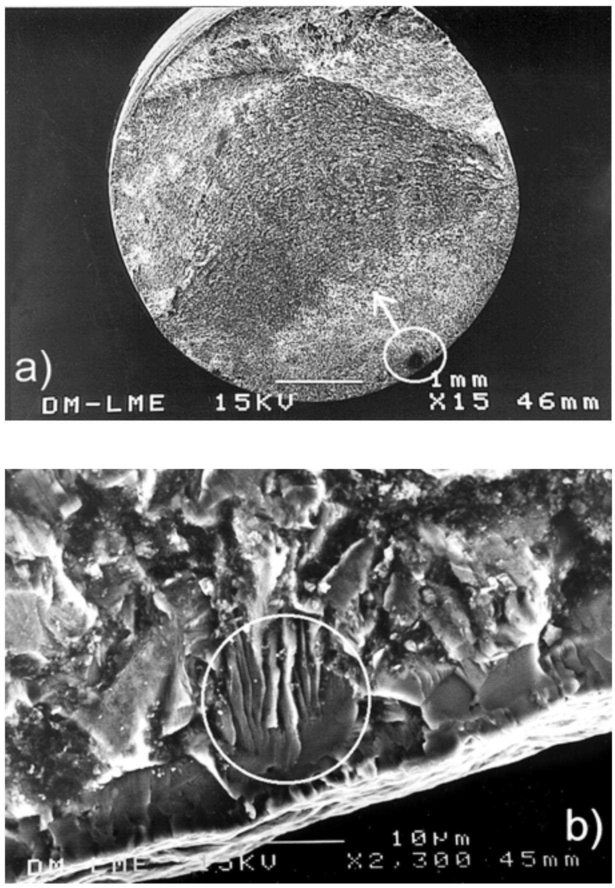

Figure 2 (a) General view and (b) detail of the fractography of the sample Ti64_900_1 tested with a deformation of $\pm5\cdot10^{-3}$.

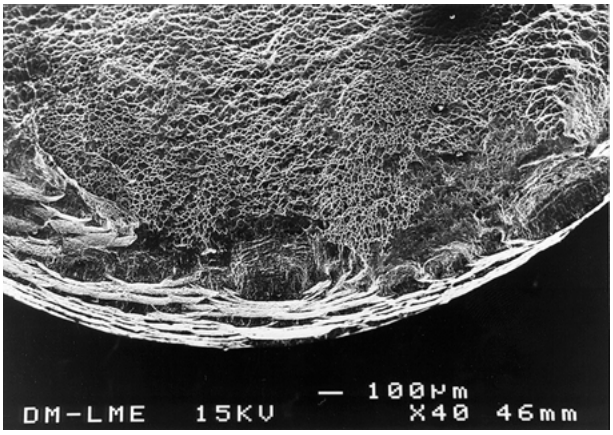

Figure 3 Fractography of the central region of the sample Ti64_900_1 tested with a deformation of $\pm 6 \cdot 10^{-3}$.

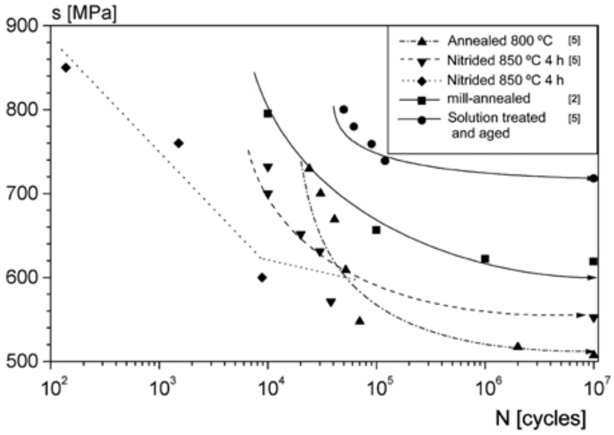

Figure 4 Comparison of fatigue behavior results.

one. The effects of the nitrided layer could even be positive, as the residual stress in the sample surface could slow down crack nucleation. A possibly better explanation is the important difference of Young's modulus between Ti6Al4V (100 GPa) and titanium nitrides (300–400 GPa) [5]. When high strains are imposed in a nitrided sample the nitrided layer cannot follow the strain because of its brittleness, and a crack nucleates and grows in this layer. When the imposed strains are lower, the nitrided layer stands the strain. In this case, the compressive forces of the layer prevent crack nucleation on the surface, improving the fatigue resistance of the nitrided sample.

Comparison of the nitrided sample results with fatigue tests of solution treated and aged (STA) samples results [5] shown in Fig. 4, exhibits a clear disadvantage for nitrided samples, mostly due to the fact that STA treatment offers really good improvements in fatigue life for the Ti6Al4V alloy [4].

It is important to note that some ways exist in order to improve Ti6Al4V fatigue life. The formation of more suitable microstructures (STA-like) by means of heat treatment after the nitriding process could be an interesting one, as it could add the best properties of both treatments.

Another method for improving fatigue life is the application of a shot peening treatment [1, 6]. This treatment produces good improvements in fatigue life, but it also increases surface roughness. Besides, the effect on the nitrided layer should be studied.

The good properties of the nitrided Ti6Al4V, as well as its excellent resistance to corrosion in saline medium [7] allows to think about some applications of nitrided Ti6Al4V alloy as biomaterial, mainly in prostheses and dental implants, in order to improve wear resistance without a remarkable reduction of other properties.

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Received 14 May
and accepted 21 May 2001