Influence of UFG structure formation on mechanical and fatigue properties in Ti-6Al-7Nb alloy

V V Polyakova¹, V N Anumalasetty², I P Semenova¹, R Z Valiev¹ ³
¹ Ufa State Aviation Technical University, K. Marx Street 12, 450 000, Ufa, Russia
² Carpenter Technology Corporation, R&D Bldg.68, POBox: 14662, Reading, PA, 19612-4662, USA
³ Laboratory for Mechanics of Bulk Nanostructured Materials, Saint Petersburg State University, Universitetsky pr. 28, 198504, Peterhof, Saint Petersburg, Russia
Email: Vnurik@gmail.com

Abstract. Ultrafine-grained (UFG) Ti alloys have potential applications in osteosynthesis and orthopedics due to high bio-compatibility and increased weight-to-strength ratio. In current study, Ti6Al7Nb ELI alloy is processed through equal channel angular pressing-conform (ECAP-Conform) and subsequent thermo-mechanical processing to generate a UFG microstructure. The fatigue properties of UFG alloys are compared to coarse grained (CG) alloys. Our study demonstrates that the UFG alloys with an average grain size of ~180 nm showed 35% enhancement of fatigue endurance limit as compared to coarse-grained alloys. On the fracture surfaces of the UFG and CG samples fatigue striations and dimpled relief were observed. However, the fracture surface of the UFG sample looks smoother; fewer amounts of secondary micro-cracks and more ductile rupture were also observed, which testifies to the good crack resistance in the UFG alloy after high-cyclic fatigue tests.

1. Introduction

Modern traumatology and orthopedics are constantly searching for new ways to improve methods of treatment aimed at reducing the osteosynthesis time through development and production of biologically and mechanically compatible implants for regeneration of the damaged bone tissue, which, in turn, imposes certain requirements on the implant material. It is crucial that the material is biocompatible and hypoallergenic so as not to cause the rejection of the implant by human organic tissues. At the same time, it must have high strength and fatigue endurance in order to not fail under static and dynamic loads while in service, and also have sufficient ductility for torsional strain and bending loads during the installation [1,2].

Due to a unique combination of low elastic modulus, corrosion resistance and good biocompatibility, titanium-based alloys are widely used in medicine as structural materials for implants of a large range of application [3]. For example, in orthopedics and osteosynthesis, alloys with higher strength than in pure titanium are used, such as low-alloyed (α+β) titanium materials – Ti-6Al-4V and Ti-6Al-7Nb. In particular, the Ti-6Al-7Nb alloy (ISO 5832-11, ASTM F 1295) was specially developed for medicine as a Ti-6Al-4V analogue, which contains vanadium that is histotoxic in the ionic form [4,5]. However, despite its undeniable advantages and successful
application, as regards strength and fatigue properties, the Ti-6Al-7Nb alloy is slightly ranks below stainless steel, which has traditionally been used for osteosynthesis due to a variety of mechanical properties controlled by different thermomechanical treatments [6].

Improvement of functional properties in low-alloyed (α+β)-titanium materials is possible due to development of new techniques of enhancing their structural strength by replacing the typical schemes with new more efficient processing schemes. At present time in materials science the techniques of severe plastic deformation (SPD) of metallic materials are progressively developed, which allow producing samples of different geometry with an ultrafine-grained (UFG) structure and high mechanical properties [7,8,9,10]. In particular, in order to obtain elongated semi-products shaped as rods for the fabrication of intramedullary nails, fixing elements and cerclage wire etc. from titanium alloys, the SPD by the scheme of continuous equal channel angular pressing (ECAP-C) can be used in combination with conventional metal forming techniques [11,12,13,14,15,16]. This paper deals with the effect of SPD including ECAP-C and drawing on the microstructure, mechanical and fatigue properties of (α+β)-titanium alloys, using the example of the Ti-6Al-7Nb alloy.

2. Material and methods of research

Hot-rolled and annealed (in the (α+β)-zone) Ti-6Al-7Nb rods (ASTM F 1295) with a diameter of 12 mm and a length of 500 mm were used as the initial material. The Ti-6Al-7Nb rods have the following chemical composition: Ti – basis, Al – 5.82 %, Nb – 6.90 %, Fe – 0.18 %, O – 0.16 %, C – 0.008 %, N – 0.003 %, Ta < 0.01. The temperature of polymorphic transformation was 1002 °C. The samples were processed by thermo-mechanical treatment including equal channel angular pressing via the “Conform” scheme with an accumulated strain of 4.2 and following drawing from 12 to 5 mm in diameter. The thermo-mechanical treatment was implemented in the temperature range 0.2Tmelt to 0.4Tmelt. The temperature was controlled with a thermocouple and a noncontact laser pyrometer. During the processing semifluid oil was used. It consists of surface-active compound, functional additions and powdered crystalline graphite. As a result one-meter rods with 5 mm in diameter were produced.

For microstructural studies a light microscope (LM) «Olympus GX 51 and a transmission electron microscope (TEM) JEM 2100 EX were used. TEM investigations were performed on thin foils cut from the transverse and longitudinal section of the rods. The thin foils were prepared by electrolytic polishing in an alcohol solution of methanol and butanol with the addition of HClO4 on a Tenupol 5 Struers at -30 °C. The average size of structural elements was determined from the dark-field images. The statistical analysis was performed using software of the electron microscope JEM 2100. At least 200 grains were used to calculate the average grain size.

Mechanical properties were determined through the tensile tests of the samples at room temperature on an Instron 1114. The traverse speed was 1 mm/min. The force measurement accuracy was 1%. Cylindrical test samples with a gage diameter of 3 mm were cut from the central part of the rod in the longitudinal direction. Axial high cycle fatigue (HCF) tests in stress controlled mode were conducted with tension-
compression and symmetrical loading cycle $R = -1$, frequency $f = 30$Hz on the
servohydraulic fatigue testing machine Instron 8801.

3. Results and Discussion

3.1. Microstructural studies

Fig. 1 shows typical optical images of the investigated material in the initial and SPD-
processed states. The Ti-6Al-7Nb alloy in the initial state is characterized by a coarse-
grained (CG) globular structure, mainly consisting of the primary $\alpha$-phase grains with
an average size of $5.5 \pm 0.4 \mu m$ (bright fields in Fig. 1a). This structure is typical for
the ($\alpha+\beta$)-titanium strained at temperatures in the ($\alpha+\beta$)-area.

![Fig. 1. The Ti-6Al-7Nb microstructure: a) Initial condition. Transverse section. LM.
b, c) structure of rods (5 mm) produced by ECAP-C and drawing. LM. b) transverse,
c) longitudinal section.](image)

Thermomechanical treatment of the Ti-6Al-7Nb alloy led to a duplex structure
formation. The duplex structure consisted 12% of primary $\alpha$-phase fine particles with
an average size $\sim 1.5 \mu m$ (bright fields in Fig. 1b, 1c) and 88% of the ultrafine ($\alpha+\beta$)-matrix. In the longitudinal section the orientation of boundary surfaces is in the
direction of strain. In general, the alloy is characterized by an anisotropic linearly
oriented structure with an orientation axis parallel to the drawing axis.

![Fig. 2. Microstructure of the UFG Ti-6Al-7Nb alloy produced by ECAP and drawing (ø 5): a, b, c) bright-field image, a) transverse section, b, c) longitudinal section. In Figure a, location 1 indicates the grains with high-angle equilibrium boundaries, and location 2 indicates the grains with high-angle non-equilibrium boundaries of high dislocation density.](image)

TEM analysis of the microstructure (Fig. 2) showed that the Ti-6Al-7Nb alloy in the
($\alpha+\beta$)-field is characterized by an UFG structure with fragmented low-angle grain
boundaries of elongated morphology. Quantitative analysis showed that in the transverse section the average grain/sub-grain size was 180 ± 30 nm, and in the longitudinal section the size ranged from 500 to 1500 nm (Fig. 2a, 2b). In general, several types of grains can be distinguished in the structure. The first type is grains with non-equilibrium boundaries formed by the accumulation of dislocations and their rearrangement during straining. Within such grains the dislocations are organized either randomly, or they form walls and pile-ups. The second type of grains is characterized by low dislocation density and equilibrium high-angle boundaries, as evidenced by the banded contrast (Fig. 2a). In the primary α-phase grains cellular dislocation structures and subgrains are observed. Diffraction patterns obtained from α-grains showed the azimuthal blur of spots that indicates significant micro-distortions of the lattice during straining (Fig. 2c).

3.2. Tensile test results and fatigue properties

The UFG structure formation with the grain size of ~180 nm in the Ti-6Al-7Nb alloy led to an increased strength by comparison with the CG material. Typical "stress–strain" curves derived from the tensile testing are shown in Figure 3a. The ultimate tensile strength (UTS) of the alloy is increased by ~45% (from ~1020 to ~1460 MPa) with thermomechanical treatment. This holds true also for the yield strength (YS) which reached a value of ~1355 MPa. Although the elongation to failure of the samples with the UFG microstructures decreased ~2% in comparison to the CG structure (13 to 11%).

![Fig. 3. a) Uniaxial tension stress-strain curves of CG and UFG Ti-6Al-7Nb. b) Axial HCF curves for Ti-6Al-7Nb with CG and UFG structures.](image)

This mechanical behavior is typical for many bulk ultrafine-grained and nanostructured materials due to the physical nature of the plastic deformation in metals, when the mechanisms of dislocation nucleation and migration are hampered by the ultrafine grain and high defects density [7]. It is known that under low strains in the conditions of high-cycle fatigue tests the HCF curve obeys Basquin’s law expressed as follows \( \frac{\sigma_f}{E} (2N_f)^b \), where \( \sigma_f \) - fatigue strength coefficient, \( E \) - elastic modulus, \( b \) - fatigue strength exponent and \( N_f \) - number of cycles to failure [17]. The strength increase in metallic materials due to the
UFG structure formation leads to an increase in $\frac{\sigma'}{E}$ [18], which gives reason to expect enhanced fatigue properties in the UFG Ti-6Al-7Nb alloy. Axial fatigue test results ($R=-1$) (Fig. 3b) showed that the formation of the UFG structure in Ti-6Al-7Nb, along with high strength, also increased the endurance limit by 35% in comparison to coarse-grained analogue (700 and 450 MPa on the basis of $10^7$ cycles). Limited fatigue life at comparable levels of stress increased more than tenfold.

3.3. Relief features of the fractured samples
To determine the features of fatigue fracture of the Ti-6Al-7Nb alloy with a CG and an UFG structure, the fracture relief was analyzed after axial fatigue tests. Fig. 4 displays a typical view of Ti-6Al-7Nb sample fracture after axial HCF tests. In both states on the fracture surface (Fig. 4) three typical zones of fatigue failure can be distinguished: 1. initiation and stable extension of the crack, 2. accelerated crack growth, 3. static fracture [19].

Fractography of the fatigue fracture zone of the Ti-6Al-7Nb alloy in the initial condition indicates that the crack in the stable growth area developed with a quite flat quasi-brittle relief, with typical striations and secondary micro-cracks. The distance between the striations was 380 nm (Fig. 5a). The zone of accelerated crack growth can be characterized by irregular rough relief with macro-striations, the distance between the markings being 600 nm. Secondary cracks are also seen (Fig. 5b). Static fracture has typical dimple relief representing ductile fracture. The average dimple
size ranges from 5 to 15 µm (Fig. 5c), which is obviously due to different size parameters of the alloy initial structure.

**Fig. 5.** SEM Micrographs of fracture patterns of fatigue fracture of the Ti-6Al-7Nb alloy: a, b, c) coarse-grained initial material, HCF tested at a stress of 525 MPa; d, e, f) UFG material, HCF tested at a stress of 730 MPa. The arrows show the direction of crack growth.

Fracture patterns of the samples with an UFG structure reveal some differences with the coarse-grained sample relief. In the zone of stable crack growth one can observe marked galling of the material due to friction during the test. However, upon closer examination, both brittle cleavage and facets with micro-striations spaced at ~ 90 nm
can be distinguished (Fig. 5d). The zone of accelerated crack growth is characterized by a mixed relief: ductile dimple fracture alternates with facets of quasi-cleavage with striations spaced at ~ 190 nm (Fig. 5 e). The static fracture relief has a typical ductile dimple nature. The average dimple size was 2.2 ± 0.4 µm (Fig. 5f).

Kinetics of crack development can be considered from the point of view of the work expended on the material failure. This work can be divided into two components – work of crack initiation and work of crack propagation. Due to the lengthy fatigue process, in the microstructure, the structural changes take place that are associated with the accumulation of microplastic strain. In fact, because of high defect density, in UFG materials early localization of micro-strain occurs. Therefore, the crack should initiate earlier than in CG materials due to the mechanisms of interaction of different dislocation structures. Therefore, one can assume that the work of crack initiation in the UFG Ti-6Al-7Nb alloy is less than in the CG analogue.

It should be noted that the following factors influence the crack growth rate in the zone of stable growth: frequency of cycling, cycle asymmetry and the test environment. Microstructural characteristics and the scale factor do not have a substantial impact [20,21]. In the stable zone of CG Ti-6Al-7Nb typical micro-mechanisms are observed – ductile striations and quasi-brittle fracture. Unfortunately, the material galling in UFG samples limits the comparative analysis to a certain extent, but in both states in the zone of stable growth (Fig. 5 a and d) secondary micro-cracks are observed, however, their opening in CG samples is more intensive (Fig. 5a).

The patterns of accelerated fatigue crack growth are strongly influenced by the material microstructure [21]. Great differences in the reliefs of fractured samples with CG and UFG structures are observed. Above all, on the fatigue fractures of an UFG sample the signs of dimple relief are observed, indicating a ductile nature of failure. Moreover, the spacing between the striations in the UFG alloy is 3 times less than in the CG alloy, which may denote a lower crack propagation rate [20]. This may be due to a great extent of grain and sub-grain boundaries in UFG Ti-6Al-7Nb. In this case, the work of crack propagation in UFG samples is larger than in CG ones.

Conclusions

1. It was established that the thermomechanical treatment of the Ti-6Al-7Nb alloy including continuous equal channel angular pressing and drawing results in the formation of an anisotropic UFG structure with grains/sub-grains oriented along the deformation, the transverse size of which was 180 nm.

2. It was shown that the UFG structure formation by reducing the structural parameters tenfold and increasing the dislocation density can considerably enhance the strength of Ti-6Al-7Nb from 1020 to 1460 MPa while maintaining the ductility of 11%, and as a result, enhance the endurance limit from 450 to 700 MPa on the base of 10^7 during the HCF axial fatigue test.

3. Systematic studies on the mechanisms of fatigue crack propagation in the investigated UFG alloy Ti-6Al-7Nb demonstrated: in contrast to the CG analogue, in which the fracture micro relief in the zone of accelerated crack propagation is associated mainly with the striation relief, in the UFG sample a ductile dimple fracture is observed, along with the fracture by a mechanism of quasi-cleavage with fatigue striations.
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