

# ENHANCED FATIGUE STRENGTH OF ULTRAFINE-GRAINED Ti-6Al-7Nb ELI ALLOY: MICROSTRUCTURAL ASPECTS AND FAILURE PECULIARITIES

S.A. Gabitova, V.V. Polyakova and I.P. Semenova

Ufa State Aviation Technical University, K. Marx 12, Ufa 450000, Russia

Received: April 20, 2012

**Abstract.** This paper deals with the cyclic deformation behavior of the ultrafine-grained (UFG) Ti-6Al-7Nb alloy with extra low content of impurities (ELI) produced by severe plastic deformation (SPD). The SPD processing comprised equal channel angular pressing (ECAP) and subsequent thermomechanical treatment and was aimed at improvement of mechanical properties of the alloy. Formation of a UFG structure with the average phase grain size of  $250 \pm 50$  nm led to enhancement of fatigue endurance limit by 40% as compared to the coarse-grained state. The presented microstructural investigations demonstrated that the UFG microstructure significantly influences the crack paths and is very stable in the course of cyclic deformation. A high potential for prospective use of the UFG alloy in biomedical and engineering applications was discussed.

## 1. INTRODUCTION

Titanium low-alloyed alloys are widely applied as structural materials in modern medicine. It is predetermined by their exceptional corrosion resistance in various atmospheric conditions. On their surface there is generated an oxide film, which not only protects, but also possesses distinct osteoconductive properties. Titanium alloys also possess high strength-to-weight ratio and lower elasticity modulus as compared to other materials, in particular steels and cobalt-chromium alloys. It is known that the Ti-6Al-4V ELI alloy is widely used in dentistry for fabrication of dental implants, in orthopedics – for fabrication of endoprosthesis with cementless fixation, for surgical instruments, *etc.* However, it contains vanadium, which is histotoxic in the form of ions, and which in case of micromotion can come out of the alloy composition in a free form. Therefore, in recent years the Ti-6Al-7Nb alloy (Protasul-

100) containing niobium, which is harmless for tissues, has been used. It is acknowledged by implantologists as one of the best alloys. Its chemical composition, microstructure and mechanical properties were registered under the Swiss standard SN056512 in 1987 [1].

In the course of exploitation implants experience intensive cyclic and static loads. Obviously, for their fabrication deformed titanium alloys should be used, as they, in contrast to cast material, possess higher reserve of strength and fatigue endurance, and also more condensed structure, which contribute to reduction of healing time [1]. Therefore, enhancement of fatigue properties of low-alloyed titanium alloys by means of replacement of conventional processing schemes by new more productive ones is topical. In this work the object of investigations is the Ti-6Al-7Nb ELI alloy. Due to the low content of  $\beta$ -phase, it is not possible to achieve considerable

Corresponding author: S.A. Gabitova, e-mail: lana\_gabitova@mail.ru

strengthening of this alloy by means of quenching and ageing. The best effect can be achieved by deformation and thermo-mechanical processing [2].

In this work with the view of considerable enhancement of mechanical properties by means of formation of ultrafine-grained (UFG) structure in the Ti-6Al-7Nb ELI alloy there was used severe plastic deformation (SPD) by equal-channel angular pressing (ECAP) technique, its main advantage over other SPD techniques is the possibility to retain initial dimensions of billets [3]. Subsequent thermo-mechanical processing enables to produce billets with various shapes and dimensions and also contributes to additional strengthening of the alloy [4].

This work is devoted to investigation of influence of UFG structure on fatigue properties and fracture mode in the Ti-6Al-7Nb ELI alloy, processed by ECAP and subsequent extrusion.

## 2. MATERIALS AND INVESTIGATION TECHNIQUES

For investigations hot-rolled rods with a diameter of 30 mm of the Ti-6Al-7Nb ELI alloy were used. The alloy chemical composition was as follows: Ti-basis; 5.63 mass.% Al; 6.75% Nb; 0.005% Ta; 0.1% Fe; 0.01% Cu; 0.16% O.

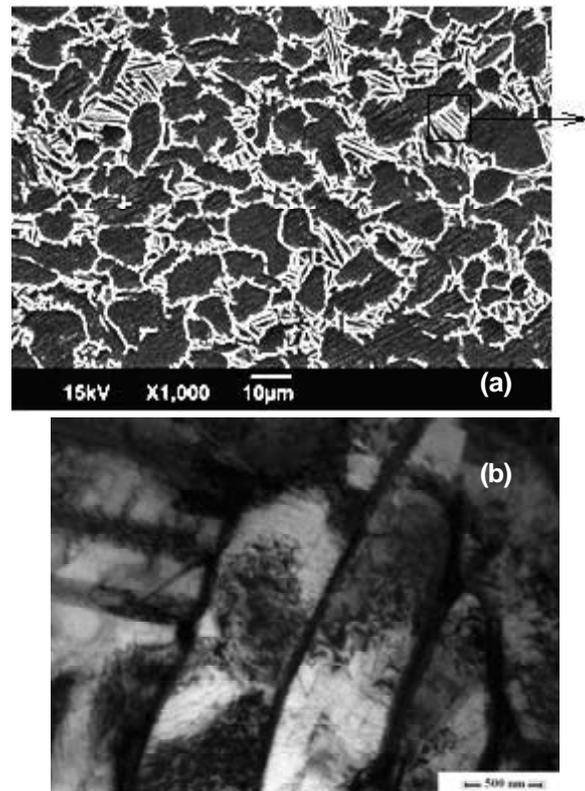
The UFG condition was formed with the help of combined thermo-mechanical processing of the Ti-6Al-7Nb ELI alloy, comprising ECAP at a temperature of 600 °C, 6 passes, die angle  $\Phi = 120^\circ$ , and subsequent extrusion (4 passes at a temperature of 300 °C, 1 pass at room temperature) [5].

Fatigue tests were performed on smooth samples under conditions of rotational bending with a frequency of  $f = 50$  Hz, symmetrical loading cycle ( $R = -1$ ) and the number of loading cycles  $N_b = 10^7$  cycles [6]. The billet microstructure was investigated with the help of transmission electron microscopy (TEM). The sample fracture relief was analyzed by means of scanning electron microscopy (SEM).

## 3. INVESTIGATION RESULTS AND DISCUSSION

### 3.1. Microstructure and mechanical properties of the Ti-6Al-7Nb ELI alloy after SPD

In the coarse-grained (CG) condition the alloy microstructure is characterized by equiaxed grains of primary  $\alpha$ -phase with an average size of 14  $\mu\text{m}$  and  $\beta$ -transformed structure with plate pack length of 8  $\mu\text{m}$ , the volume fraction of which makes 49% (Fig. 1a). Fig 1b displays the secondary  $\alpha$ -phase



**Fig. 1.** Microstructure of the Ti-6Al-7Nb alloy in a coarse-grained condition, (a) SEM; (b) TEM.

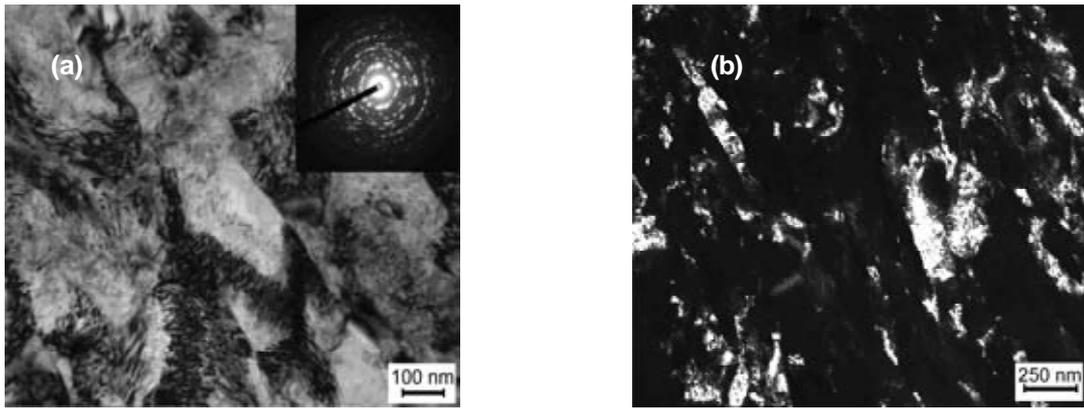
plates on which pile-ups of dislocations can be observed as a result of hot rolling of initial rods.

Combined processing of the Ti-6Al-7Nb ELI alloy led to formation of microstructure, oriented along the direction of deformation by extrusion. Herewith severe plastic deformation contributed to reduction of primary  $\alpha$ -phase grains to 7  $\mu\text{m}$  and new subgrains formation in them. After SPD processing a UFG structure was formed with an average size of grains/subgrains of  $250 \pm 20$  nm, defined in dark-field images (Fig. 2b). A diffraction pattern taken from this area is characterized by spots located uniformly in a circumferential direction, which testifies to formation of a grain structure with high-angle boundaries. Azimuthal blurring of spots testifies to high inner stresses and developed substructure (Fig. 2a).

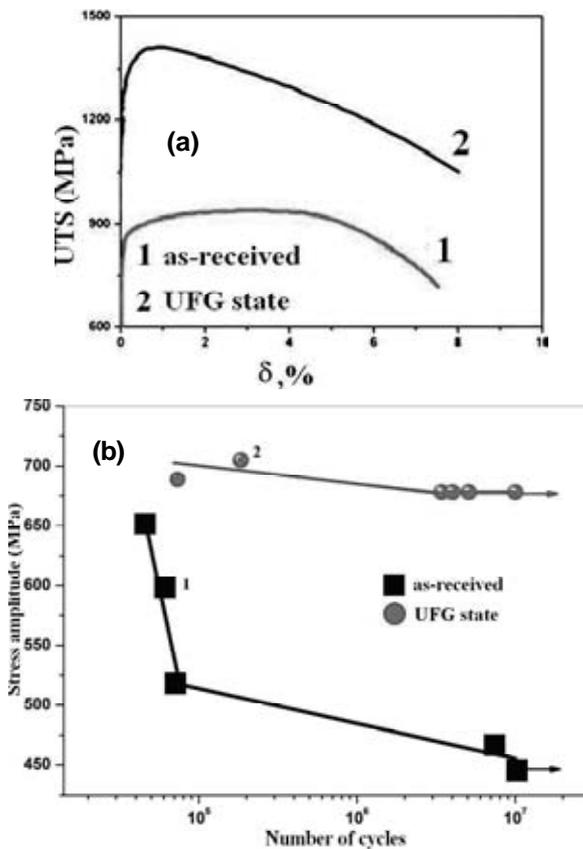
Due to the fact that the UFG structure volume fraction in the alloy is over 80%, the alloy can be related to nanostructured materials [4].

UFG structure formation in the Ti-6Al-7Nb ELI alloy led to increase of strength from 943 to 1415 MPa, the relative elongation almost did not change and made 8% (Fig. 3a).

Results of fatigue tests of samples in the as-received condition and after combined SPD processing are represented in Fig. 3b. It is observed that in a CG condition the samples possess low endur-



**Fig. 2.** Microstructure of the UFG Ti-6Al-7Nb ELI alloy: a) bright-field; b) dark-field image. Longitudinal section. TEM.

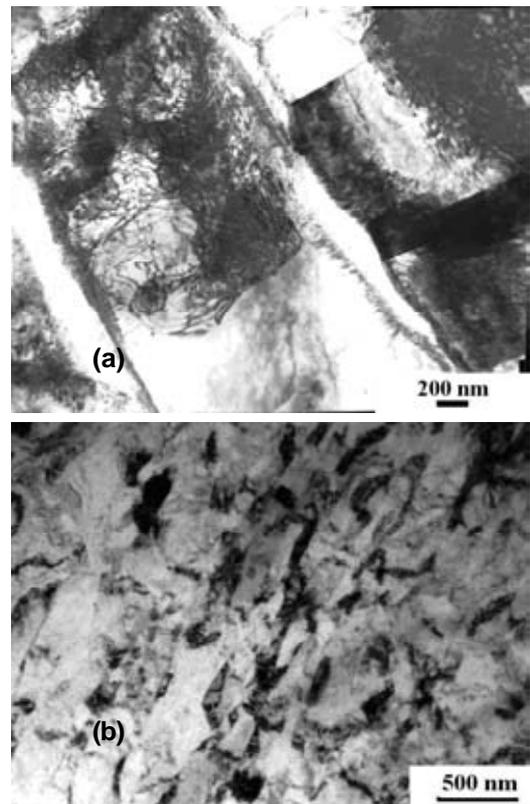


**Fig. 3.** a) mechanical properties of the Ti-6Al-7Nb ELI alloy before (1) and after (2) combined processing; b) dependence of the number of cycles on stress amplitude in CG (1) and UFG (2) conditions.

ance limit, which relates to low strength of the alloy (Fig. 3a). UFG structure formation and, consequently, high strength in the alloy enabled to increase the endurance limit from 450 to 680 MPa.

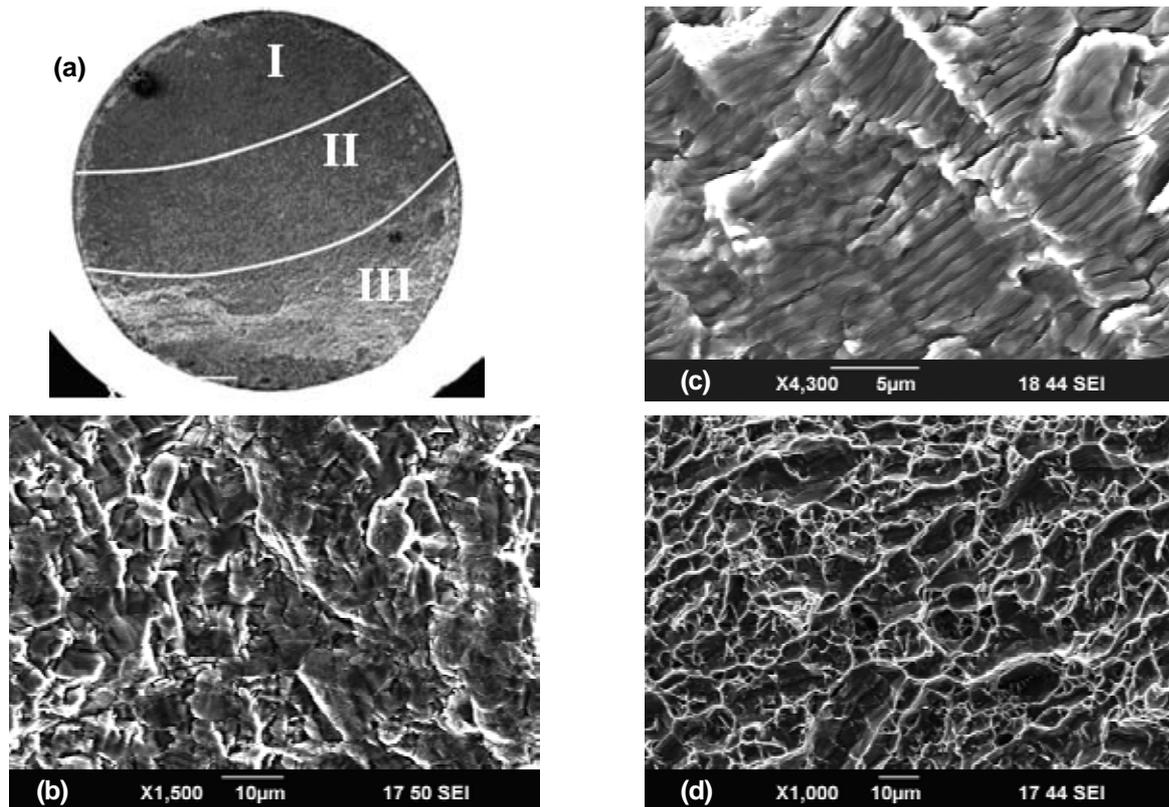
### 3.2. Influence of the UFG structure on the failure mechanisms of the alloy

Studies of the CG alloy microstructure after fatigue tests were conducted on the samples that did not



**Fig. 4.** Microstructure of the Ti-6Al-7Nb ELI alloy in the longitudinal section after fatigue tests: in CG state (a); in UFG state (b).

fail on the basis of  $10^7$  cycles. As a result of impact of the cyclic deformation a typical cellular dislocation structure was formed in the bodies of grains and  $\alpha$ -phase plates (Fig. 4a). Assessment of the dislocation density with the help of TEM microstructure images of CG samples demonstrated that it increases by almost 4-times, which testifies to cyclic strengthening of the material due to formation of new dislocations. Unlike CG samples, the UFG samples do not demonstrate significant changes in the structure (Fig. 4b). The  $\alpha$ -phase grains have an elongated shape, and their size is on average 250



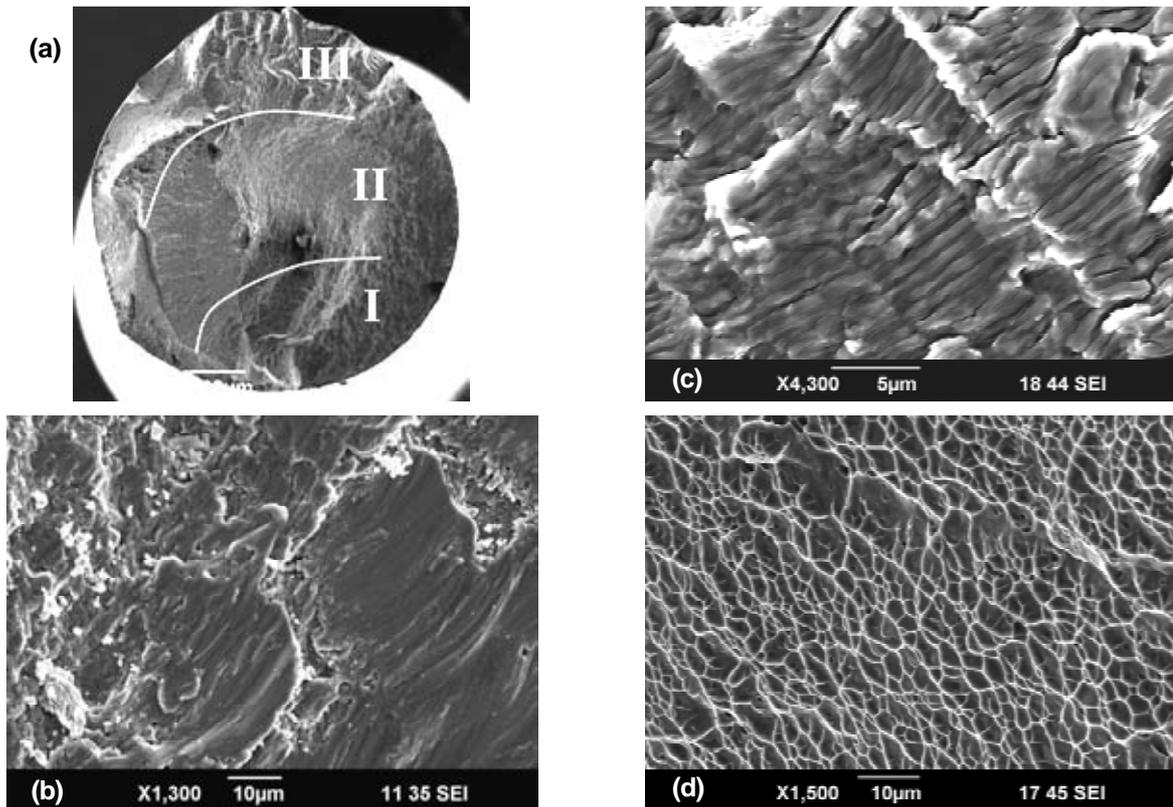
**Fig. 5.** CG sample fracture surface relief after tests under  $\sigma_{\max} = 517$  MPa and number of cycles  $N = 7.1 \times 10^4$ : a) general view; b) area of formation and stable extension of crack; c) area of crack propagation; d) static fracture area.

nm. It can be assumed that there is some reduction in the total dislocation density and their redistribution during cyclic deformation, which testifies to some softening of the UFG alloy. It is evident that ultrafine grains are too small for a cellular substructure to be formed in them. Such phenomena were observed in Cu, Ti, Ti-6Al-4V ELI, where the UFG structure was considered as more stable in comparison with the coarse-grained one [7].

In order to determine the peculiarities of fatigue fracture of the Ti-6Al-7Nb ELI alloy in CG and UFG conditions, fractographic analysis of the fracture relief was performed. It was established that in all the samples the fatigue cracks are located at right angle to the loading direction. On the sample fracture surface there may be distinguished three typical zones, which include: I – area of initiation and stable extension of crack; II – area of crack propagation and III – fast fracture area. It is observed that the area of fatigue fracture of samples occupies almost the most part of the total surface of fracture. Let us consider relief of the fractured samples N1 and N2 in CG and UFG conditions, correspondingly, with the test basis of approximately  $10^5$  cycles (Figs. 5 and 6).

In a CG sample of the Ti-6Al-7Nb ELI alloy in the area of initiation and stable extension of fatigue crack there appear cleavage facets with fatigue striations. There are observed secondary microcracks between the facets (Fig. 5b). The area of propagation of crack is characterized by coarse striated relief with secondary microcracks. The distance between fatigue microstriations makes 350 nm (Fig. 5c). Static fracture – is a typical ductile dimple fracture, the dimples have elongated form (Fig. 5d).

In the relief of stable crack extension area of Ti-6Al-7Nb ELI alloy sample in UFG condition, as well as in CG condition there is observed galling of material as a result of friction in the course of testing (Fig. 6b). The area of propagation of crack is characterized by mixed relief: there is transition from striation relief with a distance between striations of 60 nm (which is by almost 6 times less than in a CG sample) to ductile-dimple relief. With that secondary microcracks are not observed (Fig. 6c). The relief of static fracture area has a typical ductile dimple character. Dimples mostly have an equiaxed shape (Fig. 6d), their size is much less than in a CG sample. It is observed that in UFG samples the distance between fatigue striations is smaller than



**Fig. 6.** Fracture surface relief of the UFG Ti-6Al-7Nb ELI alloy after tests under  $\sigma_{\max} = 705$  MPa, number of cycles  $N = 18.4 \times 10^4$  cycles: a) general view; b) area of formation and stable extension of crack; c) area of crack propagation; d) static fracture area

in CG samples, which may testify to a lower rate of fatigue crack propagation. On the whole, there may be defined several main factors influencing the increase of the UFG Ti-6Al-7Nb ELI alloy endurance life: firstly, small grain size, which increases work of crack initiation; secondly, sufficient crack ductility at propagation of fatigue crack in a UFG sample, which is related to high extension of grain and subgrain boundaries [8].

#### 4. CONCLUSIONS

1. It was demonstrated that formation of UFG structure in the Ti-6Al-7Nb allows significantly increasing strength and, consequently, endurance limit from 450 to 680 MPa on the basis of  $10^7$  cycles under the conditions of rotation bending.

2. There were revealed peculiarities of fatigue fracture of UFG and CG alloy. It was demonstrated that the fracture surface of the UFG and CG alloy is characterized by fatigue striations and dimpled relief. Lack of secondary microcracks and more ductile rupture testifies to good crack resistance in the UFG alloy after cyclic tests.

3. Unlike CG alloy, the UFG samples do not show significant changes in the structure after fatigue tests; this testifies to its stability under cyclic stresses.

#### REFERENCES

- [1] D.M. Brunette, P. Tengvall, M. Textor and P. Thomsen, *Titanium in medicine* (Springer, 2001) p. 1019.
- [2] B.A. Kolachev, V.I. Elagin and V.A. Livanov, *Metal science and thermal processing of non-ferrous metals and alloys. Testbook for higher professional institutions (4<sup>th</sup> edition, reviewed)* (MISIS, 2005), p. 432.
- [3] R.Z. Valiev and I.V. Alexandrov, *Bulk nanostructured metallic materials: processing, structure and properties*, (Academkniga, 2007), p.398.
- [4] L.R. Saitova, I.P. Semnova, G.I. Raab and R.Z. Valiev // *Physics and technology of high pressures* **14** (2004) 19.
- [5] V.V. Nurgaleeva, I.P. Semenova, G.I. Raab and R.Z. Valiev // *Metal forming* **11** (2008) 28.

- [6] GOST 25.502-79. *Strength analysis and testing in machine building. Methods of metals mechanical testing. Methods of fatigue testing.*
- [7] L.R. Saitova, H.W. Hoepfel, M. Goeken, I.P. Semenova, G.I. Raab and R.Z. Valiev //

*Materials Science and Engineering A* **503**  
(2009) 145.

- [8] V.F. Terentiev, *Fatigue of metallic materials*  
(Nauka, 2002) p.248.