Enhanced fatigue properties of Ti-6Al-4V alloy turbine blades via formation of ultra-fine grained structure and ion implantation of surface

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Abstract. An overall approach to enhance service properties of Ti alloys is based on material nanostructuring in its volume and surface layer. In this work ultrafine-grained (UFG) structure is formed in Ti-6Al-4V alloy through equal channel angular pressing via the Conform scheme with subsequent drawing. Samples modelling the shape of blades are prepared. Their surface is subjected to ion implantation with N^+. Fatigue tests are performed in the conditions imitating the stress-strain state of blades. It is shown the increasing the resistance of a high-cycle fatigue of blades due to formation of the UFG structure and subsequent surface modification in the material. The fatigue behaviour of the UFG alloy subjected to ion implantation is discussed.

1 Introduction

Development of modern branches of mechanical engineering imposes high requirements to service properties of structural materials and items produced out of them. In many cases it is important to ensure a set of properties, for example a combination of high fatigue strength, corrosion and erosion resistance along with a low specific weight of such items as high-load parts of power units and aircrafts. Ti alloys are widely used in practice for this purpose [1]. Ti alloys have a much higher specific strength as compared to refractory steels and Ni alloys [1, 2]. Today Ti-6Al-4V alloy is the most widely used one due to favourable alloying [1, 2].

As is known, there is generally a correlation between the fatigue endurance limit and the ultimate tensile strength for different materials [3] Recent studies testify to an increase in the fatigue properties of metals and alloys through formation of ultrafine-grained (UFG) structures using severe plastic deformation (SPD) processing [3, 4]. In most cases, this improvement is connected with enhancement of the monotonic strength as a result of microstructural refinement. The formation of a bulk UFG structure in Ti alloys by SPD techniques allows increasing their specific strength, fatigue resistance, fatigue life, which creates possibilities to enhance service properties of items made out of these materials [5-7]. Alongside with that, the surface condition plays an important role in resistance of parts operating in extreme conditions (high cycle loads, aggressive environment, elevated temperature, erosion impact, etc.) to external loads. Among successful and promising techniques of surface treatment in structural materials are the techniques connected with impact of concentrated energy flows, to which ion implantation belongs [8-13].

The physical entity of ion implantation is introduction (implantation) of an alloying element in the surface layer of a part as a result of bombardment by ions with high kinetic energy [9, 10]. In particular, the surface modification of conventional Ti-6Al-4V alloy by ion implantation with N^+ enables improving its tribological [11], corrosion properties [12], creep rupture strength at 600°C [13].

As is known that ionized atoms or molecules of an alloying substance in the surface layer form a complex dislocation substructure, and ions introduced in the crystalline lattice of a metal base form solid solutions or new chemical compounds [9, 10]. Unlike coarse-grained (CG) materials, nucleation and accumulation of new dislocations are constrained in ultrafine grains with a high dislocation density [14]. Probably, this can affect the formation mechanisms and the modified layer parameters in the UFG Ti alloy surface and, therefore, the level of its service properties.

Thus, the aim of this work is to study the influence of consecutive impact of SPD-processing and ion modification of the surface on the fatigue properties of Ti-6Al-4V alloy. To form a bulk UFG structure in the alloy, equal channel angular pressing via the Conform scheme (ECAP-C) is used. To study the fatigue resistance, samples modelling a gas turbine engine (GTE) compressor blade are produced. The surface of the samples is subjected to low-energy implantation with N^+ ions.

2 Material and experimental procedure

The investigations are carried out on Ti-6Al-4V alloy (Ti-basis, Al – 6.6 %; V – 4.9 %; Zr – 0.02 %; Si – 0.033 %; Fe – 0.18 %; C – 0.007 %; O_2– 0.17 %). The temperature of polymorphic transformation (TPT) is 975±5 °C. The initial billets with a diameter of 15 mm and 1000 mm long are subjected to heat treatment (HT) by quenching from T=950°C (heating during 20 min) and consequent 4-hour annealing at 675°C [15]. Further the billets are subjected to 6 passes of ECAP-C on a die-set with the channels intersection angle ψ=120° (e = 4.5) at 550°C (the die-set heating is 350°C). The subsequent drawing at 450°C is performed till a rod diameter of 12 mm (ε~35%), from which samples are produced for fatigue tests (Fig. 1). After the treatment, the samples are subjected to annealing at 500°C C for 1 hour to reduce the total density of dislocations, internal elastic stresses [7].
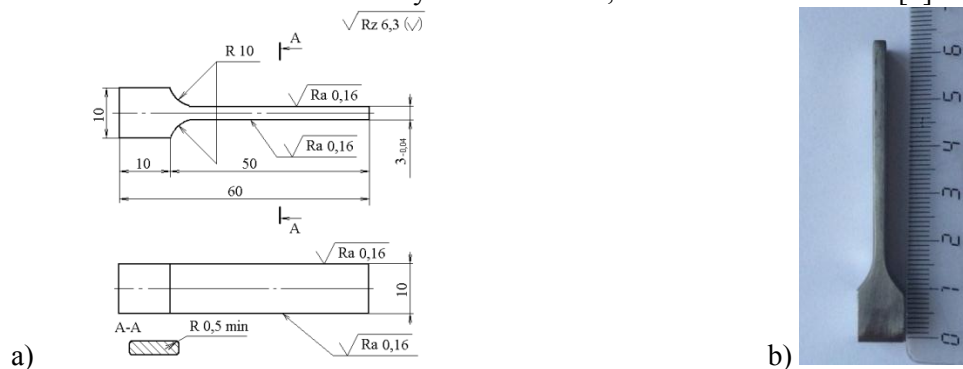

Figure 1 The scheme (a) and the image (b) of the fatigue sample.

The microstructure is studied by optical and transmission electron microscopy (TEM) on a JEM-200B microscope with an accelerating voltage of 200 kV. The experimental X-ray data are produced using a diffractometer DRON-4-07, copper radiation was employed. The distribution of elements in the surface layer is studied with the help of Auger electron spectroscopy [16]. Auger profiling of samples in depth is performed via surface sputtering with ions of Ar^+ with energy of ~ 3 keV. The quantitative analysis of the surface composition is performed by comparing the intensities of sorted peaks of elements with account of the data on their atomic sensitivity factors [16].

Cylindrical samples with a gauge length of 15 mm and diameter of 3 mm are subjected to tensile testing on an Instron tensile machine at room temperature and the strain rate $\acute{\varepsilon} = 10^{-3}$ s^{-1}. The fatigue sample surface is subjected to electrolyte-plasma treatment, which is performed at a voltage of 250-270 V and T=95°C in the electrolyte on the basis of fluoride salts [17] to ensure the required surface roughness. The subsequent ion implantation (ii) with nitrogen N^+ is carried out at an accelerated voltage of E= 300 eV and a dose of $2x10^{19}$ ions/cm^2 [18]. The fatigue tests are carried out on the electrodynamic stand VEDS-400A on the basis of $2x10^7$ cycles under the symmetric (R=-1) bending in the first vibration mode. The vibration frequency varies from 930 to 1020 Hz, the maximum stress of the cycle varies from 500 to 640 MPa.

3. Results
3.1 Microstructure and mechanical properties of Ti-6Al-4V after SPD processing

Figure 2 displays the microstructure images of the initial (CG) Ti-6Al-4V alloy and that after the combined SPD - processing (HT+ECAP-C+drawing+annealing). The microstructure of the initial rod is a typical bimodal one with a size of primary α-phase grains of (10±5) μm and areas with a plate (α+β) structure. The primary α-phase fraction is about 65% (Fig.2a). The β-phase fraction does not exceed 12 % according to the X-ray results. After the combined SPD-processing the fraction of primary α-phase grains decreases to 25%, and its grain size reduces down to 5 μm (Fig. 2b).
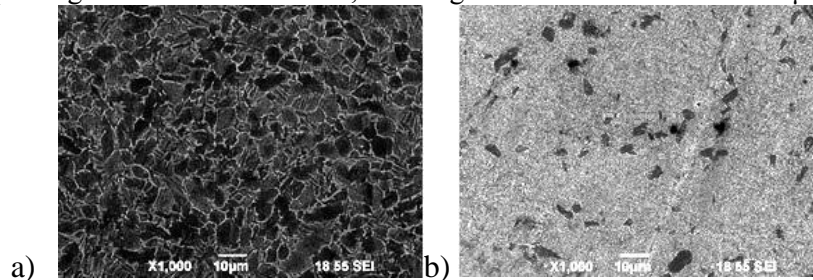

a)   b)

Figure 2 Microstructure of the Ti-6Al-4V alloy: (a) in as-received state and (b) after SPD-processing: transversal section.

Figure 3 displays TEM images of the billet microstructure in the transversal and longitudinal sections after SPD processing. The microstructure is mainly of grain character with clearly observed boundaries, which is connected with redistribution of the dislocation structure and formation of more defect-free equilibrium grain boundaries after the final annealing. The average size of structural elements is 200±20 nm (Fig. 3a). Grain elongation is observed along the strain direction in the ratio 1:2 in the longitudinal section of a billet (Fig. 3 b, c). The total dislocation density in the billet after SPD is about 4.5×10^{15} m^{-2}, which is much higher than the dislocation density in the initial alloy $(0.19 \times 10^{15}$ m$^{-2})$.

It is seen from Table 1 that the UFG structure formation in the alloy results in enhancement of the ultimate tensile strength (UTS) from 1030 to 1460 MPa due to considerable reduction of the α-phase grain/subgrain size from 10 to 0.2 μm, which correlates well with the previously obtained results for this alloy subjected to conventional ECAP with subsequent thermo-mechanical treatment [5]. Some reduction of the relative elongation (from 15 to 8%) is also typical of Ti alloys after SPD [5].
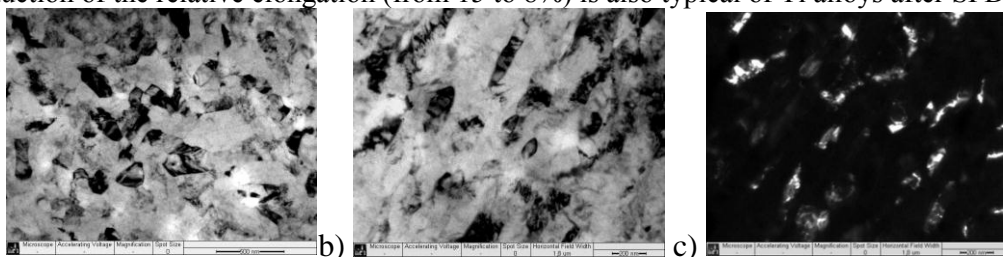

b)   c)

Figure 3 TEM images of the Ti-6Al-4V alloy microstructure after SPD-processing in transversal (a) and longitudinal (b, c) sections.

Table 1 Mechanical properties of the Ti-6Al-4V alloy before and after SPD-processing

State	Average grain size, D, μm	UTS, MPa	YS 0.2, MPa	Elong.,%
As-received	10±5	1030	850	15,0
SPD-processing	0.20±0.02	1460	1380	8,5

3.2 Ion implantation of the surface of UFG Ti-6Al-4V alloy blade samples.

Atomic $N^{+,}$ introduced in the surface of UFG samples, is confirmed by an X-ray diffraction pattern (Fig. 4). The value of the modified layer is estimated using the Auger spectroscopy [16]. Fig. 5

displays the concentration profiles of alloying elements and *N* in at. % in depth of the sample surface. On the average, the depth of N^+ penetration in the surface does not exceed 6 μm. As is known that the value of the layer modified with N^+ in Ti alloy at low-energy ion implantation can vary from 4 to 10 μm depending on the structure state of the surface and radiation dose [8-10].
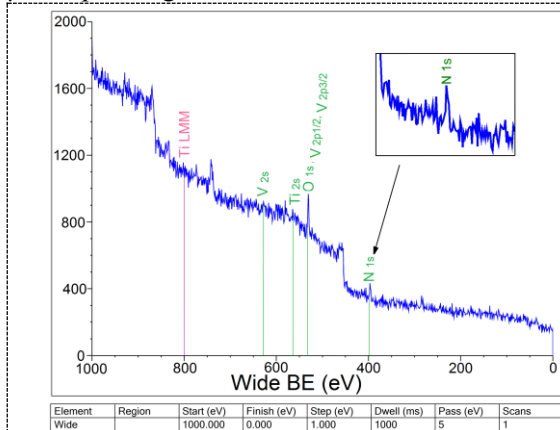

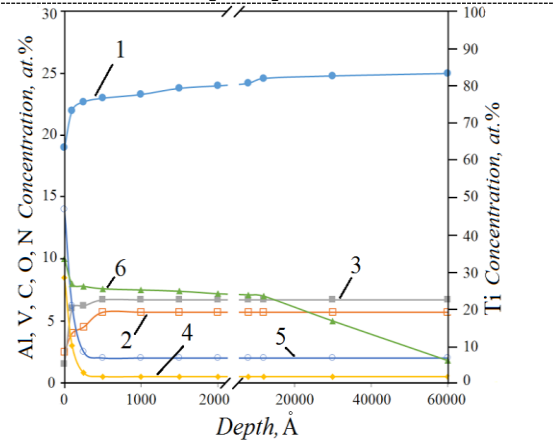| **Figure 4** *X*-ray diffraction pattern of the UFG samples surface after N^+ ion implantation. | **Figure 5** Concentration profiles of alloying elements in depth of the sample surface: **1**-Ti**; 2**–Al**; 3**–V**; 4**–C**; 5**–O**; 6**-N |

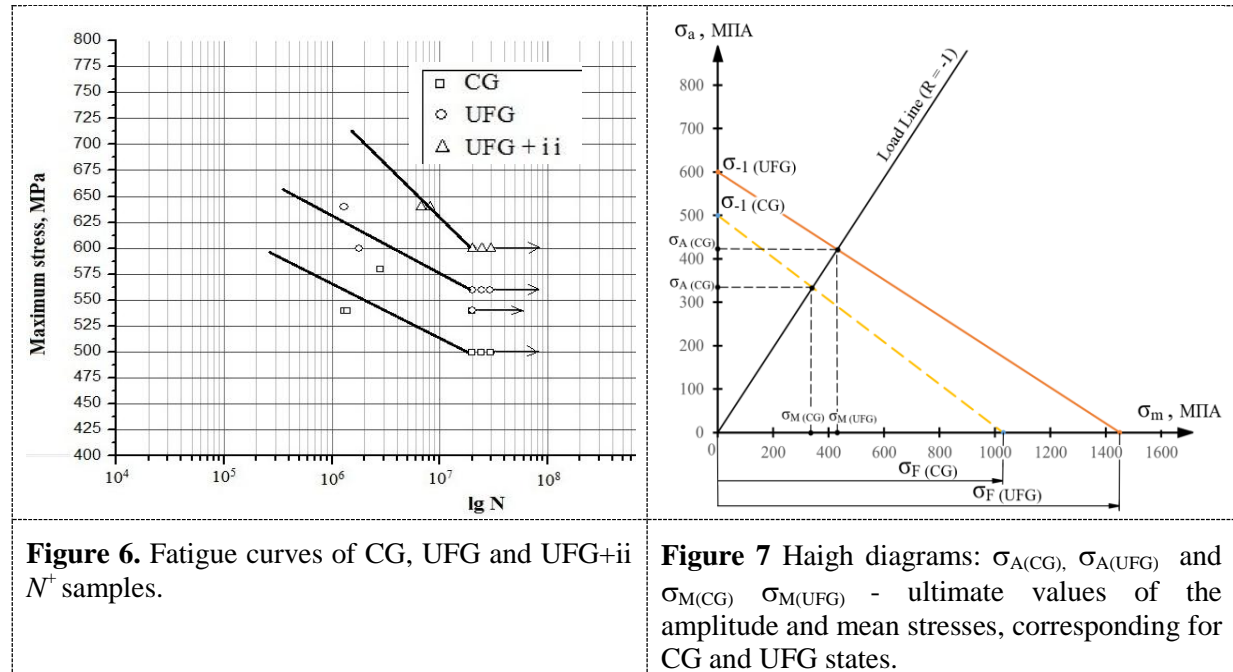*3.3 Fatigue tests of UFG Ti-6Al-4V alloy samples with ion implantation of the surface.*

Fig. 6 displays the fatigue test results of sample-items from CG and UFG Ti-6Al-4V alloys on the basis of 2×10^7 cycles. The main increment in the fatigue strength is observed as a result of formation of the UFG structure in it (from 500 to 570 MPa), i.e. approximately by 15%. The similar result was achieved in [15], where the fatigue endurance limit of CG Grade 5 with a bimodal structure and UFG states were 600 and 690 MPa respectively on the basis of 10^7 cycles of standard samples with a round section. Preliminary tensile tests of UFG samples with N^+ ion implantation show that the mechanical properties at room temperature practically do not change and are within the statistical spread. The surface modification of sample by ion N^+ gave an additional increment in the fatigue strength, which was 600 MPa.

4 Discussions

In this work the ECAP-Conform technique applied in combination with drawing and annealing allows achieving effective refinement of α-grains in the Ti-6Al-4V alloy and forming a UFG structure with an average size of 200 nm and, thus, enhancing the UTS from 1030 to 1460 MPa. As it is mentioned above, the strength enhancement in Ti-6Al-4V allows increasing the fatigue endurance limit at room temperature, which is testified in previous works [5, 15]. The main difference of the current work from the previous ones is evaluation of the fatigue resistance of samples imitating the shape of a GTE blade (Fig. 1). The results of bench tests of such UFG alloy samples on the enhanced basis up to 2×10^7 cycles in the conditions close to operating ones regarding frequency and stresses, also showed improvement of fatigue resistance (from 500 to 570 MPa) (Fig. 6).

The influence of UFG Ti alloy surface modification with N^+ ions on the fatigue strength should be noted. On the one hand, the mechanical properties at room temperature practically did not change. However, after ion implantation the fatigue strength tends to improvement and it is 600 MPa (Fig. 6). This can be explained by the fact that the positive effect of surface modification is conditioned by alteration of the crack initiation mechanism. In particular it was shown in [10] that fatigue crack initiation in Ti-6Al-4V alloy samples occurred on the surface. In case of radiated samples fatigue cracks initiated at a distance of 25-150 μm from the surface. In this work the main contribution in the

fatigue strength of UFG alloy is made by grain refinement, i.e. grain boundary strengthening mechanism.

Figure 6. Fatigue curves of CG, UFG and UFG+ii N^+ samples.	**Figure 7** Haigh diagrams: $\sigma_{A(CG)}$, $\sigma_{A(UFG)}$ and $\sigma_{M(CG)}$ $\sigma_{M(UFG)}$ - ultimate values of the amplitude and mean stresses, corresponding for CG and UFG states.

In engineering calculations of the design integrity and evaluation of the fatigue behavior of an item, probabilistic methods based on the Goodman line [19] in Haigh diagrams and corresponding fatigue safety factors are used. These diagrams allow seeing what stress amplitude material and/or an item can suffer without failing at the given mean stress. With use of the experimental data obtained in this work, the diagrams are plotted to determine the ultimate values of the amplitude σ_A and mean stress σ_M, shown in Fig. 7. Table 2 lists the values of σ_F (UTS) and fatigue endurance limit σ_{-1} for the CG and UFG alloys studied in this work. Fig. 7 shows that the Goodman lines for the UFG sample and item are higher than those for the CG state. This testifies to the fact that the fatigue safety factor at the given mean stress σ_m will be higher in the item with the UFG structure. For example, for the σ_m =300 MPa, the amplitude σ_a is close to the ultimate value $\sigma_{A\,(CG)}$ for the CG alloy as against the UFG alloy, for which the value σ_a is much lower than the ultimate one $\sigma_{A(UFG)}$ (500 MPa) (Fig. 7). The fatigue safety factor S_f is determined via the formula [19]: $S_f = \dfrac{1}{\frac{\sigma_a}{\sigma_{-1}} + \frac{\sigma_m}{\sigma_f}}$, where σ_a is the amplitude stress,

σ_m – the mean stress, σ_f is the material strength (in this case, it is UTS); σ_{-1} - the fatigue endurance limit at the stress ratio R=-1. It is seen from Table 2 that at the mean stress σ_m=300 MPa, S_f is 0.64 for the CG alloy and 1.41 i.e. higher by 30% for the UFG one.

Table 2 Evaluation fatigue safety factor S_f at $\sigma_a = \sigma_m$=300 MPa for CG and UFG Ti-6Al-4V alloy

	Endurance limit σ_{-1}, MPa	σ_f (UTS), MPa	Mean stress σ_m, MPa	Stress amplitude σ_a, MPa	fatigue safety factor, S_f
CG	500	1030	300	300	1.12
UFG	600	1460	300	300	1.43

Thus, the above considered experimental data testify to a principal possibility for enhancement of service properties of items manufactured from Ti-6Al-4V with an UFG structure with subsequent ion modification of the surface with N^+. It is known that parts of GTE from Ti alloys are exploited at temperatures up to 450°C. One can assume that in these exploitation conditions, creation of a layer modified with N^+ in the UFG alloy surface at elevated temperatures can cause improvement of the creep strength. A similar effect was observed in [13], where ion implantation N^+ of Ti-6Al-4V alloy

with resulted in improvement of the creep strength at 600°C. However, it requires more careful studies, which are planned to be performed by the authors in the future. Such a comprehensive approach to Ti-6Al-4V alloy hardening is promising for manufacturing of GTE items, for example, compressor blades, to which high requirements on operating properties at elevated temperatures are imposed.

5 Conclusions

1. It is established that formation of the UFG structure in Ti-6Al-4V with the average size of grains/subgrains of 200 nm by ECAP-Conform technique with subsequent drawing results in enhancement of the UTS at room temperature to 1460 MPa with a relative elongation of about 8 %.
2. It is shown that consecutive impact of SPD and N^+ ion implantation of Ti-6Al-4V sample surface ensures increase of the fatigue resistance in the conditions imitating the stress-strain state of GTE blades by no less than 20%. The fatigue safety factor S_f for the given acting stress increases by 30%.
3. The produced results demonstrate the perspectives for overall hardening of Ti-6Al-4V, which includes formation of a UFG structure in the volume by SPD techniques and subsequent modification of the surface with N^+ ions to increase the service properties of GTE items.

Acknowledgments

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