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## SUPERPLASTIC BEHAVIOR OF ULTRAFINE-GRAINED Ti-6Al-4V ELI ALLOY PRODUCED BY SEVERE PLASTIC DEFORMATION

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*This paper reports the results of investigation of mechanical behavior of the ultrafine-grained (UFG) Ti-6Al-4V alloy processed by equal-channel angular pressing (ECAP) and extrusion at temperatures in a range of 500–800 °C. The UFG alloy reveals superplastic behavior at relatively low temperatures down to 600 °C (elongation 240%). The features of microstructure change of the UFG alloy under superplastic straining conditions that contribute to additional strengthening of the alloy are shown.*

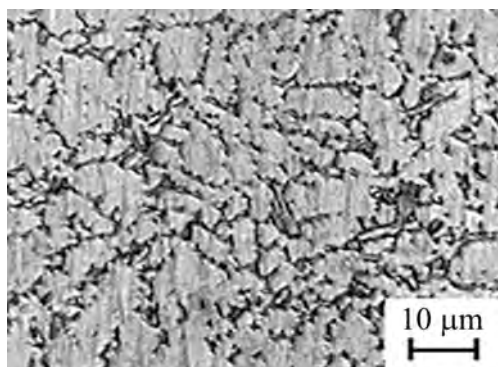
### Introduction

Superplastic deformation of metals rouses large scientific and practical interest among the researchers particularly in terms of the possibility to process complex-shaped articles [1]. Among structural factors that influence superplasticity the grain size should be outlined first of all, the reduction of which may result in superplasticity manifestation at relatively low temperatures and high strain rates [2]. Therefore, the development of methods of severe plastic deformation (SPD) opened new opportunities for enhancing superplastic properties in metals [2]. In recent years, superplastic properties were demonstrated at low temperatures and/or high strain rates in the UFG Al alloys [3–5], intermetallic compounds NiAl [6,7] and other nanostructured materials processed by high-pressure torsion (HPT) and ECAP.

In earlier works [8–10], the low-temperature superplasticity of the titanium alloy Ti-6Al-4V with UFG structure processed by HPT was demonstrated as well. However, these studies were conducted on small samples. The results of investigations of mechanical behavior of the UFG Ti-6Al-4V alloy carried out at temperatures in a range of 500–800 °C are presented in this paper considering large practical potential of the given alloy for structural applications in medicine and engineering; the alloy was processed by ECAP and warm extrusion, combination of which enables to produce bulk UFG semi-products for practical use.

### Materials and methods of investigation

The tests were conducted on the rods 40 mm in diameter from the Ti–6Al–4V ELI alloy (Intrinsic Devices Company, USA) of the following composition: Ti – base, Al – 6.0%; V – 4.2%; Fe – 0.2%; C – 0.001%; O<sub>2</sub> – 0.11%; N<sub>2</sub> – 0.0025%;



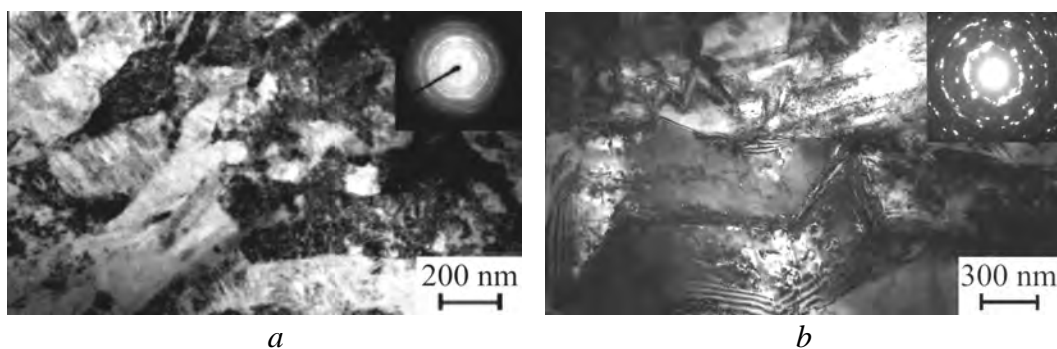
**Fig. 1.** Microstructure of the Ti–6Al–4V ELI alloy in the as-received state. Longitudinal section, OM

H<sub>2</sub> – 0.002%. This alloy with low impurity content is applied for the production of medical implants. The temperature of polymorphic transformation ( $T_{PT}$ ) in the alloy constitutes 960°C. The microstructure of the alloy in the as-received state was globular with an average size of  $\alpha$ -grains 8  $\mu\text{m}$  in cross section, 20  $\mu\text{m}$  in longitudinal section (Fig. 1). The samples 150 mm in length were subjected to straining in 2 stages: ECAP in a die-set with channels intersection angle  $\varphi = 120^\circ$  at a temperature of 600°C via route  $B_C$  and multicycle extrusion at 300°C

with cumulative elongation ratio  $\lambda = 4.2$  [12]. The view of the sample 18 mm in diameter and 300 mm in length is presented in Fig. 2. The microstructure of the samples was studied by means of optical microscopy (OM) and transmission electron microscopy (TEM). Mechanical tensile tests were carried out on an «Instron» machine at room temperature using a strain rate of  $\dot{\epsilon} = 10^{-3} \text{ s}^{-1}$ , and at elevated temperatures in a range of 500–800°C using strain rates of  $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$  and  $10^{-4} \text{ s}^{-1}$ . Test samples with the diameter of working part equal to 3 mm were cut from the central part of a rod along its longitudinal axis. Not less than 3 samples were tested for each state.



**Fig. 2.** Semi-product from the Ti–6Al–4V ELI alloy



**Fig. 3.** Microstructure of the Ti–6Al–4V ELI alloy: *a* – after ECAP and extrusion, *b* – after ECAP, extrusion and annealing at 600°C for a period of 1 h. Longitudinal section, TEM

Experimental results and discussion

Fig. 3,*a* presents the image of the alloy microstructure after ECAP and extrusion. It is seen that SPD leads to a significant grain refinement and formation of complex UFG structure in which grains/subgrains have irregular shape and contain a large number of lattice defects – microtwins, dislocations and others. There exists some inhomogeneity of microstructure characterized by the presence of very small grains/subgrains less than 100 nm in size along with large grains of 400 to 600 nm. Grain boundaries are indistinct and do not have clear boundary contrast (Fig. 3,*a*).

Table presents the results of mechanical tests of the UFG samples that revealed considerable strengthening of the alloy at room temperature in comparison with the initial state due to significant grain refinement. Relative elongation of the sample has lower values (8%) owing to structural features of SPD materials and physical nature of their plastic deformation when the mechanisms of dislocation generation and movement are not effective in ultrafine grains [2].

Table  
Mechanical properties of the Ti-6Al-4V ELI alloy in various states at room temperature

| Alloy state              | Ultimate tensile strength, MPa | Yield stress, MPa | Relative elongation, % |
|--------------------------|--------------------------------|-------------------|------------------------|
| Initial                  | 940                            | 840               | 16                     |
| After ECAP and extrusion | 1300                           | 1250              | 8                      |

UFG structure of the produced samples is stable up to a temperature of 600°C; heating at higher temperature revealed considerable grain growth to which microhardness fall testifies (Fig. 4). No considerable changes in grain growth were observed after heating of the samples at 600°C (Fig. 3,*b*).

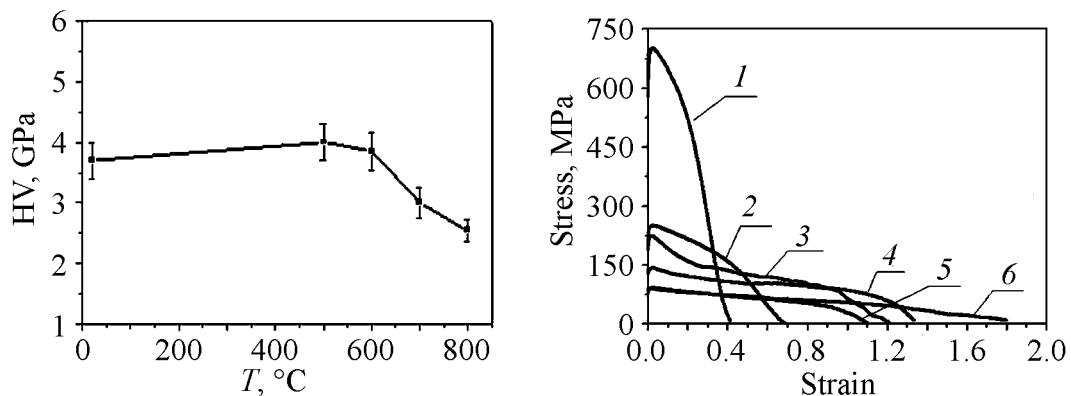
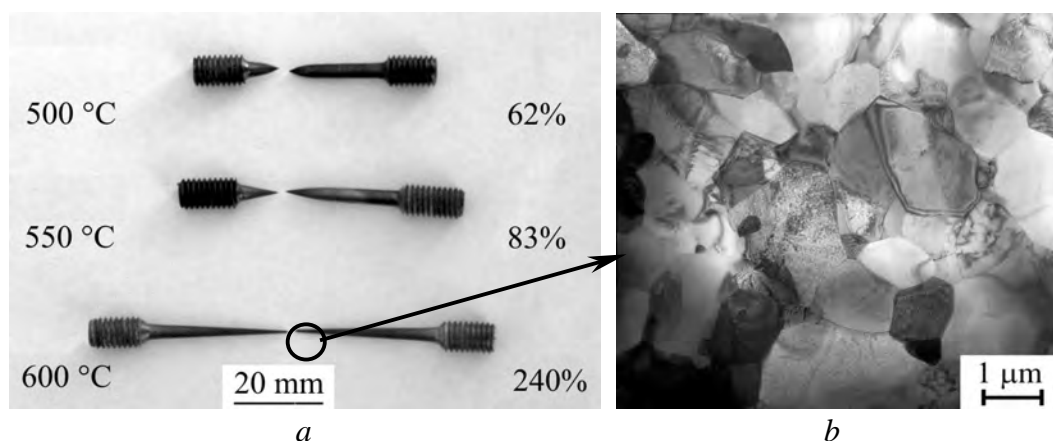


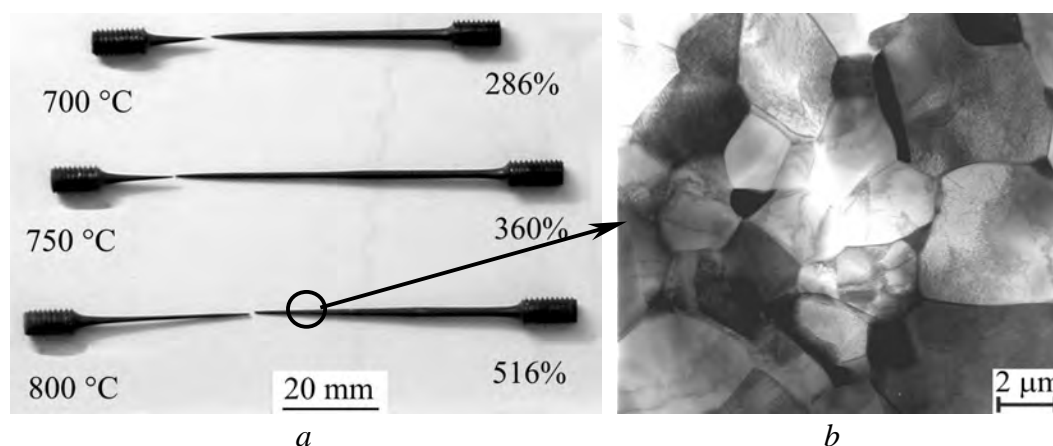
Fig. 4. Diagram showing the behavior of microhardness of the UFG alloy depending on the temperature of heating

Fig. 5. Stress-strain curves of coarse-grained and UFG alloy Ti-6Al-4V ELI in different conditions of tension: 1 – UFG, 500°C, 10<sup>-4</sup> s<sup>-1</sup>; 2 – as-received, 700°C, 10<sup>-2</sup> s<sup>-1</sup>; 3 – UFG, 600°C, 10<sup>-4</sup> s<sup>-1</sup>; 4 – UFG, 700°C, 10<sup>-2</sup> s<sup>-1</sup>; 5 – as-received, 800°C, 10<sup>-2</sup> s<sup>-1</sup>; 6 – UFG, 800°C, 10<sup>-2</sup> s<sup>-1</sup>

Fig. 5 presents the curves in arbitrary «stress-strain» coordinates after tension of the samples in the initial coarse-grained (CG) and UFG states at temperatures of 500, 600°C with a strain rate of  $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$  and temperatures of 700 and 800°C with a strain rate of  $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$ . At a temperature of 500°C and strain rate  $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$  the UFG alloy shows severe strengthening and low elongation value (62%). Elevating the temperature up to 600°C leads to a change in the shape of a tensile curve of the samples that becomes closer to superplastic behavior when flow stress decreases almost by a factor of three in comparison with a temperature of 500°C, and elongation reaches 200%. At tension with a strain rate of  $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$  for the CG and UFG states the strength reduces with temperature increase, and at a temperature of 700°C the UFG samples reveal the features of high strain rate superplasticity (elongation 268%) and strength common to coarse-grained samples. At 800°C elongation of the UFG alloy is maximum and reaches 516%.



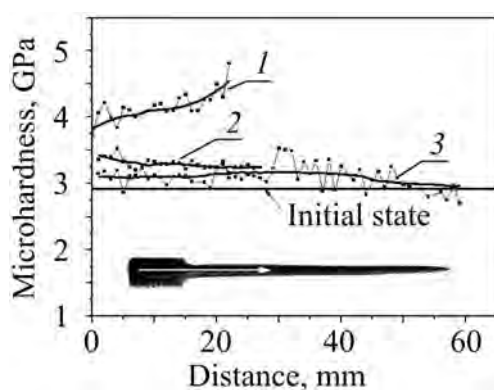
**Fig. 6.** Samples of the UFG Ti-6Al-4V ELI alloy after tension in the 500–600°C temperature range and  $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$  and microstructure in the indicated area of the sample; longitudinal section. (TEM was performed at the University of Erlangen)



**Fig. 7.** Samples of the UFG Ti-6Al-4V ELI alloy after tension in the 700–800°C temperature range and  $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$  and microstructure in the indicated area of the sample; longitudinal section. (TEM was performed at the University of Erlangen)

The view of the samples after tension in UFG state at temperatures of 700–800°C and  $\dot{\epsilon} = 10^{-2} \text{ s}^{-1}$  and at temperatures of 500 and 550°C and  $\dot{\epsilon} = 10^{-4} \text{ s}^{-1}$  is shown in Fig. 6,*a* and 7,*a*. Fig. 6,*b* presents the microstructure of the alloy after superplastic deformation at 600°C, the inspection of which reveals qualitative changes as compared to the structure after ECAP and extrusion. The grains about 500 nm in size with well-defined boundaries are clearly seen in the image of the microstructure. Despite considerable elongation of the sample (240%), the grains have equiaxed shape. As compared to the initial deformed state after ECAP and extrusion, a slight growth of their size is observed. Dislocation density within the grains has reduced significantly and does not exceed  $10^{14} \text{ m}^{-2}$ . The UFG alloy reveals also superplastic behavior at tension with a higher strain rate of  $10^{-2} \text{ s}^{-1}$  and higher temperatures. In addition, the microstructure of the samples after high strain rate deformation also undergoes characteristic changes that are followed by slight grain growth at 800°C (Fig. 7,*b*).

Microstructural changes that occurred in the samples after superplastic straining influenced the values of microhardness that was measured along the length of the sample from head to the point of rupture (Fig. 8). The recovery and grain growth in the head and neck of the sample subjected to straining at elevated temperatures (800°C) lead to a fall in microhardness as compared to the UFG state at room temperature and its higher homogeneity along the length of the sample. However, at low temperatures (500–550°C), when grain growth practically does not occur in the neck of the sample, hardening is observed which can be caused by further refinement of the microstructure of the alloy. The possibility of enhancing strength by means of straining in conditions close to superplastic ones was demonstrated on the samples of the UFG alloy after preliminary straining at 500°C; the samples after further straining at room temperature revealed strength amounting to 1510 MPa preserving sufficient ductility.



**Fig. 8.** Diagram showing the behavior of microhardness along the length of the samples after tension for various temperatures: 1 – 500°C,  $10^{-4} \text{ s}^{-1}$ ; 2 – 500°C,  $10^{-2} \text{ s}^{-1}$ ; 3 – 800°C,  $10^{-2} \text{ s}^{-1}$

Thus, straining in the conditions of low-temperature superplasticity of UFG materials can be used for processing articles of a complex shape providing at the same time the enhancement of mechanical properties at room temperature. Stressed state at localization of strain in conditions of uniaxial tension is close to, for example, the conditions of isothermal forging of the samples that enables to produce complex-shaped articles. The use of this approach enables efficient production of medical implants with qualitatively new service properties. This work is under development at our Institute.

### Conclusions

The Ti–6Al–4V ELI alloy after SPD including ECAP and extrusion demonstrates high mechanical properties at room temperature and superplasticity at temperatures above 600°C. The features of superplastic behavior of this UFG alloy consist in the formation of equiaxial structure with more «perfect» high-angle grain boundaries. Superplastic straining may result in additional strengthening of the alloy providing that the grain sizes are preserved in UFG structure at relatively low temperatures. This approach is very promising for processing of complex-shaped articles with high mechanical properties, particularly enhanced strength and ductility.

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