Effect of ultrasonic pretreatment on the superplasticity of titanium alloy Ti-6Al-4V processed by ECAP

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Abstract. Improving the superplasticity characteristics of alloys widely used in industry, such as titanium alloy Ti-6Al-4V, is an important practical problem. Processing by the methods of severe plastic deformation can reduce the temperature of superplasticity. However, the material exhibits high flow stresses. In this work we studied the effect of preliminary ultrasonic treatment with an amplitude of oscillating tension-compression stresses of 100 MPa on the characteristics of superplasticity of the Ti-6Al-4V alloy processed by equal channel angular pressing (ECAP). It is shown that high-frequency oscillating stresses resulted in the reduction of the flow stress, the increase in elongation and the strain rate sensitivity coefficient during tensile tests in the conditions of superplasticity.

1. Introduction
Metals and alloys with ultrafine-grained (UFG) and nano-scale structure exhibit high strength at ambient conditions and high ductility at elevated temperatures [1]. For example, titanium alloy Ti-6Al-4V processed by ECAP demonstrated an elongation of about 240% during isothermal deformation at 600 °C with a strain rate of 10^{-4} s^{-1} [2]. At the same time, low temperature superplastic deformation usually occurs at high flow stresses, which limits the possibilities of its practical applications. Ultrasonic treatment (UST) is one of the metal processing techniques, which allows modifying the defect structure of materials. It has been shown that UST of UFG metals results in a relaxation of the nonequilibrium structure of grain boundaries, the release of internal strains, and increases the ductility and impact toughness of these materials [3,4]. Imposing axial ultrasonic vibrations to the samples during superplastic deformation tests was shown to reduce the flow stress and increase the elongation to failure [5]. It is of a significant interest to study the effect of preliminary treatment of UFG alloys by ultrasound on their superplastic deformation. In the present paper, we study the changes in the structure and superplastic properties of the Ti-6Al-4V alloy processed by ECAP due to the UST with a stress amplitude of 100 MPa.

2. Experimental materials and procedure
Bars of titanium alloy Ti-6Al-4V were subjected to 6 passes of ECAP via route Bc at a temperature of 600 °C in a tool with an intersection angle of channels 120° (ε_{sum} ≈ 4) (marked as ECAP condition). Samples with a length of 40 mm were cut from the ECAP-ed material. Several samples were then subjected to UST with a frequency of 20 kHz. They were tightly clamped in the middle part of a cylindric steel waveguide with a length of 128 mm that is equal to the half-wavelength for this
frequency, so that a standing wave is formed in the waveguide. The amplitude of oscillating tension-compression stresses in the mid cross section of the samples was equal to 100 MPa. These samples will be further marked as “ECAP+UST samples”, while the ones tested directly after ECAP as “ECAP samples”.

Dog-bone samples with a gauge length of 10 mm, a width of 3.4 mm and a thickness of 1.5 mm were used for tensile tests. The tensile tests were carried out in isothermal conditions at a temperature of 600 °C with initial strain rates \( \mu = 10^{-4} - 10^{-3} \text{s}^{-1} \) in an Instron 5982 electromechanical-testing machine. Relative elongation to failure \( \delta \), true stress \( \sigma \) and true strain \( \varepsilon \) on the stage of the uniform elongation, and the strain rate sensitivity coefficient \( m \) at \( \varepsilon = 0.05 \) were determined.

The structure of the samples was studied in their longitudinal section by scanning electron microscopy (SEM) using a TESCAN MIRA 3 LMH FEG microscope in the regimes of back-scattered electron imaging (BSE) and electron back-scattered diffraction (EBSD) and by transmission electron microscopy (TEM) using a JEM 2000EX microscope.

3. Experimental results and discussion

The microstructure of the investigated samples is shown in figure 1. No differences in the microstructure of the samples subjected to ECAP and ECAP+ UST were detected. Elongated primary \( \alpha \)-grains with sizes in the longitudinal and cross directions 15±2 and 6±1 µm, respectively, and a mixture of fine crystals with sizes less than 1 µm of the secondary \( \alpha \)- and \( \beta \)-phases were observed. Volume fractions of the primary \( \alpha \)-phase and \( \beta \)-phase were about 25% and 9%, respectively. Reliable EBSD structural maps were not obtained, probably due to the high level of internal stresses in the ECAP and ECAP+UST samples (figure 1b). Room temperature mechanical properties of the ECAP and ECAP+UST samples were nearly the same: the ultimate strength and elongation to failure were 1250 MPa and 12%, respectively.

![Figure 1. BSE (a) and TEM (b) images of microstructure of Ti-6Al-4V alloy subjected to ECAP (a) and ECAP+UST (b).](image)

Preheating of the samples before tensile tests at 600 °C leads to the development of recovery and recrystallization processes [6]. Fine misoriented fragments of the \( \alpha \)-phase were observed in the BSE images (figure 2 a, c) and in the orientation maps (figure 2 b, d). Apparently, the UST promoted the development of relaxation processes and facilitated the formation of new grain boundaries during preheating of the ECAP-ed alloy Ti-6Al-4V. The fractions of high-angle boundaries were 0.38 and 0.53 in the preheated samples in ECAP and ECAP+UST conditions, respectively.

This difference in the microstructure provided differences in the properties of the investigated samples during superplastic deformation. The results of tensile tests are presented in figures 3-5. The
shapes of $\sigma - \varepsilon$ curves were not typical for the classical superplastic flow (figure 3). The presence of strain softening stages in $\sigma - \varepsilon$ curves assumes that certain relaxation processes occurred in the alloy during testing. UST resulted in a decrease in the intensity of softening processes during superplastic deformation. A steady state flow stage was observed during tensile tests with initial strain rates of $1.7 \times 10^{-4}$ s$^{-1}$ and lower; at higher strain rates, the stage of strain localization followed after the strain softening stage.

**Figure 2.** Microstructure of the preheated samples of the Ti-6Al-4V alloy subjected to ECAP (a, b) and ECAP+UST (c, d); (a,c) – BSE images, (b,d) – orientation maps.

**Figure 3.** Typical true stress – true strain curves of tensile deformation of UFG Ti-6Al-4V after ECAP and ECAP+UST.
ECAP-ed Ti-6Al-4V alloy subjected to UST demonstrated lower values of the flow stress in initial stages of deformation (figures 3, 4) and higher values of strain rate sensitivity (figure 5) and elongation to failure (figure 4).

![Figure 4](image1.png) ![Figure 5](image2.png)

**Figure 4.** Effect of UST on the flow stress and elongation to failure of UFG Ti-6Al-4V alloy  
**Figure 5.** Effect of UST on the strain rate sensitivity coefficient, $m$

More detailed structural studies are needed to understand the nature of the effect of ultrasonic treatment on the superplastic behavior of the Ti-6Al-4V alloy. In particular, the complete, incomplete and pseudopartial wetting of grain boundaries by the second solid phase discussed in [7] can explain the observed effect of the UST on high temperature deformation of the alloy.

4. **Conclusions**

Structural studies demonstrated that UST accelerated the recovery and recrystallization processes in the ECAP-ed alloy Ti-6Al-4V during the preheating of the samples at 600 °C. This led to the formation of a finer-grained structure compared to an alloy that was not subjected to UST. It was shown that high-frequency oscillations with a stress amplitude of 100 MPa resulted in the reduction of the flow stress, increase of elongation and the strain rate sensitivity coefficient during tensile tests in the conditions of low-temperature superplasticity.

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**References**

[1] Valiev R Z, Zhilyaev A P and Langdon T G 2013 *Bulk Nanostructured Materials: Fundamentals and Applications* (Hoboken: Wiley)  
[2] Valiev R Z, Islamgaliev R K and Semenova I P 2007 *Mater. Sci. Eng. A* 463 2  
[3] Samigullina A A, Nazarov A A, Mulyukov R R, Tsarenko Yu V and Rubanik V V 2014 *Rev. Adv. Mater. Sci.* 39 48  
[4] Samigullina A A, Mulyukov R R, Nazarov A A, Mukhametgalina A A, Tsarenko Y V and Rubanik V V 2014 *Lett. Mater.* 4 52  
[5] Myshlyaev M M, Shpeizman V V, Klubovich V V, Kulak M M and Lyu Y 2015 *Phys. Solid State* 57 2039  
[6] Humphreys F J and Hatherly M 2004 *Recrystallization and Related Annealing Phenomena*, 2nd ed (Amsterdam: Elsevier)  
[7] Gornakova A S, Straumal B B, Nekrasov A N, Kilmametov A and Afonikova N S 2018 *J. Mater. Eng. Perf.* 27 4989