



# Superplastic deformation behavior of Ti-4Al-2V alloy governed by its structure and precipitation phase evolution

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## ABSTRACT

Superplastic deformation development in pseudo- $\alpha$  Ti-4Al-2V alloy (volume fraction of the  $\beta$  phase  $< 2\%$ ) with different initial grain structure was studied at low-temperature region (673–1173 K). It is shown that in the coarse-grained state of the alloy superplastic deformation does not realize in the investigated temperature range. At the same time, an alloy with an ultrafine-grained and fine-grained structure exhibits the development of superplasticity with maximum elongations to failure 620% and 300%, respectively. In this case, the temperature interval of realization of the superplastic flow of the ultrafine-grained alloy is shifted to the region of lower temperatures (approximately 250 degrees) in comparison with the fine-grained counter-part. It was found that the nonmonotonic temperature dependence of elongation for the ultrafine-grained alloy is determined by its specific structure and phase evolution during superplastic deformation due to considerable grain growth. It is shown that superplastic deformation is accompanied by intense precipitation of second phase particles at grain boundaries and in the grain bulk. It is assumed that this is due to the activation of grain boundary diffusion fluxes and, as a consequence, to the accelerated redistribution of alloying elements in the bulk and at the grain boundaries.

## 1. Introduction

Titanium alloys find numerous applications in machine and aircraft industries owing to their unique physical and mechanical properties [1–5], which depend on the microstructure formed during deformation and heat treatment. It is known that plastic strain localization in titanium alloys with low thermal conductivity leads to the formation of so-called zones of intense flow at elevated temperatures, making the macro- and microstructure highly inhomogeneous [6]. At temperatures above  $0.6 T_m$  ( $T_m$  is the melting point), titanium alloys undergo intense surface oxidation in air with the formation of a brittle alpha-case layer [5,7]. Consequently, low temperature processing of these alloys, particularly under superplastic forming conditions, becomes especially relevant. Superplastic forming often produces a homogeneous structure throughout the billet volume without additional multipass processing and interpass annealing [8,9].

Superplastic flow occurs in polycrystalline materials with a grain size of less than  $10 \mu\text{m}$ , usually at temperatures about  $0.5 T_m$  and strain rates ranging from  $10^{-5}$  to  $10^{-2} \text{ s}^{-1}$  [8,9]. Therefore the material must have a prepared fine globular structure. Another feature of ( $\alpha + \beta$ ) titanium alloys is that they can go over to the superplastic state without special preparation of their structure under certain temperature and

strain rate conditions [10,11]. This “natural” superplasticity is a result of phase transformations and high diffusion mobility of atoms, due to which the fine globular structure is formed in the alloys by the time of the onset of superplastic deformation and/or during deformation [8]. The initial structure and phase composition of the alloys can also strongly affect the development of plastic flow and the value of elongation to failure [8,9,12]. It is therefore of practical importance to investigate the evolution of the structure and phase composition, as well as the plastic flow in titanium alloys deformed in the moderate temperature range ( $0.4\text{--}0.5 T_m$ ) in which low temperature superplasticity can occur.

To date, most studies on superplastic deformation of titanium alloys have been carried out for ( $\alpha + \beta$ ) alloys [8,9], including the alloys with ultrafine-grained structure ( $d < 1 \mu\text{m}$ ) [13–15]. It is known that owing to the presence of the second phase the fine-grained structure favorable for superplastic deformation is stable [8,16], diffusion-controlled phase transformations occur at deformation temperatures, and the lamellar microstructure can rapidly transform into globular one [10,12,17,18]. A more ductile  $\beta$ -phase prevents the formation of porosity during superplastic deformation [19]. Hence the mechanism of superplastic flow in pseudo- $\alpha$  titanium alloys may have its own features, first of all due to a small volume fraction of the second ( $\beta$ ) phase.

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Pseudo- $\alpha$  titanium alloys show much promise as materials for aircraft industry [2,4], chemical industry, and medicine [20] owing to their high fatigue properties and creep resistance at elevated temperatures, as well as to increased corrosion resistance in various corrosive environments [21]. In recent years, there has been an increased interest in deformation processing of such alloys, including under superplasticity conditions [22–24]. The cited authors found for alloys with high initial  $\beta$ -phase content ( $> 6\%$ ) that superplasticity in pseudo- $\alpha$  titanium alloys, including those with fine-grained structure, occurs in the temperature range 1073–1173 K ( $0.5$ – $0.6 T_m$ ). As noted above, at such temperatures ( $> 1073$  K) the oxidation of titanium alloys with rapid growth of the oxide layer is observed which leads to a reduction in their ductility. It is therefore of great interest to study whether low temperature superplasticity (at temperatures below  $0.6 T_m$ ) can occur in pseudo- $\alpha$  titanium alloys with low  $\beta$ -phase content ( $< 2\%$ ) and different initial structure.

## 2. Material and experimental procedures

The material for investigation was commercial pseudo- $\alpha$  Ti-4Al-2V alloy (Russian alloy PT-3V) with the chemical composition Ti – 4.66 wt % Al – 1.92 wt % V [17]. Ultrafine-grained structure was produced by severe plastic deformation of the original coarse-grained alloy using multidirectional forging [17] on an IP-2000 pressing machine in the temperature range 1073–723 K. The ultrafine-grained alloy was then annealed at 973 K for 1 h to obtain a fine-grained structure.

Tensile tests of dumbbell specimens with the gauge part measured  $5 \times 1.7 \times 0.8 \text{ mm}^3$  were carried out using a PV-3012M machine equipped with a tensometric load measuring system, with simultaneous recording of flow curves in the time–load coordinates. The specimens were cut by electric spark machining. Before testing, an about 100  $\mu\text{m}$  thick layer was removed from the specimen surface by mechanical grinding and subsequent electrolytic polishing. Tension was performed in vacuum of  $10^{-2}$  Pa with an initial strain rate of  $6.9 \cdot 10^{-3} \text{ s}^{-1}$  in the temperature range 673–1173 K. The spread in elongation to failure in specimens did not exceed 10%. To study the evolution of structural-phase state of the alloy during superplastic deformation the tensile tests of some samples were interrupted at 100% of strain.

The surface microstructure was examined by scanning electron microscopy using a Quanta 200 3D microscope with a tungsten filament and the Pegasus EBSD system. In so doing, the specimens were polished in order to remove a thin layer of  $\sim 10 \mu\text{m}$  and chemically etched to reveal the microstructure. Electron microscopy studies of thin foils were carried out on a JEM-2100 microscope at the Shared Use Center NANOTEH of ISPMS SB RAS. Foils for electron microscopy were prepared by a standard procedure using a Micron-103 jet polishing machine with an electrolyte of the following composition: 20%  $\text{HClO}_4$  + 80%  $\text{CH}_3\text{CO}_2\text{H}$ . The sizes of the grain-subgrain structure elements were determined from the dark-field image. No less than 200 measurements were sampled. The phase composition was studied using a Shimadzu XRD-6000 diffractometer using  $\text{CuK}\alpha$  radiation. Metallographic studies were performed on an Olympus GX 71 optical microscope.

## 3. Experimental results

### 3.1. Flow behavior

Comparative studies of the plastic deformation of Ti-4Al-2V alloy within the above given temperature range were conducted for three states. The coarse-grained alloy has a martensitic structure with the grain size about 700  $\mu\text{m}$  (Fig. 1). The fine-grained alloy exhibits a globular structure with the average grain size about 6  $\mu\text{m}$ . The average size of the grain-subgrain structure in the ultrafine-grained alloy is 0.25  $\mu\text{m}$  (Fig. 1). The volume fraction of the  $\beta$ -phase in the Ti-4Al-2V alloy in all states is approximately 1–2% and is not detected by X-ray

diffraction analysis. As it was shown in Ref. [25], the formation of the ultrafine-grained structure in the alloy significantly increases its mechanical properties at room temperature in comparison with the coarse-grained state (tensile strength 1170 and 715 MPa, respectively).

The study of the temperature dependence of the mechanical properties of tensile Ti-4Al-2V alloy revealed that they strongly depend on the structural state of the alloy. Fig. 2 shows the flow curves for the ultrafine- and fine-grained Ti-4Al-2V alloy in the temperature range 823–1173 K. As one can see, the ultrafine-grained alloy exhibits higher elongation to failure and lower flow stresses at all temperatures as compared with the fine-grained state. Moreover, the ultrafine-grained alloy has high superplastic properties with an about 350% elongation to failure already at 873 K, whereas the same quantity in the fine-grained alloy is about 50% (Fig. 2). Noteworthy is curve 5 in Fig. 2a which shows a flow stress drop to several MPa in the ultrafine-grained alloy at a temperature of 1173 K.

The coarse-grained Ti-4Al-2V alloy demonstrates the highest tensile strength and yield strength at temperatures above 873 K. However, the alloy ductility under the studied conditions does not exceed 60%. Thus, the alloy with the initial coarse-grained structure does not become superplastic even at 1173 K (Fig. 3, curve 1).

The fine-grained Ti-4Al-2V alloy exhibits rather high ductility in the temperature range 973–1173 K (Figs. 2 and 3). The relative elongation of the specimens under the used tensile test conditions increases from 100% to 300% (Fig. 3b, curve 2). According to the temperature dependence of the strength characteristics of the alloy, its strength in the ultrafine-grained state is much higher at 673 K than in other states (Fig. 3a). With increasing test temperature, the ultrafine-grained alloy undergoes rapid softening, while its elongation to failure increases sharply and reaches about 100% at 823 K (Fig. 3b, curve 3). The experimental data in Fig. 3 illustrate that the superplastic flow behavior of the ultrafine-grained Ti-4Al-2V alloy is distinguished by a decrease in the relative elongation of specimens at 973 and 1073 K, and by a sharp increase at 1173 K. However, the flow stress–strain curves have no specific features (Fig. 2a). Additionally, the ductility of the ultrafine-grained alloy at the test temperature 1173 K is twice as high as that for the fine-grained state. It can be assumed that these effects are associated with the structural-phase state evolution in the deforming alloy.

Thus, the Ti-4Al-2V alloy with ultrafine- and fine-grained structure undergoes superplastic deformation in the considered conditions. If we assume that superplastic flow occurs at a specimen elongation to failure of more than 100%, then the superplasticity temperature of the ultrafine-grained Ti-4Al-2V alloy is by approximately 250 degrees lower than in the fine-grained state (Fig. 3, cf. curves 2 and 3). The coarse-grained alloy undergoes no superplastic flow.

### 3.2. Microstructure evolution

As was suggested above, the superplastic deformation features of Ti-4Al-2V alloy may be related to the structure and phase evolution. We studied the structural-phase state of the alloy deformed plastically to 100% at various temperatures. Fig. 4 shows micrographs of the surface of the gauge part of ultrafine-grained specimens deformed superplastically at 873 and 973 K. It is seen that there are precipitates at grain boundaries after deformation. The grain size in the alloy deformed at 973 K is much larger than that at 873 K. According to EBSD analysis, the average grain size after 100% deformation is 1.24 and 0.87  $\mu\text{m}$ , respectively, and the grains are equiaxed (Fig. 4).

X-ray diffraction line broadening analysis of dislocation density in the ultrafine-grained alloy showed that the density decreases from  $1.4 \cdot 10^{14}$  to  $5 \cdot 10^{12} \text{ m}^{-2}$  as a result of 100% superplastic deformation, which corresponds to the annealed (recrystallized) state [26]. Transmission electron microscopy also revealed a low density of dislocations in the bulk of grains after superplastic deformation (Fig. 5a), which, together with the globular grain shape, indicates that the main deformation mechanism in the considered conditions is grain boundary

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