INTRODUCTION

The increase in demand for titanium alloys in the chemical and aerospace industries inspired a buildup of investigations in the field of improving their utilization properties. Grain refining to submicron dimensions (size of grains $d < 1 \mu m$) is known to be an effective way of improving the strength and utilization properties of metal polycrystals at low homologic temperatures [1, 2]. For that matter, methods of deformation and thermomechanical processing are actively developed today for obtaining ultrafine-grained structure in commercial titanium alloys [1, 2]. One of the often used techniques of formation of ultrafine-grained structure in titanium alloys is such an SPD (severe plastic deformation) method as three-dimensional pressing at elevated temperatures [3], making it possible to decrease the degree of deformation required for obtaining the ultrafine-grained structure with the size of elements of 0.1–0.5 $\mu m$. Formation of the ultrafine-grained structure in a Ti–6Al–4V alloy is shown to lead to growth of its strength properties by approximately a factor of 1.5, an increase in the hydrogen embrittlement resistance, and a decrease in the temperature transition to the superplastic state by 200–300 K.

MATERIALS AND METHODS

As a material for the investigation, commercial two-phase $\alpha + \beta$ alloy Ti–6Al–4V (VT6 alloy grade) of the following composition in wt % was used: 6.6 Al + 4.9 V + 0.02 Zr + 0.033 Si + 0.18 Fe + 0.007 C + 0.17 O + 0.01 N + 0.002 H; the balance is Ti. The structure of the initial Ti–6Al–4V alloy was fine-grained with the grains 6 $\mu m$ in average size. The volume fraction of $\beta$ phase in the alloy was 13 vol %. An ultrafine-grained state in the Ti–6Al–4V alloy was achieved by two methods: a method combining reversible hydrogen alloying and hot working. The initial dimensions of deformed samples in the first case were the following: diameter of 20 mm, height of 50 mm; in a method combining reversible hydrogen alloying and hot pressing, they were 20 mm and 35 mm, respectively. The three-dimensional pressing was carried out under a gradual decrease in deformation temperature from...
to 773 K in the process of five cycles (one cycle corresponded to three upsettings by ~50% at the same temperature [5]). In a case of the method combining reversible hydrogen alloying and hot working, the ultrafine-grained structure in the Ti–6Al–4V alloy was formed in accordance with a schedule including hydrogen pre-alloying, water quenching from 1223 K, single-stage compression by 80% at the temperature of 973 K, and hydrogen degassing in vacuum at the temperature of 873 K.

Hydrogen alloying of the alloy up to concentrations of 0.2–0.33 wt % (hereinafter, the hydrogen concentration in the alloy is stated in wt %) was carried out in gas environment by a Siverst-type apparatus at temperature of 973 K. The hydrogen concentration in the samples was measured by a RHEN 602 gas analyzer with 0.001% accuracy.

Electron-microscopic investigations of thin foils were carried out using an EM-125K transmission electron microscope. The dimensions of structural elements were measured according to photos of the microstructure by the secant method. The phase composition and lattice parameters of phases of the alloy were defined by the X-ray diffraction method using a Shimadzu XRD-6000 diffractometer in Cu$K_\alpha$ radiation.

The mechanical properties of flat dog-bone shaped samples with bodies of gage dimensions of $15 \times 3 \times 1 \text{ mm}^3$ were investigated with a PV-3012M machine within the temperature range of 293–923 K. The experiments at elevated temperatures were carried out in a vacuum of $10^{-2} \text{ Pa}$. The samples with and without a notch on their bodies were investigated. The notch depth was 0.7 mm, the angle was 50 degrees, and the corner radius of the notch was 0.25 mm. The billets were cut by electric spark to obtain the samples. The surface of the samples before the testing was first abraded and then electropolished.

**EXPERIMENTAL RESULTS AND DISCUSSION**

A representative electron-microscopic image of the ultrafine-grained structure of the Ti–6Al–4V alloy after three-dimensional pressing according the mode described above is shown in Fig. 1. It is evident that an entangled deformational contrast is observed and some structure elements are poorly distinguished on the bright-field image of the structure (Fig. 1a). Quite a number of reflections uniformly spaced on a circle are observable in the microdiffraction image of such a structure recorded from the area of 1.8 $\mu\text{m}^2$ (Fig. 1a). Thus, many reflections have azimuthal blurring. Such a type of microdiffraction is typical of ultrafine-grained materials with high misorientations between the elements of grain/subgrain structure, nonequilibrium grain boundaries, and internal fields of compressive stresses [1, 2]. The dimensions of the elements of the grain/subgrain structure of the Ti–6Al–4V alloy defined from the dark-field image (Fig. 1b) after complete cyclic process of pressing vary between 0.05 and 0.5 $\mu\text{m}$ (Fig. 1c). The average size of the elements of the ultrafine-grained structure is 0.18 $\mu\text{m}$. X-ray diffraction analysis shows that the Ti–6Al–4V alloy stays two-phased in the process of forming an ultrafine-grained condition by the three-dimensional pressing. However, the volume fraction of the $\beta$ phase decreases when the number of pressing cycles increases and is ~7% after the last cycle of pressing (Table 1).

An electron-microscopic image of a representative area of the ultrafine-grained structure of the Ti–6Al–4V alloy made by the method combining reversible hydrogen alloying and hot pressing and also the correspond-