CONDENSED-STATE PHYSICS

INFLUENCE OF HYDROGENATION ON EVOLUTION OF
SUBMICROCRYSTALLINE STRUCTURE OF Ti–6Al–4V ALLOY
UPON EXPOSURE TO TEMPERATURE AND STRESS

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The influence of hydrogenation on the growth and size distribution of submicrocrystalline grain-subgrain structure elements of Ti–6Al–4V alloy is investigated by methods of transmission electron microscopy at temperatures 673–973 K. The kinetics is investigated and the energy of activation of growth of grain-subgrain alloy structure elements is determined in the processes of free annealing and stretching. The activation effect of deformation and hydrogen diffusion on migration of grain boundaries and growth of submicrocrystalline grain-subgrain structure elements is established.

Keywords: Ti–6Al–4V titanium alloy, stress, hydrogen diffusion, growth of submicrocrystalline grain-subgrain structure elements, activation energy.

INTRODUCTION

A number of phenomena associated with diffusion of substitutional and interstitial impurity atoms from external or internal sources along grain boundaries are observed in polycrystalline materials at elevated temperatures. Among such phenomena are migration of boundaries and recrystallization (activated recrystallization) initiated by impurity diffusion [1–4]. Nano- and submicrocrystalline materials obtained by methods of severe plastic deformation which are promising for application as structural ones have extended and often nonequilibrium grain boundaries and increased grain boundary diffusion coefficients [5, 6]. Therefore, the evolution in such submicrocrystalline materials of migration of boundaries and recrystallization caused by impurity diffusion along grain boundaries can introduce essential corrections to the temperature intervals of their structural stability and structure-sensitive properties. Of particular interest are investigations of the influence of hydrogen diffusion on the stability of structure of submicrocrystalline metal materials. First, hydrogen can easily penetrate into metal materials from the environment [7]. Second, possessing high diffusion mobility in comparison with other interstitial impurities, it can be redistributed inside of the material volume under the influence of elastic pressure fields [8]. This leads to formation of internal diffusion hydrogen flows. It seems likely that they initiate migration of boundaries and growth of grains. However, the problem of the influence of hydrogen flows along grain boundaries on the stability of the structure and properties of submicrocrystalline materials obtained by methods of severe plastic deformation has not yet been considered in the literature.

The present work is aimed at investigation of the influence of hydrogenation on the recrystallization and growth of grains during annealing and tensile strain in a submicrocrystalline structure formed by methods of severe plastic deformation on an example of Ti–6Al–4V titanium alloy.

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MATERIAL AND METHODS OF RESEARCH

We investigated two-phase (α + β) Ti–6Al–4V alloy (mark VT6) of the following composition (hereinafter the hydrogen concentration in the alloy is indicated in mass%): 6.6Al + 4.9V + 0.02Zr + 0.033Si + 0.18Fe + 0.007C + 0.17O + 0.01N + 0.002H, the rest titanium. In the initial state the Ti–6Al–4V alloy had fine-grained structure with average grain size of 6 μm. The volume fraction of the β-phase in the alloy was 13 vol.%. The submicrocrystalline state of Ti–6Al–4V alloy was obtained by the method of pressing with change of the deformation axis and gradual decrease of the deformation temperature from 1073 to 873 K [9].

The alloy was hydrogenated to concentration of 0.24 mass% in a gas medium using a Siverst-type setup at a temperature of 873 K. The hydrogen concentration in specimens was measured by a RHEN 602 gas analyzer with error of 0.001%.

Electron microscopic analysis of thin foils was performed using an ÉM-125K transmission electron microscope. An OLYMPUS GX71 optical microscope was used for metallographic studies. Sizes of structural elements were measured from photographs of the microstructure by the secant method. The phase structure of the alloy was determined by the method of x-ray structural analysis on a Shimadzu XRD-7000 diffractometer in CuKα radiation at temperatures 293–873 K.

Annealing and tensile tests were performed in vacuum of 10^{-2} Pa using a PV-3012M machine equipped with a tensometric system of load measurement with automatic recording of stress-strain flow curves in load–time coordinates. The initial rate of sample stretching was 2.2·10^{-3} s^{-1}. Test specimens shaped as double blades with working part sizes 15 × 3 × 1 mm were cut from billets by the electrospark method. Before testing, specimen surfaces were subjected to mechanical grinding and electrolytic polishing.

RESULTS AND DISCUSSION

The typical structure of the examined submicrocrystalline Ti–6Al–4V alloy is shown in Fig. 1. On electron diffraction patterns of an area of 1.4 μm² (Fig. 1a), a large number of reflections was observed uniformly spaced along a circle, which testifies to the presence in the structure of a great number of grains in unit volume and significant degrees of their misorientations. The complex strain-induced contrast caused by high density of dislocations and presence of extinction contours did not allow us to determine the grain sizes from the bright-field image. The average size of grain-subgrain elements of the alloy structure retrieved from the dark-field image (Fig. 1b) was 0.41 μm.

To choose the temperature of hydrogenation of the Ti–6Al–4V alloy, the thermostability of the obtained submicrocrystalline structure was estimated at temperatures 823–1023 K. Annealing for an hour in the indicated temperature interval demonstrated that the size of the submicrocrystalline structure elements of the alloy remained unchanged up to temperature of 923 K. At a temperature of 923 K, an increase in sizes of grain-subgrain structure elements was observed. However, the alloy structure remained submicrocrystalline. Thus, judging by the character of changes of the structure element size (disappearance of the elements with the smallest size and occurrence of the elements with larger sizes), the collective recrystallization was observed during heating and annealing of the submicrocrystalline Ti–6Al–4V alloy. Growth of structure elements till sizes exceeding one micron takes place during annealing at 1073 K. Hydrogenation of submicrocrystalline Ti–6Al–4V alloy at a temperature of 873 K for 15 min with heating and cooling rates of 12 deg/min does not change the average size of grain-subgrain structure elements. The average size of grain-subgrain structure elements of the hydrogenated alloy determined from the dark-field image was 0.42 μm.

By the methods of x-ray structural analysis it was established that the submicrocrystalline Ti–6Al–4V–0.002H alloy at temperatures in the range 293–973 K contains only α- and β-phases (curve 1 in Fig. 2). The volume fraction of the β-phase calculated from the x-ray diffraction pattern was about 9 vol.%. Precipitations of hydrides arose in specimens after hydrogenation in addition to the α- and β-phases at room temperature. This was testified by the change in the relationship between the intensities of the (100), (002), and (101) reflections of the α-phase of Ti–6Al–4V–0.24H alloy compared to the corresponding relationship of these reflections in the Ti–6Al–4V–0.002H alloy (see curve 2 in...