ISSN 1063-7850, Technical Physics Letters, 2017, Vol. 43, No. 1, pp. 61–63. © Pleiades Publishing, Ltd., 2017.
Original Russian Text © V.I. Betekhtin, A.G. Kadomtsev, M.V. Narykova, O.V. Amosova, V. Sklenicka, 2017, published in Pis'ma v Zhurnal Tekhnicheskoi Fiziki, 2017, Vol. 43, No. 1, pp. 38–44.

Defect Structure and Mechanical Stability of Microcrystalline Titanium Produced by Equal Channel Angular Pressing

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Received July 16, 2016

Abstract—It is established that increases in nanoporosity and the proportion of high-angle grain boundaries in the process of equal-channel angular pressing are the main structural factors leading to reduction in mechanical stability (durability) of microcrystalline titanium during long-term tests under creeping conditions.

DOI: 10.1134/S1063785017010047

The wide area of application of titanium is evoking increased interest in the study of the structure peculiarities of this metal in its high-strength microcrystalline state formed as a result of severe plastic deformation (SPD). It is known that the high mechanical properties after SPD are determined basically by the size of the grains and condition of their boundaries. Reduction of the size of grains during SPD leads to an increase in the volume fraction of their boundaries, where high concentrations of defects (dislocations, vacancies, nanopores, etc.) and high internal stresses are localized [1, 2]. Because of this, the nano- and microcrystalline metals produced by SPD are inherently nonequilibrium, and so the problem of their mechanical stability, especially during prolonged stress, is important both for fundamental research and in terms of application [3]. In [4, 5], it was shown that a significant influence on the mechanical stability is exerted by two structural factors: nanoporosity formed during SPD and high-angle boundaries ($\varphi > 15°$) causing a high level of internal stress. In this paper, we investigate the contribution of these two factors to mechanical stability (durability during the test in the creeping mode) of microcrystalline titanium produced by equal channel angular pressing (ECAP).

VT1-0 titanium with an impurity content ≈0.3% was selected for the study. The ECAP was performed by route B_c with cyclic rotation of the workpiece around the axis of the channel by 90° after each cycle and the angle of intersection of the channels of 120° at 673 K [6].

For mechanical testing, samples were used that had been prepared after different numbers of ECAP passes. The samples had a length of the uniformly deformable part of 15 mm with a cross-sectional area of 3 × 2 mm^2. The prepared samples were tested at $T = 673$ K and $\sigma = 15$ MPa until rupture, and the time to failure (durability) was determined. Additionally the microhardness and its variation depending on the number of passes in ECAP were determined on initial (before testing in creep conditions) samples.

The density of the samples and its change during ECAP caused by pore formation, among other things, were determined by triple hydrostatic weighing. The parameters of the pores were found with the help of a modified method of X-ray scattering in the field of ultralow angles using a high (1.5 GPa) hydrostatic pressure for identifying the void nature of the scattering inhomogeneities [7]. The sizes of grains and their distribution by disordering were determined using transmission and scanning electron microscopy and backscattering of electrons.

Consider the obtained experimental data. It was established that the densities of samples of titanium in the original (before ECAP) state and after two, four, and eight passes were 4.5127 ± 0.0003, 4.5117 ± 0.0005, 4.5060 ± 0.0006, and 4.5100 ± 0.0005 g/cm^3, respectively. Thus, there is a clear trend toward growth "loosening" of the titanium (determined by the level of nanoporosity, among other factors) with an increase in the number of passes. The effect of high hydrostatic pressure, as studies have shown, leads to a significant increase of density. For example, after four passes in ECAP, the density increased from 4.5065 to 4.5100 g/cm^3 because of the applied hydrostatic pressure. The action of hydrostatic pressure allowed us to identify the nature of the increased intensity of the small angle scattering that occurs after ECAP (Fig. 1, curves *1* and *2*). It is seen that the intensity of the scattering is markedly reduced after the action on samples

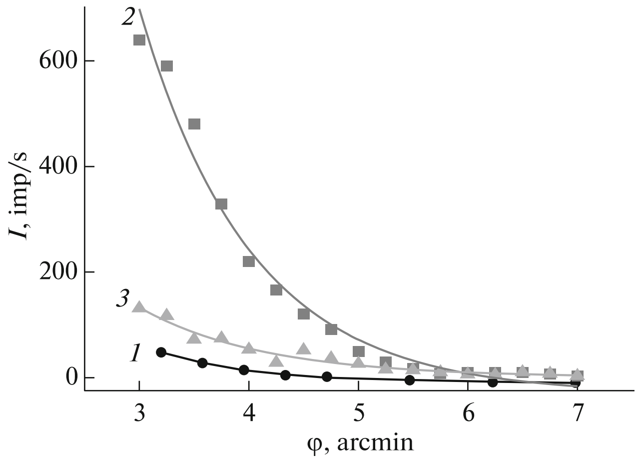

Fig. 1. The intensity of small-angle x-ray scattering for the samples of titanium: (*1*) coarse-grain (initial) condition, (*2*) after four passes of ECAP, and (*3*) after four passes of ECAP and the impact of a hydrostatic pressure of 1.5 GPa.

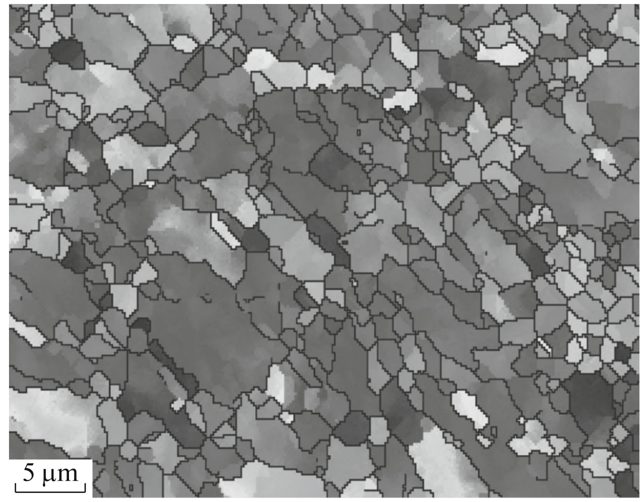

Fig. 2. Microstructure of titanium after eight passes of ECAP.

of high hydrostatic pressure (Fig. 1, curve *3*). Assuming (based on the data of [5, 7]) that the observed effect is due to a decrease (healing) of the porosity, the sizes of pores and the contribution of their healing in the density were determined in accordance with [8]. It was established that pore sizes of ≈15–20 nm and their volume fraction are in good agreement with the density change after the application of pressure.

Consider the data on the influence of number of passes during ECAP on the grain sizes and, especially their distribution, in disordering. The measurements performed with high statistics showed that the average grains size in the initial state and after two, four, and eight passages were 15, 2.15, 1.25, and 0.75 μm, respectively. A grain structure after eight passes is shown in Fig. 2 as an example. The hydrostatic pressure, as shown by structural studies, was not affected by the grain size. No effect of pressure on the distribution of grains by disordering was detected either; for example, in Fig. 3 the distributions by disordering (a) before and (b) after the effect of pressure for titanium obtained as a result of two passes of ECAP are shown. At the same time, the portion of high angle boundaries (high angle grain boundaries, HABG) grows significantly with an increase in the number of passes. Thus, if for two passages the HABG is ~33%, then after eight passes it is ~57%. Note that the lack of an effect of pressure on the distribution of grains by disordering was observed earlier for pure Al and Cu–0.2 wt % Zr alloy [4].

Let us consider and analyze the data of mechanical tests. Previously, it was found that under the chosen test conditions, the durability of titanium on the creep in the initial state and after two, four, and eight passes is ≈140, 78, 60, and 38 h [6]; i.e., even after the transition to the microcrystalline state, the durability significantly decreased. Similar patterns were obtained for a range of metals and alloys in [9, 10]. After exposure to pressure leading to healing of nanoporosity, an increase in durability was observed. However, the effect of a significant increase in durability was detected only for the samples obtained with a small number of passes; with an increased number of passes, it practically disappeared. Thus, the durability of the samples of titanium after two, four, and eight passes and the healing effect of the pressure was 98, 62, and 39 h, respectively. It is seen that, after only two passes, the healing of the pores markedly increased the durability (from 78 to 98 h).

The data obtained allow us to conclude that healing of the nanopores effectively increases the durability only in samples with a relatively small portion. At an increase in the high angle boundaries, their negative impact under sustained loading, obviously, completely negates the effect of healing of the nanopores that is positive for mechanical stability.

At the same time, the formation of a microcrystalline disordered structure under ECAP and the increase of the portion of high angle boundaries significantly increases the ultimate strength, yield strength, microhardness, i.e., "short-term" characteristics of the mechanical properties that are not associated with the duration of action of the load. Indeed, for the Ti studied in this work, the microhardness in the initial state and after four and eight passes in ECAP is 1060, 2950, and 3150 MPa.

Thus, it follows from the obtained data that the nanopores and high-angle boundaries have different effects on the strength characteristics. Nanoporosity probably has practically no effect on the "short-term" durability characteristics, and the increase in the high angle boundaries (with reduction in the sizes of the grains) in ECAP leads, as has been noted [1, 2] to an improvement in these strength characteristics.

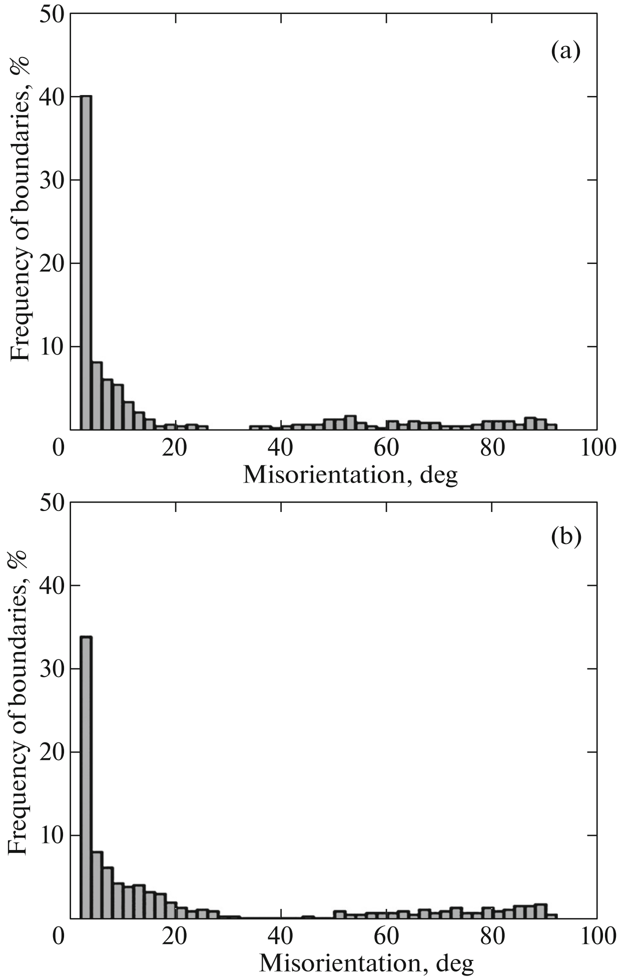

Fig. 3. The distribution of grains as function of disordering for microcrystalline titanium after two passes of ECAP (a) before and (b) after exposure to hydrostatic pressure of 1.5 GPa.

However, the transformation of titanium in the microcrystalline state with high characteristics of ultimate strength and microhardness led to lower mechanical stability (durability) in long-term tests.

Taking into account these and previously obtained results from structural research [5–7, 9], the following explanation for the above-considered data on the influence of ECAP on different characteristics of strength can be given. The high-angle grain boundaries (sources of high internal stresses) and nanopores formed during large plastic deformations (including during ECAP) have a negative impact on the durability of microcrystalline metals and alloys that are tested in the regime of creeping or fatigue. These structural defects are obviously the nucleation "centers" for the development of fracture during long-term loading under creep conditions. Note that increasing the volume fraction of grain boundaries in the material during SPD can also have a negative impact on durability when tested in the creep regime, as the development of deformation and fracture occurs mainly at the borders. However, the role of the first two factors is predominant.

The results are of undoubted interest also from the practical point of view, as they allow us to choose the right number of passes of ECAP to obtain optimal mechanical properties, especially under creep and fatigue tests, i.e., under long-term tests that determine the performance of such an important structural material as titanium.

Acknowledgments. This study was performed under a grant from the Russian Science Foundation, project no. 15-12-30010. We authors express their gratitude to staff members of GATA (Ufa) G.K. Raab and S.N. Faizova for conducting ECAP of titanium.

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Translated by N. Petrov