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Evolution of the defective structure during long-term loading of ultrafine-grained titanium VT1-0 obtained by intensive plastic deformation

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For high-strength titanium VT1-0, the ultrafine-grained structure of which was obtained by mechanical-thermal treatment using the methods of helical and longitudinal rolling followed by annealing to relieve stress of the first kind. The effect of long-term loading in the creep mode at elevated temperature on the size and shape of grains has been studied. A similar study was previously carried out for recrystallized coarse-grained titanium transferred from the ultrafine-grained state by isothermal annealing. Based on the data obtained in the work and the previous results of the authors, the factors affecting the mechanical stability (durability) of ultrafine-grained metals obtained under severe plastic deformation were analyzed.

Keywords: creep, durability, nanopores, ultrafine-grained metals, titanium, recrystallization.

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1. Introduction

A widespread method for the making of ultrafine-grained (UFG) states of metals and alloys with very high mechanical properties is intensive plastic deformation (IPD) [1–4], which consists in the maximum plastic deformation in quasi-hydrostatic conditions. The defective structure which forms during IPD has a number of peculiarities which hinder the wide practical application of high-strength UFG-materials, particularly during their long-term loading (operation).

One of these peculiarities is related to the fact that this UFG-structure, which conditions the high values of strength characteristics, is unstable [5,6]. UFG-structure evolution during long-term loading in the creep condition (particularly at elevated temperatures) or fatigue condition can cause a size increase and a shape change of crystal grains and, consequently, a degradation of high mechanical properties. An equally important peculiarity, which should be taken into consideration when analyzing the structure of UFG-materials, is the formation of damage such as nano-sized pores, usually localized within the grain boundaries, during their making by the method of Equal channel angular pressing (ECAP) under IPD. Such nanopores forming under IPD were found in the studies of titanium and several other metals [7–14]. Their formation can be related both to the coagulation of a high concentration of non-equilibrium vacancies, forming under a plastic deformation, and to the known dislocation-disclination mechanisms of microdiscontinuity formation. It is also possible that nanopores in case of IPD act as channels for the dissipation of some of the huge energy transferred to the material. From this viewpoint, the formation of nanopores facilitates the making of UFG-metals and alloys.

The previous studies have demonstrated that nanopores forming under IPD exert virtually no impact on strength characteristics of UFG-materials under short-time loading (tensile strength, yield strength, microhardness) [15–17]. However, under long-term loading (creep, fatigue) these „initial“ nanopores (formed during making) can become „sources“ of damage which significantly reduces the durability of high-strength UFG-metals and alloys, i.e., their service life. Nanopores localized within grain boundaries can also affect the intergranular slipping and evolution of grain sizes and shape. It has been found that a decrease of the concentration of „initial“ nanopores (e.g., by their healing under a high hydrostatic pressure or a change of the IPD condition) makes it possible to increase durability under cyclic loading or during a creep test [16,17].

The present paper deals mainly with the results of the studies of grain size evolution for UFG commercially pure titanium (VT1-0 alloy) during a creep test. We analyze the relation of the grain size change to loading duration (durability).

2. Materials and research methods

The main works were performed using commercial titanium VT1-0 with the total impurity content (C, N, Fe, O, H, Al, Si) equal to ~ 0.3 wt.%. Mechanical-thermal treatment of the alloy under study was performed using the optimized mode described in [18] with helical and lengthwise-cross rolling, followed by grade rolling to the diameter of 8 mm with a finishing isothermal annealing at 623 K for three hours to relieve stresses of the first kind. Flat samples with the working part length of 22 mm and cross-sectional area of 0.9 mm^2 were cut out from the blanks, obtained by such treatment, for a creep test.

Creep tests were carried out under tension in the range of 210–460 MPa at 623 K. This temperature was chosen due to the following: as has been shown earlier [19], free annealing of the UFG-titanium, studied in the present paper, at the given temperature for tens of hours does not lead to a significant change of crystalline grain sizes and shape. Depending on chosen stress, time to failure (durability) varied from 10 to 10^5 s.

Structural studies for grain size determination were performed by the electron backscatter diffraction (EBSD) method using scanning electron microscopy. This method was used to obtain crystallographic orientation charts and to plot grain size distribution histograms. The grain distribution was also studied in terms of their aspect correlation in order to obtain more exhaustive information. After mechanical polishing the sample surface was treated by a low-energy argon beam to relieve stresses after polishing. Shooting was performed at the accelerating voltage of 20 kV, the sample surface being oriented at 70 degrees to the electron beam direction. Data was processed using the TSL OIM Analysis software version 6.21 (EDAX Company).

The volume of damage under creep was estimated by the precision method of density measurement with the accuracy of 10^{-4} using a Simadzu AUW 120D analytical balance with a SMK-301 add-on device. This method allows for determining of sample density with the required high accuracy for estimating the sample density change after various impacts. The relative error of density determination $\Delta\rho/\rho$ did not exceed 0.01%. Information about the presence and parameters of nanopores was obtained by the method of small angle X-ray scattering (modernized by the authors) which was used in the previous papers on identification of nanoporosity forming under IPD [7,16,17].

In order to reveal the peculiarities of defective structure evolution during the creep of titanium in the UFG-state, similar studies were conducted for the same titanium transformed from the UFG to the rather coarse-grained state (CG). To do so, the UFG-titanium samples were annealed for an hour at 823 K. The electron microscope studies have shown that if the average grain size of UFG-titanium was $\sim 0.2\,\mu$m, the titanium CG was $\sim 2.3\,\mu$m.

FG titanium after tests at $T = 623$ K, % of the total number

Grain size, nm	Before tests, %	$\tau = 10^2$ s,	$\tau = 8 \cdot 10^4$ s,
< 200	23	24	30
200–400	43	39	31
400–1000	31	34	30
> 1000	3	3	9

3. Experimental data and analysis

The preliminary estimates have shown that the UFG samples fail at 460 MPa, and CG-titanium samples fail at 250 MPa under tension with a constant loading rate. Taking these values into account, creep tests for UFG titanium were performed in the stress range of 270–420 MPa, and 200–240 MPa for CG-titanium. Within the given ranges, durability of UFG- and CG-titanium, depending on stress, varied by almost four orders: from 10 to 10^5 s.

Structural studies were performed for ten UFG-titanium samples whose durability, depending on applied stress, varied from ten seconds to several tens of hours.

The typical results of the study of the grain size on surface area dependence for the UFG-samples with a considerably different durability are shown in Fig. 1, 2.

It is clearly seen from the figures that there are two peaks (maximum) of the grain size with an increase of the time to failure: one peak at $d \leq 0.2\,\mu$m and the other at $d \geq 1\,\mu$m, while the surface area occupied by nanosized grains decreases with an increase of durability and the surface area occupied by of submicron grains increases.

Additional information to confirm the above-mentioned results was obtained when studying the grain distribution (in percent of their total number) for titanium samples with a different durability (see the Table).

We have also obtained grain distribution in terms of the aspect correlation. It was found that the distribution has separate narrow bands when the test time increases, but maximum 85% of the grains have a 1–3 aspect correlation, i.e. the grain shape is redistributed within the specified range.

As already stated, the peculiarities of grain size change during the creep test have been also studied for the titanium samples transformed into the CG-state. Fig. 3 shows the typical grain size on surface area dependence for the CG-titanium samples tested at different stresses at which durability changed from $3 \cdot 10^2$ to $1.2 \cdot 10^4$ s. (It should be noted that the durability virtually did not affect the appearance of the dependence in Fig. 3).

The obtained data shows that CG-titanium grains in the size range of ~ 1–$9\,\mu$m retain rather an equilibrium shape during creep and their average size of ~ 2.3–$3\,\mu$m is virtually unchanged.

The following conclusions can be made based on the analysis of all the obtained data.

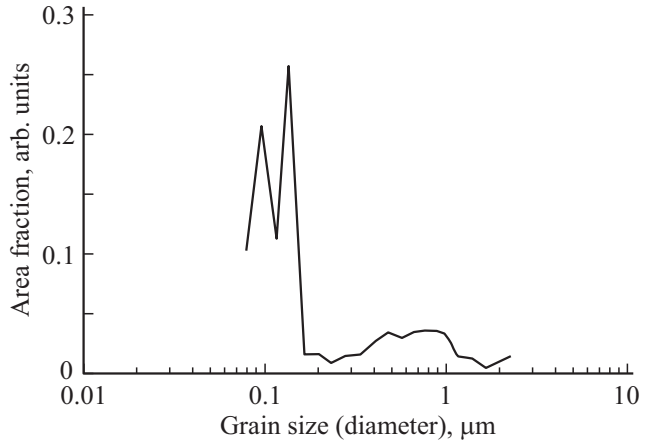

Figure 1. Grain surface area on grain size dependence for UFG VT1-0 after durability tests at $T = 623$ K, $\sigma = 330$ MPa, $\tau = 100$ s.

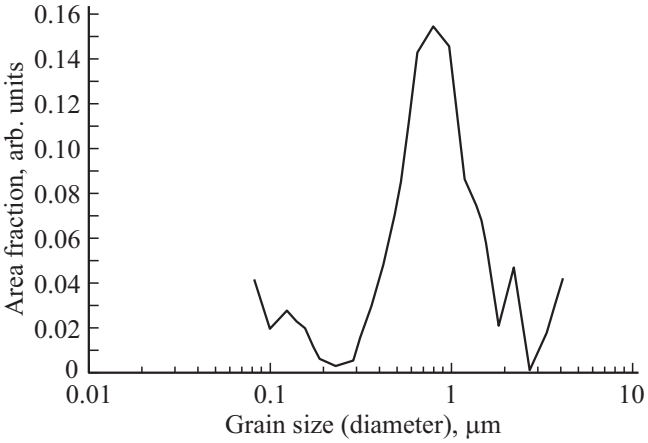

Figure 2. Grain surface area on grain size dependence for UFG VT1-0 after durability tests at $T = 623$ K, $\sigma = 310$ MPa, $\tau = 8 \cdot 10^4$ s.

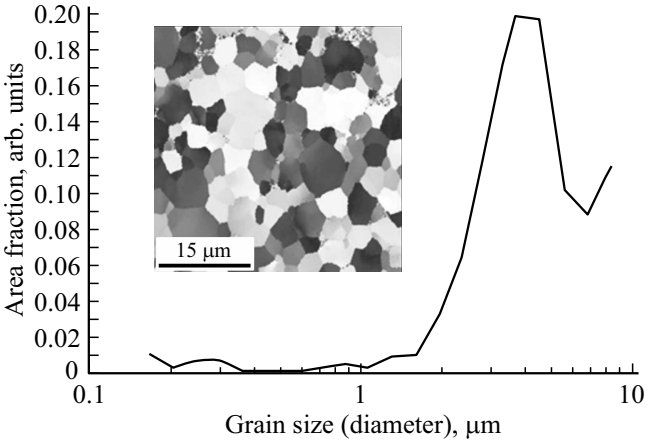

Figure 3. Misorientation Euler angle distribution charts and grain surface area on grain size dependence for CG VT1-0 after durability tests at $T = 623$ K, $\sigma = 230$ MPa, $\tau = 1.2 \cdot 10^4$ s.

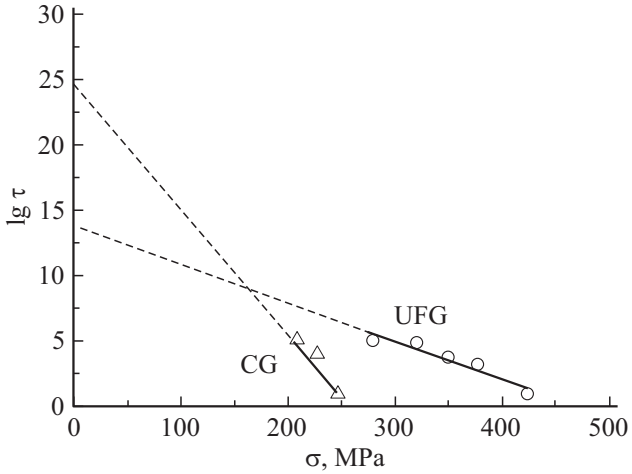

Figure 4. Durability under creep tension for UFG and CG VT1-0 at $T = 623$ K.

Firstly, the fraction of nanosized and sub-microcrystalline grains (100–400 nm) remains predominant ($\geq 90\%$) irrespective of load action time (durability). This means that the studied commercially pure titanium, subjected to mechanical-thermal treatment by a combination of various rolling types and subsequent annealing, retains a high strength (conditioned by the said structure) under long-term loading in the studied interval of external impacts. This conclusion is confirmed by the results of a study of time dependence of UFG-titanium strength obtained by the authors earlier [19].

Secondly, a tendency to the formation of elongated micron-size grains is observed with an increase of load action time (durability). It can be assumed that such elongated grains form due to dynamic recrystallization of some nano- and sub-microcrystalline grains.

The revealed tendency makes it possible to assume that, when stress decreases below 270 MPa and durability increases to 10^6–10^9, the durability of UFG- and CG-titanium will first be identical, and then CG-titanium will be more resistant to sustained loads.

Indeed, it follows from the data of [19] that the $\lg\tau - \sigma$ dependence for UFG-titanium under extrapolation must first intersect with the similar dependence for CG-titanium, and then will be below it. This is due to the fact that the slope of the $\lg\tau - \sigma$ dependence for UFG is two times gentler than for CG-titanium. The latter is evident from Fig. 4.

The obtained result is probably caused by two factors.

Firstly, by dynamic recrystallization during long-term loading of UFG-titanium. The second factor, evidently the more important one, is due to the fact that UFG-titanium has a nanoporosity which formed during IPD.

The small angle X-ray scattering and densimetry data has shown that nanopores sized several tens of nanometers form in the UFG-titanium after the aforesaid mechanical-thermal treatment. The CG-titanium did not have such pores in the „initial“ state (prior to creep tests) (they probably healed

during the making of the CG-structure due to annealing at 823 K). Therefore, it appears that the presence and development of nanopores at stresses below 240 MPa and an increase of creep test time (durability) to 10^6-10^9 s may lead to the following: mechanical stability (durability) of UFG-titanium will be lower than that of its CG-counterpart.

It should be noted that a preliminary estimate of the energy of activation of creep damage development was 80 and 60 kkal/mol, respectively, for CG- and UFG-states [19]. These activation energy values make it possible to assume that the development of damage (including nanoporosity) for the CG-state is limited by bulk diffusion, and for UFG-titanium — by grain-boundary diffusion.

The obtained data is consistent and complements the results of the authors' previous papers [7,16,17,19,20]. As distinct from this, the authors of [20] have established that durability after a creep test at 673 K for UFG-titanium, obtained by another method — IPD (Equal channel angular pressing — ECAP), is considerably smaller than that for its coarse-grained counterpart (before IPD). This result was explained by an increase of the UFG-titanium grain size during creep and an intensive growth of the „initial" nanopores (formed under IPD), which are localized in the new recrystallized grain boundaries.

The authors of [17,21–24] have found that the durability of Al-Sc and Cu-Zr alloys[1], tested in the creep mode at 673 K, after 10–12 runs of ECAP becomes $(5-10^2)$ times smaller than that for their „initial" coarse-grained counterparts (before ECAP). This result was associated with a higher concentration of vacancies (and, consequently, nanopores) which formed near the particles of the second phase [19] in the course of ECAP [17].

Thus, the nanoporosity forming during IPD is an important factor that can reduce the mechanical stability of high-strength UFG-materials, made under IPD, during their long-term loading.

A similar impact of nanopores, forming under IPD by the method of equal channel angular extrusion, was observed by the authors under cyclic loading of UFG-titanium [21].

4. Conclusions

1. It has been established that the UFG-structure, which formed in titanium during mechanical-thermal treatment using a combination of helical and lengthwise-cross, as well as grade rolling with finishing isothermal annealing, remains stable under creep at 623 K and stresses within 270–420 MPa. This ensures the mechanical stability of high-strength titanium under its long-term loading.

2. The analysis of the obtained data also indicates that the observed decrease of mechanical stability (durability) of metals with the UFG-structure, which can occur under certain long-term loading conditions, is due to dynamic recrystallization and, in particular, the development of nanopores which originate in the course of UFG-structure formation.

3. It appears that the development of scientifically grounded ways and methods for reduction of levels of nanoporosity, forming under IPD, will make it possible to increase mechanical stability of the UFG-structure and the scope of practical application of high-strength UFG-metals and alloys.

[1] The said alloys contained nanoparticles of Al_3Sc and Cu_2Zr_6 [17,20].

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Conflict of interest

The authors declare that they have no conflict of interest.

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