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Evolution of nanoporosity, Young's modulus and microplastic properties of nanostructured titanium VT1-0 under creep

© M.V. Narykova, B.K. Kardashev, V.I. Betekhtin, A.G. Kadomtsev

Ioffe Institute, St. Petersburg, Russia
E-mail: Maria.Narykova@mail.ioffe.ru

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This study investigates the evolution of nanoporosity, Young's modulus, and microplastic properties of nanostructured (NS) titanium VT1-0 under creep. The NS titanium samples were fabricated using a combination of transverse screw rolling and longitudinal bar rolling with additional nitrogen cooling at the final stage of deformation. Variations in elastic and microplastic properties of NS titanium VT1-0 were examined for the first time in testing for tensile creep lifetime. The dependence of density reduction (nanopore formation) on the degree of deformation was determined.

Keywords: ultrafine-grained titanium, nanostructured titanium, VT1-0, durability, Young's modulus, creep, nanopores.

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Ultrafine-grained (UFG) metallic materials obtained by techniques of intense plastic deformation are distinguished by strength characteristics (tensile strength, creep strength, microhardness) higher than those of coarse-grained analogues. These improvements are provided by various strengthening mechanisms, including strengthening via grain boundaries, which makes UFG materials promising for using in constructional elements. However, their practical applications require resistance to long-term loads. Modern studies reveal significant contradictions in data on the UFG metal creep: grain refinement may either increase or decrease the material durability depending on the structure's peculiar features and loading conditions [1–3]. These contradictive results highlight the need to study the relationship between the UFG material microstructure, deformation parameters (temperature, stress), and internal characteristics (alloy purity, defect density). Though some studies on creep in pure metals and alloys with the UFG structure were performed as far back as in the early 2000s [4,5], key issues related to the structure evolution and damage accumulation under long-term loading still remain unresolved.

One of the structural factors negatively affecting the behavior of UFG materials under long-term loading is nanoporosity [6,7]. The nanopores' volume fraction depends on the method of fabricating the UFG structure (the number of passes in equal-channel angular pressing or applying counterpressure), post-deformation treatment (low-temperature annealing, applying high hydrostatic pressure), the presence of inclusions, etc. These defects not only serve as stress concentrators, but also reduce elastic characteristics, e. g. Young's modulus [8,9].

To ensure correct analysis of experimental data on creep, the following conditions are necessary: structural stability of the material, low temperatures, and „moderate" stresses. Therefore, we have chosen for research commercial titanium VT1-0 in which impurities have a stabilizing influence on grain boundaries due to the „braking" effect [10].

This paper presents the results of studying the kinetics of microdiscontinuities accumulation and evolution of elastic and microplastic properties of NS titanium VT1-0 during the tensile creep test at the temperature ensuring its structural stability.

Titanium alloy VT1-0 was studied in the NS state obtained by the method combining transverse screw rolling with longitudinal bar rolling [11]; at the final stage, deformation was accomplished in liquid nitrogen. The VT1-0 rolling jointly with additional cooling provides significant refinement of grains. The conducted structural studies showed that the average grain size in NS titanium is 117 nm. The fraction of NS titanium grains 100 nm and less in size exceeds 30 %. The same fraction is composed of grains up to 150 nm, which, in general, allows defining this state as nanostructured [12] in which the decisive role in formation of the material properties is played by nanoscale components.

Samples of NS titanium were tested for tensile strength in the creep mode at the temperature of 350 °C. Earlier it was shown that testing at this temperature do not induce significant growth of grains, and the NS state is preserved [13]. In this work, four samples were selected for research: prior to testing (No 1), after loading at $\sigma = 352$ MPa and for time t of 13 800 (No 2) and 20 000 s (No 3) not led to destruction, and destroyed sample (No 4) tested at $\sigma = 590$ MPa for $t = 300$ s. Each sample had the shape of I-type flat blade with the working part 22 mm long and 0.9 mm thick. Numbers of samples under study, test conditions, deformation ε, density ρ and density reduction (loosening) $\Delta\rho/\rho$ are given in the table.

Using the acoustic method employing a composite piezoelectric vibrator, the following elastic and microplastic prop-

Test conditions (stress σ and time t), deformation ε, density ρ and density reduction $\Delta\rho/\rho$ for the samples of NS titanium VT1-0

Sample No	σ, MPa	t, s	ε, %	ρ, g/cm^3	$\Delta\rho/\rho$, %
1	0	0	0	4.4896	0
2	352	13800	3.4	4.4706	0.42
3	352	20000	3.5	4.4598	0.66
4	590	300	6.7	4.4200	1.56

erties were determined: Young's modulus E, decrement δ, and microplastic flow stress σ_d. Detailed description of this technique is given in [14,15]. On the completion of the durability test, the wide part of the sample (blade) was removed for further studies. The samples for acoustic studies were rods approximately 22 mm long having rectangular cross section of 0.9×3 mm. The tests were conducted at the frequency of about 100 kHz in a wide range of oscillatory deformation amplitude ε which included the linear (amplitude-independent) and nonlinear (microplastic) regions. The elastic modulus was calculated as

$$E = 4\rho l^2 f^2, \quad (1)$$

where l is the sample length, ρ is the sample density, f is the oscillation frequency. Relative error in determining the sample natural frequency by the double-vibrator method was $\sim 10^{-3}$, relative error in determining the elastic modulus was $\sim 4 \cdot 10^{-3}$.

Acoustic measurements in a wide range of amplitudes allow evaluation of the material mechanical (microplastic) properties in the stress–strain coordinates typical of conventional mechanical tests. In this case, oscillatory stress amplitudes $\sigma = E\varepsilon$ (Hooke's law) are plotted on the ordinate axis, while nonlinear inelastic deformation $\varepsilon_d = \varepsilon(\Delta E/E)_h$ is plotted on the abscissa axis. Here $\Delta E = E(\varepsilon) - E_i$ is the amplitude-dependent Young's modulus defect, E_i is the Young's modulus measured at low ε in the amplitude-independent region.

Fig. 1 presents amplitude dependences of Young's modulus $E(\varepsilon)$ and decrement $\delta(\varepsilon)$; Fig. 2 shows the diagrams of microplastic deformation of the NS titanium samples.

Fig. 1 shows that, after the NS sample No 2 is deformed by 3.4 %, the Young's modulus decreases relative to the initial (prior to testing) state: from 104 to 102 GPa. An increase in the load duration (at the same stress of 352 MPa) leads to an increase in modulus E to 104 GPa. The highest value of $E = 110$ GPa corresponds to the destroyed state (sample No 4). The modulus decrease in sample No 2 may have at least two sources. The first one is a decrease in the sample density according to (1) associated with development of the material nanopores. Density variation $\Delta\rho/\rho$ determined by the densitometric method was 0.42 % (see the Table). The second source is an increase in the dislocation density which typically occurs in the case of plastic deformation of metallic materials. For the sample

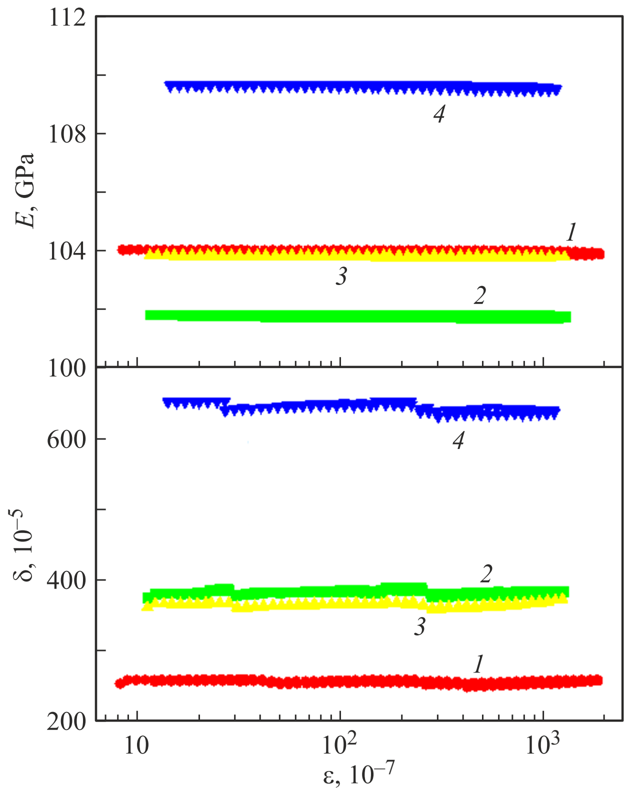

Figure 1. Amplitude dependences of Young's modulus E and decrement δ in NS titanium VT1-0 (the curve numbers correspond to the sample numbers). The measurements were performed at room temperature.

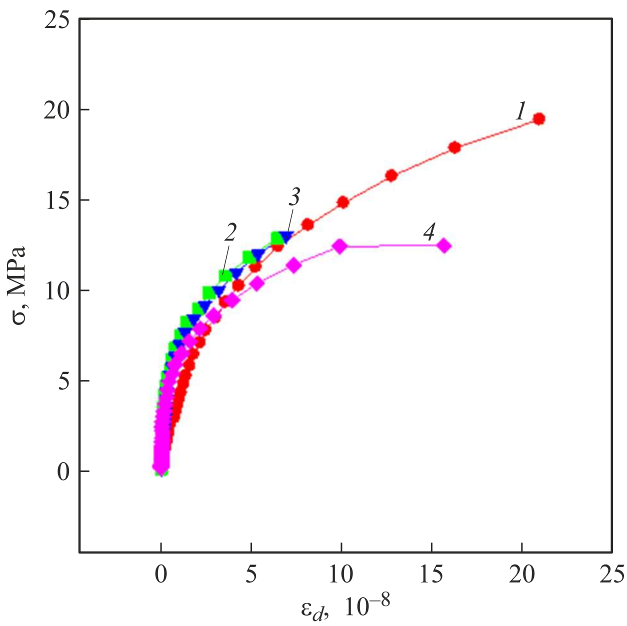

Figure 2. Microplastic deformation diagrams of NS titanium VT1-0 samples (the curve numbers correspond to the sample numbers).

led to destruction (No 4), density reduction by another 0.9 % is observed; at the same time, there takes place an increase in the Young's modulus by almost 6 % (relative to sample No 3).

Fig. 3 presents for clarity the Young's modulus dependence on density reduction (nanopore formation) in the NS titanium sample ($E_0 = E_i$ is the Young's modulus of sample No 1 prior to testing). Evidently, first the modulus decrease is observed; its growth is observed as the deformation (and density reduction) increases. Similar dependences, namely the modulus decrease at the initial stage of deformation and increase with increasing deformation degree, were previously revealed for microcrystalline alloys based on aluminum (Al−0.2 % Sc) [16] and titanium (Ti−26Nb−7Mo−12Zr) [17]. For instance, an increase in the number of equal-channel angular pressing passes for the Al−0.2 % Sc alloy and in the deformation degree during rolling for the titanium alloy resulted in an increase in modulus E.

Non-monotonic behavior of the modulus for NS titanium (Fig. 3) may be explained by that one of the factors that can lead to the modulus increase is the growth of internal stresses [18]. Such stresses may arise in metallic materials under plastic deformation both in mechanical testing and during formation of the UFG structure by intense plastic deformation. The obtained result is consistent with studies of other authors where deformation (cyclic or monotonic) caused high internal stresses in UFG materials [19,20]. In those studies, large inelastic reverse deformations were observed in UFG samples after unloading, while unloading of coarse-grained-structure samples was almost fully elastic. The modulus increase shown in Fig. 3 is most probably caused by an increase in internal stresses. The influence of crystallographic texture [21] which could get formed in metallic materials during the tensile creep test is also possible [22].

Let us consider variations in logarithmic decrement δ (Fig. 1) under deformation of the NS titanium samples. Its successive increase during the tensile creep test may be explained by two reasons: first, it is known that, under plastic deformation of metals, the density of dislocations increases, which should make increasing the decrement; second, when a certain region (especially near the fracture zone) gets deformed, the structure „refining"(decrease in the average grain size) is possible [23]. Along with this, there takes place an increase in the total length of grain boundaries where the ultrasonic energy is dissipated, which should also induce an increase in decrement. The most significant increase in the amplitude-independent decrement is characteristic of the destroyed sample No 4 whose deformation is maximal. Note here that, when ε increases (Fig. 1), no amplitude dependence of the decrement is observed on all the four samples, contrary to the clearly manifested dependence $E(\varepsilon)$, due to which nonlinear inelastic deformation occurs (Fig. 2). This most likely means that dislocation attenuation in the tested samples is significantly weaker than the grain boundary attenuation: the modulus decrease in crystals and coarse-crystalline materials is typically accompanied by a considerable increase in decrement.

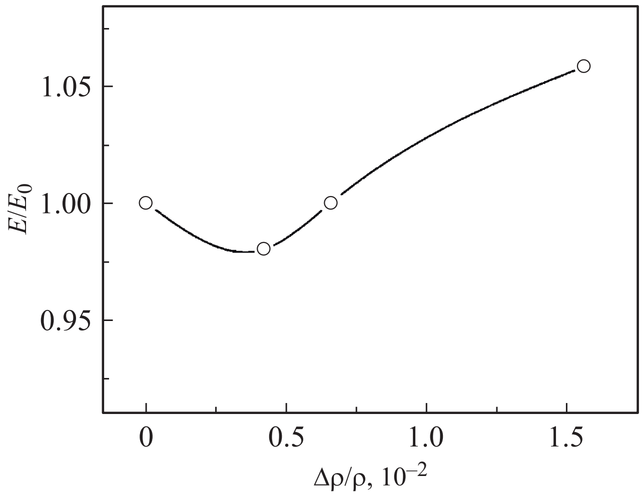

Figure 3. The Young's modulus as a function of density reduction (nanopore formation) for NS titanium in tensile creep testing.

The NS titanium microplastic deformation diagrams obtained from dependences $E(\varepsilon)$ are shown in Fig. 2. Evidently, behaviors of the $\sigma(\varepsilon_d)$ curves for samples No 2 and 3 are identical. As compared with sample No 1, those samples exhibit slightly higher σ values up to microdeformation $\varepsilon_d = 5 \cdot 10^{-8}$. Note in addition that sample No 4 is somewhat „stronger" at very low microdeformation ε_d (up to $\sim 2 \cdot 10^{-8}$)) than the initial sample. However, when $\varepsilon_d = 7 \cdot 10^{-8}$, the σ value for sample No 4 is considerably lower than that for other samples (about 11 MPa), i. e., the sample No 4 deformation (at the micro level) by the same value $\varepsilon_d \approx 7 \cdot 10^{-8}$ needs application of a minimal stress. All this may be associated with nonuniformity of the structure formed along the sample length in the process of creep testing.

Thus, the paper presents for the first time the results of studying elastic and microplastic properties of NS titanium VT1-0 obtained by complex rolling with cooling in nitrogen at the final stage of the sample fabrication. Evolution of the elastic modulus and logarithmic decrement after different stages of deformation under creep tension has been studied. It is shown that the main factor in the elastic modulus increase with decreasing densitometric density in samples subjected to tensile creep tests is emergence of high internal stresses; the level of decrement is therewith determined mainly by ultrasonic energy dissipation at the grain boundaries.

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Conflict of interest

The authors declare that they have no conflict of interests.

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