

MODIFICATION OF STRUCTURE AND PROPERTIES OF NANOSTRUCTURED VT1-0 TITANIUM ALLOY UNDER ULTRASONIC INFLUENCE

E.S. Savchuk, V.I. Sokolenko, E.V. Karaseva, A.V. Mats, V.A. Frolov
National Science Center “Kharkov Institute of Physics and Technology”, Kharkiv, Ukraine
E-mail: vsokol@kipt.kharkov.ua

The effect of ultrasonic influence on the structure evolution and creep of nanostructured titanium alloy of the grade VT1-0 obtained by the method of severe plastic deformation has been studied. It is shown that ultrasonic influence with a frequency of $f = 20$ kHz and an amplitude of 65 MPa leads to relaxation of internal stresses in the nanostructured VT1-0 alloy due to the formation of an equilibrium structure of boundaries without noticeable grain growth. In this case, the mechanical properties of the alloy change as follows: the tensile strength increases, while maintaining the yield strength and ductility. The reason for the observed effect may be the low stacking fault energy of the VT1-0 alloy, which complicates the course of relaxation processes, as well as the rearrangement of the structure during creep with the formation of new deformation boundaries, which are more resistant to tensile loads.

PACS: 62.20.Hg, 61.72.Ff, 61.10.-i

INTRODUCTION

In recent years, much attention has been paid to the development of a new approach to improving the properties of metals and alloys based on their nanostructuring by severe plastic deformation (SPD) [1]. Commercially pure titanium is widely used in various industries, including as a structural material for nuclear power engineering due to its high strength, high radiation resistance, and corrosion resistance in active media [2]. The formation of the nanostructured state using SPD makes it possible significantly increase the ultimate strength of titanium [3, 4], while maintaining, and even increasing, its positive properties, for example, biocompatibility [5].

Now, a large amount of research has been carried out on the structure and mechanical properties of titanium obtained by SPD, however, the problem of increasing the plasticity and thermomechanical stability of nanostructures titanium while maintaining high strength remains the actual.

It is known that in nanostructures materials obtained by SPD, grain boundaries that are in a nonequilibrium state and have an increased free volume create a high level of internal stresses and, as a result, nanomaterials have low plasticity and thermal stability [6–9].

Low-intensity ultrasonic influence (USI) is an effective way to decrease the level of inhomogeneities of internal stress fields, reduce the metastability of the structural-phase state, and, consequently, improve the physical and mechanical properties of various heterogeneous materials [10].

To relax the stresses of the nonequilibrium structure of nanomaterials obtained by SPD, a promising method for improving the properties can be USI.

An ultrasonic wave, passing through a material, interacts with defects of various types and causes changes in the structure, which depend on the parameters of USI: intensity, temperature, duration, etc. By varying the parameters of USI, it is possible to obtain a structure in the material that has the necessary properties.

Previously, on the example of materials with an HCP lattice (Zr, Zr1Nb alloy), we have shown [11, 12] that as a result of low-intensity ultrasonic action on a nanostructure state of deformation origin, the level decreases and the spectrum of internal stresses in the bulk of the material is equalized, while maintaining the size factor and increasing the homogeneity of the structure. Ultrasonic treatment makes it possible to maintain a higher strength of the material, while increasing plasticity, material homogeneity and greater structural stability during plastic flow under creep conditions.

When choosing structural materials, much attention is paid to the study of creep characteristics, since most industrial structures operate under static loading conditions.

The purpose of this work is to study the patterns of creep and evolution of the structure of commercially pure titanium alloy of the grade VT1-0, obtained by the method of severe plastic deformation by rolling, and the effect of ultrasonic influence on the characteristics of the material.

MATERIAL AND PROCESSING METHOD

The commercial pure titanium alloy of the grade VT1-0 of industrial production was investigated, the amount of impurities of which does not exceed 0.3%. In order to study the effect of ultrasonic treatment on the structure and properties of the VT1-0, the following processing modes were studied:

1. MT-1 – rolling deformation at the room temperature, strain (ϵ) was $\epsilon = 3.0$;
2. MT-2 – MT-1 + USI at 300 K.

The part of strained samples was subjected to low-intensity USI ($f = 20$ kHz) at $T = 20$ °C by the method described in [13]. The amplitude of the ultrasonic shear stresses was 65 MPa, the duration was 30 min.

For studying the material defect structures, the electrical resistivity (R) was measured by 4-th points scheme at $T = 20$ °C after each treatment. A measuring error did not exceed $\pm 0.05\%$.

The structure evolution monitoring was carried out by electron microscopy. Creep tests were carried out in the step loading regime, the elongation measurement accuracy was $5 \cdot 10^{-5}$ cm.

RESULTS AND DISCUSSION

Fig. 1 shows the dependences of the creep rate at $T = 20$ and 350 °C on the true stress of the VT1-0 alloy samples in various structural states.

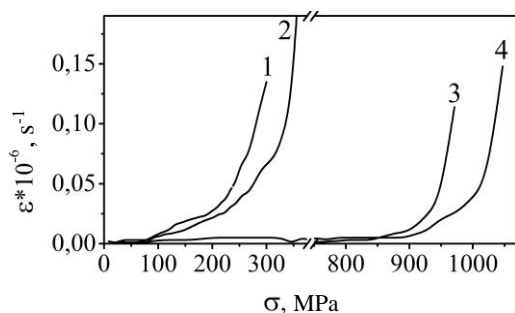


Fig. 1. Creep rates as a function of the applied stress at $T = 20$ (3, 4) and 350 °C (1, 2) of VT1-0 alloy specimens after processing modes: MT-1 – 1, 3; MT-2 – 2, 4

It can be seen, that after ultrasonic treatment of the nanostructured VT1-0 alloy (MT-2), the tensile strength at the test temperature $T = 20$ °C increased by $\sim 8\%$ compared to MO-1, while maintaining the yield strength and plasticity.

At the test temperature $T = 350$ °C, the tensile strength increases by $\sim 20\%$, while the yield strength and plasticity do not change.

It was shown in [8, 9] that, as a result of low-intensity ultrasonic action, the material is softening because of the relaxation of internal stresses in the bulk of the material due to a number of factors. High-frequency alternating action generates a large number of vacancies, which stimulates non-conservative dislocation glide. Moreover, the dissipation of vibration energy occurs mainly at the interfaces, which can lead to the formation of an equilibrium state of the boundaries, as well as to a decrease in the level of local stresses. In this case, the equilibrium grain boundaries are more resistant to subsequent mechanical and thermal effects under creep conditions.

In contrast to the effect of ultrasonic action on the properties of nanostructure Zr and Zr1Nb samples [11, 12], nanostructured titanium alloy is characterized by an increase in the tensile strength, while maintaining the yield strength and plasticity.

After deformation by rolling and USI, the values of the relative electrical resistivity ($R_{300\text{ K}}/R_{77\text{ K}}$) of samples of the VT1-0 alloy were calculated. Determining the value of the residual electrical resistivity makes it possible to control the change in the defective state of the material. Studies have shown that USI leads to an increase in residual electrical resistivity by $\sim 10\%$, which indicates a decrease in defectiveness and a decrease in the level of internal stresses in samples of the VT1-0 nanostructure alloy.

Structural studies have shown that after rolling at $T = 20$ °C to deformation $\varepsilon = 3.0$, the structure with a

grain size of 55 to 140 nm was formed. Grains with a size of ~ 100 nm dominate; and as the fact the nanostructure was formed (Fig. 2,a). The concentration of the boundary phase reaches a significant value: the width of traces of the boundaries on electron microscope images can be ~ 25 nm. The density of dislocations in the body of grains is very low and is at the limit of accuracy of the device determination.

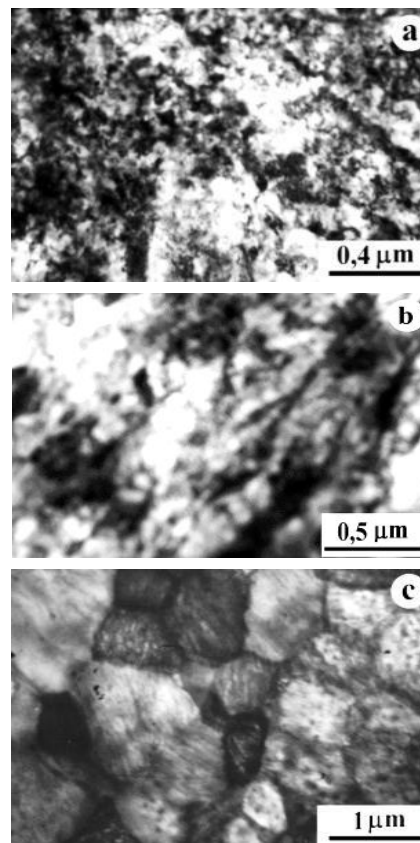


Fig. 2. TEM images of VT1-0 alloy after processing modes and creep: a – MT-1; b – MT-1+creep at 20 °C ($\sigma \sim 0.9\sigma_B$); c – MT-1+creep at 350 °C ($\sigma \sim 0.9\sigma_B$)

The imposition of a tensile load in the creep regime at $T = 20$ °C on the deformation nanostructure of the VT1-0 alloy leads to the destruction of most of the boundaries and the formation of a cellular and fragmented structure with a fragment size of $\sim 0.05 \dots 0.15$ μm . New boundaries are formed by dislocations that appeared during the scattering of unstable deformation boundaries (see Fig. 2,b).

In the process of creep at $T = 350$ °C of VT1-0 nanostructures samples, the recrystallized structure with an average grain size of ~ 1 μm in place of the nanostructure was formed (see Fig. 2,c).

The main result of ultrasonic influence is a significant reduction in the overall level of internal stresses, while maintaining the size factor and increasing the homogeneity of the structure. This is evidenced by the absence of bending contours at the boundaries in bright-field images and the absence of strands between reflections in microdiffraction patterns (see Fig. 3,a).

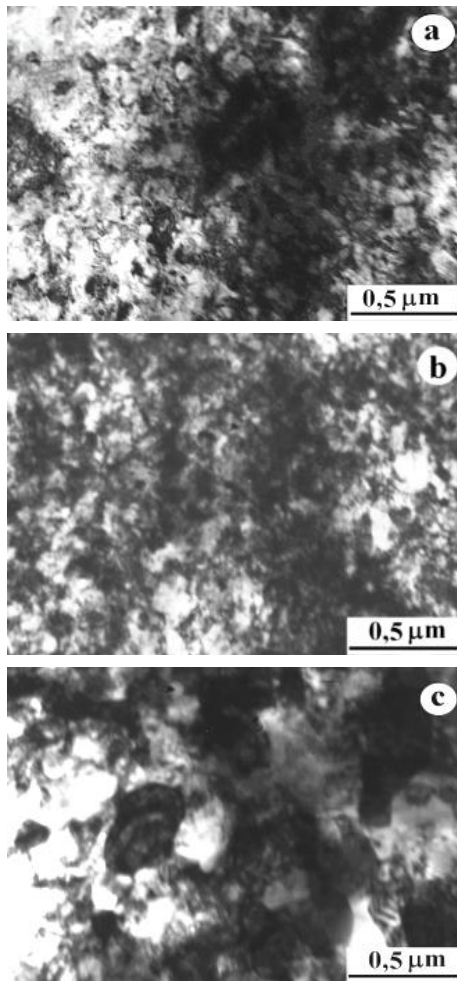


Fig. 3. TEM images of VT1-0 alloy after processing modes and creep: a – MT-2; b – MT-2+creep at 20 °C ($\sigma \sim 0.9\sigma_B$); c – MT-2+creep at 350 °C ($\sigma \sim 0.9\sigma_B$)

There are realized translational modes in the body of subgrains in the formation of cells through the process of creep at $T = 20\text{ °C}$ of a relaxed structure. The deformation boundaries turn out to be more resistant to tensile loads (see Fig. 3,b).

The deformation is localized at the boundaries of nanograins in the form of clusters of dislocations. Microtwins (twin stacking faults) are formed in the body of nanograins, which are typical for cold free deformation, since their formation requires two times less energy than conventional stacking faults. They are additional barriers for slipping dislocations.

Increasing the test temperature in the creep mode to $T = 350\text{ °C}$ leads to unfinished primary recrystallization. The size of new grains is about $\sim 0.2 \dots 1\ \mu\text{m}$. Polygonal boundaries are observed in the body of some grains (see Fig. 3,c). Deformation is also carried out by developing translational modes. However, an additional reserve of uniform plasticity is achieved due to thermally activated structural rearrangement with the formation and displacement of high-angle and low-angle polygonal boundaries. Recrystallization nuclei are formed at the triple junctions of deformation boundaries – the places of maximum distortion.

Thus, structural studies indicate a decrease in the overall level of internal stresses, which is associated

with the development of return processes and the restructuring of the structure.

To determine the mechanisms of deformation during the creep of the VT1-0 nanostructured alloy, the activation volume (V) was measured and calculated by using the formulas of the theory of thermally activated plastic deformation [17]. The obtained value for samples after both treatments (MT-1 and MT-2) is about $V \sim 1.5 \cdot 10^{-21}\ \text{cm}^3$ at the stress near the yield point. This allows us to conclude that the process of plastic deformation is controlled by impurities and point defects. As the applied stress increases, the activation volume decreases at all test temperatures.

According to the theory, this means that deformation defects also control the plastic flow of the material, and therefore, the mechanism of strain hardening works [17], or the localization of plastic deformation increases during creep [18]. Since in our case the tensile strength increases, while maintaining the yield strength and plasticity, it can be assumed that both of the above processes can be responsible for the development of deformation during creep.

According to structural studies, in the process of creep of VT1-0, simultaneously with the process of the dislocations slipping, reverse processes develop at the grain boundaries and the structure is rearranged. We have shown [14–16] that the plastic flow of a nanostructures material obtained by SPD is due to the combined action of several mechanisms: intragranular slip of dislocations, cross slip, climbing and annihilation of dislocations at grain boundaries, as well as diffusion creep and grain boundary slip. The contribution of each of these mechanisms to the deformation of the material depends on the test temperature, the level of internal stresses, and the state of the grain boundaries.

Based on the stress dependence of the creep rate (see Fig. 1), it can be concluded that the contribution to the deformation of the slip mechanisms inside the grains and near the boundaries in VT1-0 specimens after USI is greater than in the deformed state. Fig. 1 shows that the length of the stage of a smooth increase in the creep rate with increasing stress, which corresponds to the development of slip and climbing of the dislocations inside the grains and at boundaries, is longer for specimens after USI than for specimens not subjected to ultrasonic treatment.

Structural studies also show that a decrease in the level of internal stresses due to USI leads to a slowdown in the processes of recrystallization and the development of polygonization. It is known that at the lower level of internal stresses, a recovery process occurs, leading to the formation of a polygonal structure. In this case, the energy of the system decreases, and the polygonization process can compete with the recrystallization process, delaying it and increasing the stress or temperature level to start it [19]. Thus, at stresses near the ultimate strength ($\sigma \sim 0.9\sigma_B$), the recrystallization is unfinished, i.e. the completely ordered structure has not yet formed and this may lead to an increase in the tensile strength.

It is interesting that, according to the data on the change in the residual electrical resistance and

structural studies, there is a decrease in the overall level of internal stresses in the VT1-0 nanostructures alloy after USI, but the yield strength under creep conditions, i.e. indicator of the magnitude of internal stresses at the macrolevel, does not change at both test temperatures.

Possibly this means that when we analyzing the results, it is necessary to take into account not only the characteristics of obstacles, changes and rearrangements of the structure, but also the parameters of the dislocation itself, such as its splitting, which is controlled by the stacking fault energy.

It is known [20] that the stacking fault is one of the most common defects in the HCP crystal lattice, and its presence can significantly effect on the processes of plastic deformation, hardening, fracture, recrystallization, the nature of phase transformations, etc. in metallic materials. The stacking fault energy, associated with the features of the electronic structure of the material, determines the magnitude of the splitting of a dislocation, and the magnitude of the splitting effects all processes of dislocation rearrangements.

The splitting of dislocations largely controls the mobility of dislocations, their capability to overcome various type of obstacles, the character of direct interaction with other defects, which ultimately determines the level of structurally sensitive properties, primarily mechanical ones [21].

It is known [21] that unsplit and split dislocations interact with point defects in different ways, as a result of which, a decrease in the stacking fault energy can lead to difficulty in moving point defects to the boundaries of nanograins and slow down the processes of dislocation climbing associated with vacancy flows, which will complicate the process of restoring the structure of boundaries.

Taking this into account, it should be expected that the influence of the excess density of point defects on mechanical properties will be different in metals with different stacking fault energies from softening to hardening in crystals with a large number of split dislocations, i.e. characterized by low stacking fault energy.

It is known [21], that the stacking fault energy of titanium is ~ 20 mJ/m², which is an order of magnitude less than that of zirconium ~ 220 mJ/m², and therefore the return processes and recovery of the boundary structure in titanium and zirconium can proceed differently.

CONCLUSIONS

The effect of ultrasonic influence on the evolution of the structure and creep of commercially pure nanostructured titanium alloy of the grade VT1-0 obtained by SPD by rolling has been studied.

It is shown that ultrasonic influence with a frequency of $f = 20$ kHz and an amplitude of 65 MPa leads to the relaxation of internal stresses of the nanostructured VT1-0 alloy due to the formation of an equilibrium boundary structure without noticeable grain growth.

Studies of creep at $T = 20$ and 350 °C of nanostructured VT1-0 alloy showed that USI leads to an increase in tensile strength, while maintaining the yield strength and plasticity. This may be due to the rearrangement of the structure and the formation of new deformation boundaries (cell walls, polygonal boundaries, microtwins), which are more resistant to tensile loads.

In addition, the low stacking fault energy of the VT1-0 alloy can make it difficult for point defects to move to the boundaries of nanograins and slow down the recovery processes associated with vacancy flows, which complicates the reconstruction of the structure of boundaries and leads to a slowdown in recrystallization processes and the development of polygonization.

REFERENCES

1. V.N. Voyevodin, I.M. Neklyudov. *Evolution of structure phase state and radiation resistance of structural materials*. Kiev: "Naukova dumka", 2006, p. 376.
2. I.V. Gorynin, V.V. Rybin, S.S. Ushkov, O.A. Kozhevnikov. Titanium alloys as promising reactor materials // *Radiation materials science and structural strength of reactor materials*. 2002, p. 37-45.
3. S.V. Zherebtsov, G.S. Dyakonov, A.A. Salem, V.I. Sokolenko, G.A. Salishchev, S.L. Semiatin. Formation of nanostructures in commercial-purity titanium via cryorolling // *Acta Materialia*. 2013, v. 61, p. 1167-1178.
4. V.V. Stolyarov, Y.T. Zhu, I.V. Alexandrov, et al. Influence of ECAP routes on the microstructure and properties of pure Ti // *Mater. Sci. Eng.* 2001, v. A 299, p. 59-67.
5. K.V. Kutniy, O.I. Volchok, I.F. Kislyak, M.A. Tikhonovsky, G.E. Storozhilov. Obtaining of pure nanostructured titanium for medicine by severe deformation at cryogenic temperatures // *Mat.-wiss. u. Werkstofftech.* 2011, v. 42, N 2, p. 114-117.
6. R.Z. Valiev, I.V. Aleksandrov. *Nanostructured materials got an intensive plastic deformation*. M.: "Logos", 2000, 271 p.
7. V.V. Rybin. *Large plastic deformation and fracture of metals*. M.: "Metallurgy Publ.", 1986, 224 p.
8. E.V. Karaseva, A.V. Matz, V.I. Sokolenko, V.A. Frolov. Effect of structural instability on creep of zirconium subjected to severe plastic deformation // *Problems of Atomic Science and Technology. Series "Vacuum, Pure Materials, Superconductors"*. 2014, N 1(89), p. 106-109.
9. E.V. Karaseva. Influence of structural instability at the creep characteristics of constructing materials // *Problems of Atomic Science and Technology. Series "Physics of Radiation Effect and Radiation Materials Science"*. 2015, N 5(99), p. 130-133.
10. I.M. Neklyudov, V.I. Sokolenko, V.M. Netysov. Development of Methods of the Directional Change and Properties of Construction Materials by Stimulation of Relaxation Processes in the NSC "KIPT": The Review Dedicated to the 80th Anniversary of the National Scientific Centre

“Kharkov Physical-Technical Institute” // *Progress in Physics of Metals*. 2008, v. 9, N 2, p. 171-193.

11. V.I. Sokolenko, V.M. Gorbatenko, E.V. Karaseva, A.V. Mats, E.S. Savchuk, V.A. Frolov. Ultrasound influence on creep nanostructured Zr // *Problems of Atomic Science and Technology. Series “Vacuum, Pure Materials, Superconductors”*. 2016, v. 101, N 1, p. 41-44.

12. E.V. Karaseva, V.I. Sokolenko, A.V. Mats, E.S. Savchuk, V.A. Frolov. Effect of ultrasonic impact treatment on the creep characteristics and evolution of the Zr1Nb alloy nanostructure // *Functional Materials*. 2018, v. 25(3), p. 458-462.

13. I.A. Gindin, G.N. Malik, I.M. Neklyudov. Ultrasonic influence on paramets of monocystals Cu hardening // *News of High Schools, Physic*. 1972, N 2, p. 51-56.

14. I.F. Borisova, I.N. Butenko, E.V. Karaseva, D.G. Malyhin, A.V. Mats, V.I. Sokolenko, V.A. Frolov. Texture formation peculiarities of zirconium in condition of large plastic deformation and its influence on characteristics of creep in the temperatures range 300...700 K // *Problems of Atomic Science and Technology. Series “Physics of Radiation Effect and Radiation Materials Science”*. 2009, N 2(93), p. 100-105.

15. E.V. Karaseva, D.G. Malykhin, A.V. Mats, V.I. Sokolenko. Creep of Zr1Nb alloy in different structural state in temperature range 300...700 K // *Problems of Atomic Science and Technology. Series “Physics of Radiation Effect and Radiation Materials Science”*. 2011, N 4(74), p. 45-48.

16. E.S. Savchuk, V.I. Sokolenko, E.V. Karaseva, A.V. Mats, V.A. Mats, V.A. Frolov. Creep of VT1-0 alloy in different structural states // *Problems of Atomic Science and Technology. Series “Vacuum, Pure Materials, Superconductors”*. 2021, N 1(101), p. 41-44.

17. A. Evans, R. Rawlings. Thermally activated processes in crystals // *Phys. Stat. Sol.* 1969, v. 34, N 9.

18. T.Yu. Yakovleva. *Localization of plastic deformation and fatigue of metals*. Kiev: “Naukova dumka”, 2003, p. 236.

19. S.S. Gorelik *Recrystallization of metals and alloys*. M.: “Metallurgy”, 1978, 567 p.

20. Ya.D. Vishnyakov. *Packing defects in the crystal structure*. M.: “Metallurgy”, 1970, 216 p.

21. L.I. Yakovenkova, L.E. Karkina, G.L. Podchinekova. The structure of the core of a split dislocation and the energy of interaction with a vacancy in fcc crystals with different stacking fault energies // *Phys. Met. Metallography*. 1985, v. 5, p. 899-894.

Article received 21.07.2022

МОДИФІКАЦІЯ СТРУКТУРИ І ВЛАСТИВОСТЕЙ НАНОСТРУКТУРНОГО СПЛАВУ VT1-0 ЗА ДОПОМОГОЮ УЛЬТРАЗВУКУ

Є.С. Савчук, В.І. Соколенко, Є.В. Карасьова, О.В. Мац, В.О. Фролов

Досліджено вплив ультразвукової дії на еволюцію структури та повзучість технічно чистого наноструктурного титанового сплаву VT1-0, який отриманий методом інтенсивної пластичної деформації. Показано, що ультразвукова дія частотою $f = 20$ кГц та амплітудою 65 МПа призводить до релаксації внутрішніх напруг наноструктурного сплаву VT1-0 внаслідок формування рівноважної структури границь без помітного зростання зерен. При цьому механічні властивості сплаву змінюються таким чином: збільшується межа міцності при збереженні межі плинності та пластичності. Причинами ефекту, що спостерігається, можуть бути низька енергія дефекту упаковки сплаву VT1-0, що ускладнює хід релаксаційних процесів, а також перебудова структури в процесі повзучості з утворенням нових деформаційних границь, які виявляються більш стійкими до навантажень, що розтягують.