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CREEP OF VT1-0 ALLOY IN DIFFERENT STRUCTURAL STATES

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The effect of electrons irradiation with an energy of E = 10 MeV and a dose of D = $5\cdot10^{19}$ cm^{-2} on the structure evolution and creep of alloy VT1-0 in initial and nanostructured states obtained by the IPD method has been studied. It is shown that, the deformation nanostructure of VT1-0 is resistant to subsequent deformation under creep conditions at T = 20 °C and is destroyed at T = 350 °C due to the development of recrystallization processes. Irradiation by electrons with an energy of E = 10 MeV and a dose of D = $5\cdot10^{19}$ cm^{-2} has slightly effect on the mechanical characteristics of VT1-0, both in initial and nanostructured states, but leads to the conservation and stabilization of the nanostructure during creep at T = 20 °C and to the deceleration of the process of structure transformation at T = 350 °C as a result of a decrease in the level of internal stresses.

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INTRODUCTION

Currently, titanium is demanded in various industries. The technical basis for the use of titanium alloys as structural materials for nuclear power engineering are: the high strength, the high radiation resistance at operating temperatures (250...400 °C), corrosion resistance in active surroundings [1]. Low-alloy titanium alloys are more than an order of magnitude higher to alloys based on iron and chromium in the level of induced activity [2]. Titanium alloys retain high plasticity after neutron irradiation and have a low rate of its decrease as the fluence increases. According to these characteristics, they are exceeded to both low-alloy and austenitic steels [3].

For the wide use of titanium alloys as structural materials for reactor vessels, a number of studies are required. However, it can already be argued that, in terms of reliability and safety, they are a promising structural reactor vessels material. It is known [3] that technical titanium is similar in strength characteristics to traditional structural steels, but at the same time it is 45% lighter, however, there are still many questions on the physics of strength.

Since, the study of the regularities of the VT1-0 titanium nanostructure formation is of both scientific and practical interest, because nanocrystalline materials have unique mechanical properties, such as high strength, wear resistance, hardness, and high fatigue properties.

Now the most promising method for obtaining nanostructured crystalline materials is the method intense plastic deformation (IPD), in particular, deformation by rolling [4]. It is shown in [3, 5] that in the nanocrystalline state the mechanical characteristics (yield stress, ultimate strength, microhardness) of technical titanium VT1-0 reach the properties of high-strength alloyed titanium alloys. However, the nature of the change in the physical and mechanical properties during the formation of the nanostructure of titanium alloys, as well as the issue of the stability of the created nanostructure in a wide range of temperatures and deformations, has not been sufficiently studied [6, 7]. Consequently, studies of the structure and properties of nanostructured titanium alloys, as well as their dependence on temperature and acting stress, are actual.

The purpose of this work is to study the regularities of creep and evolution of the structure of commercially pure VT1-0 titanium, obtained by the IPD method by rolling, and the effect of electron irradiation on its mechanical characteristics.

MATERIAL AND PROCESSING METHOD

Investigated commercial pure titanium VT1-0 of industrial production, the amount of impurities of which does not exceed 0.3%. Samples VT1-0 for testing were made by the method electrospark cutting in the form of plates with a size of 0.3x3x50 mm.

In order to study the effect of electron irradiation on the structure and properties of the VT1-0, the following processing modes were studied:

1. MT-1 – initial state;
2. MT-2 – MT-1 + electron irradiation with an energy of E = 10 MeV and dose of D = $5\cdot10^{19}$ cm^{-2};
3. MT-3 – rolling at 20 °C, strain (ε) was ε = 3.0;
4. MT-4 – MT-3 + electron irradiation with an energy of E = 10 MeV and dose of D = $5\cdot10^{19}$ cm^{-2}.

The samples were irradiated with an electron beam with the energy of E = 10 MeV and with the dose set of D = $5\cdot10^{19}$ cm^{-2}. The accelerated electron flow was formed by a linear electron accelerator operating in a pulsed mode. The average beam current was 10 μA, the pulse duration was 3.6 μs, the transmission frequency was 6 Hz. The temperature of the samples not exceeding 90 °C and was determined on the temperature dependence of the electrical resistance of the VT1-0 alloy.

The creep tests were carried out in the step loading regime at 20 and 350 °C, the measurement accuracy was $5\cdot10^{-5}$ cm. To study the defect structure of the VT1-0 alloy, the method of measuring the electrical resistance (R) after each treatment was used. Electrical resistance was measured using a 4-point scheme by the compensation method. The R measurement error did not exceed ± 0.5%. The structure evolution control was carried out using the electron microscopic method.

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.

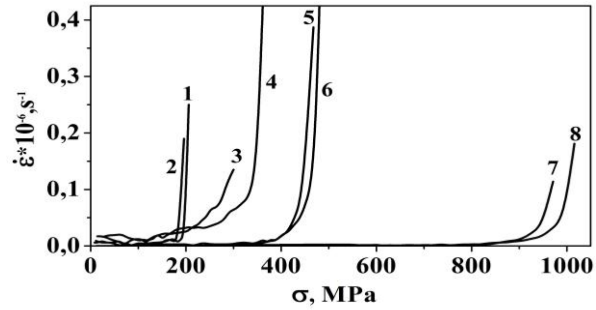

Fig. 1. Creep rates as a function of the applied stress at T = 20 °C (5–8) and T = 350 °C (1–4) of VT1-0 alloy specimens in different structural states: MT-1 = 1, 5; MT-2 – 2, 6; MT-3 – 3, 7; MT-4 – 4, 8

Analysis of the results obtained shows that IPD (MT-3) leads to an increase in the strength characteristics of the VT1-0 alloy at all test temperatures: 2 times compared with the initial state (MT-1) at the test temperature T = 20 °C and 1.5 times at the test temperature T = 350 °C. In this case, the plasticity of the rolled samples decreased from 7.7% (MT-1) to 6% (MT-3) (see Fig. 1, curves 5, 7) at 20 °C, by ~ 20%, i.e. the material has hardened At the test temperature T = 350 °C the plasticity increased 5 times from 3% (MT-1) to 19.2% (MT-3) (see Fig. 1, curves 2, 3).

After electron irradiation of the alloy VT1-0 in supply state (MT-2), the strength characteristics at 20 °C practically did not change compared to the initial state (MT-1), while the plasticity increased from 7.7 to 10% (see Fig. 1, curves 5, 6). Tests under creep conditions of irradiated samples VT1-0 in supply state (MT 2) at 350 °C showed that the strength properties and plasticity of the alloy (see Fig. 1, curves 1, 2) practically did not change.

After irradiation of rolled VT1-0 (MT-4) specimens, the ultimate strength at 20 °C slightly increased in comparison with the unirradiated state (MT-3) by ~ 40 MPa, and the plasticity increased from 6 to 7% (see Fig. 1, curves 7, 8). At T = 350 °C, an increase of the ultimate strength by ~ 60 MPa is observed for irradiated rolled samples of commercial titanium (MT-4) and an increase in plasticity from 19.2 to 20.9% (see Fig. 1, curves 4, 5) while maintaining the yield stress.

It should be noted that the creep rate of irradiated and unirradiated nanostructured samples of VT1-0 alloy (MT-3 and MT-4) increases in comparison with the creep rate of the industrial alloy samples in both states (MT-1 and MT-2) and on the dependence of the creep rate on the true stress at T = 350 °C the steps are observed, which indicates a change in the deformation mechanism with an increase of the stress (see Fig. 1, curves 3, 4).

Earlier [8–10], we studied the regularities of creep of nanostructured Zr and alloy Zr1Nb and showed that plastic flow of nanostructured materials is due to the combined action of several mechanisms: intragranular slip of dislocations, transverse slip, climb and annihilation of dislocations at grain boundaries, and slip on the boundaries. The contribution of each of these mechanisms to plastic deformation changes depending on the applied stress, so at the initial stages of creep, return processes develop at grain boundaries, and then sliping on the boundaries and restructuring of the material begins.

Analysis of thermal activation parameters of plastic deformation of nanostructured samples of VT1-0 alloy shows that the activation volume, calculated by the formulas of the theory of thermoactivated creep, decreases with increasing stress, in contrast to the dependences obtained in [8–10]. In accordance with the theory, this means that the density of deformation defects increases with an increase in stress during the creep process, and the deformation at the stage of developed plastic flow is controlled by the mechanism of thermally activated overcoming of obstacles by moving dislocations, i.e. by the mechanism of strain hardening, which leads to an increase in the ultimate strength.

The obstacles that control the process of intragrains plastic flow change as the applied stress increases. At the initial stages, these can be point defects and impurities, and with an increase in stress the forest dislocations and dislocation accumulations may be. Simultaneously with the processes of intragrains slipping, return processes develop near the grain boundaries and the slip on the boundaries appeared, as well as the rearrangement of the structure at the test temperature T = 350 °C, as evidenced by a sharp increase in the creep rate at stresses near to the ultimate strength.

Thus, irradiation by electrons with an energy of E = 10 MeV and a dose of $D = 5\cdot10^{19}$ cm^{-2} has little effect on the mechanical properties of VT1-0 both in initial state and in nanostructured state at all test temperatures. It can be noted that at T = 350 °C, there is a tendency to an increase in the strength, plasticity, and creep rate of the irradiated nanostructured VT1-0 specimens in comparison with the unirradiated specimens, and the deformation mechanism changes during the creep process.

After deformation by rolling and irradiation, the values of the relative electrical resistance ($R_{300\,K}/R_{77\,K}$) of the VT1-0 alloy samples were determined. Determination of the value of the residual electrical resistance makes it possible to control the changes in the defect state of the material. The measurement results of the residual electrical resistance are shown in Table.

It can be seen from the table that irradiation leads to a decrease in the value of $R_{300\,K}/R_{77\,K}$, which indicates the decrease in the level of internal stresses in the samples of the VT1-0 nanostructured alloy and to the increase in internal stresses in the samples of the industrial alloy, which is consistent with the changes in the creep characteristics at T = 20 °C.

The value of the residual electrical resistance of the VT1-0 alloy after various treatments

Measured characteristics	Treatment type			
	MT-1	MT-2	MT-3	MT-4
$R_{300\,K}/R_{77\,K}$	3.193	2.916	2.654	2.778

Structural studies have shown that commercially pure VT1-0 titanium in suppled state has a very heterogeneous structure. In the body of grains, there are microvolumes with a small number of dislocations, separate dislocation accumulations, less often – with a cellular structure. A feature of the structure can be considered a high concentration of packing defects (Fig. 2,a).

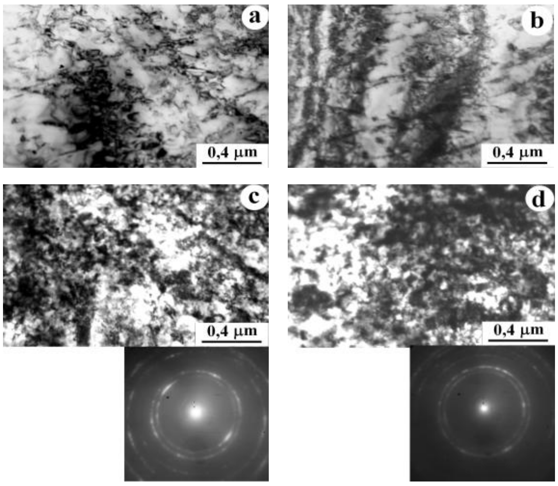

Fig. 2. TEM images of VT1-0 alloy after follows treatments: a – MT-1; b – MT-2; c – MT-3; d – MT-4

The main consequence of electron irradiation of the initial VT1-0 structure can be considered the appearance of bending deformations of the crystal lattice and an increase in the width of packing defect. Elastic distortions are evidenced by extended contours in electron microscopic images, the appearance of which is due to the synchronous displacement of the lattice atoms from the equilibrium positions. Partial relaxation of distortions occurs through the formation of misfit dislocations. They are visible in and along the contours. The reason for the expanding of packing defects may be the active absorption by their of the complexes “vacancy – interstitial impurity atom” formed during the irradiation (see Fig. 2,b).

The transformation of the VT1-0 structure during cold rolling (20 °C) is characteristic of metals and alloys with a low (< 10 erg/cm^2) packing defect energy. It is known [5], that twinning dominates at degrees of deformation up to ~ 25%. The twin width is about 0.15…1 μm. With an increase in the degree of deformation, the slipping develops in the body of twins. Dislocations form the plane clusters. With the accumulation of power, such dislocation “charges” destroy the twins boundaries, sharply increasing the degree of structural defects. By the time the deformation reaches 60%, the extended boundaries of the dislocation-disclination genesis limit the microvolumes of the texture orientation.

During further deformation, rotational plastic flow modes develop, forming at $\varepsilon = 3.0$ the structure with a grain size of 55 to 140 nm. Grains with a size of 100 nm dominate, i.e. in fact, the nanostructure was formed, which is confirmed by the dark-field image on Fig. 2,c. The concentration of the boundary phase reaches a significant magnitude and the width of the boundaries traces in electron microscopic images can be ~ 25 nm. There are practically no dislocations in the body of grains (see Fig. 2,c).

Irradiation of the nanostructure formed after rolling leads to relaxation of internal stresses both in the boundaries and in the grains themselves. The boundaries are more clearly marked, and the grains were broken into blocks or cells, separated by low-angle dislocation boundaries. The total length of the boundaries of different power increased by 2.5…3 times (see Fig. 2,d), while, as can be seen in the dark-field image, the nanostructure was conserved.

Deformation in the creep regime at T = 350 °C of the initial samples is accompanied by the formation of the cellular structure (Fig. 3,a).

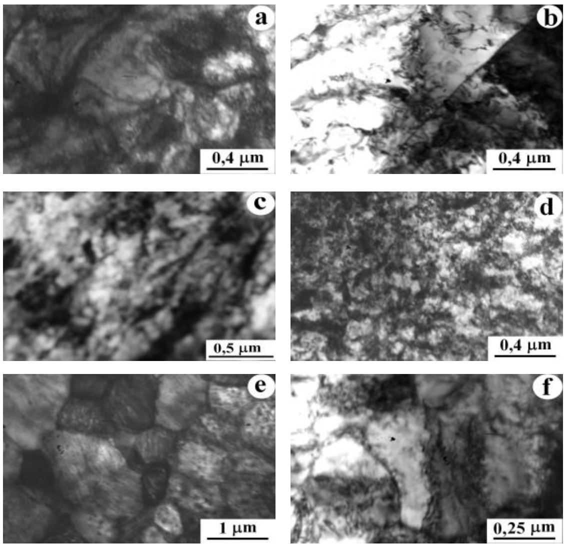

Fig. 3. TEM images of VT1-0 alloy after follows treatments: a – MT-1+creep at 350 °C ($\sigma \sim 0.9\sigma_B$); b – MT-2+creep at 350 °C ($\sigma \sim 0.9\sigma_B$); c – MT-3+creep at 20 °C ($\sigma \sim 0.9\sigma_B$); d – MT-4+creep at 20 °C ($\sigma \sim 0.9\sigma_B$); e – MT-3+creep at 350 °C ($\sigma \sim 0.9\sigma_B$); f – MT-4+creep at 350 °C ($\sigma \sim 0.9\sigma_B$)

In the irradiated initial samples, such a structure does not have the time to form during the creep process. The applied mechanical and thermal energy leads to the emission of dislocations and their partial annihilation (see Fig. 3,b).

The superposition of the tensile load in the creep regime at 20 °C on the deformation nanostructure leads to the destruction of most of the boundaries and the formation of a cellular and fragmented structure with a fragment size of 0.05…0.15 μm. New boundaries are formed by dislocations that appeared during the disintegration of unstable deformation boundaries (see Fig. 3,c).

The plastic flow of nanostructured irradiated samples (MT-4) at 20 °C was realized within grain boundaries (see Fig. 3,d). Under conditions of a limited volume of structural elements of deformation, it quickly obtained a collective character, initiating disclination effects in the body of grains. This is evidenced by the comminution of the grains (20…100 nm). The rotations of grains are possible, taking into account the quasi-amorphous state of the boundaries when the limiting concentration of dislocations is obtained with overlapping of their cores. It should be noted that the nanostructure is conserved (see Fig. 3,d).

During creep at 350 °C of rolled VT1-0 samples (MT-3), the recrystallized structure with an average grain size of ~ 1 μm was formed in place of the nanostructure (see Fig. 3,e).

Thus, the development of hardening processes during creep may be connected with the fact that the restructuring of the structure begins at an earlier stage of deformation than was observed in [6–8].

In the creep process at 350 °C of nanostructured irradiated samples (MT-4), recrystallization appearsed unfinished due to the activation of polygonization processes with the formation of closed and open dislocation boundaries (see Fig. 3,f). This may be due to the fact that the level of internal stresses in the nanostructured material decreased after irradiation.

It is known from the theory [11] that the recrystallization process can immediately begin at a sufficiently high level of energy reserved in the metal during deformation. At a lower level of internal stresses and made of heating, the return processes takes place, leading to the formation of a polygonal structure. In this case, the energy of the system decreases and the process of polygonization can compete with the process of recrystallization, delaying it and increasing the temperature of its onset [11].

Thus, irradiation of the nanostructured VT1-0 by electrons with an energy of E = 10 MeV and a dose of $D = 5\cdot10^{19}$ cm^{-2} leads to the decrease in the level of internal stresses and the slowdown in the transformation processes of the nanostructure in creep conditions.

CONCLUSIONS

The effect of electron irradiation with an energy of E = 10 MeV and a dose of $D = 5\cdot10^{19}$ cm^{-2} on the creep and structure evolution of the samples of commercial titanium VT1-0 of industrial production and samples obtained by the IPD method by rolling was studied.

It is shown that, at all test temperatures, the strength characteristics of VT1-0 in the nanostructured state increase by 1.5…2 times in comparison with the characteristics of industrial alloy. The deformation nanostructure is resistant to subsequent deformation under creep conditions at the test temperature of 20°C and is destroyed at the test temperature of 350 °C due to the development of recrystallization processes.

Irradiation by electrons with an energy of E = 10 MeV and a dose of $D = 5\cdot10^{19}$ cm^{-2} of nanostructured samples of titanium VT1-0 weakly effects on its mechanical properties at a test temperature of 20 °C, but leads to the conservation and stabilization of the nanostructure as a result of the decrease in the level internal stresses.

Thus, commercially pure titanium VT1-0 in a nanostructured state with high strength characteristics and sufficient plasticity has well perspectives for the practical application under irradiation conditions at the temperature about 20 °C.

Irradiation by electrons with an energy of E = 10 MeV and a dose of $D = 5\cdot10^{19}$ cm^{-2} of the VT1-0 samples in the nanostructured state reveals a tendency to increase the strength, plasticity and creep rate at the test temperature of 350 °C compared to the characteristics of the unirradiated rolled samples. This is due to the deceleration in recrystallization processes and the development of polygonization processes due to the decrease in the level of internal stresses in the nanostructured material after irradiation, i.e. the process of structure transformation decelerated in creep conditions.

REFERENCES

1. 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.
2. A.M. Parshin, V.A. Zhukov, A.P. Petkova Features of radiation cold-brittleness and ways to improve the performance of reactor pressure vessel materials // *Nuclear Power Plant Construction*. 2002, v. 282, p. 258-266.
3. A.M. Parshin, O.E. Muratov, O.A. Kozhevnikov. On the use of titanium alloys for pressurized water reactor vessels // *Problems of Atomic Science and Technology. Series "Physics of Radiation Effect and Radiation Materials Science"*. 2005, N 3(86), p. 179-181.
4. 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.
5. R.Z. Valiev, E.V. Kozlov, Yu.F. Ivanov, J. Lian, A.A. Nazarov, B. Baudelet. The nanostructure of Ti after deformation // *Acta Metallurgical Mater*. 1994, v. 42, p. 2467-2473.
6. 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.
7. A.N. Dovbnya, V.A. Mats, V.I. Sokolenko. Influence of electron irradiation on the nanostructural state of deformed zirconium // *Problems of Atomic Science and Technology. Series "Physics of Radiation Effect and Radiation Materials Science"*. 2012, N 5, p. 36-39.
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, Supreconductors"*. 2014, N 1(89), p. 106-109.
9. E.V. Karaseva, A.V. Mats, V.A. Mats, E.S. Savchuk, V.I. Sokolenko, D.V. Tiтov. Effect of electronic radiation on the evolution of nanostructure and creep of Zr at the temperature of 670 K // *Problems of Atomic Science and Technology. Series "Vacuum, Pure materials, Supreconductors*". 2020, N 1(125), p. 91-95.
10. E.V. Karaseva, S.V. Malykhin, A.V. Mats, V.A. Mats, E.S. Savchuk, V.I. Sokolenko. Influence of electron irradiation on the structure evolution and creep of Zr1Nb alloy at 380 °C // *Problems of Atomic Science and Technology. Series "Physics of Radiation Effect and Radiation Materials Science*". 2021, N 2(132), p. 55-59.
11. S.S. Gorelik. *Recrystallization of metals and alloys*. M.: "Metallurgy", 1978, p. 567.

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ПОЛЗУЧЕСТЬ СПЛАВА ВТ1-0 В РАЗЛИЧНЫХ СТРУКТУРНЫХ СОСТОЯНИЯХ

Е.С. Савчук, В.И. Соколенко, Е.В. Карасева, А.В. Мац, В.А. Мац, В.А. Фролов

Изучено влияние электронного облучения с энергией E = 10 МэВ и дозой D = $5 \cdot 10^{19}$ см$^{-2}$ на эволюцию структуры и ползучесть сплава ВТ1-0 в состоянии поставки и наноструктурном состоянии, полученном методом ИПД прокаткой. Показано, что деформационная наноструктура устойчива к последующей деформации в условиях ползучести при T = 20 °С и разрушается при T = 350 °С вследствие развития процессов рекристаллизации. Облучение электронами с энергией E = 10 МэВ и дозой D = $5 \cdot 10^{19}$ см$^{-2}$ слабо влияет на механические характеристики ВТ1-0 как в состоянии поставки, так и в наноструктурном состоянии, однако приводит к сохранению и стабилизации наноструктуры в процессе ползучести при T = 20 °С и замедлению процесса трансформации структуры при T = 350 °С в результате снижения уровня внутренних напряжений.

ПОВЗУЧІСТЬ СПЛАВУ ВТ1-0 У РІЗНИХ СТРУКТУРНИХ СТАНАХ

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

Вивчено вплив електронного опромінення з енергією E = 10 МеВ і дозою D = $5 \cdot 10^{19}$ см$^{-2}$на еволюцію структури та повзучість сплаву ВТ1-0 у стані постачання і наноструктурном стані, отриманого методом ІПД прокаткою. Показано, що деформаційна наноструктура стійка до подальшої деформації в умовах повзучості при T = 20 °С і руйнується при T = 350 °С внаслідок розвитку процесів рекристалізації. Опромінення електронами з енергією E = 10 МеВ і дозою D = $5 \cdot 10^{19}$ см$^{-2}$ слабо впливає на механічні характеристики ВТ1-0 як у стані постачання, так і в наноструктурному стані, проте призводить до збереження і стабілізації наноструктури в процесі повзучості при T = 20 °С і до уповільнення процесу трансформації структури при T = 350 °С у результаті зниження рівня внутрішніх напружень.