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# CREATION OF VARIOUS STRUCTURES, INCLUDING NANOMESTER SCALE, AND INVESTIGATION OF MECHANICAL AND DISSIPATIVE PROPERTIES OF VT6 TITANIUM ALLOY

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With the use of cryogenic deformations by rolling and quasi-hydroextrusion, as well as annealing up to 723 K, various structures, including nanometer ones, were created in ultrafine-grained (UFG) titanium alloy of the VT6 grade, and mechanical and dissipative properties were studied. The deformation regimes for the formation of a nanostructured state with a high level of strength characteristics without loss of plasticity are established. The revealed features of the temperature dependences in the range of 100...600 K of internal friction and shear modulus in the nanostructured state in comparison with the UFG state are analyzed.

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## INTRODUCTION

In recent years, much attention in the study and practical application of the plan has been paid to nanocrystalline materials. They are characterized by a grain size of $d \leq 100$ nm and have a high level of structural and functional properties, which is not achieved for coarse-grained materials. In bulk metals and alloys, nanometer-scale structures can be obtained through large plastic deformations, using traditional forming methods such as rolling, drawing, extrusion, as well as more complex methods – equal-channel angular pressing, screw extrusion, torsion under load, all-round isothermal forging [1–3].

he suppression of recovery processes with a decrease in the deformation temperature makes it possible to create structures in bulk crystals with a significantly higher defect concentration and degree of dispersion compared to deformation at room and higher temperatures. This makes it attractive to use low-temperature deformation effects to create nanostructures. For the first time, a nanostructured state in a metal (copper) was obtained at the NSC KIPT more than half a century ago using rolling deformation at 20 and 4.2 K [4].

Titanium and titanium alloys are promising structural and functional materials for wide application and considerable attention is paid to the creation of nanoscale structures in them (see, for example, [5–7]).

In addition to using separate pressure treatment methods, an effective technique for structure dispersion is the combination of various methods, which are characterized by different stress diagrams. In particular, it was shown in [8] that the combination of severe plastic deformation according to the upsetting–extrusion–drawing scheme with cryogenic quasi-hydroextrusion (QHE) made it possible to create nanocrystalline titanium (grain size 75 nm) of increased purity with high mechanical properties (ultimate strength $\sigma_B$ = 930 MPa, plasticity $\varepsilon \approx 12\%$).

The aim of this work was to study the use of a combination of large plastic deformations at elevated and cryogenic temperatures for the formation of various structures in a titanium alloy of the VT6 grade, including nanoscale structures, and to establish the relationship between physical and mechanical properties and the structural state.

## MATERIAL AND RESEARCH METHODS

An industrial titanium alloy of the VT6 grade was studied, the chemical composition of which, in accordance with GOST 19807-91, is given in Table 1.

Table 1

Chemical composition in % titanium alloy of the VT6 grade

| Fe | C | Si | V | N | Ti | Al | Zr | O | H | Other impurities |
|---|---|---|---|---|---|---|---|---|---|---|
| up to 0.6 | up to 0.1 | up to 0.1 | 3.5…5.3 | up to 0.05 | 86.45…90.9 | 5.3…6.8 | up to 0.3 | up to 0.2 | up to 0.015 | 0.3 |

The high-temperature mechanical-thermal treatment of the alloy included the following operations: forging (deposit) at 900 °C, heating to 1000 °C, cooling into water, all-round isothermal forging at 700, 650, and 600 °C [5]. Then samples and billets were cut out from the workpiece for research and subsequent low-temperature deformation by rolling and QGE.

Low-temperature (77 K) deformation by rolling was carried out on laboratory rolls with a roll diameter of 80 mm. The use of various processing modes and methods (rolling rate and reduction fraction, number of passes, shelling of billets into soft metal, which prevents the occurrence of macrostresses in the surface layer, etc.) made it possible to achieve high total degrees of deformation without disturbing the continuity of the material and to exclude intermediate annealing. The QGE deformation at 77 K was carried out on the device described in [9]. The microstructure of the VT6 alloy was studied using an EMV 100BR electron microscope.

The mechanical characteristics of the samples were defined under conditions of active tension at a rate of $1\cdot10^{-3}$ $s^{-1}$. The shear modulus G and internal friction (or vibration decrement) $\gamma$ were measured using a reverse torsion pendulum at a frequency of ~ 0.4 Hz in a vacuum of $10^{-3}$ Torr. The experiments were carried out in the temperature range 100…700 K in the amplitude-independent region. The sample deformation amplitude was $1\cdot10^{-5}$.

## RESULTS AND DISCUSSION

***The microstructure characteristics of specimens strained by rolling.*** After high-temperature mechanical-thermal treatment, the alloy had a structure with a grain size of 150…200 nm (Fig. 1,a), which can be characterized as ultrafine-grained (UFG). The character of the microdiffraction pattern taken from an area of 2 $\mu m^2$ confirms the high degree of dispersion of this structure.

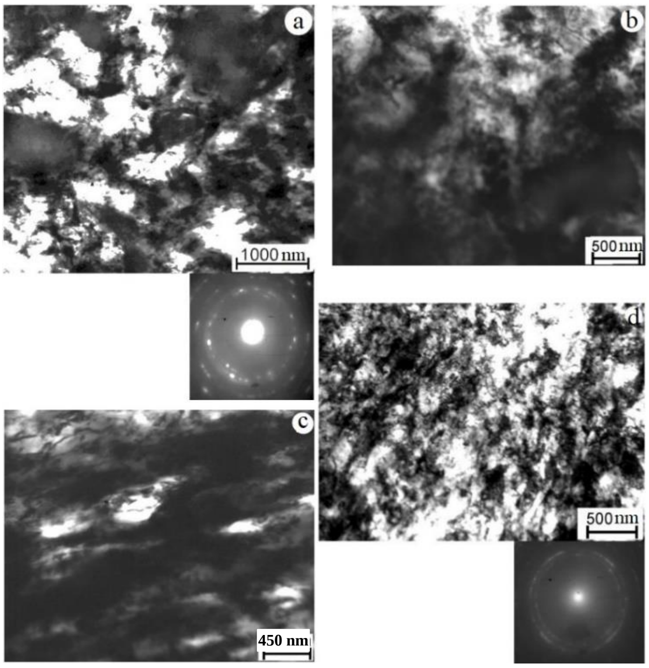


*Fig. 1. Structure of the VT6 alloy: UFG state (a); after deformation by rolling at 77 K by 30 (b); 50 (c), and 60% (d)*

During low-temperature rolling, the UFG structure becomes unstable. After deformation up to 30%, a significant part of the boundaries is destroyed, and dislocation pileups are observed in their place (see Fig. 1,b). A further increase in the degree of deformation leads to the formation of a substructure characteristic of hcp deformed metals with low stacking-fault energy. Such substructure represents subgrains somewhat elongated along the rolling direction. At $\delta = 50\%$, the subgrain size in diameter is 200 nm (see Fig. 1,c). An increase in the degree of deformation to 60% leads to a significant refinement of the substructure – up to 70 nm (see Fig. 1,d).

***The mechanical characteristics***. Fig. 2 shows the tensile curves of the VT6 alloy samples in the UFG state and after deformation by rolling and QHE at 77 K to various degrees, and also for comparison – of coarse-grained (15…20 μm) material.

Table 2 shows for the VT6 alloy in various states the values of proportional limit $\sigma_{pl}$, ultimate strength $\sigma_B$, uniform elongation $\varepsilon_u$ and elongation to failure $\varepsilon_f$, averaged over several samples.

Table 2 and Fig. 1 show that the change in the mechanical properties of the VT6 alloy with a preliminarily formed UFG structure depends on the type and degree of subsequent deformation. In general, low-temperature deformation leads to an increase in strength characteristics. Attention is drawn to a slight decrease in $\sigma_{pl}$ of the sample deformed by rolling up to 30%. The decrease in the proportional limit should be associated with the destruction of the grain boundaries of the UFG structure and with the formation of dislocation clusters (see Fig. 1,b) with lower values of the starting stresses of dislocation sources.

For QHE deformation in the range up to 21% and rolling up to 60%, the normalized values of the growth rate $\sigma_{pl}$ and $\sigma_B$ coincide with a good degree of accuracy and amount to ≈ 1.8 and ≈ 0.48, respectively. Such a significant excess of the rate of strain hardening in the case of QHE deformation in comparison with rolling indicates a more intense generation and accumulation of deformation defects in the QHE process due to the specifics of the deformation diagram. This is confirmed by the behavior of plasticity: for specimens deformed by QHE, the values of $\varepsilon_p$ and $\varepsilon_f$ are lower in comparison with the characteristics of specimens deformed by rolling (see Table 2).

A further increase in the degree of QHE deformation ($\delta > 21\%$) leads to the formation of microcracks in the body of the grains. This should be attributed to the emergence of a high level of internal stresses, the relaxation of which is difficult due to low temperatures. It can be assumed that the achieved value of the ultimate strength $\sigma_B \sim 1400$ MPa corresponds to the maximal strain hardening of the VT6 alloy.

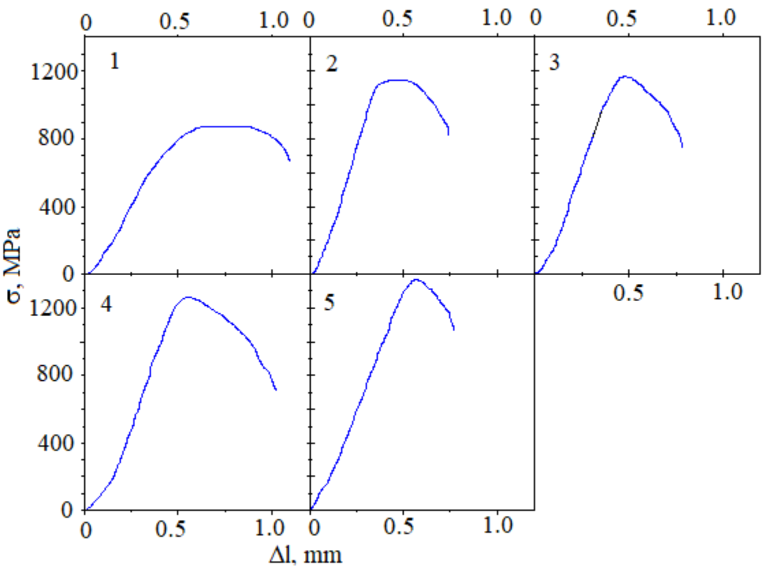


*Fig. 2. Tensile curves of the VT6 alloy samples: 1 – coarse-grained; 2 – UFG; 3 – after rolling by 30%; 4 – after rolling by 60%; 5 – after QHE by 21%*

Table 2

| State | $\sigma_{pl}$, MPa | $\sigma_B$, MPa | $\varepsilon_u$, % | $\varepsilon_f$, % |
|---|---|---|---|---|
| Coarse-grained | 700 | 900 | 10 | 15 |
| UFG | 1083 | 1130 | 3 | 10 |
| UFG + QHE at 77 K, δ = 7% | 1180 | 1200 | 1 | 8.6 |
| UFG + QHE at 77 K, δ = 23% | 1260 | 1360 | 1 | 8 |
| UFG + rolling at 77 K, δ = 30% | 900 | 1160 | 3 | 13 |
| UFG + rolling at 77 K, δ = 60% | 1150 | 1250 | 1.5 | 11 |

***Dissipative characteristics and elastic moduli***. Temperature dependences of low-frequency internal friction γ and shear modulus G of VT6 alloy in various states are shown in Figs. 3–8.

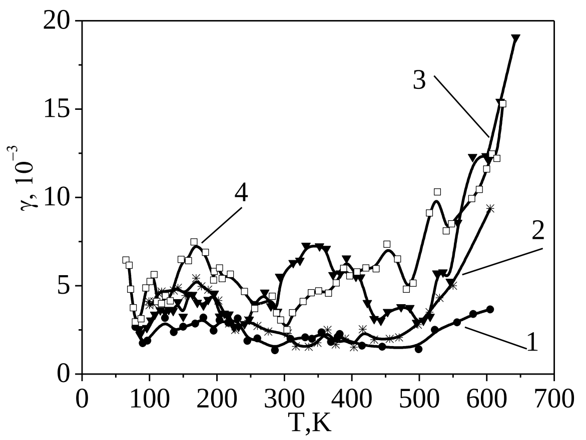


*Fig. 3. Temperature dependences of internal friction of the VT6 alloy: 1 – UFG state; 2 – after rolling by 42%; 3 – after rolling by 60%; 4 – after QHE by 21%*

In the UFG state, the temperature dependence of internal friction is characterized by weakly pronounced background rises in the temperature ranges of 100…250 and 500…600 K (see Fig. 3, curve 1). For the UFG alloy after rolling by 42 and 60% and CGE by 21%, a wide peak appears on the temperature dependence of internal friction in the range of 100…230 K. The occurrence of deformation peaks in this temperature range is usually associated with relaxation processes predetermined by the motion of dislocations in the presence of point defects (impurities, vacancies) in an alternating stress field created by measuring internal friction. We can assume that CGE and cryogenic rolling caused the appearance of new complexes of the dislocation-point defect type.

For a sample deformed by rolling by 60%, in the temperature range of 230…500 K, a broad peak of internal friction of a complex shape is observed. This peak seems to be a superposition of several peaks with different activation energies (see Fig. 4). A similar peak appears after deformation by QHE by 21% (see Fig. 4,b).

Using formula (1), one can estimate the activation energies E of relaxation processes responsible for the appearance of peaks:

$$E = RT_{\max} \ln \frac{k_B T_{\max}}{\hbar \omega_{\max}}, \qquad (1)$$

where $T_{max}$ is the temperature of the maximum; $\omega_{max}$ is the frequency at which the maximum is fixed; $k_B$ is the Boltzmann constant; $\hbar$ is Planck's constant. The characteristic values of activation energies calculated by formula (1) are shown in Fig. 5.

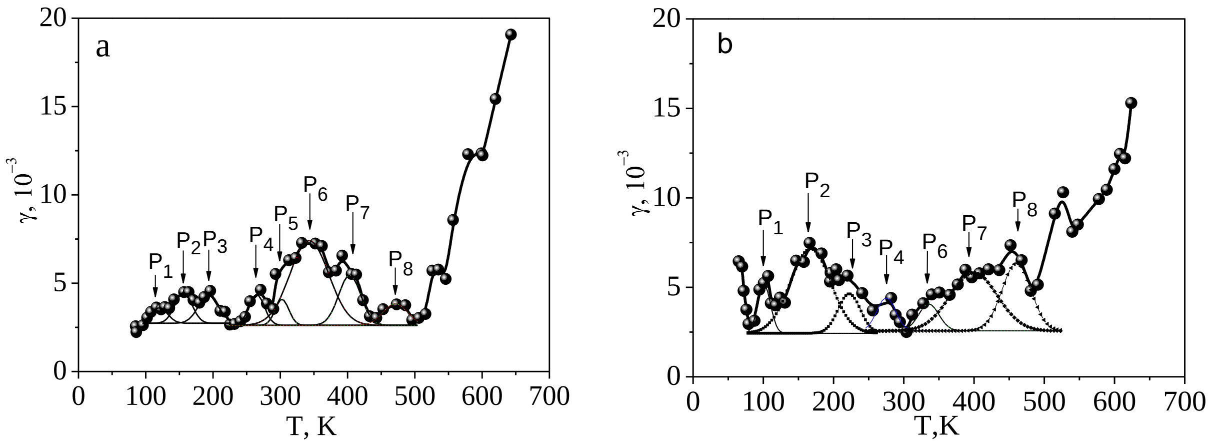


*Fig. 4. Decomposition into peaks of the experimental curves of internal friction of the VT6 alloy deformed at 77 K by rolling by 60% (a) and QHE by 20% (b)*

The temperature dependence of the natural frequencies of the oscillatory system, obtained by measuring the internal friction of the samples under study, was used in the calculation.

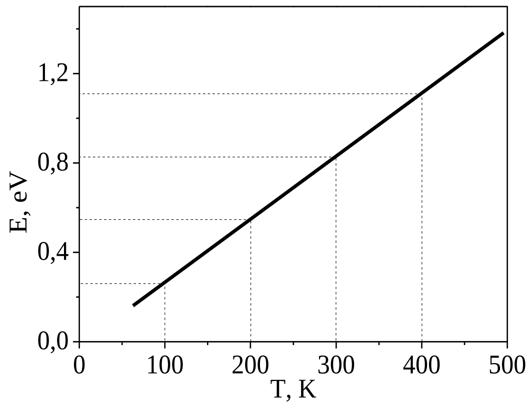


*Fig. 5. Temperature dependence of the activation energy of relaxation processes, calculated for the measurement frequency range of 0.2…0.3 Hz*

It should be noted that rolling by 42% did not lead to the appearance of an internal friction peak in the range of 300…500 K. Apparently, a necessary condition for the appearance of peaks in this temperature range is the presence of high fields of elastic stresses created as a result of deformation to large degrees. The presence of interfaces of different types in a strongly strained material, a high density of dislocations and their clasters, and complexes of various point defects complicates the unambiguous interpretation of the nature of the maxima. An interesting fact is that the method of deformation affects the spectrum of internal friction in the range of 300…500 K.

While maintaining the temperature position of the peaks, their intensity is different. For samples deformed by rolling and deformed by QHE, the main peak is observed at 350 K (E = 0.9 eV) and at 470 K (E = 1.35 eV), respectively. This indicates differences in the spectrum of the resulting deformation defects and their complexes.

Upon heating to 500 K, samples deformed both by QHE and by rolling, the peaks in the region of 300…500 K disappear. The low recovery temperature of the temperature dependence of internal friction to that characteristic of the initial state indicates that point defects are involved in the relaxation processes responsible for the appearance of peaks in the range of 300…500 K. These can be both vacancies created during deformation, and impurity atoms, as well as complexes of point defects (divacancies, vacancy-impurity atom complexes, etc.).

Point defects and their complexes can contribute to the spectrum of internal friction independently, as well as when interacting with dislocations of different types and different slip systems. The disappearance of peaks in the region of 300…500 K upon heating to 500 K indicates a decrease in their amount as a result of annihilation and entry into sinks, and may also be a consequence of partial removal of internal stresses.

After rolling by 42 and 60%, with an increase in the measurement temperature, starting from 470 and 510 K, respectively, a significant increase in the vibration decrement γ is observed. The same increase in γ at T > 470 K is typical for an alloy deformed by QHE by 20%. It is known [10] that at $T = 0.3…0.5\ T_{melt}$, the temperature dependences of the internal friction of metals and alloys can show maxima associated with grain boundaries. The formation of new nonequilibrium interfaces leads to a shift of the peak towards low temperatures [11].

The observed stage of the increase in the decrement of oscillations, apparently, is the beginning of the low-temperature branch of the grain-boundary maximum. The lower temperature of the beginning of the low-temperature branch of the grain-boundary maximum of internal friction compared to the UFG state may indicate the formation of new dissipation elements in the form of non-equilibrium interfaces in the process of deformation by rolling and QGE, i.e., additional refinement of the structure. A more intensive growth of the low-temperature branch of the grain-boundary maximum for deformed samples (see Fig. 3) is appropriate to associate with the greater length of the non-equilibrium interfaces. For a material deformed by rolling by 60%, these are the boundaries of the nanograin structure (see Fig. 1).

Fig. 6 (curve 1) shows the temperature dependence of the shear modulus of the UFG alloy VT6.

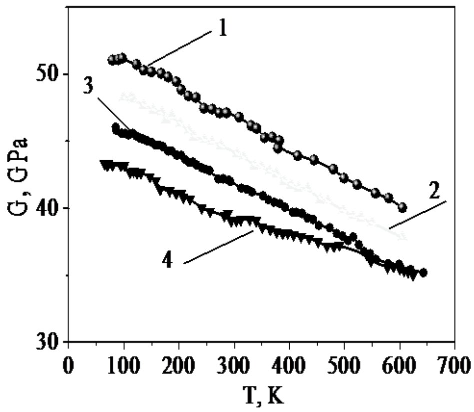


*Fig. 6. Temperature dependences of the shear modulus of the VT6 alloy: 1 – UFG state; 2 – after rolling by 42%; 3 – after rolling by 60%, and 4 – after QHE by 20%*

The observed decrease in the shear modulus with increasing measurement temperature (293…600 K) reaches ~ 15%. As is known [12], internal stresses, dislocations, point defects, and grain boundaries can contribute to elastic moduli. Deformation by cryorolling by 42 and 60% leads to a decrease in the shear modulus (curves 2 and 3). In this case, the effect is more significant with an increase in the degree of deformation. The greatest reduction in the shear modulus is achieved as a result of QHE by 21% (curve 4). Since the change in the shear modulus can serve as a measure of the defectiveness of the material, therefore, the most distorted structure with a high level of internal stresses is created as a result of the QHE by 21%, which is in accordance with the results of measurements of the strength characteristics.

Heating to 650 K of the VT6 alloy deformed by rolling did not lead to a significant change in the shear modulus (see Fig. 7). In this case, the peak of internal friction in the range of 300…500 K practically disappears (see Fig. 8, curve 2). Such a change in the spectrum of the temperature dependence of internal friction can be associated with the presence of point defects of deformation origin in the deformed alloy and their annealing upon heating to the specified temperature. It can be concluded that point defects created by rolling deformation by 60% do not make a significant contribution to the shear modulus.

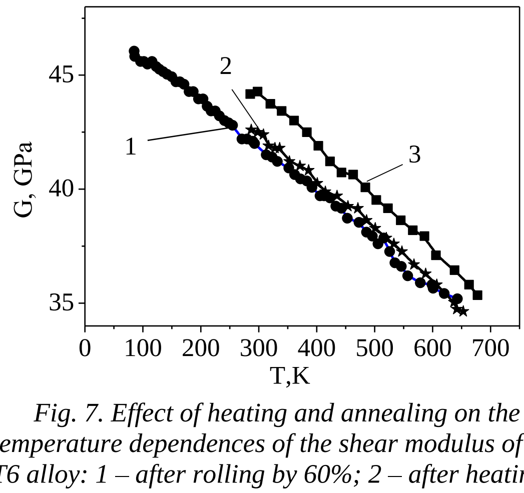


*Fig. 7. Effect of heating and annealing on the temperature dependences of the shear modulus of the VT6 alloy: 1 – after rolling by 60%; 2 – after heating up to 650 K; 3 – after annealing at 723 K, 30 min*

Annealing at 723 K for 30 min led to an increase in the shear modulus. However, a complete return to the shear modulus characteristic of the UFG state was not observed, which follows from a comparison of curve 3 in Fig. 7 and curve 1 in Fig. 6. Additional information on the nature of structural changes as a result of annealing in this mode can be obtained from measurements of internal friction.

The temperature dependence of internal friction shows a shift of the low-temperature branch of the grain boundary maximum towards higher temperatures (see Fig. 8, curve 3). This should be associated with the occurrence of polygonization processes at T = 723 K in the nanostructure created by low-temperature deformation.

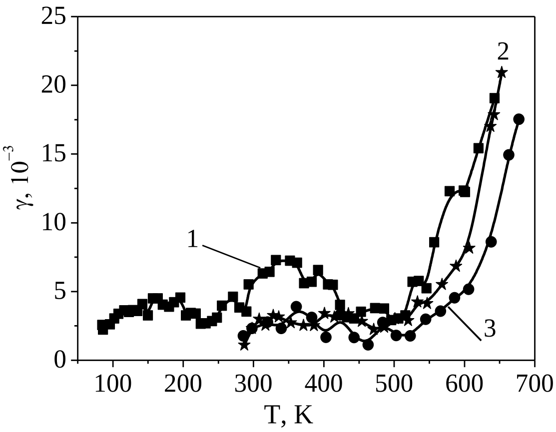


*Fig. 8. Effect of annealing on the internal friction of the VT6 alloy: 1 – rolling by 60%; 2 – heating up to 650 K; 3 – annealing at 723 K, 30 min*

## CONCLUSIONS

With the use of cryogenic deformations by rolling and QHE, as well as annealing up to 723 K, various structures, including nanometer ones, were created in ultrafine-grained titanium alloy VT6, and mechanical and dissipative properties were studied.

It is shown that, as a result of cryogenic deformation by rolling by 60%, an additional refinement of the structure of the VT6 alloy to the nanometer scale and a significant increase in strength properties without loss of plasticity occurs.

The optimal degrees of QHE deformation at 77 K are established, as a result of which the ultimate strength value $\sigma_B$ ~ 1400 MPa, corresponds to the maximal strain hardening of the VT6 alloy, is achieved.

Compared to the UFG state, the VT6 alloy in the nanostructured state is characterized by:

– a wide maximum of internal friction in the range of 100…230 K, which is associated with the appearance of new complexes of the dislocation-point defect type;

– a wide maximum of internal friction in the range of 300…450 K, the nature of which is difficult to establish due to the presence of high fields of internal stresses in the material, high density of dislocations and point defects;

– increase of grain-boundary internal friction in the temperature range 500…600 K;

– reduction of the shear modulus in the temperature range of 500…600 K by ~ 5000 MPa.

It is shown that point defects, which appeared in the VT6 alloy as a result of cryogenic rolling, do not make a significant contribution to the shear modulus.

The shift of the low-temperature branch of the grain-boundary maximum of internal friction towards higher temperatures observed in the VT6 nanostructured alloy after annealing at 723 K is associated with the occurrence of polygonization processes.

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## СТВОРЕННЯ В ТИТАНОВОМУ СПЛАВІ ВТ6 РІЗНИХ СТРУКТУР, ЩО ВКЛЮЧАЮТЬ НАНОМЕТРОВІ, І ДОСЛІДЖЕННЯ МЕХАНІЧНИХ І ДИСИПАТИВНИХ ВЛАСТИВОСТЕЙ

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Із застосуванням кріогенних деформацій прокаткою та квазігідроекструзією, а також відпалу до 723 K в ультрадрібнозернистому (УДЗ) титановому сплаві марки ВТ6 створені різні структури, що включають нанометрові, та досліджено механічні та дисипативні властивості. Встановлено режими деформації для формування наноструктурного стану з високим рівнем характеристик міцності без втрати запасу пластичності. Проаналізовано виявлені особливості температурних залежностей в інтервалі 100…600 K внутрішнього тертя і модуля зсуву в наноструктурному стані порівняно з УДЗ-станом.