Low-temperature Mechanical Relaxation in Commercially Pure Titanium

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EXPERIMENTAL PROCEDURE, SAMPLE CHARACTERISTICS

The temperature dependences of the decrement δ(T) and the dynamic Young's modulus E(T) of nano- and ultrafine-crystalline Ti of technical purity VT1-0 (Grade 2) were studied in the temperature range of 5 - 325 K using the method of mechanical resonance spectroscopy. The measurements were carried out at frequencies of 1.4 - 3.7 kHz of bending vibrations. The studied samples had the form of thin plates 4x0.3x22 mm, they were cut from larger blanks in two directions: parallel and perpendicular to the rolling direction. The substructural state of the samples was formed by deformation at temperatures of 100 and 290 K to true strain values of $\varepsilon = 1.2 - 1.9$; subsequent annealing at 525 K, 720 K and 940 K; holding at 300 K for 7 years. Electron microscopic studies have shown that the intragranular substructure of Ti after deformation at 290 K is characterized by high-density dislocation clusters, which leads to the appearance of numerous bending extinction contours (Fig. 1a), indicating a high level of internal stresses. The size of such areas is from fractions of a micron to several microns. In the substructure of the cryodeformed material, Coherent Scattering Regions (CSR) with a size of 30-50 nm predominate (Fig. 1b). After cryodeformation $\varepsilon = 1.2$, CSR are observed mainly in the form of clusters; with an increase in the degree of deformation, the clusters disintegrate and the CSR are distributed quite uniformly [1, 2]. The genesis of the CSR is due to the processes of multiple re-splitting of titanium under low-temperature deformation conditions [3]. Cryomechanical treatment of titanium leads to strong fragmentation of grains, in which the CSR size of crystallites is ~ 36 nm. Subsequent annealing at 525 K slightly increases the average crystallite size to 43 nm. At the same time, annealing at this temperature almost completely removes the internal stresses that have arisen in the material after cryorolling. Annealing at 720 K leads to a twofold increase in the average grain size (up to 70 nm), annealing at a temperature of 940 K leads to recrystallization and the disappearance of the nanostructured state of the samples.

EXPERIMENTAL RESULTS

The creation of deformation microstructures leads to the appearance of a peak P_1 on the δ(T) dependences at a temperature of 230K (Fig. 3). On the E(T) dependence, a diffuse step corresponds to it. An increase in the degree of deformation leads to a broadening and an increase in the amplitude of the peak P_1; in nanocrystalline samples, the amplitude of the peak P_1 is significantly higher. A series of annealings at 525K, 720K and 940K successively reduces (up to disappearance) the peak height and localization temperature. The peak P_1 is wider than the Debye peak and is frequency-dependent; with an increase in the frequency of mechanical vibrations of the sample, it shifts to the region of higher temperatures, which indicates its thermally activated nature. Estimates of the activation parameters of P_1 (activation energy $U_0 = 0.38$eV and the period of attempts $\tau_0 = 2 \cdot 10^{-13}$ s) are consistent with [4]. The combination of P_1 properties allows us to speak of its dislocation-deformation nature and high structural sensitivity of the relaxor system responsible for the occurrence of the peak. The differences in the nature of the formed microstructures of the material, caused by different deformation mechanisms at 100K and 290K, allow us to speak about the absence of a connection between this relaxation resonance and the intragranular microstructure. The experimentally obtained values of the activation parameters are typical for the so-called Koiwa-Hashiguti peaks [5] associated with the process of thermally activated detachment of a dislocation segment from a local structural defect, which can be impurity and interstitial atoms, vacancies. In this case, U_0 has the meaning of the activation energy of detachment, and τ_0 is the oscillation period of the dislocation segment directly interacting with the defect. Nanostructured samples have a number of features. Firstly, in the region of 43-78 K, a relaxation peak of acoustic absorption P_2 is observed. An increase in the degree of cryodeformation leads to a narrowing of the peak P_2 and a decrease in the temperature of its localization. Annealing at 525 K reduces the height and temperature of the localization of the peak P_2. After annealing at 720K, the P_2 peak is practically not observed. The P_2 absorption peak is also frequency-dependent; with an increase in the frequency of mechanical vibrations of the sample, it shifts to the region of higher temperatures, which indicates its thermally activated nature. Estimates of the activation parameters of the P_2 peak are $U_0 = 0.03$ eV and $\tau_0 = 2 \cdot 10^{-11}$ s. Such values of the activation parameters are characteristic of the process of overcoming the Peierls relief by dislocations via the mechanism of thermally activated nucleation of paired kinks, i.e. this peak is similar to the Bordoni peaks in fcc crystals [6]. It should be noted that a peak similar to P_2 was recorded in nanostructured Zr [7] obtained by intense plastic deformation. Secondly, in cryodeformed samples in the nanostructured state, the dynamic elastic modulus E is $\Delta E = 0.8$-1.2% lower than in the same samples after recrystallization annealing, and the value of ΔE is greater the greater the degree of cryodeformation. Annealing at 525K reduces ΔE, and after annealing at 720K, the low-temperature parts of the temperature dependences E(T) of cryodeformed and recrystallized samples practically coincide. Such behavior of the modulus is probably associated with the formation and evolution of the nanostructured state [8-9]. The temperature dependences of the logarithmic decrement of oscillations δ(T) and the dynamic Young's modulus E(T) in samples cut parallel and perpendicular to the rolling direction are qualitatively similar, and minor quantitativedifferences are probably associated with the presence of texture.

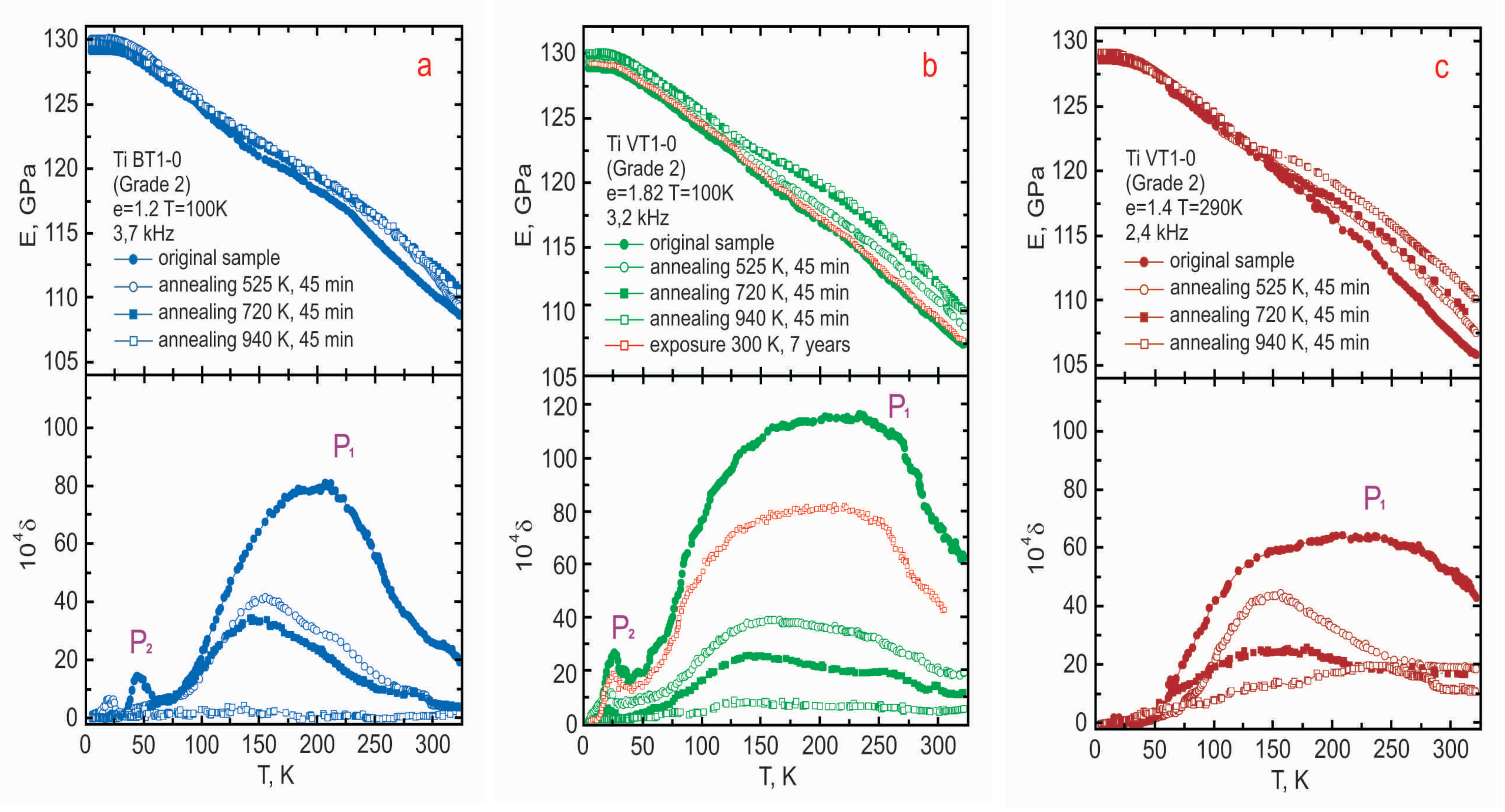

Fig. 3 Temperature dependences of the decrement δ(T) and dynamic Young's modulus E(T) of nano- (a, b) and ultrafine-crystalline (c) titanium VT1-0 (Grade 2).

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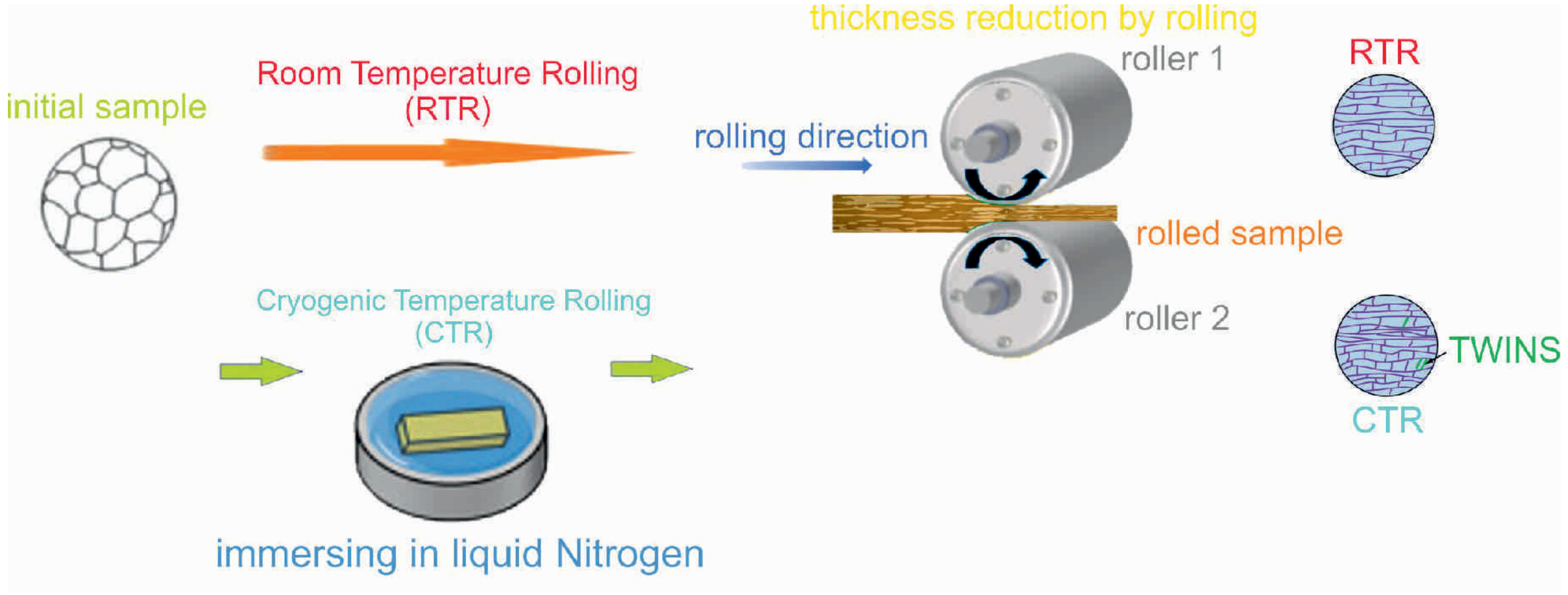

Diagram of the rolling-induced deformation state formation in VT1-0 titanium

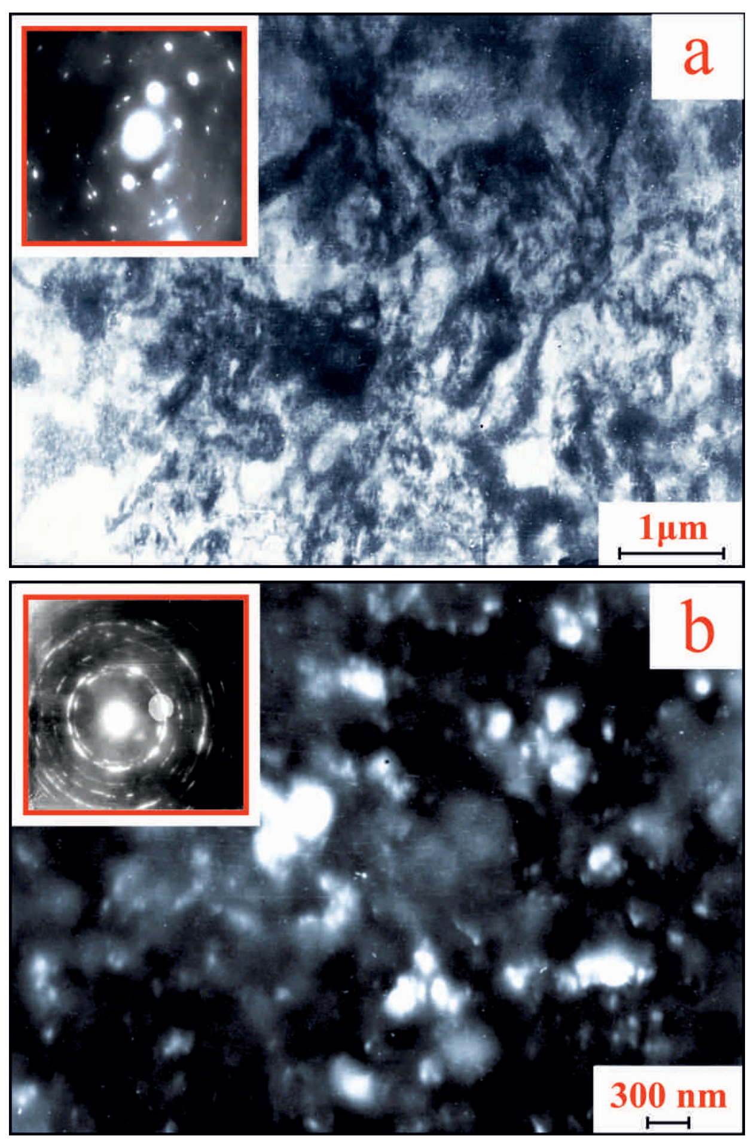

Fig. 1 Dark-field electron microscopic image of technical grade Ti VT1-0 (Grade 2) after deformation $\varepsilon = 1.8$: a) at 290 K; b) at 100 K.

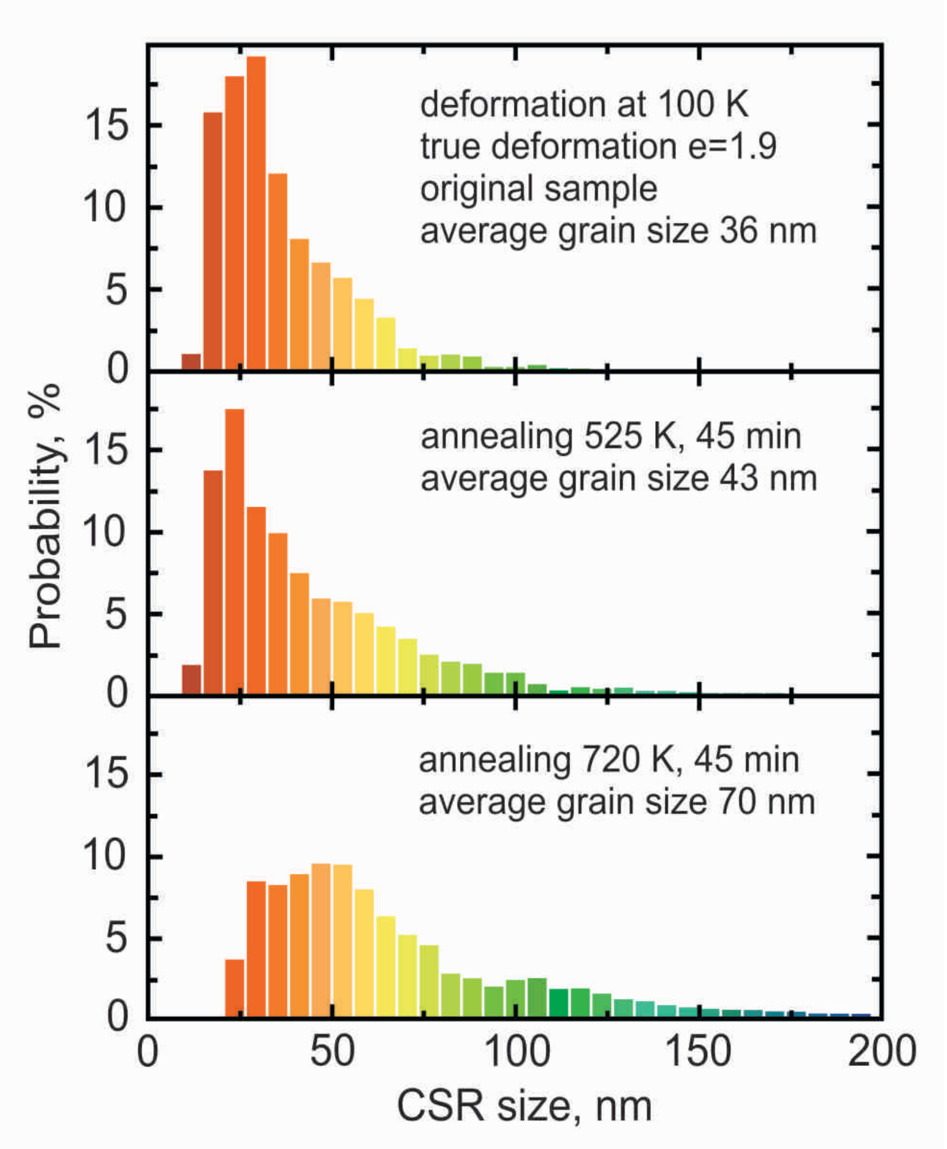

Fig. 2 Histograms of the distribution of the sizes of the CSR in the Ti VT1-0 (Grade 2) sample after cryodeformation $\varepsilon = 1.9$ and subsequent annealing.

Structural parameters of the VT1-0 titanium specimen after CTR from X-ray diffraction. The size L of regions exhibiting X-ray coherent scattering in crystallites, as well as the level of microdeformation εΔ and the unit cell parameters

e	L, nm	$\Delta\varepsilon \cdot 10^{-3}$	parameters cell		$\rho_L \cdot 10^{14}$, m^{-2}	$\rho_Z \cdot 10^{14}$, m^{-2}	$<\rho> \cdot 10^{14}$, m^{-2}
			a, Å	*c*, Å			
0	282	0.6	2.958	4.711	0.37	0.15	0.23
0.12	182	2.3	2.964	4.699	0.9	2.13	1.39
0.6	110	0.3	2.998	4.729	2.48	0.04	0.3
1.2	55	3.9	2.967	4.709	9.9	6.1	7.78
2	64	3.8	2.972	4.709	7.32	5.78	6.51

CONCLUSIONS

1. The temperature dependences of the logarithmic decrement of oscillations δ(T) and the dynamic Young's modulus E(T) of nanostructured and ultrafine-crystalline Ti of technical purity VT1-0 (Grade 2) were studied.
2. It was found that severe plastic deformation by rolling leads to strong fragmentation of the grain of the original material. In this case, in the substructure of the material deformed at 100 K, Coherent Scattering Regions (CSR) with a size of 30-50 nm predominate, while after deformation at 290K the CSR size is from fractions of a micron to several microns.
3. Severe plastic deformation leads to the occurrence of relaxation resonance P_1 with an activation energy of $U_0 = 0.38$eV and a period of attempts $\tau_0 = 2 \cdot 10^{-13}$ s at a temperature of 230K. The set of properties of P_1 allow us to associate it with the Koiwa-Hasiguti relaxation process.
4. The formation of a nanostructured state during cryodeformation is accompanied by a reduction in the dynamic elastic modulus $\Delta E = 0.8 - 1.2$ % across the entire temperature range studied. The magnitude of ΔE increases with the degree of cryodeformation. Recrystallization annealing at 940K results in the modulus returning to its original values.
5. The nanostructured state of the samples is characterized by the presence of a relaxation peak P_2 in the range of 43-78K with an activation energy of $U_0 = 0.03$eV and an attempt period of $\tau_0 = 2 \cdot 10^{-11}$s. The combination of P_2 properties allows us to state that this peak is similar to the Bordoni peaks in fcc crystals.
6. The mechanical spectroscopy data indicate the stability of the nanostructured state formed during cryomechanical treatment for a long time (7 years).