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Structure relaxation in thin filamentary germanium crystals

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In filamentary germanium crystals previously strained by torsion, the shape and structure return associated with reverse motion of screw dislocations towards the sources thereof have been revealed under action of thermal field and elastic one generated only by a constant uniaxial tensile loading. A change in mechanisms controlling the dislocation mobility and the plastic straining in the microcrystal has been found.

В предварительно пластически деформированных кручением одного знака нитевидных кристаллах германия при воздействии термического и упругого, порожденного только постоянной одноосной нагрузкой растяжения, полей обнаруживаются возврат формы и структуры, связанные с реверсивным движением винтовых дислокаций к своим источникам. Обнаружена смена механизмов, контролирующих подвижность дислокаций и пластическую деформацию в микрокристалле.

A solid surface is known to act as a dislocation source or as a dislocation runoff, or as a barrier for the dislocation motion [1–3]. This results in various surface effects [2–9] that influence considerably the mechanical properties of bulk and filamentary single crystals. The surface effects are as a rule dimensional, and it is just the filamentary crystals (FC) that are unique model samples to observe and study thereof [4, 5]. The two linear dimension being microscopic, the FC specific surface is one to three decimal orders larger as compared to the bulk crystals. In this connection, the peculiarities of plastic strain associated with the forward [4, 5] and reverse [10, 11] motion of dislocations interacting strongly with the free surface are seen most clearly. In the FC, the effect of dislocation post-action is more pronounced as compared to the bulk single crystals. The stress relaxation under annealing of plastically strained FC is due mainly to the plastic shape changes in the near-surface layers and is associated with the motion of dislocations interacting strongly with the free surface. These phenomena are universal in character and are observable in FC with both low [12, 13] and high Peierls barriers after the previous mechanical straining by torsion [13, 14], bending [4, 12], and tension [4, 5]. This unique property of FC makes it possible to study the thin structure of kinetics and energy characteristics of the processes [4–11], and to discriminate the contributions of dislocations interacting strongly and weakly with the free surface to the plastic shape change [4, 5], all that using one and the same sample.

The growing, selection, the study of shape and structure return and internal friction (IF) in the p-Ge<111> FC were carried out according to procedures described in [10–15]. As the samples, Ge FC with the growth axis oriented along <111>, of 3 to 15 μm in dia. and 1.5 to 3.0 mm working length, the conicity being less than 10^{-3}. The torsion angle measurement error at the return was less than $1'$, that of temperature, ±5 K. The axial stresses at the constant tensile loading were 10^4 to 10^5 Pa. The previous plastic straining by uniaxial torsion was carried out at a rate of

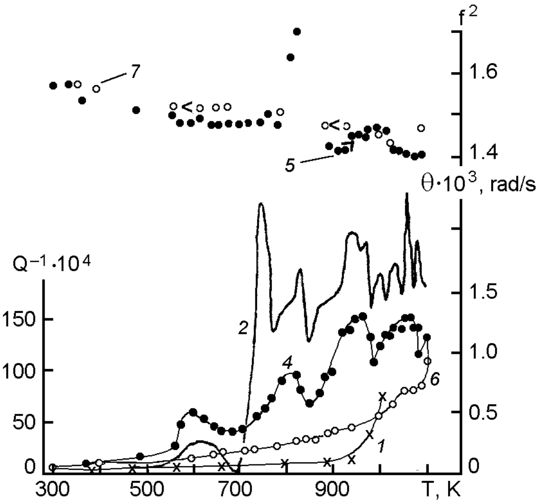

Fig. 1. Temperature dependences of Q^{-1} (*1*, *4*, *6*), $\dot{\theta}$ (2), f^2 (*5*, *7*) for a Ge <111> FC strained plastically by torsion. D = 8 μm, γ = 0.035 %, isochoric annealing rate 0.1 K/s.

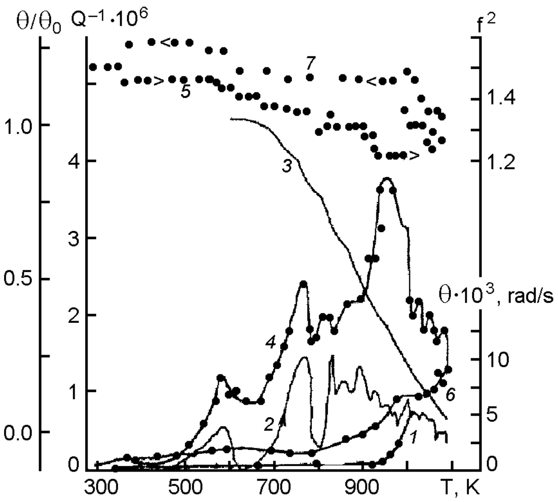

Fig. 2. Temperature dependences of Q^{-1} (*1*, *4*, *6*), $\dot{\theta}$ (*2*), θ/θ_0 (*3*), f^2 (*5*, *7*) for a Ge <111> FC strained plastically by torsion. D = 8 μm, γ = 0.12 %, isochoric annealing rate 0.1 K/s.

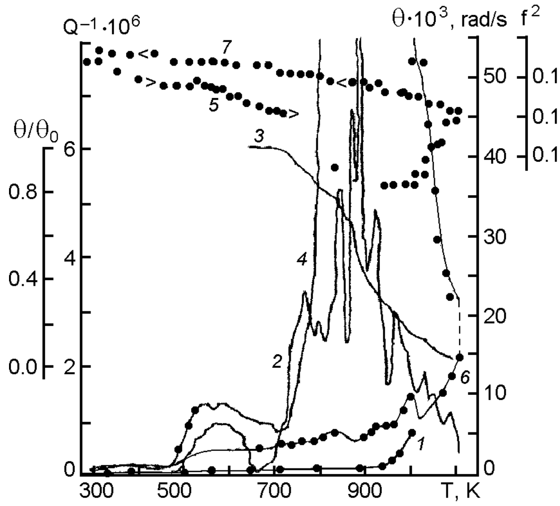

Fig. 3. Temperature dependences of Q^{-1} (1, 4, 6), $\dot{\theta}$ (2), θ/θ_0 (3), f^2 (5, 7) for a Ge <111> FC strained plastically by torsion. D = 8 μm, γ = 0.42 %, isochoric annealing rate 0.1 K/s.

$5{\cdot}10^{-5}$ s^{-1} at 1000 K and can be repeated multiply. The FC was twisted through a preset angle θ_0 by introducing dislocations of the same sign therein, starting from the surface, preferably the screw type ones [4]. Then, the stressed sample was cooled down to room temperature, thus providing the freezing of dislocations. The pre-strained dislocation structure was controlled by the IF method either immediately within the experimental setup or by x-ray diffraction (XRD), selective etching, and metallography. The uniaxial constant tensile loading due to torsional micropendulum generates the uni-sign stresses σ_p with the long-range stresses σ_d in the dislocation accumulations and the image force field stresses σ_l. The total stress $\sigma_c = \sigma_p + \sigma_d + \sigma_l$ favors the reverse dislocation motion towards the surface sources and annihilation thereof, thus making it possible to study the kinetics and dynamics of the dislocation ensemble decay within the FC volume that was generated due to the previous plastic straining by torsion. The shape and structure return of the pre-strained FC was judged from its detwisting angle θ under isochoric annealing at a rate of 0.1 to 0.5 K/s.

The high crystal structure perfection in the FC selected to be studied is evidenced by the temperature dependences $Q^{-1}(T)$ shown in Figs. 1–3 (curves 1). A specific feature of the initial thin Ge FC consists in the low (1 to $5{\cdot}10^{-5}$) $Q^{-1}(T)$ background value, absence of any peaks in the $Q^{-1}(T)$ and squared frequency $f^2(T)$ (not shown) within a wide (300 to 900 K) temperature range. These experimental facts agree well with the previous studies [4–11] and evidence the absence of defects able to dissipate appreciably the elastic vibration energy in the initial FC. Above 900 K (curves 1), the $Q^{-1}(T)$ and $f^2(T)$ dependences for initially dislocation-free FC show anomalies that indicate the microscale plasticity appearance under the axial tensile loading (10^4 to 10^5 Pa) and the alternate-sign vibrations generated by the torsion micropendulum of the IF measuring unit.

In the $Q^{-1}(T)$ dependences for Ge FC pre-strained by torsion, some singularities are seen in the 300 to 900 K temperature range (see curves 4 for the forward run and 6 for the reverse one) as compared to the initial samples (curves 1). Moreover, the thin structure of the return rate $\dot{\theta}$ (curves 2) has been studied for previously unstrained samples within the 300 to 1100 K range. The full return dependences $\theta/\theta_0(T)$ have been obtained (curves 3; not shown in Fig. 1). A good correlation of anomalies is established in the full $Q^{-1}(T)$ (curves 4) and $\dot{\theta}(T)$ (curves 2) dependences. Three temperature ranges have been discriminated conventionally where the activation volumes and energies have been measured for the return and plastic shape change processes in the FC caused by the reverse dislocation slip towards the sources thereof. The experimental data are summarized in the Table. The calculated Peierls stress (σ_p) for Ge FC has been found to be of about $2{\cdot}10^9$ Pa.

The experiments have shown that the dislocation mobility during the reverse motion towards the surface sources is influenced, along with the imaging forces, by the dislocation interaction forces within the accumulation and the internal stresses due to the axial loading. The structure studies using the IF and XRD have shown that the dislocation density is lowered during the annealing, however, no full structure return is attained in most cases. This is evidenced by the data obtained using XRD, selective etching, and IF. The Ge FC pre-strained plastically by torsion at $T < 0.7T_m$ revealed neither the plastic strain localization nor a neck even after a high-temperature ($>0.8\ T_m$) and de-twisting through several radians.

In Ge FC pre-strained plastically by torsion through $\gamma \leq 1$ % at $T \sim 0.7T_m$, neither the shape return (de-twisting) nor the IF changes were revealed at room temperature for a prolonged period ($\geq 2{\cdot}10^6$ s). This indicates that at 300 K, the dislocations in Ge FC are "frozen" and do not contribute to the elastic vibration damping. At higher temperatures, the shape return is started at shorter observation times. The onset temperature of the plastic shape change (T_n) depends on the FC geometric size and on the pre-straining extent. The T_n coincides with the onset temperature of the Q^{-1} increase and the f^2 decrease. The highest dislocation return rate $\dot{\theta}$ answers to the temperature position of the IF peaks. As the initial FC shape is recovered at high temperatures, the initial state of Q^{-1} and f^2 becomes returned as well as the sample structure that is judged from the decreased asterism of the Laue spots. After about 95 % recovery of the initial FC structure, essentially no peaks are observed in the $Q^{-1}(T)$ curves, however, there is no full return of Q^{-1} and structure. This is caused by that the FC is subjected to axial tension and high temperature in the experimental conditions that favor the dislocation generation in individual local microvolumes due to the internal stress relaxation. However, a complete return of the crystal structure is revealed in fully unloaded FC at temperatures close to T_m.

Table. Energy characteristics of dislocational shape return of a Ge <111> FC

T, K	H, eV	$9{\cdot}10^{22}$, cm^3
400–650	1.0–1.5	3.0–13.0
700–850	1.7–2.4	8.0–27.0
850–1100	2.5–4.1	27.0–140.0

The possibility of an essentially complete shape return in thin FC makes it possible to use repeatedly one and the same sample to study Q^{-1}, $f^2(T)$, $\dot{\theta}(T)$, etc., under different conditions of the plastic pre-straining, what is of a special importance in accumulation of statistical data on variations of a physical quantity for the samples with the same impurity content.

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Структурна релаксація у тонких ниткоподібних кристалах германію

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У ниткоподібних кристалах германію, попередньо пластично деформованих крутінням одного знаку, під впливом термічного та пружного, породженого тільки постійним одновісним навантаженням розтягу, полів виявлено повернення форми та структури, пов'язане з реверсивним рухом гвинтових дислокацій до своїх джерел. Виявлено зміну механізмів, що контролюють рухомість дислокацій та пластичну деформацію у мікрокристалі.