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Electroplastic effect associated with the dislocation generation in the initially dislocation-free silicon filamentary crystals

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Experiments on local high-intensity electric current pulse action on mechanical properties of a Si filamentary crystal made it possible to reveal a novel type of electroplastic effect associated with the generation of dislocations.

Эксперименты по локальному высокоинтенсивному воздействию импульсом электрического тока на механические свойства нитевидных кристаллов Si позволили обнаружить новую разновидность электропластического эффекта, связанного с зарождением дислокаций.

The electroplastic effect is known to be manifested as an additional plastic strain stimulated by electric current [1]. The effect is manifested in macroscale only at high stresses near to the easy yield and in the crystals [1–5] that show a considerable yield (several per cent and even several tens per cent) also in the absence of the current. As to dislocation-free, elastically strained and low-plastic crystals, the effect was not observed up to now, most likely due to its low value [4].

The bulk Si crystals are essentially brittle at room temperatures. This seems to be the reason why the elecrtoplasic effect did not observed therein at 300 K, in spite of numerous attempts to subject the samples to both constant and pulse currents at the density values ranging from the measuring to destroying ones [6].

In filamentary Si crystals or whiskers (FC), the macroscopic electroplastic effect phenomena was revealed first only in special experimental conditions at high temperatures (about 1050 K) under relatively high tensile loads (about 0.5 to 100 MPa) and constant currents of 1 to 10 A/mm^2 density [7]. In such conditions, the Si FC show an appreciable plasticity in the no-current state. Recently, the first results have been reported on the effect of a series of current pulses and axial tensile loading on the Si FC structure and properties. The purpose of this work is to obtain further data on the study conditions and procedure as well as on the electroplastic effect nature and properties in semiconductors under action of single current pulses.

As the experimental samples, used were the initially dislocation-free p-Si <111> FC of about 2 to $7 \cdot 10^{-2}$ mm in dia. and about 3 to 5 mm length with a low conicity (about 10^{-3}) shaped as a regular hexagon in the cross-section. The FC were grown in a quartz ampoule from Si of KDB and EKDB grades using the gas transport chemical reaction [9]. In the course of growing, the FC were doped with Pt, Au, B, In, and compounds thereof up to preset specific resistance values. The experimental samples and controls were prepared from one and the same FC by subdividing it mechanically into several pieces. Thus, the samples with an essentially the same impurity content were obtained. As the controls, the initial samples were used as well as the FC subjected

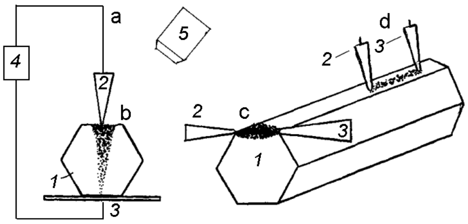

Fig. 1. Schematic view of setup for subjecting a FC to single electric pulses (a) and the current pulse passage schemes (b—d).

to a mechanical action by external stress of 1.0 to 10.0 MPa. The structure of the initial and treated samples was examined using X-ray diffraction (XRD), etching, metallography, internal friction, and rotary creep by techniques described in [10]. To study the electric current effect on the structure and properties of the samples, the Si FC were included into electric circuit by creation of ohmic contacts as described in [10], the current guides being made of Pt microwire of 17 to 25 μm in diameter.

The experimental setup is shown schematically in Fig. 1a. The Si FC, 1, is placed between two electrodes, 2 and 3, made of graphite or another material that does not contaminate substantially the FC. Each electrode can be moved along three orthogonal coordinate axes by manipulators and can be brought into contact with the FC surface in any preset point, thus providing a constant mechanical loading or a cyclic one and the sample inclusion into closed electric circuit. Another butt of each electrode 2 and 3 is connected to the controlled power source 4. The voltage V on the electrodes 2 and 3 is measured by a voltmeter at an error of about 0.1 %; the current I by a milliampermeter at an error of ≤1 %; the resistance, to within about 0.1 %, the instruments being integrated into the setup. The preparations and monitoring of the electric pulse action are carried out using an optical microscope 5.

The experimental samples were subjected to single electric pulses at 300 K (see Fig. 2) flowing perpendicularly (b, c) or in parallel (d) to the growth axis near the FC surface (c, d) or in its volume (b), with or without a constant (b–d) or cyclic (b) mechanical stress of 1 to 10 MPa. The electric voltage without the mechanical action was 1 to 20 V, thus providing the pulse current values of $0.02 \le I_i \le 0.6$ A and the respective current densities of $20 \le j_i \le 700$ A/mm^2.

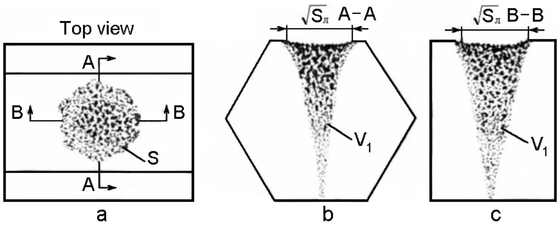

Fig. 2. Surface relief distortion (a) and the dislocation cluster shapes (b, c) in the current channel in a p-Si<111> FC.

At the side facet of the Si FC, in the contact zone, an artificial point surface defect has been revealed accompanied by local changes in the surface relief topography. The area, S_l, of the locally distorted free surface amounted several tenths per cent to several per cent of the side facet area, S_{fc}, and depended on the experimental conditions. Using the selective etching, the etch pits have been found at the side facet of the Si FC in the artificial point surface defect localization zone. The pits have been identified as dislocation ones. This is confirmed by the following experimental facts. No pits were observed prior to the current pulse action, since only initially dislocation-free samples were selected. After the pulse action, the etching pits were localized only within the artificial defect zone while those were absent on the rest of the whole side facet. A gradient of the etch pit density was observed from the defect periphery to its center. The pits were arranged in rows oriented along the slip traces. The pits itself had the shape and orientation corresponding to the dislocation ones. Using the alternate polishing and selective etching, a gradient of the pit density along the FC radius was revealed, the maximum density being observed at the FC surface and decreasing in its depth. This fact evidenced that under the current pulse, along with the surface defect S_l (a), the crystal structure became distorted within a microvolume V_l (b, c) that is localized under the surface defect and is a dislocation cluster with a density gradient. The dislocation cluster microvolume V_l (b, c) showed an elongated shape oriented mainly along the current flow channel. The microvolume geometry, size, shape, orientation, etc. depend substantially on the temperature, the characteristics of FC, electrode, and the current pulse. As the current density was decreased, the size of the crystal structure deterioration zone both at the FC surface and in the volume was diminished.

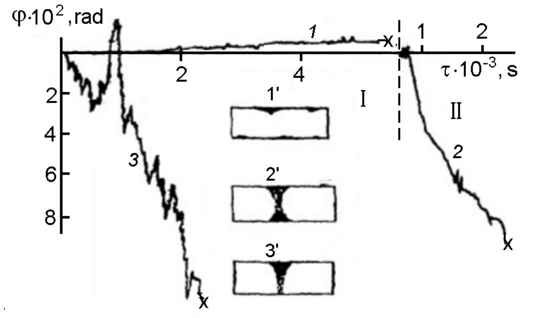

Fig. 3. Kinetic curves of the rotary creep component under uniaxial tension in a p-Si<111> FC and temperatures (K): 1350 (*1*), 1300 (*2*, *3*). d = 21 μm. $<\sigma_p>$ = 6.4·105 Pa. "x" denotes the sample failure.

The XRD structure examination has shown the presence of a strongly distorted region, in contrast to the initially dislocation-free FC, thus confirming the supposed dislocation cluster formation. The annealing at about 1070 K during about 10 h results in a partial structure recovery while the further anneal at 1270 K for about 15 h favored the essentially complete structure recovery. Thus, the dislocation in the cluster have been believed to be mobile.

The additional data on the structure and properties of the formed dislocation cluster were obtained using the rotary creep method (Fig. 3) that is highly sensitive to the structure imperfections. In the current-treated FC (curve 3), unlikely the initial dislocation-free ones (curve 1), the rotary creep onset is shifted towards lower temperatures by 30 to 50 K. Moreover, the experimental creep curves $\varphi(t)$ of FC containing an artificial defect show neither incubation period (the creep onset delay t_z) nor the stopping creep region typical of the initial FC (curve 1, scheme 1′). The $\varphi(t)$ dependence for the current-treated FC containing local dislocation cluster (scheme 3′) reflects the developing continuous creep process that finishes with the FC destruction. This agrees well with the $\varphi(t)$ dependence (curve 2) for the samples containing a local dislocation cluster (scheme 2′) that is generated by the combined effect of the temperature and mechanical load in the absence of electric pulse. This fact allows to conclude that the dislocations generated by the combined effect of the current, temperature, and mechanical load affect the FC plasticity in the same manner as those generated by the combined thermal and mechanical action [11]. The anneal of the current pulse treated FC under absence of external stresses favors the essentially complete recovery of its mechanical properties. These experimental results were confirmed by the internal friction examination (Fig. 4).

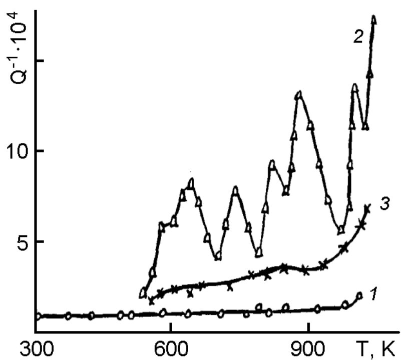

Fig. 4. Temperature dependences of internal friction in Si FC. Samples: initial, dislocation-free (*1*), pretreated withelectric pulses (dislocation generation) (*2*), after high-temperature annealing and partial dislocation output (relaxed structure) (*3*). d = 21 μm, γ_0 = $4.4\cdot10^{-5}$, $<\sigma_r>$ = $9\cdot10^5$ Pa, f = 4,6 Hz.

In Si FC loaded (by bending, torsion, tension) at 300 K, a plastic strain jump is clearly seen at the moment of the current pulse passing. Since the initial FC is dislocation-free, the observed strain jump can be explained only under assumption that a dislocation is generated in the elastically strained sample. In this connection, the observed plastic strain jump is an experimental fact of considerable importance that not only confirms the effect of the electrically stimulated defect generation but also evidences a novel type of electroplastic effect that was not observed before. The electroplastic effect becomes more pronounced as the load or temperature rises, or under combined action of both. The plastic strain jump can be supposed to be associated with the collective injection of dislocations from the FC surface into the subsurface layers at the current pulse action instant [12–15]. The effect has been found to depend also on the pulse current density j_i as well as on other experimental conditions. For all the Si FC exhibiting the plasticity jump, a threshold voltage V_p (and thus the pulse current density) below which no effect is observed. The effect value is increased in parallel with V_p and the V_p value is individual for each FC. This evidences the statistical distribution of defects responsible for the effect nature in the FC.

To explain the effects observed, let the processes be considered occurring at the contact site of the metal (Pt) electrode with the Si FC. First let the strain be considered in the contact area due to interaction of the solid surfaces being in contact. Let R be the curvature radius of the Pt electrode. As a result of attraction forces between the surface atoms of the electrode and FC, a contact zone of the radius r is formed surrounded by the elastic strain field. The strain can be estimate under assumption that a dislocation is formed in the contact area having the Buergers vector

$$\mathbf{b} \sim r^2/R. \qquad (1)$$

The characteristic values of elastic strains $\mathbf{u}$ and stresses τ in that zone are

$$u \sim \mathbf{b}/r, \ \sigma \sim \mu \cdot \mathbf{b}/r, \qquad (2)$$

respectively, where μ is the effective elastic modulus of the system in contact. Taking into account that the shear moduli of Si and Pt are approximately the same, μ can be adopted to be equal to that of Si. The elastic strain U in the contact zone is

$$U \sim \sigma \cdot u \cdot r^3 \sim \mu\mathbf{b}^2 \cdot r = \mu \cdot r^5/R^2. \qquad (3)$$

The system surface energy within the contact zone is decreased, the decrease can be estimated as

$$U_\sigma = -\pi r^2 \cdot \sigma, \qquad (4)$$

where σ is the surface energy density difference between the crystals being not in contact and those in contact. To find the contact zone radius r, the minimum total energy is to be determined. We obtain

$$r = (\sigma \cdot R^2/\mu)^{1/3}. \qquad (5)$$

Assuming $\sigma \sim 0.1\ \mu{\cdot}a$ (a being the lattice constant), we obtain

$$r \sim (0.1a \cdot R^2)^{1/3} = 10^3 a, \qquad (6)$$

where R is taken to be about 1 μm. The obtained contact zone radius is one decimal order smaller than the observed distorted relief area on the FC surface. It can be shown, however, that when the electrode comes into contact with the crystal, dislocation loops arise in the latter. To that end, let the characteristic strain in the contact zone be determined:

$$u \sim (\sigma/\mu \cdot R)^{1/3} \quad \sim 10^{-2}. \qquad (7)$$

We obtain for the stress τ

$$\tau = \mu \cdot u \sim 5 \cdot 10^8 \mathrm{Pa} \qquad (8)$$

that exceeds the experimental value of the plastic staining stress of the FC. Thus, dislocation loops arise in the FC under contact with the metal electrode. The number of those loops seems to be rather small and those cannot go out of the crystal after the electrode is removed.

It is to note that a similar contact mechanism of the metal electrode and the FC can result from the Schottky layer formation. This layer is due to a partial electron transition from the electrode to the contact zone of FC. This effect, however, must be similar to that considered above in the order of magnitude.

Another mechanism of elastic stress formation in the contact zone is based on the electric field formed in the FC under an external stress. The electric field strength is

$$E = V/r, \qquad (9)$$

where V is the electric voltage. The electric field pressure, τ, on the FC surface is

$$\tau = E^2/8\pi = V^2/8\pi r^2. \qquad (10)$$

The total (electric plus elastic) energy in the contact zone is

$$U = \frac{\mu \cdot r^5}{R^2} - \frac{V^2}{8\pi \cdot r^2}\upsilon, \qquad (11)$$

where υ is the volume compression of the FC under electric tension pressure. We can assume that

$$\upsilon \sim \pi \mathbf{b} r^2. \qquad (12)$$

By differentiation of the (12) for r, we obtain

$$r = (V^2 \cdot R^2/\mu)^{1/3}. \qquad (13)$$

Substituting the numerical values $V = 10$ V and $\mu = 7{\cdot}10^{10}$ Pa into (13), we obtain $r \sim$ 1 μm coincident in the order of magnitude with the distorted surface area of the FC observed in the experiment. The elastic stress caused by the electric field tension is estimated as

$$\tau = (V^2 \cdot \mu^2/R)^{1/3} \sim 0.1\mu. \qquad (14)$$

These high values show that the electric field is sufficiently strong to be the source of dislocations resulting in the field-induced plastic properties.

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Електропластичний ефект у початково бездислокаційних ниткоподібних кристалах кремнію, пов'язаний з генерацією дислокацій

А.П.Єрмаков, Б.М.Даринський, А.І.Дрожжин

Експерименти з локального високоінтенсивного впливу імпульсом електричного струму на механічні властивості ниткоподібного кристалу кремнію дозволили виявити новий різновид електропластичного ефекту, пов'язаного з зародженням дислокацій.