HIGH-TEMPERATURE CREEP OF SILICON SINGLE CRYSTALS

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HIGH-TEMPERATURE CREEP OF SILICON SINGLE CRYSTALS

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According to all studies, the creep of homeopolar crystals (see, for example, ([1–3])) differs fundamentally from the creep of metals: whereas for the former the initial segment of the creep curve is characterized by an incubation period and a gradual increase in the deformation rate, followed by a stationary stage, which is replaced by a creep stage with a continuously decreasing rate, for the latter (see ([4,5])), on the contrary, the deformation first slows down, then a stationary stage occurs, and afterward there begins a stage of accelerated creep, ending in fracture of the metal. In the first case, the creep rate is determined directly by the resistance of the lattice to the motion of dislocations, and only in the last stage is it affected by dislocation interactions, whereas in metals this effect already determines the creep rate in the first stage. The question naturally arises whether the difference between the creep of homeopolar and metallic crystals is connected with the different type of interatomic bonds in them, or whether it is explained by the fact that, in the case of homeopolar crystals, only the initial stage of the process has been studied. In the present work an attempt was made to obtain an answer to this question.

Creep tests were carried out under conditions of uniaxial compression along the $\langle 111 \rangle$ direction in an argon atmosphere at temperatures of $900 \div 1300^\circ$ and stresses of $2 \div 10$ kg/mm^2, on specimens in the form of a rectangular parallelepiped ($6 \times 3.4 \times 2.6$ mm^3), prepared from an n-type ingot with a dislocation density of the order of 10^3 cm^{-2}. The specimens were first polished mechanically and then, to remove the work-hardened layer (100 μ), chemically in a mixture of hydrofluoric (1 h) and nitric (7 h) acids. The accuracy of load measurement was 10 g, and that of deformation $5 \cdot 10^{-5}$. During each experiment the temperature change did not exceed 10°, and the change in stress was no more than 2%.

Before loading, the specimens were heated for one hour at the test temperature. After the test was completed they were cooled under load in order to avoid redistribution of dislocations. The dislocation structure was studied by transmission in a JEM-150 electron microscope on thin silicon films prepared by the method described in ([6]), and by the method of selective chemical etching in a solution of chromium trioxide (1 h), distilled water (1 h), and hydrofluoric acid

Fig. 2. Boundary of misoriented blocks (steady-state stage of creep)

Figure 1: Fig. 2. Boundary of misoriented blocks (steady-state stage of creep)

Fig. 1. Creep curves of silicon single crystals. Temperature 1100°. Stresses 2 kg/mm^2 (1 and 2; curve 1 characterizes the early stages of creep) and 10 kg/mm^2 (3). The cross denotes fracture of the specimen.

Figure 2: Fig. 1. Creep curves of silicon single crystals. Temperature 1100°. Stresses 2 kg/mm^2 (1 and 2; curve 1 characterizes the early stages of creep) and 10 kg/mm^2 (3). The cross denotes fracture of the specimen.

(2 h). To obtain reliable data on the regularities of creep in silicon crystals, the results obtained on several specimens deformed under identical conditions were averaged.

Figure 1 shows typical creep curves of silicon for the investigated range of temperatures and stresses. At low stresses, creep of the specimens initially developed in a manner analogous to the data of works ([1–3]) (curve 1), but then, in contrast to those works, the deformation rate became constant, and for the overwhelming part of the time the specimens deformed at this rate (curve 2).

The duration of the characteristic portions of the creep curves decreased with in–

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Fig. 2. Boundary of misoriented blocks
(steady-state stage of creep)

with the magnitude of the applied stress and the test temperature. At sufficiently high stresses the initial portions of the process disappeared, and the creep curve became similar to the creep curves of metals: the deformation of the specimens under loading was followed by unsteady, steady-state, and accelerated stages (curve 3). The process ended with fracture of the specimen. The deformation in the steady-state stage was usually 3–8%. Structural studies showed that the initial portions of creep are characterized by a rapid transition from the motion of individual dislocations in intersecting slip planes to a cellular structure formed by tangled dislocation networks with numerous dislocation dipoles and loops in the region of retarded creep. Steady-state creep of the specimens corresponded to a disoriented block structure (Fig. 2, see insert facing p. 559) with a large number of hexagonal dislocation networks forming block boundaries. The block size was about 2μ. The dislocation density within the blocks was of the order of 10^8 cm^{-2}. According to (7, 8), this structure is similar to the structure of metals deformed in the steady-state stage of creep at moderate temperatures.

Fig. 1. Creep curves of silicon single crystals. Temperature 1100°. Stresses

2 kg/mm^2 (1 and 2; curve 1 characterizes the early stages of creep) and 10 kg/mm^2 (3). The cross denotes fracture of the specimen.

Thus, the creep curve of homeopolar crystals described in the literature is far from complete. It should be supplemented by two stages, one of which proceeds at a constant rate and is the longer of the two, while the other is characterized by accelerating deformation and ends in fracture of the material. The portion of constant deformation rate at the beginning of the process (see Fig. 1, curve 1), interpreted in works on the creep of homeopolar crystals as a steady-state stage, is not such a stage, but is a constituent part of unsteady creep. In this short portion the dislocation structure changes sharply. At the same time, throughout the second portion of constant deformation rate the characteristic block structure remains unchanged. It is precisely this portion that corresponds to the steady-state stage of creep. Consequently, typical creep curves of homeopolar crystals and of metals are qualitatively similar, except for the features at the very beginning of the process.

The change in the rate of steady-state creep ($\dot{\varepsilon}$) of silicon single crystals as a function of the test conditions is illustrated in Fig. 3. It is seen that, in semilogarithmic coordinates, the dependence of the rate $\dot{\varepsilon}$ on the reciprocal temperature $1/T$ is represented by a family of straight lines, each corresponding to a definite value of the external stress. Extrapolation of these straight lines to $1/T = 0$ showed that they converge at $\lg \dot{\varepsilon}_0 \approx 11$.

From the results obtained it follows that, in the investigated range of external conditions, the steady-state creep rate of silicon obeys the kinetic equation

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp\left(-\frac{U_0 - v\sigma}{kT}\right). \tag{1}$$

In this equation the height of the energy barrier is $U_0 = 5.6$ eV; the activation volume is $v = 2.7 \cdot 10^{-21}$ cm^3; σ is the external stress. It is known ([9], [10]) that an analogous dependence describes the steady-state creep of many metals under uniaxial tension in the region of moderate temperatures and elevated stresses. Apparently, the indicated commonality in the description of the steady-state creep of silicon and metals is due to the similarity of the structural state of these materials.

In ([9], [10]) the conclusion was drawn that the activation energy U_0 corresponds to the sublimation energy. In ([11]) it is noted that the value of U_0 is close both to the sublimation energy and to the activation energy of certain mechanisms of plastic deformation associated with the interaction of dislocations at block boundaries. In the case of silicon, the parameter $U_0 = 5.6$ eV is comparable both with the sublimation energy (4.87 eV ([12])) and with the activation energy for self-diffusion of vacancies (5.13 eV ([13]))*, but it considerably exceeds the barrier for the motion of dislocations in the Peierls force field (2.2 eV ([14])). An estimate analogous to that performed in ([11]) showed that the barriers impeding

Fig. 3. Temperature dependence of the rate of steady-state creep of silicon at stresses of 2 kg/mm^2 (1), 4 (2), 6 (3), 8 (4), and 10 kg/mm^2 (5)

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the motion of dislocations are associated not with dislocations inside blocks, but with dislocations of interblock boundaries. Thus, the steady-state creep of silicon cannot be limited by the motion of dislocations in the force field of the crystal lattice (as occurs at the beginning of the process), but is determined by the collective interaction of dislocations at block boundaries, apparently by the same mechanism that is responsible for the steady-state creep of metals at moderate temperatures.

Fig. 3. Temperature dependence of the rate of steady-state creep of silicon at stresses of 2 kg/mm^2 (1), 4 (2), 6 (3), 8 (4), and 10 kg/mm^2 (5).

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* Elucidation of the physical nature of the energy barrier U_0 is the subject of further investigations.

Note: Figure translations are in progress. See original paper for figures.