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**ABSTRACT:** The results of an experimental check of the assumptions underlying the theory of diffusional creep [1, 2] are presented (i. e., the assumptions that a small viscosity coefficient exists at the boundaries and that the boundaries are effective as vacancy sources and sinks). Direct measurements of boundary slip and comparison of the profiles of bent specimens with those predicted by the theory indicated that the above assumptions are valid for only a few (approximately 20%) of the crystallites. This means that purely diffusional creep is not the dominant mechanism involved in high-temperature straining. However, our results do not exclude the possibility of quite effective directional self-diffusion in the case of a few grains with what appears to be the most favorable orientations relative to their neighbors. The shape of the  $\epsilon$ - $\sigma$  curve seems to indicate that the principal creep process involved, under these conditions, is that of dislocation migration.

Straining as a result of directional self-diffusion can proceed successfully only upon fulfillment of the following two conditions.

1. As was shown by Lifshits [3], in the general case of a crystal with grains of arbitrary shape, diffusional creep is necessarily accompanied by slip along the crystallite boundaries. Hence, the macroscopic viscosity coefficient (or tensor, in the case of structural anisotropy) breaks down into the two components  $\eta_0$  and  $\eta_1$ , the first of which is determined by diffusional processes within the grains, while the second is related to the properties of the boundaries. If the boundaries behave as a fluid layer with the viscosity coefficient  $\eta_b$ , then  $\eta_1$  is of the form  $\eta_1 = \eta_b l/a$ . Here  $l$  is the grain size and  $a$  is the thickness of the "amorphized" boundary layer which is on the order of the interatomic distance, in the case of pure metals.

In estimating the diffusional creep rate, the term allowing for resistance to boundary slip is usually neglected by assuming that

$$\frac{\eta_1}{\eta_0} = \frac{\eta_b l}{\eta_0 a} \ll 1. \quad (1)$$

It is uncertain whether this condition is fulfilled in reality. If inequality (1) does not, in fact, hold, creep must be restricted, not only by volume self-diffusion, but also by boundary slip. The creep rate can then be much smaller than the expected value.

2. The Nabarro-Herring theory [1, 2] involves the assumption that  $\eta_0$  is determined solely by the vacancy diffusion parameters in the crystallite bodies and not by the rate of vacancy formation at the boundaries. It is uncertain whether the boundaries between arbitrarily oriented crystallites satisfy this requirement, i. e., whether they are capable of absorbing and emitting an infinite number of vacancies without experiencing any alteration in their structure.

**The experiment.** We carried out our experiment on a material with a microstructure such that the diffusional straining of the grains and boundary slip could proceed independently. This enabled us to measure both  $\eta_0$  and  $\eta_1$  directly.

The specimens took the form of industrial copper rods whose transverse cross sections measured  $140 \times 100 \mu$ . The specimens were first annealed at  $1060^\circ \text{C}$ , after which the individual grains occupied the entire transverse cross section of the specimen (the specimens acquired a "bamboo-like" structure).

The grain size was  $200 \mu$ . Both the annealing and subsequent testing were conducted in a helium atmosphere. The test temperature was  $1040 \pm 3^\circ \text{C}$ . Two series of tests were carried out. In the first series we investigated creep under tension. The working length of the specimens was 60 mm. The applied stresses lay in the  $(2-20) \cdot 10^5 \text{ dyn/cm}^2$  range. A correction was made for surface tension, which was assumed to equal  $1800 \text{ dyn/cm}$  [4]. After 100 hr under tension we determined the total elongation of the specimens and the shear strains along the boundaries of individual grains. The accuracy of the measurements was  $\pm 1 \mu$ . The measurements were carried out only at boundaries with sufficiently large shearing stresses. Since boundary slip and diffusional

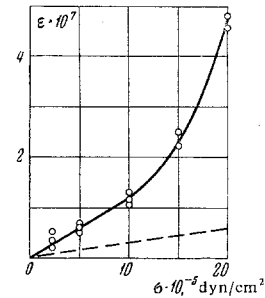


Fig. 1

straining in the case of "bamboo-like" specimens must proceed independently, we determined the quantity  $\eta_b/a$  appearing in (1) directly from the magnitude of the boundary slip by means of the expression  $\tau_s = \eta_b l/at$ . Here,  $\tau_s$  are the shearing stresses at the boundary,  $l$  is the boundary displacement, and  $t$  is the time.

In the second series of tests, we investigated the creep of specimens after sagging under their own weight. Specimens of length 14 mm were placed on two supports 12 mm apart. The maximum tensile or compressive stresses in the specimens were close to  $7 \cdot 10^5 \text{ dyn/cm}^2$ . The test period was 60 hr. After this time had elapsed, we determined the changes in shape of the specimens by means of an interferometer. By comparing the resulting experimental beam profile with the theoretical profile computed on the basis of the diffusional theory, we were able to test the validity of the second assumption underlying the latter.

**Evaluation of the role of boundary slip.** Figure 1 shows the measured creep rate under tension as a function of the applied stress. The dashed curve represents the same function as predicted by the diffusional model. The latter curve was constructed from the standard [1-3,5] expression

$$\dot{\epsilon} = N \frac{D\omega\sigma}{kTAL}, \quad (2)$$

where  $D$  is the self-diffusion coefficient,  $\omega$  is the volume of the atom,  $k$  is Boltzmann's constant,  $T$  is the absolute temperature, and  $A$  is the specimen thickness.

Because of its weak dependence on  $A/L$ , the coefficient  $N$  was assumed to equal that obtained in [5] for nearly cubical grains ( $N = 12$ ). The values of  $D$  were taken from [6]. The coefficient  $\eta_0$  computed from the above expression turned out to be  $3.2 \cdot 10^{13} \text{ P}$ .

We see from the figure that, for  $\sigma > 10^6 \text{ dyn/cm}^2$ , the relationship between the creep rate and the stress is described by a power law, so that the strain in this range is not described by the diffusional theory. For  $\sigma \leq 10^6 \text{ dyn/cm}^2$ , the creep rate, as a function of stress, agrees sufficiently well with theory, although the experimental values of  $\dot{\epsilon}$  exceed the theoretical ones more than threefold. All our measurements of the boundary displacements were confined to this linear range.

Our measurements indicated that the shear along most of the boundaries did not exceed the measurement error. The absence of visible

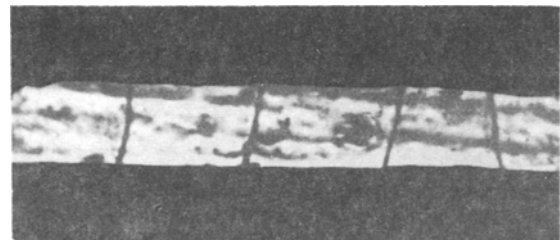


Fig. 2

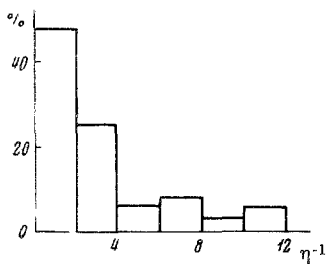


Fig. 3

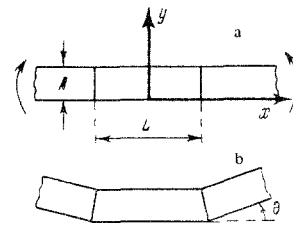


Fig. 4

slip is apparent from the photograph (Fig. 2) showing the microstructure of one of the specimens following creep. Slippage was distinctly evident only over a few of the boundaries and did not exceed  $6 \mu$  even for specimens subjected to  $\sigma = 10^6$  dyn/cm<sup>2</sup>. The minimum values of  $\eta_s/a$ , computed, with allowance for possible measurements errors, lay in the range  $(1.3-17.5) \cdot 10^{14}$  P/cm for various boundaries.

Figure 3 shows the relative number (%) of grain boundaries exhibiting this ratio of viscosity coefficients  $\eta^{-1}$  (plotted along the axis of abscissas). (For convenience of construction, we plotted not the ratio  $\eta = \eta_s L / \eta_0 a$  itself but rather the reciprocal quantity along the horizontal axis.) We see that condition (1) was fulfilled for not more than 20% of the boundaries, and that, for most of them, coefficients  $\eta_1$  and  $\eta_0$  were at least of the same order of magnitude. An even larger disparity between inequality (1) and experiment was noted when the experimentally determined  $\eta_0$  was used.

Thus, in the case of a massive crystal or arbitrary structure, the diffusional creep rate must be considerably smaller than the value obtained without allowance for resistance to boundary creep, even if condition (2) is fulfilled.

We note that our values of  $\eta_s/a$  were considerably in excess of the estimates given in [3, 5]. For example, Lifshits [3] assumes that if the grain boundary is a layer of amorphized material,  $\eta_s \sim kT/Da$ . Under our experimental conditions for copper, this would yield  $\eta_s/a \sim 7 \times 10^{10}$  P/cm, a value four orders of magnitude smaller than those actually obtained. The reason for these discrepancies apparently lies in the fact that, in estimating  $\eta_s$ , the authors of [3, 5] assumed that boundary slip can proceed independently of the straining of the crystallites themselves. On the other hand, Kennedy [7] discusses the numerous considerations which contradict this view and shows that the magnitude of the boundary shear strains is closely related to that of the strains experienced by the grains themselves.

The effectiveness of boundaries as vacancy sinks. In order to evaluate the effectiveness of grain boundaries as absorbers of vacancies or atoms, we must be able to separate the diffusional strain from all other strains in comparing experiment with theory. To this end let us consider the diffusional straining of a beam with grain size  $L$  and thickness  $A$  acted on by the moments  $M$ . The structure of the beam and the positions of the coordinate axes are shown in Fig. 4a. For simplicity we assume that the diffusional flows from the side surfaces are small, so that the straining is two-dimensional. In addition, we assume that the flow is steady. The redistribution of vacancies immediately upon application of the load is negligibly small.

Let us denote the expression  $\mu_a - \mu_* - \mu_0$  by  $\mu$ . Here  $\mu_a$  and  $\mu_*$  are the chemical potentials of an atom and a vacancy at some point of the crystal and  $\mu_0$  is the chemical potential of an atom in the unstressed crystal. If the intercrystalline layers are effective sources and sinks, the boundary conditions are

$$\mu \Big|_{y=A} = 0, \quad \frac{\partial \mu}{\partial x} \Big|_{x=0} = 0, \quad \frac{\partial \mu}{\partial x} \Big|_{x=L} = \psi \left(1 - 2 \frac{y}{A}\right).$$

Moreover,

$$\int_0^{1/2 A} \mu dy \Big|_{x=L} = -\frac{\omega \sigma_0 A}{4}, \quad \left(\sigma_0 = \frac{M}{V}\right).$$

Here  $\psi$  is some constant to be determined and  $W$  is the moment of resistance of the cross section. As is shown in [8],  $\mu$  satisfies the equation  $\Delta \mu = 0$  for real values of the stresses.

The solution of the equation implies that diffusional flows, which act to rotate one grain relative to another, arise in the beam (Fig. 4b). The angle  $\theta$  of this rotation is given by

$$\theta \approx \text{tg } \theta = N \frac{D \sigma_0 \omega t}{k T A^2}, \quad N = \frac{\pi^3}{8} \left( \sum_{n=1}^{\infty} \frac{1}{(2n)^3} \text{cth} \frac{n \pi L}{A} \right)^{-1} \quad (3)$$

For  $L \geq A$ , we have  $N \approx 25.8$ .

Thus, directional self-diffusion gives rise only to deflections along the boundaries but does not strain the grains themselves. This enables us to isolate the purely diffusional effects in the bending conditions from the strains occasioned by other mechanisms such as dislocation migration.

In our experiments, bending took place under the weight of the specimen alone, so that the stressed state in the central portions of the specimens sufficiently approximated the stressed state assumed in the above derivation. The value of this angle  $\theta$ , given by the theoretical formula for the conditions of the experiment, turned out to be  $1.9^\circ$ . The experimental error was  $0.2^\circ$ . Our measurements showed that  $\theta$  ranged from 0 to  $2.2^\circ$  for various pairs of grains. No noticeable rotation occurred at most of the boundaries. The proportion of grain pairs with an angle  $\theta$  considerably in excess of the measurement error bracket was approximately 25%.

The above results indicate that the conditions necessary for diffusional straining are not fulfilled for most crystallites. A considerable portion of the grain boundaries in our experiments could not be regarded as infinite nor could they be regarded as sufficiently effective vacancy sources and sinks.

#### REFERENCES

1. F. R. N. Nabarro, "Deformation of crystals by the motion of single ions," Reports of a Conference on the Strengths of Solids, Phys. Soc., London, 1948.
2. C. Herring, "Diffusional viscosity of polycrystalline solids," J. Appl. Phys., vol. 21, p. 437, 1950.
3. I. M. Lifshits, "The theory of diffusional-viscous flow of polycrystalline solids," ZhETF, vol. 44, no. 4, p. 1349, 1963.
4. M. C. Inman, D. McLean, and H. R. Tipler, "Interfacial free energy of copper-antimony alloys," Proc. Roy. Soc. A, vol. 273, p. 538, 1963.
5. G. B. Gibbs, "Fluage par diffusion dans les solides polycrystallins," Mem. Scient. rev. metallurg., vol. 62, no. 10, p. 780, 1965.
6. A. Kuper, H. Letaw, Jr. L. Slifkin, E. Sonder, and S. T. Tomizuka, "Self-diffusion in copper," Phys. Rev., vol. 96, no. 5, 1954.
7. A. J. Kennedy, "Processes of creep and fatigue in metals," Oliver and Boyd, Edinburgh and London, 1962.
8. K. K. Ziling, "Behavior of cadmium whiskers close to the melting point," PMTF [Journal of Applied Mechanics and Technical Physics], no. 3, p. 118, 1965.

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