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A cell theory for stage IV work hardening of metals and semiconductors

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Résumé. — Le durcissement dans les métaux cubiques à faces centrées et les cristaux de structure diamant est analysé dans un modèle cellulaire. Selon l'énergie des fautes d'empilement et la température, la guérison dynamique peut s'effectuer soit par glissement dévié, soit par montée. Les dislocations subsistantes provoquent un redurcissement de stade IV dont l'origine est double et correspondant à deux fourchettes de température avant que l'autre mécanisme dynamique de guérison achève le durcissement.

Abstract. — Work hardening of fcc metals and diamond structure crystals is considered in a cell model. Depending on stacking fault energy and temperature dynamic recovery can occur by cross slip or climb. The remaining dislocations cause a rehardening stage IV of two different origins and in two temperature ranges before the other dynamic recovery mechanism terminates work hardening.

1. Introduction.

The discovery of a new work hardening stage, called stage IV, which follows the well known stage III of dynamic recovery in metals and semiconductors, and is sometimes followed itself by another dynamic recovery stage V, has revived interest in work hardening theory [1-9]. The conditions under which a stage IV is observed differ greatly according to the temperature of deformation. There is a high temperature stage IV_H and a medium to low temperature stage IV_L ; the work hardening rates θ_{IV} are in the former case at least 10 times larger than in the latter. In copper single crystals stage IV_H has recently be discovered above 700 K, $\dot{\epsilon} = 2 \times 10^{-3} \text{ s}^{-1}$. [8]. In silicon and germanium single crystals the temperature has to be close to the melting temperature and the strain rate not too high in order for the work hardening rate to rise again in the course of uniaxial compression [10]. In metals and alloys on the other hand a stage IV_L appears only at temperatures below half the melting temperature and only at large strains which are attained in a torsion or compression test [4]. Such tests have been performed on a variety of polycrystalline metals and alloys in a number of recent theses by Alberdi [11], Hughes [12] and Rollet [13].

Due to the possible complications in a large strain experiment on a polycrystalline aggregate a number of « conventional » causes for a rehardening stage have partly been discussed [14] and discarded [4]. Such as :

grain refinement : Since the grain size decreases at large strains stage, IV_L might be due to grain boundary strengthening following the Hall-Petch relation. Several workers have found however that such a grain size effect vanishes at large strains ;

deformation bands as regions of slip inhomogeneity or plastic instability were not observed in connection with stage IV (see also [9]) ;

texture might change the active slip systems and therefore hardening. This was looked for in the semiconductor experiments and not found. In metals grains reorient to a stable texture, thus this effect should saturate and not lead to new hardening ;

the misorientation between cells or subgrains increases with strain. The corresponding boundaries could act as barriers to dislocation motion. Rollett finds no correlation between misorientation and hardening and considers the former to be a by-product of large strains ;

specimen shape changes were also studied as a possible course of extra hardening in metals and semiconductors, but found not to be responsible for a stage IV ;

second phase particles in overaged alloys provide a special case of stage IV_L hardening as these materials do not form the usual cells during stage III but a special particle-related microstructure. This case will not be considered further in this paper as also Rollett *et al.* [4] develop a separate model for hardening in these alloys ;

slip on non-compact planes such as $\{112\}$ and $\{113\}$ in copper is associated with stage IV_H by Anongba *et al.* [8]. Since in the model discussed below we associate θ_{IVH} with climb there is an element of non-compact slip as already discussed for « fanning slip lines » seen in Ni50%Co above 600 K by Pfaff [15]. Our model anyway is independent on the particular type of slip plane the dislocation uses.

We are left with the microstructure as developed during stage II straining as the only possible cause of stage IV hardening. Rollett *et al.* [4] start from the areal glide model of Kocks [16] and the « debris » left after extensive glide of this sort in stage II. Stage III is then introduced by a Voce-type of stress-strain behaviour which leads to an asymptotic saturation stress σ_{III_s} and an empirical work hardening law

$$\theta_{III} = \theta_{II}(1 - \sigma/\sigma_{III_s}).$$

This is interpreted by thermally activated dynamic recovery without identifying it with a particular dislocation process like cross slip which would lead to a saturation stress of the form

$$\ln \sigma_{III_s} / \sigma_{so} = \frac{kT}{A} \ln \dot{\epsilon}_0 / \dot{\epsilon}.$$

We would like to interpret stage IV in the following in the cell model of Nix *et al.* [2] and some recent modification of it. Rollett *et al.* [4] criticize the application of this model and a similar one of Prinz and Argon [1] to the large strain/low temperature stage IV_L in metals because of the climb contribution it contains in the basic equations for the evolution of the cell walls. We have in fact applied a simplified but analytically tractable form of the Nix model to the case of the stage IV_H and would like to investigate in the following its applicability to the low temperature stage IV_L . Essentially the cell model of work hardening as one based on two state

variables (dislocation densities) is then a complete description of the work hardening of pure fcc and diamond structure crystals — beyond stage I where cells do not exist yet.

2. Description of the cell structure.

Figure 1 shows edge dislocation walls of thickness L_c , spacing L_c which bound cells in one direction. They form the places at which new dislocation loops are nucleated and in which their edge components are finally deposited. The walls are somewhat transparent to incoming dislocations — at least in the form of a relay race of forced emission into the next cell as the slip lines or mean free paths of edge dislocations are 10 times longer than the cell dimension, L_c . L_c itself decreases with increasing flow stress as

$$L_c \approx 10 \mu b / \tau_c . \tag{1}$$

This is called the principle of similitude [17] and is derived from a minimization of the strain energy of finite dislocation walls [18] or is suggested by an analysis of dislocation trapping and annihilation on parallel slip planes [19]. The walls contain dislocation segments of length ℓ which can act as Frank-Read sources at a stress $\tau = Gb/\ell$. So $\ell = L_c/10$ and a whole train of dislocations, spaced ℓ apart, will leave one of the walls and traverse the cell before it meets the next wall. Some of the dislocations will push through it ; the last one perhaps being trapped in the wall and so on. Occasionally two such trains and their edge dislocations coming from opposite directions and wall sources will meet at a slip plane spacing d smaller than the passing distance $d_p = \frac{\mu b}{8 \pi (1 - \nu) \tau}$. In that case they will trap each other into dipoles which might trap further dislocations on parallel planes to the nucleus of a new dislocation wall between two existent ones. This is considered to be the mechanism of adjustment of the cell size to the increasing stress expressed in equation (1). If edge dislocations of opposite sign pass each other at a spacing $d < d_a \approx 10\text{-}20 \text{ \AA}$ they collapse into rows of vacancies (« debris »)

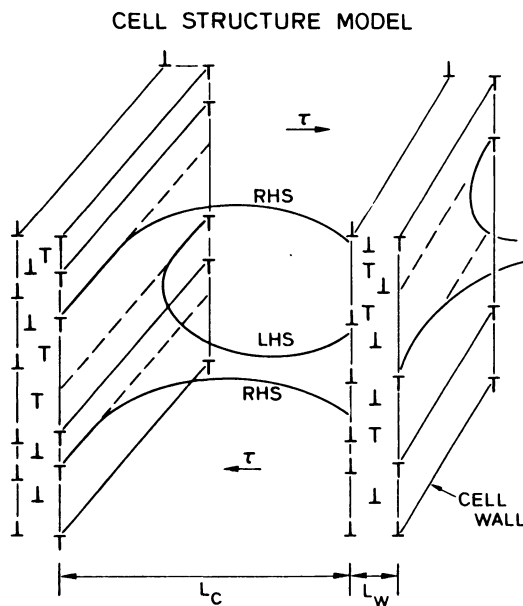


Fig. 1. — Cell structure model of stage IV (2D) after [2, 18].

[20] and are annihilated. We consider the debris as the result of a process limiting the edge dislocation density in the walls while Rollett *et al.* [5] assume that it causes stage IV_L hardening altogether.

The model of figure 1 does not describe 3D-cells yet which are now introduced as proposed by Nix [21] in figure 2. Different slip systems operate in neighbouring cells so that their screw components form stable cross-grids parallel to the plane of the paper while the edge components 1 and 2 run over distances $\Lambda_1 = \Lambda_2 = \Lambda$ in the 45° directions in this plane. The slip distance of the screw is $\Lambda_s = L_c$. Their « walls » are sharp and have a small internal stress field relative to the edge walls which are diffuse and contain dislocations of both signs in addition to the geometrically necessary ones which are responsible for misorientations $\pm \theta$ between neighbouring subgrains.

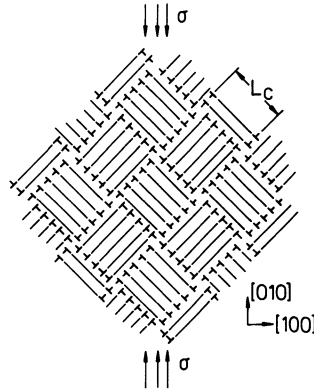


Fig. 2. — 3D-cell structure model, for slip geometry of NaCl (after Nix [21]).

3. Evolution of the dislocations densities and internal stresses.

The flow stress is carried by internal stresses of the dislocations in the cell interior ; $\tau_c = \mu b \sqrt{\rho_c}$, and the edge walls $\tau_e = \mu b \sqrt{\rho_e}$, but only to a negligible extent by the cross-grid screw dislocation walls. They are much sharper than the edge walls although not fully stress-free at spacings closer than the grid size. We expect that the applied stress is carried by cell interior (cell size $L_c \gg L_e$) and edge walls (width L_e) in proportion to their area fractions, see references [2, 7], i.e.

$$\tau = \tau_c \frac{L_c}{L_e + L_c} + \tau_e \frac{L_e}{L_e + L_c} \approx \tau_c + \frac{L_e}{L_c} \tau_e . \tag{2}$$

The flow stress is mainly carried by dislocation movement in the cell interior

$$d\varepsilon = d\varepsilon_c \gg d\varepsilon_e, d\varepsilon_s .$$

The flow stress components evolve from accumulation (+) and annihilation processes (-) as follows : inside a cell

$$\left. \frac{d\tau_c}{d\varepsilon_c} \right|_+ = \frac{d\tau_c}{d\rho_c} \cdot \frac{d\rho_c}{d\varepsilon_c} = \frac{\mu b}{2\sqrt{\rho_c}} \frac{1}{b\Lambda} = \frac{\mu}{200} \tag{3}$$

considering the empirical relation between mean free path Λ and cell size discussed above and the principle of similitude, equation (1). Actually equation (3) describes work hardening in

stage II where cell walls have not formed yet and there is no dynamical recovery either. This equation is no first principle description of stage II but contains results of slip line length measurements as does Seeger's [22] model. A thermal component of the flow stress is not considered because we are concerned either with high temperatures or with large strains. Cells form as soon as dynamical recovery sets in whereby one kind of segments of a dislocation loop partially annihilates while the other is set free to move into the wall or boundary. This was recognized by Seeger in 1955 for stage II in fcc metals. The mutual annihilation of parallel screws by cross slip happens inside the cells and not in the screw walls where the remaining orthogonal screw dislocations mutually attract each other to form a square net. So again in the cells

$$\frac{d\tau_c}{d\varepsilon_c} \Big|_- = \frac{d\tau_c}{d\rho_c} \cdot \frac{\dot{\rho}_c}{\dot{\varepsilon}} \quad (4)$$

and $\dot{\rho}$, the static recovery rate of screws, is described either in Seeger's model [22] or in Escaig's [23]

$$-\dot{\rho}_c = \frac{\rho_c}{w} 2 L_c \nu_0 \exp(-W_{cs}(\tau)/kT) \quad (5)$$

with w being the spacing of cross slip sites along a screw, ν_0 an attack frequency. $W_{cs}(\tau)$ is a logarithmically stress dependent activation energy in Seeger's model, a linearly stress dependent in Escaig's.

Cross slip of screws is very difficult relative to climb of edges in the case of high temperature deforming semiconductors. Thus this recovery term is absent in their stages III and IV_H. The edge walls become more dense by the deposition of 2 segments per loop that cannot penetrate them (fraction β)

$$\frac{d\tau_e}{d\varepsilon_c} \Big|_+ = \frac{d\tau_e}{d\rho_e} \frac{d\rho_e}{d\varepsilon_e} = \frac{\mu b}{2 \sqrt{\rho_e}} \cdot \beta \cdot \frac{2}{bL_e} = \frac{\mu^2 b \beta}{\tau_e L_e} \quad (6)$$

The width L_e of these walls is taken to be constant or, following Nix, to become smaller with increasing strength and decreasing temperature

$$L_e = \text{const} \quad (6a)$$

or

$$L_e = \frac{\pi b}{\tau_e} \left(\frac{T_m}{T} \right)^2 \quad (6b)$$

The edge walls thin out by climb according to a typical static recovery law

$$\frac{d\tau_e}{d\varepsilon_c} \Big|_- = \frac{d\tau_e}{d\rho_e} \cdot \frac{\dot{\rho}_e}{\dot{\varepsilon}} \approx - \frac{D}{2 \dot{\varepsilon}} \frac{\mu^2 b}{kT} \cdot \left(\frac{\tau_e}{\mu} \right)^3 \quad (7)$$

where D is an effective diffusion coefficient. In metals below half the melting temperature T_m this term is absent. The screw nets are assumed to be relatively stress free and resistant to dynamical recovery, at least in stages II and IV_H,

$$\text{so } \frac{d\tau_s}{d\varepsilon_c} \Big|_+ = \frac{d\tau_s}{d\varepsilon_c} \Big|_- = 0.$$

The mean free path of screws is also confined to one cell diameter, much smaller than that of edges, if screws do not annihilate altogether within the cell by cross-slip.

4. Stage IV_L in fcc metals — no climb but cross slip.

The total work hardening rate is by differentiation of equation (2)

$$\theta = \frac{d\tau_c}{d\varepsilon} \Big|_+ + \frac{d\tau_c}{d\varepsilon} \Big|_- + \frac{d}{d\varepsilon} \left(\frac{L_c}{L_c} \right) \tau_c. \quad (8)$$

At the end of stage III, at the stress $\tau_{\text{III}s}$ (see Fig. 4), the work hardening rate $\theta_{\text{II}} = \frac{d\tau_c}{d\varepsilon} \Big|_+$ would just be compensated by the dynamical recovery rate $(d\tau_c/d\varepsilon) \Big|_-$ according to equation (5). Thus near this point the work hardening rate is

$$\theta_{\text{IV}}^{\tau_{\text{III}s}} = \frac{d\tau_c}{d\varepsilon} \Big|_+ + \frac{L_c}{L_c} + \tau_c \frac{d}{d\varepsilon} \left(\frac{L_c}{L_c} \right). \quad (9)$$

Making use of equation (6), (6a) and (1)

$$\theta_{\text{IV}}^{\tau_{\text{III}s}} = \frac{b\mu^2\beta}{L_c\tau_e} + \frac{\tau_c L_c}{10\mu b} \cdot \frac{d\tau_c}{d\varepsilon} = \frac{\mu\beta\tau_c}{\tau_e 10}. \quad (10)$$

Note that with $d\tau_c/d\varepsilon = 0$ also L_c does not decrease with further strain as is indeed found by Zehetbauer *et al.* [9] for copper deformed in torsion.

At $\tau_{\text{III}s}$ the remaining term in equation (10) is the first describing continuous deposition of edges in the wall. The internal stress τ_c there cannot increase forever as already Essmann and Mughrabi [20] have recognized. Two edges of opposite sign which are closer than about 10 Å interact more strongly than the theoretical strength of the lattice and collapse into an array of vacancies. Thus we put $\tau_c^{\text{max}} = \mu/10$. Thus equation (10) turns into

$$\theta_{\text{IV}}^{\tau_{\text{III}s}} = \beta\tau_c. \quad (11)$$

This is a reasonable description of stage IV_L in metals as will be discussed below. If we had used the alternative equation (6b) instead of (6a), equation (9) would get the additional term

$$\Delta\theta_{\text{IV}}^{\tau_{\text{III}s}} = \frac{\tau_c}{L_c} \frac{dL_c}{d\tau_e} \frac{d\tau_e}{d\varepsilon} \Big|_+ = - \frac{\tau_c}{L_c} \frac{L_c}{\tau_e} \frac{b\mu^2\beta}{L_c\tau_e} = - \frac{b\mu^2\beta}{L_c\tau_e} \quad (12)$$

which exactly compensates $\theta_{\text{IV}}^{\tau_{\text{III}s}}$ of equation (10) to zero. An inverse proportionality of L_c and τ_e therefore does not seem to be realistic assumption. This is already evident by comparison with a pure twist boundary which has little internal stress ($\tau_s \Rightarrow 0$) while it is very sharp ($L_s \Rightarrow 0$).

5. Stage IV at high temperatures — no cross slip but climb.

Dynamical recovery in the elemental semiconductors and also in fcc metals proceeds at high temperatures preferentially by climb [10, 15]. During this process edge dislocations move into walls (stage III) where they partially annihilate while screw dislocations form networks [3] in stage IV_H. The work hardening process thereby is relocated from the cell interiors $(d\tau_c/d\varepsilon)_+$ to the walls $(d\tau_e/d\varepsilon)_+$ but we cannot directly compensate θ_{II} in the cells by

$(d\tau_c/d\varepsilon)_-$ in the edge walls. Only at a later stage of this « perestrojka » when the walls are becoming thin and the work hardening rate $\theta \ll \theta_{II}$, the dislocation balance in the edge walls becomes visible as a minimum in the work hardening rate (near τ_{III_s}). There from equations (10), (7) and (6a)

$$\theta_{IV}^{\tau_{III_s}} = \frac{b\mu^2\beta}{L_c\tau_e} + \frac{\tau_e L_c \mu}{10\mu b 200} - \frac{D}{2\varepsilon} \frac{\mu^2 b}{kT} \left(\frac{\tau_e}{\mu}\right)^3 \frac{L_c}{L_c} \tag{13}$$

For small τ_e i.e. $\tau_c \approx \tau \gg \frac{L_c}{L_c} \tau_e$ (near τ_{III_s}) from equations (3) and (6)

$$\tau_e^2 = \frac{400\beta b}{L_e} \tau \mu \tag{14}$$

and therefore

$$\theta_{IV}^{\tau_{III_s}} \approx \frac{3}{200} \sqrt{\frac{\mu\tau L_c \beta}{b}} - 400 \frac{D}{\varepsilon kT} \sqrt{\frac{\beta^3 b^3 \tau^5}{\mu L_e}} \tag{15}$$

This describes a $(A\sqrt{\tau} - B\tau^{5/2})$ minimum in $\theta(\tau)$ where B is strongly increasing with temperature while A is a constant.

6. Experimental situation and discussion.

In *metals* at medium temperatures Rollett *et al.* [4] have reviewed the experimental situation and concluded that $\theta_{IVL} = c\tau_{III_s}$ describes it well with $c = 0.05...0.10$ (Fig. 3). Our equation (11) with $\beta = 0.1$ yields this order of magnitude considering that $\tau_c \approx \tau/10$ since in equation (2) $(L_c/L_c) \cdot \tau_e$ for $L_e = 100 b$, $L_c = 10^4...10^5 b$, $\tau_e \approx \mu/2$ is of the same order as

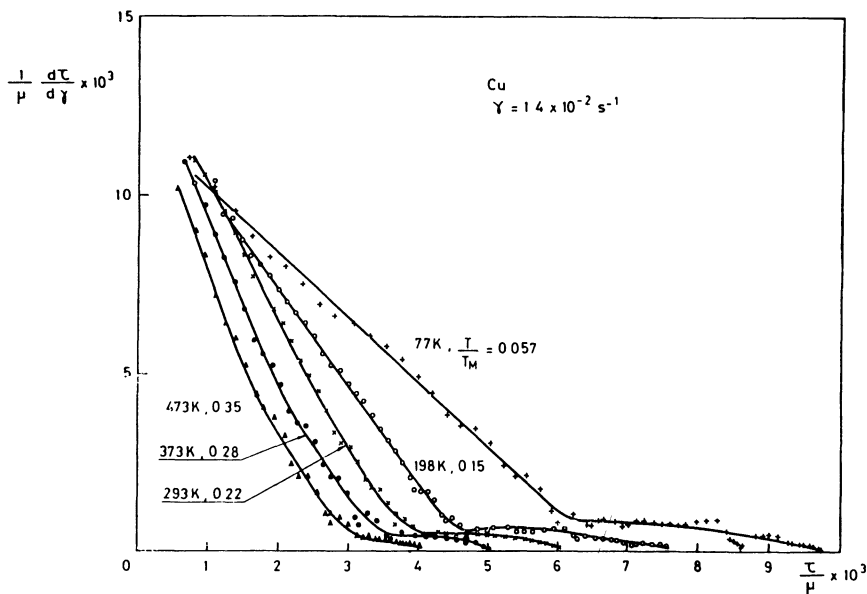


Fig. 3. — Work hardening rates vs. reduced flow stress in torsion for polycrystalline copper after Alberdi [11] (low reduced temperatures).

τ_c . Note that θ_{IVL} does not further increase with τ — it is constant (also with respect to $\dot{\epsilon}$) — at least in single-phase materials. Only the value of θ_{IVL} at the starting stress τ_{III_s} of stage IV increases with this stress, the reason being that the wall strength is then constant but the cell size is inversely proportional to τ_{III_s} . In the high temperature case of copper and the *semiconductors* (Figs. 4, 5) the relation of the increasing section of the $\theta(\tau)$ curve near the minimum is indeed a parabolic one with $\theta_{IVH} = 10 (N^{1/2}/mm) \tau^{1/2}$ compatible with equation (15) with $L_e = 100 b$. An analysis also of the temperature dependent tail for Ge of $\theta(\tau)$ in stage III in terms of equation (15) is given by Haasen *et al.* [24]. The work hardening rate θ_{IVH} for copper measured by Anongba *et al.* [8] is plotted according to equation (15) in figure 6. The slope is, as for Ge, $9 \sqrt{\text{MPa}}$, but there is an intercept of the $\theta(\sqrt{\tau})$ line of the order of the CRSS of copper at these temperatures. Fcc metals or semiconductors are considered here not because of a particular binding but they represent well studied classes of crystals of different reduced stacking fault energy

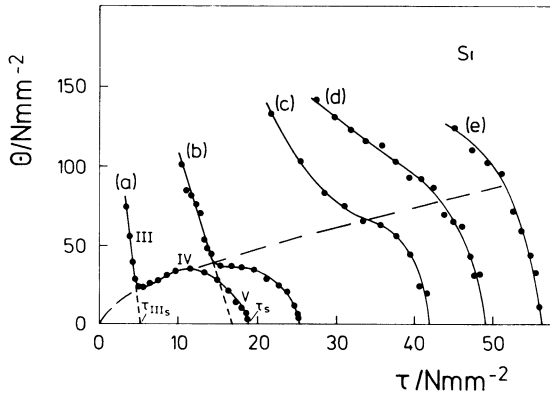


Fig. 4. — Work hardening rate of silicon single crystals for various temperatures after Siethoff *et al.* [10]: (a) $T = 1\,300^\circ\text{C}$, $\dot{\epsilon} = 0.51 \times 10^{-3} \text{ s}^{-1}$; (b) $T = 1\,200^\circ\text{C}$, $\dot{\epsilon} = 4.8 \times 10^{-3} \text{ s}^{-1}$; (c) $T = 1\,000^\circ\text{C}$, $\dot{\epsilon} = 0.48 \times 10^{-3} \text{ s}^{-1}$; (d) $T = 1\,000^\circ\text{C}$, $\dot{\epsilon} = 1.2 \times 10^{-3} \text{ s}^{-1}$; (e) $T = 1\,000^\circ\text{C}$, $\dot{\epsilon} = 12 \times 10^{-3} \text{ s}^{-1}$.

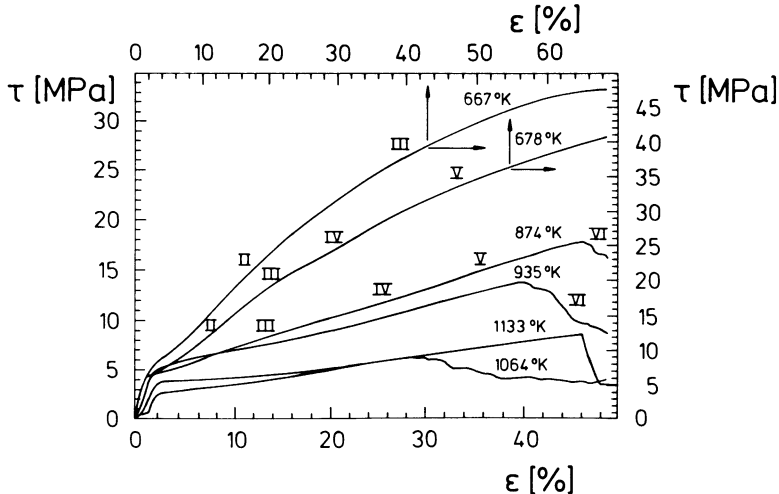


Fig. 5. — Work hardening curves of copper single crystals at high temperatures after Anongba *et al.* [8].

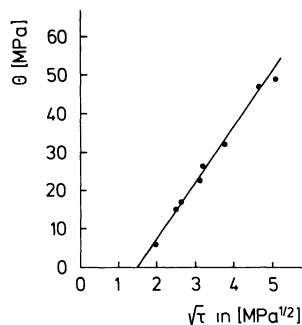


Fig. 6. — Stage IV work hardening rates of figure 5 vs. square root of flow stress.

($\gamma/\mu b$). If this quantity is large then cross slip is easy and v_c vs. This and the reduced temperature (T/T_m) are responsible for the preference of a particular dynamical recovery mechanism, cross slip or climb, in stage III. The other one then follows in stage V (not considered here). That there is a rehardening stage in-between them has to do with the increasing strength of the cell walls in our model. It does not describe so far how precisely a cell structure forms out of the less structured dislocation arrangement governing stage II. Bcc metals with their relatively easy cross slip should behave like fcc at low T

References

- [1] PRINZ F. and ARGON A. S., *Acta Met.* **32** (1984) 1021.
- [2] NIX W. D., GIBELING J. C. and HUGHES D. A., *Met. Trans.* **16A** (1985) 2215.
- [3] BRION H. G., and HAASEN P., *Philos. Mag.* **A 51** (1985) 879.
- [4] ROLLETT A. D., KOCKS U. F. and DOHERTY R. H., Los Alamos Natl. Lab. Report (1987) p. 211.
- [5] ROLLETT A. D., KOCKS U. F., EMBURY J. D., STOUT M. G. and DOHERTY R. D. (Pergamon, Oxford) *Proc. ICSMA 8* (1988) 433.
- [6] HAASEN P., *Cz. Jl. Phys.* **B 28** (1988) 494.
- [7] HAASEN P. (Pergamon, Oxford) *Proc. ICSMA 8* (1988) 343.
- [8] ANONGBA P., BONNEVILLE J. and MARTIN J. L. (Pergamon, Oxford) *Proc. ICSMA 8* (1988) 265.
- [9] ZEHETBAUER M., SEUMER U. and WITZEL W. (Pergamon, Oxford) *Proc. ICSMA 8* (1988) 451.
- [10] SIETHOFF H. and SCHRÖTER W., *Z. Metallkunde* **75** (1984) 475, 482.
- [11] ALBERDI G., Thesis, Univ. of Navarra (1984).
- [12] HUGHES D. A., Thesis, Stanford Univ. (1986).
- [13] ROLLETT A. D., Thesis, Drexel Univ. (1988).
- [14] MECKING H. and GRINBERG G. (Pergamon, Oxford) *Proc. ICSMA 5* (1979) 289.
- [15] PFAFF F., *Z. Metallkunde* **53** (1962) 411, 466.
- [16] KOCKS U.-F., *Philos. Mag.* **11** (1966) 541.
- [17] KUHLMANN-WILSDORF D., *Met. Trans.* **224** (1962) 1047.
- [18] MUGHRABI H., *Mat. Sci. Eng.* **85** (1987) 15.
- [19] ESSMANN U. and DIFFERT H., *Scr. Met.* (1989) in press.
- [20] ESSMANN U. and MUGHRABI H., *Philos. Mag.* **A 40** (1979) 735.
- [21] NIX W. D. (1988) unpublished.
- [22] SEEGER A. (Springer, Berlin) *Handb. Physik VII 2* (1955).
- [23] ESCAIG B. and BONNEVILLE J., *Acta Met.* **27** (1979) 1477.
- [24] HAASEN P., ALBORN K. and SCHRÖTER W., *Isv Akad. Nauk.* **51** (1987) 749.