



Plastic deformation of icosahedral Al–Pd–Mn single quasicrystals

II. Interpretation of the experimental results

U. MESSERSCHMIDT*†, M. BARTSCH†, B. GEYER†, M. FEUERBACHER‡, and K. URBAN‡

† Max-Planck-Institut für Mikrostrukturphysik, Weinberg 2,
D-06120 Halle/Saale, Germany

‡ Institut für Festkörperforschung Forschungszentrum Jülich GmbH,
D-52425, Jülich, Germany

[Received 15 March 1999 and accepted in revised form 7 July 1999]

ABSTRACT

Experimental data on the plastic deformation of icosahedral Al–Pd–Mn single quasicrystals are discussed in terms of dislocation models. The occurrence of a yield drop followed by a range of steady state deformation is explained by the effects of dislocation multiplication and recovery on both the mobile dislocation density and the internal stress. The influence of transient phenomena during the measuring procedures of the activation parameters is estimated. The activation volume of deformation is a unique function of the stress. The activation enthalpy and the Gibbs free energy, calculated by Schoeck's formula, assume approximately constant values above 700°C but decrease strongly below this temperature. In the temperature range where steady state deformation is possible, probably the flow stress is controlled by both the friction mechanism of moving dislocations and the recovery processes taking place during deformation.

§1. INTRODUCTION

The plastic properties of icosahedral Al–Pd–Mn single quasicrystals were determined over a temperature range where the deformation is characterized by a yield drop followed by a range of almost steady state deformation (Part I, Geyer *et al.* 2000). The present paper discusses these results in terms of dislocation models of the plastic deformation.

§2. THEORY

Usually deformation experiments on quasicrystals are interpreted by using the formalism of the thermally activated dislocation motion developed for crystalline materials. With some modifications because of the properties of dislocations in quasicrystals, this formalism is adopted here. Accordingly, the plastic strain rate $\dot{\epsilon}$ is related to the dislocation velocity v by the Orowan relation

$$\dot{\epsilon} = m_s \rho b v, \quad (1)$$

*Author for correspondence. e-mail: um@mpi-halle.mpg.de

where ρ is the density of mobile dislocations. Up to now, there is no electron microscopy evidence that dislocation structures are formed during the deformation of quasicrystals which immobilize the dislocations (Feuerbacher *et al.* 1997, Schall *et al.* 1999). Thus, it is assumed throughout this paper that all dislocations are mobile. b is the length of the Burgers vector of the gliding dislocations in physical space, and m_s is the orientation factor, which relates the compressive stress σ to the shear stress τ on the activated slip plane:

$$\tau = m_s \sigma. \quad (2)$$

For small strains, m_s can be used also to relate shear strains to compressive strains, as in equation (1). In Al-Pd-Mn, the parallel components of the most frequent Burgers vectors are parallel to twofold directions, and the most frequent slip planes are perpendicular to fivefold and threefold directions of the quasicrystal lattice (Rosenfeld *et al.* 1995). For both compression directions, the largest orientation factors are close to $m_s = 0.4$. Thus, this value will be used for the following considerations. While the experimental results in Part I were presented in terms of the compressive stress σ , the theoretical considerations are expressed in terms of the shear stress τ .

Commonly, the thermally activated dislocation motion is described by an Arrhenius relationship as reviewed by Evans and Rawlings (1969). Then, the dislocation velocity v can be written as

$$v = v_0 \exp(-\Delta G/kT) \quad (3)$$

which, after inserting equation (1), yields an expression for the plastic strain rate

$$\dot{\epsilon} = (m_s \rho b v_0) \exp(-\Delta G/kT) = \dot{\epsilon}_0 \exp(-\Delta G/kT). \quad (4)$$

Here v_0 and $\dot{\epsilon}_0$ are the pre-exponential factors, ΔG is the Gibbs free energy of activation, k is the Boltzmann constant, and T is the absolute temperature. ΔG depends on the effective stress τ^* acting locally via

$$\Delta G(\tau^*) = \Delta F(\tau^*) - V(\tau^*)\tau^*. \quad (5)$$

ΔF is the Helmholtz free energy of activation, and V is the activation volume. Both ΔF and V depend on τ^* , ΔF only slightly. The thermal component τ^* of the total flow stress τ is given by (Seeger 1958)

$$\tau^* = \tau - \tau_i \quad (\text{or } \sigma^* = \sigma - \sigma_i). \quad (6)$$

The flow stress contribution τ_i is of athermal character and originates from the long-range elastic interactions between the dislocations. Using different models, these interactions yield a square root dependence of τ_i on the total dislocation density ρ as originally shown by Taylor (1934) for a random arrangement of parallel dislocations and discussed by Basinski and Basinski (1979) on the basis of experimental data,

$$\tau_i = \alpha \mu b \rho^{1/2}. \quad (7)$$

μ is the shear modulus and α is a numerical constant. In quasicrystals, little is known about the elastic interaction between dislocations, so that α is not known precisely. Even in crystals it may vary between about 0.2 and 1, depending on the actual dislocation arrangement. A value of $\alpha = 0.5$ will be used below. Applying the

above relations will lead to an equation for the (shear) flow stress:

$$\tau = \alpha \mu b \rho^{1/2} + (1/V)[\Delta F + kT \ln(\dot{\varepsilon}/m_s \rho b v_0)]. \quad (8)$$

The activation volume can be determined experimentally via

$$V = kT/m_s r, \quad (9)$$

with $r = (\Delta \tau^*/\Delta \ln v)_T/m_s$. r equals the experimental strain rate sensitivity $r_{\text{ex}} = (\Delta \sigma/\Delta \ln \dot{\varepsilon})_T$ in equation (1) of Part I if the pre-exponential factor $\dot{\varepsilon}_0$ in equation (4) and τ_i are constant during the measuring procedure. There is no direct way of determining ΔG . However, the activation enthalpy ΔH can be determined from r and temperature change experiments, using the relation (e.g., Evans and Rawlings (1969)):

$$\Delta H = kT^2(\Delta \ln v/\Delta T)_{\tau^*} = -kT^2(\Delta \sigma^*/\Delta T)_v/r. \quad (10)$$

Again, $(\Delta \sigma^*/\Delta T)_v$ equals $(\Delta \sigma/\Delta T)_{\dot{\varepsilon}}$ only if the pre-exponential factors and τ_i remain constant during the temperature change. ΔG can be calculated from ΔH by using a formula derived by Schöck (1965):

$$\Delta G = \Delta H - T\Delta S = (\Delta H + \beta \tau V)/(1 - \beta), \quad (11)$$

with $\beta = (T/\mu)(d\mu/dT)$. The RHS of equation (11) contains only measurable quantities, but is based on the assumption that the main contribution to the activation entropy ΔS originates from the temperature dependence of the shear modulus μ . This assumption is fulfilled well for elastic interactions between dislocations and defects, but it may not be fulfilled for the deformation of quasicrystals because of the phason strain field of dislocations.

As described above, the measuring procedures of equations (9) and (10) can be applied to the experimental quantities r_{ex} and $(\Delta \sigma/\Delta T)_{\dot{\varepsilon}}$ only if $\dot{\varepsilon}_0$ and τ_i are constant during the measurement process. The most important factor in $\dot{\varepsilon}_0$ which may change is the mobile dislocation density ρ . As ρ and τ_i can change only with some strain or time having passed, the incremental changes $\Delta \ln \dot{\varepsilon}$ and ΔT should be performed as rapidly as possible. As described in §2 of Part I, changing the temperature requires partial unloading, taking about 30 min. Thus, temperature change experiments will not correspond to the instantaneous changes, which are intended. Similarly, the discrepancy between original and repeated relaxation curves reported in Part I (§3.3 and, e.g., figure 4(b)) indicates that structural changes occur also during the relaxation experiments. The interpretation of the experimental results should therefore take into account these transient effects.

§3. SHAPE OF THE STRESS-STRAIN CURVE

The discussion of the stress-strain curve will be based on the kinetics of the dislocation density as observed by Feuerbacher *et al.* (1997), Schall (1998) and Schall *et al.* (1999). The last mentioned study describes three important observations.

- (1) During straining at 730°C, in the pre-yield range the dislocation density ρ increases by more than one order of magnitude, assuming a value of $\rho_{\text{uyp}} \cong 3 \times 10^{13} \text{ m}^{-2}$ at the upper yield point at an initial rate of increase of $M = (d\rho/d\varepsilon)_+ = 6.5 \times 10^{15} \text{ m}^{-2}$. It reaches a value of $9 \times 10^{13} \text{ m}^{-2}$ at the lower yield point and a maximum of $1.2 \times 10^{14} \text{ m}^{-2}$ at a strain of $\varepsilon = 5.2\%$. At larger strains, it decreases again.

- (2) During annealing at 730°C of specimens deformed at the same temperature, the dislocation density decreases rapidly within the first 15 min but remains almost constant after 45 min.
- (3) The dislocation density at the lower yield point depends strongly on the temperature.

These observations can be modelled by the kinetic equations below.

The initial decrease in the dislocation density during annealing mentioned in observation (2) can be described by

$$(d\rho/dt)_- = -q\rho^2, \quad (12)$$

with the solution

$$1/\rho = qt + C. \quad (13)$$

For ρ , equation (12) is of second order since the annihilation process requires two dislocations of opposite sign to meet. The annihilation can either appear by cross slip or climb, depending on the character of the dislocations, leading both to equation (12). As cross slip is restricted to screw dislocations, climb should control the final annihilation process. The regression analysis of the initial decrease during annealing at 730°C yields $q = 4.4 \times 10^{-18} \text{ m}^2 \text{ s}^{-1}$. The kinetic equation of the dislocation density during deformation as described under observation (1) and taking in account the dislocation generation and recovery may read

$$d\rho = w\rho ds - q\rho^2 dt, \quad (14)$$

where s is the travelling distance of the dislocations and w is a rate constant. Equation (14) assumes that the dislocations multiply during their motion, as originally suggested by Johnston and Gilman (1959). It is shown by *in situ* straining experiments in a high-voltage electron microscope (Messerschmidt *et al.* 1998) and by conventional high-voltage transmission electron microscopy (Messerschmidt *et al.* 1999c) that the multiplication is initiated by either cross slip or a dislocation reaction, as in many crystalline materials. Accordingly, the increase in the dislocation density is proportional to the area per volume ρds swept by the dislocations. There are several processes which suggest a dependence of the multiplication rate on the stress (see, e.g., Wiedersich (1962)).† With $ds = d\varepsilon/(\rho b)$ and $dt = d\varepsilon/\dot{\varepsilon}$, equation (14) becomes

$$d\rho/d\varepsilon = w\tau/b - q\rho^2/\dot{\varepsilon}, \quad (15)$$

where $w\tau/b$ is the multiplication rate M . For a complete theory, equation (15) should be combined with equation (8). A similar evolution law with other powers in ρ is applied to quasicrystal deformation by Guyot and Canova (1998). With $d\rho/d\varepsilon = 0$, equation (15) leads to a steady state dislocation density ρ_{ss} , which may be discussed in a simplified way by assuming that the stress reaches a steady state also and by

† On principle, the local stress τ^* should enter equation (14) as it causes the bowing of a pinned dislocation segment leading to a multiplication event. This paper argues that both τ^* and τ_i depend on T so that, approximately, the Cottrell–Stokes law may hold, i.e., τ^* may be approximately proportional to τ . Formally, then it makes no difference whether equation (14) and the following equation (18) are written in τ^* or τ . τ is preferred as it is closer to the measured quantity σ .

using the experimental values of M and q . This yields

$$\rho_{ss} = [\dot{\epsilon}w\tau(bq)]^{1/2} = [\dot{\epsilon}M/q]^{1/2}. \quad (16)$$

Inserting the experimental data yields $\rho_{ss} = 1.22 \times 10^{14} \text{ m}^{-2}$, which is in very good agreement with the experimental maximum value of ρ quoted above under observation (1). Equations (15) and (16) are based on climb controlled recovery. Thus, the rate constant q should contain the diffusion coefficient, or

$$q = q_0 \exp(-\Delta H_{SD}/kT), \quad (17)$$

where q_0 is a constant and ΔH_{SD} is the activation enthalpy of self-diffusion. Both the dependence of the annihilation rate on the temperature via recovery and the dependence of the formation rate of dislocations on the stress may lead to a temperature dependence of the dislocation density, as described above under observation (3). From equations (16) and (17) it follows that

$$\ln(\tau/\rho_{ss}^2) = \text{const} - \Delta H_{SD}/kT. \quad (18)$$

Plotting the experimental values of ρ and σ (instead of τ) of Schall *et al.* (1999) versus $1/T$ yields $\Delta H_{SD} \cong 4 \text{ eV}$. This value will be discussed in §6.

In the following, the stress-strain curve of Al-Pd-Mn quasicrystals in the high-temperature range, i.e., above about 630°C for specimens of a fivefold compression axis deformed at a strain rate of 10^{-5} s^{-1} , is discussed on the basis of the above dislocation kinetics. First, the athermal contribution τ_i to the flow stress is estimated from the above-quoted dislocation densities ρ by using equation (7). Data on the shear modulus were measured by Tanaka *et al.* (1996) and by Feuerbacher *et al.* (1996). In order to extrapolate values of μ from the latter data for the transition range from the unrelaxed to the relaxed modulus, a quadratic expression was used for $\mu(T)$. Its parameters were determined by using $\mu = 77.25 \text{ GPa}$ and $d\mu/dT = -19 \text{ MPa K}^{-1}$ at 500 K , and $d\mu/dT = -35.6 \text{ MPa K}^{-1}$ at 1000 K . Slightly lower values follow from the elastic constants of Tanaka *et al.* (1996). The estimation yields about 31 MPa for $\tau_{i\text{uyp}}$ at the upper yield point at 730°C , and about 54 MPa for $\tau_{i\text{lyp}}$ at the lower one, if the very frequent value of $b = 0.183 \text{ nm}$ of the length of the Burgers vectors in physical space (Rosenfeld *et al.* 1995) is used. Values of τ_i from dislocation density data of Schall *et al.* (1999) at different temperatures are presented in table 1, which shows that τ_i at the lower yield point amounts in most cases to 27% of the flow stress. Though this estimation implies a large error because of the uncertainty of the constant α , it may be concluded that the athermal part of the flow stress cannot be neglected and that it is a fixed fraction of the flow stress, i.e., that the Cottrell-Stokes law (Cottrell and Stokes 1955) seems to hold in Al-Pd-Mn single quasicrystals.

Table 1. Shear modulus μ , dislocation density ρ after Schall *et al.* (1999), athermal stress component τ_i according to equation (7), and ratio between τ_i and (shear) flow stress at the lower yield point $\tau_{i\text{lyp}}$.

T ($^\circ\text{C}$)	μ (GPa)	ρ (m^{-2})	τ_i (MPa)	$\tau_i/\tau_{i\text{lyp}}$
695	63.5	9×10^{13}	55.1	0.27
730	62.3	6×10^{13}	44.2	0.32
790	60.1	7×10^{12}	14.6	0.27
820	58.9	2.7×10^{12}	8.8	0.27

In accordance with the strong increase in the dislocation density during the first loading, the material shows considerable plastic deformation far below the macroscopic yield point, even at low temperatures. This is demonstrated by the stress relaxation curves R1a and R1b in figure 1(a) of Part I. It was shown in §3.1 of Part I that strong work-hardening occurs in this range owing to the increasing dislocation density.

A prominent feature of the stress–strain curves is the occurrence of an upper and a lower yield point, as presented in figure 1 of Part I. Such yield point effects may have several reasons. In the present case, they can be explained consistently by the substantial increase in the dislocation density discussed above. This interpretation is inherent also in the model calculations of Schall (1998). According to equation (8), the difference between the flow stresses at the upper and the lower yield points is given by

$$\Delta\tau|_{yp} = \tau_{i_{uyp}} - \tau_{i_{lyp}} + m_s r_{ex} \ln(\rho_{lyp}/\rho_{uyp})/(1 - C_r). \quad (19)$$

The factor of $1/(1 - C_r)$ will be discussed in the next paragraph. Using the above data for ρ and τ_i at 730°C as well as the values of $r_{ex} \cong 85$ MPa from figure 7(a) of Part I and of $C_r = 0.062$ from equation (3) of Part I leads to $\Delta\tau|_{yp} \cong 17$ MPa for the stress drop at the yield point. Experimentally, at 730°C there is a yield drop of $\Delta\sigma|_{yp} \cong 43$ MPa (the regression formula in §3.1 and figure 3 of Part I). Thus, taking into account the orientation factor, the calculated value $\Delta\tau|_{yp}/m_s$ agrees very well with the experimental one, so that the increase in the dislocation density between the upper and the lower yield points can account fully for the yield drop.

A plateau region of almost constant stress follows the yield drop. As shown in figure 3 of Part I, the flow stresses at the plateau of the specimens with their compression axes in fivefold and twofold orientation are almost equal. Only at low temperatures are the flow stresses of specimens with a twofold compression axis slightly lower, which may be due to the larger orientation factors of the prominent slip systems in this orientation. The plateau region corresponds to the range of maximum dislocation density, where the changes in the dislocation density are smallest. The plateau can be described by a steady state situation in equation (15) between the dislocation generation and annihilation as expressed by equation (16). Thus, the constancy of the flow stress over a relatively wide range of strains can be understood by a steady state of the dislocation density, which implies that recovery processes take place during the deformation. In this sense, recovery seems to be a prerequisite to a continuous macroscopic deformation of quasicrystals. If the temperature is too low for sufficient recovery, the specimens work-harden rapidly and fail at low strains after reaching high stresses (Messerschmidt *et al.* 1999b).

In many papers, a decrease in stress beyond the lower yield point has been reported (e.g., by Bresson and Gratias (1993) for polycrystalline Al–Pd–Mn, and by Wollgarten *et al.* (1993) for single quasicrystalline Al–Pd–Mn). It is termed work-softening and considered an essential feature of the plastic deformation of quasicrystals. In the present study, most specimens were deformed up to about 6% plastic strain, only. Within this range, the flow stress decreases only slightly (cf. figure 1 of Part I). Thus, the activation parameters were measured at a relatively well established steady state of both stress and dislocation density. This is an advantage over earlier measurements, where the activation parameters were determined at the upper yield point (Feuerbacher *et al.* 1995, Brunner *et al.* 1997), which is a range of strongly changing dislocation density.

§4. TRANSIENT EFFECTS DURING CHANGES OF THE DEFORMATION CONDITIONS

As described in §2, transient effects have to be considered in evaluating the activation parameters by the measuring procedures of the strain rate and temperature sensitivities. During stress relaxation tests, the transients are indicated by the difference between the original and repeated relaxation curves, as in figure 4(b) of Part I. During the original relaxations with decreasing stress, a decreasing non-equilibrium dislocation density will influence both the dynamic law in equation (4) and the internal stress in equation (7). It follows from equation (6) that (in terms of the measured compressive stress)

$$d\sigma/d \ln \dot{\epsilon} = d\sigma^*/d \ln \dot{\epsilon} + d\sigma_i/d \ln \dot{\epsilon}, \quad (20)$$

or

$$\left. \frac{d\sigma}{d \ln \dot{\epsilon}} \right|_T = \left. \frac{\partial \sigma^*}{\partial \ln \dot{\epsilon}} \right|_{T,\rho} + \left. \frac{\partial \sigma^*}{\partial \ln \rho} \frac{d \ln \rho}{d \ln \dot{\epsilon}} \right|_T + \left. \frac{\partial \sigma_i}{\partial \ln \rho} \frac{d \ln \rho}{d \ln \dot{\epsilon}} \right|_T. \quad (21)$$

Considering that $\partial \sigma^*/\partial \ln \rho|_{\dot{\epsilon},T} = -r$ (cf. equations (4) and (5) and the same arguments which lead to equation (9), see Schöck (1965)) and $\partial \sigma_i/\partial \ln \rho = \sigma_i/2$ (equation (7)), it follows that

$$\left. \frac{d\sigma}{d \ln \dot{\epsilon}} \right|_T = \left. \frac{\partial \sigma^*}{\partial \ln \dot{\epsilon}} \right|_{T,\rho} - \left(r - \frac{\sigma_i}{2} \right) \left. \frac{d \ln \rho}{d \ln \dot{\epsilon}} \right|_T, \quad (22)$$

or

$$r = \frac{r_{\text{ex}} - \frac{\sigma_i}{2} \left. \frac{d \ln \rho}{d \ln \dot{\epsilon}} \right|_T}{1 - \left. \frac{d \ln \rho}{d \ln \dot{\epsilon}} \right|_T}. \quad (23)$$

This formula is valid if steady state values are taken for r_{ex} and $(d \ln \rho/d \ln \dot{\epsilon})_T$. The difference between the original and the repeated relaxation curves reveals that structural changes, i.e., a recovery of the dislocation density, occur during the original relaxations. However, there is no information available about the extent to which the structure deviates from the steady state one, which corresponds to the actual strain rate. Therefore, $(d \ln \rho/d \ln \dot{\epsilon})_T$ is not known from the deformation experiments. However, a theoretical value of 1/2 follows from equation (16).

During the reloading of the specimens after the relaxations until steady state conditions are re-established, a yield point effect appears with a stress increment $\Delta\sigma|_{\text{yp}}$ as shown in figure 6(a) of Part I. Afterwards, the steady state dislocation density is restored. Figure 6(b) of Part I demonstrates that $\Delta\sigma|_{\text{yp}}$ is equal to the stress difference $\Delta\sigma|_r$ between original and repeated relaxations at the same strain rate. Thus, the difference between original and repeated relaxations and the yield drop effect during reloading are opposite faces of these structural changes. However, the dislocation density changes much less upon reloading the specimen below the stress of the original relaxation curve, i.e., before the steady state deformation is achieved, as in repeated relaxations. This is supported by the fact that the curves do not change on further repeating the relaxation tests. Steady state deformation is reached only after the yield point following the relaxation tests, which requires about 1% of strain, as shown in figure 6(a), while the strain necessary to reload the specimen for a repeated relaxation is about one order of magnitude smaller.

As $(d \ln \rho / d \ln \dot{\epsilon})_T$ should be small during repeated relaxation tests, the repeated relaxations can be used for determining the strain rate sensitivity corresponding to a constant microstructure rather than the original relaxation curves. However, since the difference between original and repeated relaxation curves depends on the relaxation time or the total decrease in the relaxation rate $\Delta \ln(-\dot{\sigma})|_t$ (see figure 4(b) of Part I), the normalized recovery parameter C_r was defined by equation (2) of Part I. If σ_i is small and $C_r = (d \ln \rho / d \ln \dot{\epsilon})_T$, equation (23) may read

$$r \cong r_{\text{ex}} / (1 - C_r), \quad (24)$$

where r_{ex} is taken from the original relaxation curves. However, if σ_i is not small, it is proposed that equation (24) can still be used as a phenomenological relation to calculate r from r_{ex} , which considers the influence of changes in ρ on τ_i and the mobile dislocation density. As shown in figure 5 of Part I, C_r increases with increasing temperature, so that recovery effects of the dislocation density can be neglected only at the very lowest deformation temperatures.

A particular transient effect is the occurrence of sections with inverse curvature in the relaxation curves of figure 4(a) of Part I. These sections appear in relaxations after loading associated with a strong increase in the dislocation density, i.e., with work-hardening. During original relaxations, this may happen before the upper yield point, or during repeated relaxations after long relaxation times of the original relaxation, or even after unloading. Clearly the rapid drop of the strain rate at the beginning of a subsequent relaxation is due to the continuation of hardening during the initial part of the relaxation.

As discussed in § 2 of Part I and in § 2 above, unloading and waiting for a new thermal equilibrium are necessary for temperature change experiments. After the temperature change, a yield drop effect occurs, which is caused not only by the change in the dislocation density owing to the new temperature but also by recovery during the waiting time necessary for adjusting the new temperature, since a similar yield drop appears also after unloading with no temperature change. Thus, the stress increments for determining the temperature sensitivity $(\Delta \sigma / \Delta T)_\dot{\epsilon}$ should not include the upper yield point. In the present study, $(\Delta \sigma / \Delta T)_\dot{\epsilon}$ was determined in § 3.4 of Part I under steady state conditions. As with the strain rate sensitivity, changes in the dislocation density and the internal stress have to be considered if the activation enthalpy is calculated according to equation (10). By analogy with equations (20)–(22), it follows that

$$\left. \frac{d\sigma^*}{dT} \right|_{\dot{\epsilon}, \rho} = \left. \frac{\partial \sigma}{\partial T} \right|_{\dot{\epsilon}} + \left(r - \frac{\sigma_i}{2} \right) \left. \frac{d \ln \rho}{dT} \right|_{\dot{\epsilon}}. \quad (25)$$

For temperature changes, $(d \ln \rho / dT)_\dot{\epsilon}$ can be obtained from the dislocation density data measured by Schall *et al.* (1999) (their figure 2(b)). Above 730°C, it amounts to -0.035 K^{-1} and between 700°C and 730°C to only about -0.01 K^{-1} . Using characteristic values at the lower yield point at 730°C, $r = r_{\text{ex}} / (1 - C_r) \cong 90 \text{ MPa}$, $r_{\text{ex}} = 85 \text{ MPa}$, $C_r = 0.062$, $\sigma_i = \tau_i / m_s = 54 / 0.4 \cong 135 \text{ MPa}$ with $\rho = 9 \times 10^{13} \text{ m}^{-2}$ (Schall *et al.* (1999), figure 2(b)), the correction term $(r - \sigma_i / 2) (d \ln \rho / dT)_\dot{\epsilon} \cong -0.7 \text{ MPa K}^{-1}$ due to changes in $\dot{\epsilon}_0$ and τ_i is remarkable with respect to the temperature sensitivity of $(\Delta \sigma / \Delta T)_\dot{\epsilon} \cong -4.5 \text{ MPa K}^{-1}$ itself (figure 7(b) of Part I). Below 730°C, the correction term should be much smaller because of the smaller value of $(d \ln \rho / dT)_\dot{\epsilon}$. The consequences of using the correction terms will be discussed next.

§ 5. THE ACTIVATION VOLUME

The activation volume is defined by equation (9). It can be calculated from the experimental strain rate sensitivity r_{ex} by using equation (24), which takes into account the transient effects due to changes in the dislocation density during the relaxation tests. If the deformation is controlled by a single process, the activation volume describes the stress dependence of the Gibbs free energy of activation ΔG in equation (4). Thus, it should be a unique function of stress, irrespective of which parameter has been changed to vary the effective stress. Within the accuracy of the experiments, r_{ex} does not depend on the strain within the range of steady state deformation. Figure 1 shows a plot of V versus the reduced stress $\tau' = (\mu_0/\mu(T)) m_s \sigma$ for specimens with a fivefold compression axis. These specimens cover the largest range of τ' . The factor $\mu_0/\mu(T)$, which normalizes the stress, is necessary for calculating ΔG from the V versus τ' curve, which will be described in the next paragraph. The same curve is obtained for specimens with a twofold compression axis within the respective range. The stress has been varied by performing the deformations at different temperatures, different strain rates at the beginning of the relaxation tests and by measuring several values of r_{ex} along the individual relaxation curves, as described in § 2 of Part I. Each symbol corresponds to data from one experiment. Figure 1 shows that all data lie along a single curve within the experimental accuracy. This curve also coincides with values taken from a single long relaxation curve measured on a specimen with a twofold compression axis at 755°C and at an initial strain rate of 10^{-3} s^{-1} (full circles). Figure 1 would be a section of the universal interaction profile controlling the dislocation mobility in Al-Pd-Mn single quasicrystals if the internal stress were constant. However, different internal

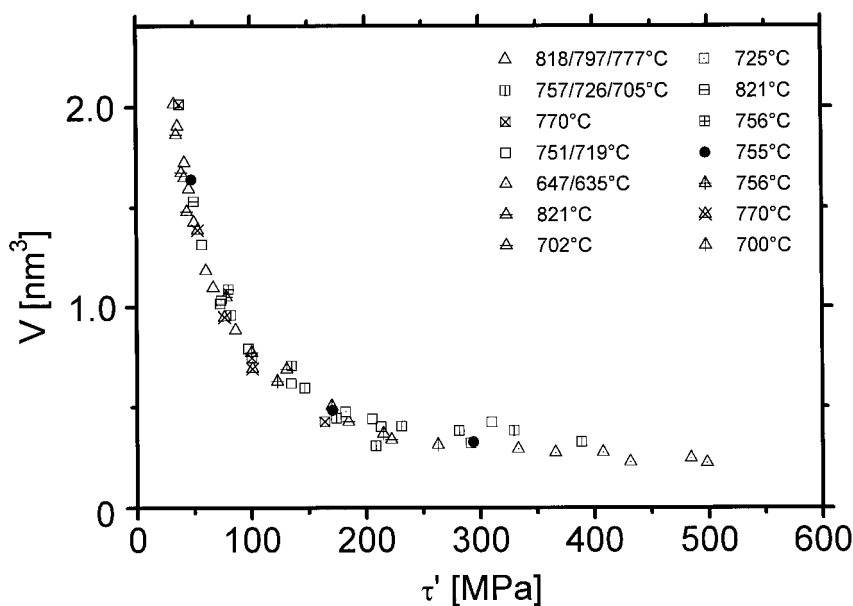


Figure 1. Dependence of the activation volume V on the reduced shear stress $\tau' = (\mu_0/\mu(T))m_s\sigma$ for specimens with a fivefold compression axis (except the full circles where there is a twofold compression axis). Each symbol corresponds to one experiment at the temperature indicated in the figure. The starting strain rates are 10^{-5} s^{-1} for triangles, 10^{-4} s^{-1} for squares and 10^{-3} s^{-1} for the circles.

stresses relate to different temperatures. Because the data points form a single curve, it can be concluded that the Cottrell–Stokes law holds.

§6. THE ACTIVATION ENERGY

The activation enthalpy ΔH is defined by equation (10). If changes in the pre-exponential factor and in the internal stress during the measuring procedure are neglected, r can be replaced by r_{ex} , and $(\Delta\sigma/\Delta T)_v$ by $(\Delta\sigma/\Delta T)_{\dot{\varepsilon}}$, leading to the experimental activation enthalpy

$$\Delta H_{\text{ex}} = -kT^2(\Delta\sigma/\Delta T)_{\dot{\varepsilon}}/r_{\text{ex}}. \quad (26)$$

Figure 2 presents a plot of ΔH_{ex} versus temperature. The values of $(\Delta\sigma/\Delta T)_{\dot{\varepsilon}}$ are taken from figure 7(b) of Part I, and those of r_{ex} are the average values from the two relaxation curves defining the stress increment $\Delta\sigma$ according to figure 4(c) of Part I. Above about 720°C, specimens with a fivefold compression axis show $\Delta H_{\text{ex}} \cong 5.7$ eV, and those with a twofold axis show $\Delta H_{\text{ex}} \cong 4.9$ eV. Below 720°C, ΔH_{ex} decreases rapidly down to about 2 eV at 640°C for the specimens with a fivefold compression axis. In the high-temperature range, the present values are slightly lower than those of Feuerbacher *et al.* (1995) but higher than those of Brunner *et al.* (1997). The difference from the data of Feuerbacher *et al.* originates mainly from the difference in measuring the stress increments from temperature changes. In the latter work, the stress increments were measured at the upper yield points after the temperature change, leading to larger values of ΔH_{ex} . No data on ΔH_{ex} are available in the literature for Al–Pd–Mn below 720°C.

Taking into account the transients, which appear during both stress relaxation experiments and temperature change tests, $(\Delta\sigma/\Delta T)_{\dot{\varepsilon}}$ in equation (26) has to be replaced by $(\Delta\sigma/\Delta T)_{\dot{\varepsilon},p}$ according to equation (25), and r_{ex} by r by either equation (23) or (24). The necessary data for σ_i and $(d \ln \rho/dT)_{\dot{\varepsilon}}$ can be taken from Schall *et*

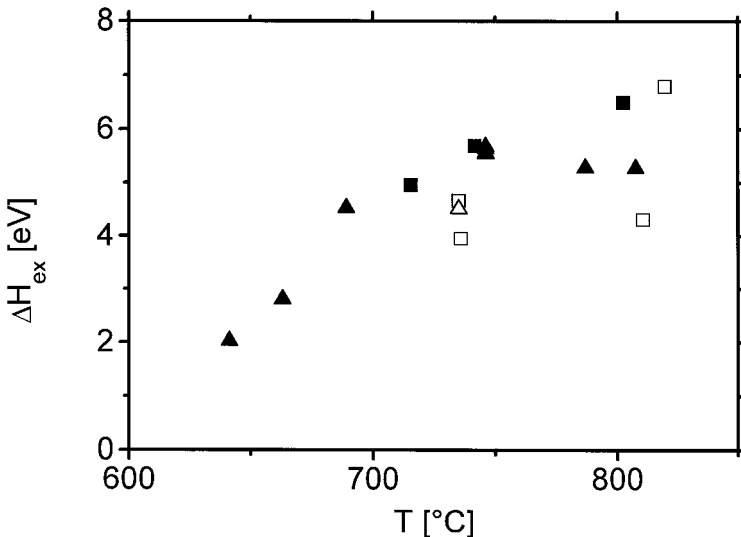


Figure 2. Experimental activation enthalpy ΔH_{ex} as a function of temperature: filled symbols, fivefold orientation of the compression axis; and open symbols, twofold orientation; triangles, strain rate of 10^{-5} s^{-1} ; and squares, 10^{-4} s^{-1} .

al. (1999) as described in §3 using equation (7) for σ_i and by the theoretical value of $(d \ln \rho / d \ln \dot{\epsilon})_T = 1/2$ or C_r (equation (3) in Part I). The corrections for the temperature sensitivity and the strain rate sensitivity compensate each other partly. The values obtained of ΔH are either about 20% lower or higher than the apparent ones of equation (26) depending on the correction of the strain rate sensitivity using either equation (23) with $(d \ln \rho / d \ln \dot{\epsilon})_T = 1/2$, or equation (24) via C_r . Because of the uncertainty in particularly σ_i , corrected values are not dealt with here. Nevertheless, the ΔH_{ex} data in figure 2 should represent the true activation enthalpy ΔH of the dislocation mobility within an accuracy of $\pm 20\%$, with the corrections being smallest at the lowest temperatures used (640°C).

The Gibbs free energy of activation ΔG can be calculated from ΔH by means of equation (11). This formula is based on the assumption that the only contribution to the activation entropy originates from the temperature dependence of the shear modulus. It contains the temperature sensitivity of the shear modulus $\beta = (T/\mu)(d\mu/dT)$. As in §3, it was taken from the data of Feuerbacher *et al.* (1996). Like ΔH , ΔG assumes approximately constant values above about 720°C . They are about 3.4 eV for specimens with a fivefold compression axis, and 2.9 eV for those with a twofold axis. Below that temperature, ΔG decreases markedly. According to equation (4), ΔG should be proportional to T if the deformation were controlled by a single thermally activated process, i.e., if the pre-exponential factor $\dot{\epsilon}_0$ had a constant value. Figure 3 shows that this condition cannot be checked above 720°C because of the large scatter of the data. Clearly it is violated below 720°C . A consideration of the transients according to equations (23)–(25) would not reduce the discrepancy.

Figure 4 plots ΔG versus the shear stress. In this plot, at least the data for the same specimen orientation should lie along a single curve, which, however, is not

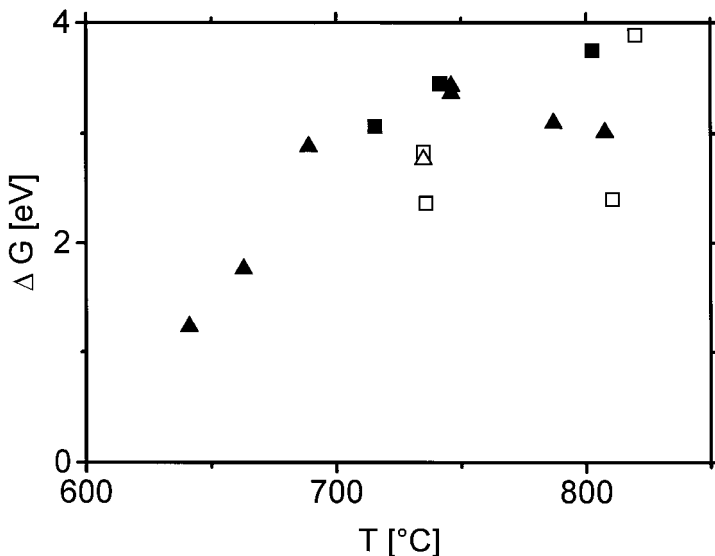


Figure 3. Temperature dependence of the Gibbs free energy of activation ΔG using Schöck's formula (equation (11)) and the temperature dependence of the shear modulus measured by Feuerbacher *et al.* (1996) (symbols as in figure 2).

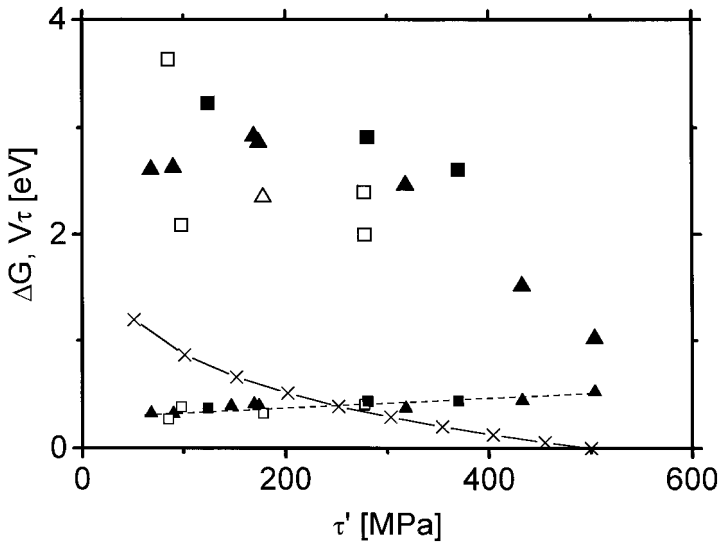


Figure 4. Dependence of the Gibbs free energy of activation ΔG on the reduced shear stress τ' with symbols as in figure 2 plus: \times and full line values calculated by the second integral in equation (25) using the data of figure 1; and small symbols and dashed line, upper limit $V\tau$ of the work term $V\tau^*$.

fulfilled well. As proposed by Cagnon (1971), alternatively ΔG can be determined by integrating the V versus τ' curve of figure 1:

$$\begin{aligned}
 \Delta G(\tau') &= \frac{\mu(T)}{\mu_0} \int_{\tau'}^{\tau'_0} V \, d\tau' \\
 &= \frac{\mu(T)}{\mu_0} \int_{\tau'_m}^{\tau'_0} V \, d\tau' + \frac{\mu(T)}{\mu_0} \int_{\tau'}^{\tau'_m} V \, d\tau' \\
 &= \Delta G(\tau'_m) + \frac{\mu(T)}{\mu_0} \int_{\tau'}^{\tau'_m} V \, d\tau'. \tag{27}
 \end{aligned}$$

τ'_0 is the limiting stress the obstacle array can sustain, and τ'_m is the maximum stress up to which the activation volume is measured. As τ'_0 itself and the course of V versus τ' between τ'_m and τ'_0 are not known, the integral is split into two parts. In figure 4, the known second integral is plotted as crosses plus line. With the exception of the unknown offset $\Delta G(\tau'_m)$, this curve should coincide with the data determined by Schöck's formula. It is obvious that the (negative) slope of this curve, which represents the activation volume, increases with decreasing stress while that based on Schöck's formula decreases. In addition, the difference between the measured values of ΔG and those predicted by integrating the $V(\tau')$ curve grows with decreasing stress. Thus, the results of determining ΔG by different methods are not consistent with each other. This indicates that the assumptions on which the formalism of thermally activated dislocation glide is based, i.e., a constant pre-exponential factor and an entropy contribution arising solely from the temperature dependence of the shear modulus, are, at least partly, not fulfilled for Al-Pd-Mn quasicrystals. Giacometti *et al.* (1999) undertook a similar analysis for Al-Cu-Fe polyquasicrystals without observing such an inconsistency. Figure 4 contains also an upper limit $V\tau$ of

the work term $V\tau^*$ (equation (5)). In comparison with ΔG , it is small and almost constant at 0.35 eV. The small work term compared with ΔG is typical of the high-temperature deformation.

§ 7. PROCESSES CONTROLLING THE DEFORMATION RATE

Three processes should be discussed which may influence the flow stress of Al-Pd-Mn quasicrystals. The first is the contribution τ_1 (or σ_1) from long-range dislocation interactions determined by equation (7). Based on the dislocation density data by Schall *et al.* (1999), this contribution is estimated in § 3 to amount to about 30% of the flow stress. Unfortunately, the constant α in equation (7) is not known exactly and the results of dislocation density measurements depend on the unloading and cooling conditions of the specimens, so that this result has a large error. The influence of changes in σ_1 during the measuring procedures of the activation parameters is discussed in § 4. The total effect of the transients amounts to about 20% of the respective quantity. It compensates partly for the activation enthalpy discussed in § 6. Up to now, no dislocation density data are available to estimate the athermal component of the flow stress below 695°C.

The second process is the friction mechanism, which controls the glide velocity of dislocations according to the Arrhenius-type relationship of equation (3). It has been proposed by Feuerbacher *et al.* (1997) and by Urban *et al.* (1998) that the thermally activated overcoming of the Mackay-type clusters is the rate-controlling process. This conclusion has been drawn from the observation that the measured activation volumes fit the size scale of the clusters. The model has been elaborated in a more quantitative way by Messerschmidt *et al.* (1999b), considering the clusters as extended obstacles according to the theory of solution hardening by Labusch and Schwarz (1991). The cluster friction model is in accordance with the viscous motion of dislocations during *in situ* observations in a high-voltage electron microscope by Wollgarten *et al.* (1995) and Messerschmidt *et al.* (1998).

The third process is recovery, which may take place during plastic deformation. It is evidenced by the static annealing experiments of the dislocation density by Schall *et al.* (1999), which can be interpreted by equations (12) and (13). Recovery manifests itself in the difference between the original and repeated relaxation curves as in figure 4(a,b) of Part I. Recovery is necessary to compensate for work-hardening to explain the steady state deformation occurring at high deformation temperatures.

There are two temperature ranges which have to be distinguished and in which the three processes contribute to the deformation characteristics in different ways. Below about 630°C, steady state deformation cannot be achieved at strain rates of 10^{-5} s^{-1} or higher. The stress increases mainly elastically above about 1000 MPa, where the specimens break. First results on the deformation processes in this low temperature range are described by Messerschmidt *et al.* (1999b).

Above 630°C, recovery prevents the stress from attaining very high values, and it allows for steady state deformation. It is a remarkable feature of the deformation of Al-Pd-Mn that over the whole temperature and stress ranges the activation volume V is a unique function of the stress (figure 1), regardless of how the stress is varied. This fact can be interpreted in two different ways. The first assumes that the activation volume V is correctly determined by equation (24) and characterizes the dynamic law of equations (4) and (5). Then, figure 1 is a universal expression of the interaction potential of the dislocation velocity-controlling process, which is assumed to be the cluster friction mechanism. In this case, the stress plotted in figure

1 corresponds mainly to the thermal stress component τ^* or, because of the Cottrell–Stokes law, it is at least proportional to it. Then, the stress dependence of the Gibbs free energy of activation can be constructed by integrating the V versus τ' curve. Apart from an unknown offset, it is shown in figure 4. These values show a different dependence on the reduced stress than do the ΔG values measured by temperature change experiments and by using the formalism of Schöck (1965). The latter values at high stresses, i.e., below about 670°C, are of a ‘reasonable’ order of magnitude. Since recovery should not be important in this range, these values should correspond to the process controlling the dislocation mobility. Then, $\Delta G + V\tau$ is a rough estimate of the total activation energy at zero stress, which amounts to about 2 eV. The values above 670°C, however, are too large and correspond to unreasonably high pre-exponential factors in equation (4). A possible explanation has been proposed by Feuerbacher *et al.* (1997) assuming that the production of phasons while the dislocations are moving in quasicrystals can be connected with contributions of configurational entropy ΔS in equation (11), in addition to those from the temperature dependence of the elastic constants considered by Schöck (1965).

In the elaborated cluster friction model of Messerschmidt *et al.* (1999a), the activation energy to overcome the individual clusters is only a fraction of the measured value. Thus, it should amount to less than 1 eV, so that the clusters do not represent strong obstacles to the dislocation motion. However, this model predicts a weak temperature dependence of the flow stress at high temperatures, which has not been observed experimentally (figure 3 of Part I).

An alternative interpretation of the unique relation between activation volume and stress starts with the statement that during the steady state deformation a dynamic equilibrium exists between work-hardening and recovery. During the relaxation tests, the dislocation structure, represented mainly by the dislocation density, depends on the deformation conditions, but the deformation under decreasing strain rates always takes place in or near the steady state. In this case, the V versus τ' curve does not describe the process controlling the dislocation velocity. In this interpretation, the flow stress should be controlled mainly by the long-range athermal component τ_i . Thus, the main difference between both interpretations lies in the different value of the athermal component of the flow stress. In the latter case, the Gibbs free energy of activation in the steady state range is the activation energy of recovery. The value of about 3.5 eV is close to the energy of about 4 eV determined in §3 (equation (18)) for the recovery of the dislocation density. However, both energies are higher than the self-diffusion energy, which should control recovery. The diffusion energy amounts to 2.32 eV for Pd (Blüher *et al.* 1998), and to 1.99 eV for Mn (Zumkley *et al.* 1997). To the authors’ knowledge, the diffusion energy of Al has not yet been determined. The slowest-moving atomic species should control the recovery process. Unreasonably high values of the activation energy are not unusual for the recovery-controlled creep of ceramic materials, see, e.g., Goméz-García *et al.* (1996) for cubic zirconia, but the reason for that is not clear. The crucial point may be that the cluster friction process implies a relatively high strain rate sensitivity, similar to that of recovery, so that it is almost impossible to separate the deformation processes under high stresses (or at low temperatures) controlled by the dynamic law of equation (4), i.e., the cluster friction mechanism, from the deformation process under low stresses (or at high temperatures) controlled by a steady state of the dislocation structure. Thus, it is possible that the deformation of Al–Pd–Mn with

increasing temperature changes gradually from a dislocation friction-controlled mode to a recovery-controlled mode.

Until now, the activation energy has been discussed on the assumption that the deformation is controlled either by the dynamic law of equation (4) or by recovery-controlled creep with the activation energy of self-diffusion (equations (16) and (17)). However, as suggested above, it is also possible that both mechanisms control the deformation simultaneously, i.e., that the mobile dislocation density ρ in equation (4) is given by the steady state dislocation density ρ_{ss} of equation (16). Together with equation (17), this yields

$$\dot{\epsilon} = \left[\frac{\dot{\epsilon} w \tau}{b q_0 \exp(-\Delta H_{SD}/kT)} \right]^{1/2} \frac{b v_0}{m_s} \exp(-\Delta G_m/kT). \quad (28)$$

The index m is added to ΔG to refer to the dislocation mobility. If the (relatively weak) dependence of τ on $\dot{\epsilon}$ is neglected, it follows that

$$\dot{\epsilon} = \frac{w \tau b v_0^2}{k_0 m_s^2} \exp \left[-\frac{2\Delta G_m - \Delta H_{SD}}{kT} \right]. \quad (29)$$

Entropy terms are not considered in the recovery process. If the dependence of the denominator of the first term on τ is neglected, the experimental activation energy is given by

$$Q_{ex} \cong 2\Delta G_m - H_{SD}. \quad (30)$$

This means that the activation energies determined in §6 cannot be interpreted simply by either dislocation mobility or recovery. A consequence of equation (29) is also that the activation volume is given by $V = V_{ex}/2$.

§8. CONCLUSION

At high temperatures, plastic deformation of icosahedral Al-Pd-Mn single quasicrystals is characterized by a yield drop followed by a stage of steady state deformation. This behaviour can be explained by the kinetics of the dislocation generation and recovery. The occurrence of recovery seems to be a prerequisite to an extensive plastic deformation.

It is estimated from the dislocation density data of Schall *et al.* (1999) that the athermal component of the flow stress τ_1 may amount to about 30% of the flow stress at all temperatures. Thus, the Cottrell-Stokes law may be valid in icosahedral Al-Pd-Mn.

The activation volume V of plastic deformation, determined from stress relaxation tests taking into account structural changes during the relaxations, is a unique reversible function of the reduced flow stress τ' .

Under the above conditions, the V versus τ' curve represents the interaction profile of the process controlling the dislocation mobility.

The Gibbs free energy of activation obtained from temperature cycling experiments and stress relaxation tests using the formula of Schöck amounts to approximately 3.4 eV above about 700°C. Below 700°C it decreases rapidly with decreasing temperature. At low temperatures, the data should represent the processes controlling the dislocation mobility. The total activation energy at zero stress then amounts to about 2 eV.

The dependence of the Gibbs free energy of activation on the stress is not consistent with values obtained by integrating the V versus τ' curve. This indicates that the assumptions on which the evaluations of the activation parameters are based are not fulfilled.

The reversible dependence of the strain rate sensitivity on the stress also can be interpreted by the steady state recovery-controlled deformation, suggesting a large contribution by the athermal component τ_i to the flow stress.

Both interpretations are combined by the assumption that there is a continuous transition from the cluster friction mechanism controlling the deformation at low temperatures to the recovery-controlled deformation at high temperatures. The activation energy may then combine both individual energies.

Further experiments will be necessary for a better understanding of the transient effects and the role of the athermal component of the flow stress.

ACKNOWLEDGEMENTS

The authors are grateful to Professor J. Kirschner for his encouraging interest in this work. They thank the Deutsche Forschungsgemeinschaft for financial support.

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