JOURNAL OF MATERIALS SCIENCE 10 (1975) 387–392

Temperature and stress dependences of creep in single crystals of lead between 20 and 300 K

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The influence of temperature and applied stress on creep in single crystals of lead has been investigated. Between 20 and 180 K a thermally activated "logarithmic" creep is observed that is easily interpreted in terms of theory based on a work hardening concept. In case recovery processes are absent the weakening of the "logarithmic" creep is determined by hardening of the crystal during the creep process and the level of effective stresses. The creep vanishes when the effective stresses become zero. The disappearance of creep at the point T_0 has been shown experimentally. The temperature T_0 is that at which critical shear stresses reach the temperature-independent branch of the yield-point curve $\tau_T(T)$. At temperatures higher than T_0 a steady-state creep is observed. At a fixed stress the creep rate is determined by the rate of recovery processes. The results obtained are described in terms of thermally activated dislocation motion.

1. Introduction

Investigations carried out in recent years have revealed the existence of an appreciable creep in metals at liquid helium temperatures (i.e. close to 4.2 K). The effect is essentially independent of temperature, and extrapolation to 0 K of the obtained curves gives non-zero creep rates. This peculiarity of low-temperature creep was observed with cadmium [1, 2], zinc [3] and [4], copper [5], aluminium [6], lead [7] and nickel [8]. In all cases, the transient-regime creep curves could be fitted to the logarithmic law

$$\epsilon = \alpha \ln(\gamma t + 1) \qquad (1)$$

where ϵ is creep strain at the moment t, the factor α depends upon temperature and stress, and γ is a constant.

The observed creep phenomena were explained on the grounds of dislocation line zero oscillations and the effect they have on the line motion through the array of local lattice barriers. From theory developed on this basis [3] an expression for the plastic strain rate can be derived which, at high temperatures, reverts to the common equation of thermally activated plasticity, i.e.

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp\left[-\frac{Q(\tau^*)}{kT}\right] \qquad (2)$$

Here $Q(\tau^*)$ is activation energy and τ^* denotes the effective stress acting on the dislocation. At low temperatures the theory predicts a weak temperature dependence of plastic strain rate with a finite value at 0 K. A characteristic value, θ, (the temperature above which thermally activated deformation mechanisms prevail and below which quantum mechanical mechanisms prevail) is involved. Theoretical estimates for θ are $\frac{1}{5}$-$\frac{1}{10}$ T_D, where T_D is the Debye temperature. Experimental measurements with various materials have given values of θ of 26 K for zinc [3] and [4], 30 K for copper [5], 38 K for aluminium [6], 8 K for lead [7] and 15 K for cadmium [2].

Investigations of creep in lead have shown the transient regime between 1.5 and 8 K to be represented by the quantum mechanical formula of Nastik *et al.* [3]. At higher temperatures the effect passes gradually in to the usual thermally activated creep. However, the highest temperature reached in the experiments [7] was only 25 K. To establish definitely the dislocation mechanisms determining low-temperature creep, it seemed necessary to analyse temperature and stress dependences over a much wider range. For that purpose the present investigation of creep in single crystals of lead between 20 and 300 K was undertaken. The results are compared with temperature dependences characteristic of lower temperatures.

2. Experimental technique

Measurements were carried out with single crystals of 99.999% pure lead that had been given the shape of a double vane with a 1.5 mm × 5 mm × 25 mm working part and heads for the tensile machine grips. Owing to the specially developed method of crystal growth [9], the samples possessed a prescribed orientation and were all formed under practically the same conditions. Hence, the tests were performed on samples that could be regarded as identical. The specimens were subject to tensile straining along an axis close to the [1 1 1] vertex of the standard triangle; Schmid's factor approached 0.4. Prior to the measurements the crystals were annealed at room temperature and chemically polished.

The equipment used for studying creep in lead was of the same type as described in [10] and [11]. Temperatures in the range 20 to 110 K were obtained by cooling the sample with liquid helium vapour whilst between 110 and 300 K it was vapour-cooled with liquid nitrogen. Creep curves were recorded automatically providing an accuracy of $\pm 4 \times 10^{-6}$ with respect to the relative strain.

To investigate the temperature dependence of creep the curves were taken at constant values of the reduced shear stress, $\tau =$ constant, and at various temperatures. For the stress dependence studies similar curves were registered at various shear stresses, τ, and constant temperature, T. Desired values of stress were achieved by a stepwise loading of the sample, with steps ~ 8 to 10 gf mm^{-2} each. Upon application of each additional load, the creep curve was recorded over 10 to 60 min.

Initial data for calculating the activation volume were obtained by changing the applied stress during creep. The increase in stress from 1 to 5 gf mm^{-2} was always performed at the same creep rate, namely $\dot{\epsilon}_1 = \text{const} = 2 \times 10^{-6}$ sec^{-1}.

Strain curves $\tau = f(\epsilon)$ were also recorded, by the additional loading method, to determine the value of the critical shear stress τ, the work-hardening coefficient ϵ and temperature dependences of the two values. The stress applied to the sample was raised by 15 gf mm^{-2} every 30 sec and the respective strain increment was registered.

3. Results and discussion

3.1. Temperature dependence of creep

Creep in lead was studied over a range of stresses corresponding to the linear hardening section of $\tau = f(\epsilon)$. The initial portion of the hardening curve, typical for the temperature range under investigation, is shown in Fig. 1. The curve was taken by the additional loading method. As is often the case with fcc crystals, the easy glide stage decreased as the temperature was raised. For this reason, the minimum stress used in the measurements was so chosen as to fall within the linear portion of the hardening curve at any temperature in the range studied. The tests have shown a "logarithmic" creep conforming to Equation 1 to occur between 20 and 200 K (see Fig. 2a and b).

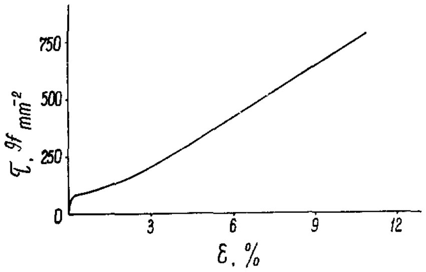

Figure 1 The hardening curve of lead single crystals, taken by the stepwise loading method, at $T = 77$ K.

From the data in Fig. 2b, the temperature dependence of factor α can be obtained. Fig. 3 shows the curve $\alpha = \alpha(T)$ corresponding to $\tau = 260$ gf mm^{-2}. As can be seen, the run of $\alpha(T)$ is non-linear as the factor is almost independent of temperature between 20 and 60 K, but then shows a roughly linear growth up to 190 K.

The creep theory based on a work-hardening concept [12] gives

$$\alpha = \frac{kT}{\mathcal{H}V} \tag{3}$$

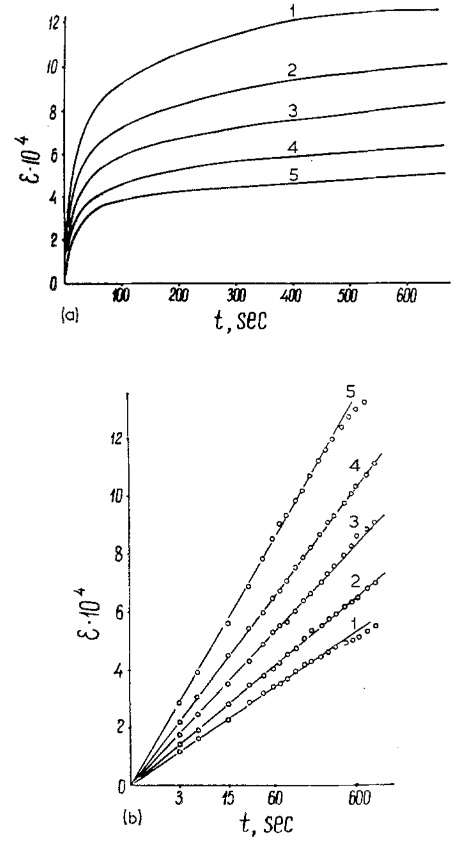

Figure 2 Creep curves represented as ϵ-t (curve a) and $\epsilon = \ln t$ (curve b). The dependences were taken at a constant deformation stress, $\tau = 260$ gf mm^{-2} at $T =$ (1) 20 K, (2) 77 K, (3) 106 K, (4) 130 K and (5) 184 K.

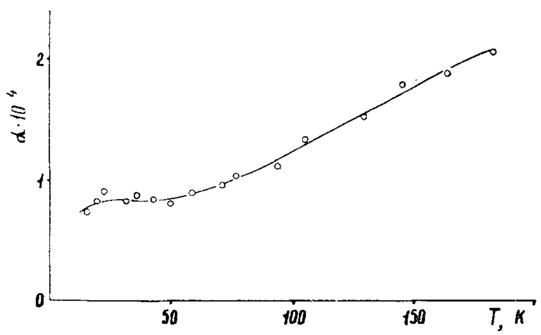

Figure 3 Temperature dependence of α at $\tau = 260$ gf mm^{-2}.

where T is temperature, k is Boltzmann's constant, $\mathscr{H}$ is work-hardening coefficient and V denotes activation volume. According to Equation 3, α should vary linearly with temperature, provided the product $\mathscr{H}V$ remains unchanged. In fact, this dependence may not be observed, as $\mathscr{H}$ and V also change with T. A comparison of the experimental curve $\alpha(T)$ with Equation 3 should be made bearing in mind this variation of the parameters. For the lead crystals used the work-hardening coefficient was independent of temperature over the range 20 to 200 K and was equal to $\sim 10^4$ gf mm^{-2} (linear stage of hardening). The temperature dependence of the activation volume V as determined at $\tau = 260$ gf mm^{-2} is given in Fig. 4a. The values presented were calculated according to the formula

$$V = kT\left(\frac{\partial \ln \dot{\epsilon}/\dot{\epsilon}_0}{\partial \tau}\right)_T \qquad (4)$$

assuming $\dot{\epsilon}_0 = \text{const}$. Fig. 4b shows the run of the product αV as obtained from the data of Figs. 3 and 4a. The value is seen to decrease linearly with decrease in temperature, tending to zero at $T \rightarrow 0$.

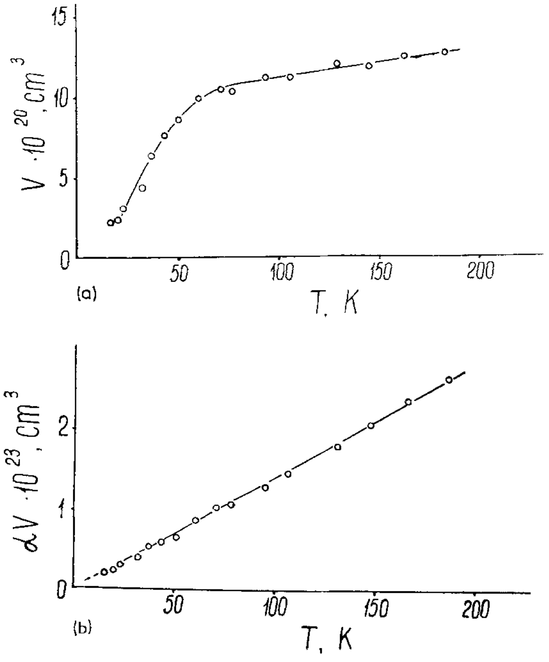

Figure 4 (a) Temperature dependence of the creep activation volume in lead at $\tau = 260$ gf mm^{-2}. (b) Temperature dependence of the product αV at the same stress.

Thus, the experimental data obtained for the temperature dependence of $\alpha(T)$ in the range 20 to 190 K are in fair agreement with Equation 3. The latter formula follows from the theory of low-temperature creep based on the work-hardening concept. It should be remembered that Equation 3 is only valid over a temperature range where representative effective stresses acting on mobile dislocations in the crystal are non-zero during the creep. To determine this range the yield point temperature dependence shown in Fig. 5 is used. Following Seeger [13] and [14], the effective stress, τ^*, is defined as the difference between the critical shear stress at a given temperature and at its athermic point. From Fig. 5 it can be seen that τ^* decreases with temperature and disappears at a T_0 within the range 180 to 220 K (more exact determination requires greater experimental detail). At stresses greater than the yield point, T_0 shifts towards higher temperatures. However, the shift is not large; for the stresses used in the present experiment it never surpassed 20 to 30 K. Thus, temperature range where the "logarithmic" creep is observed (see Fig. 3) coincides with that of positive effective stresses, ($\tau^* > 0$).

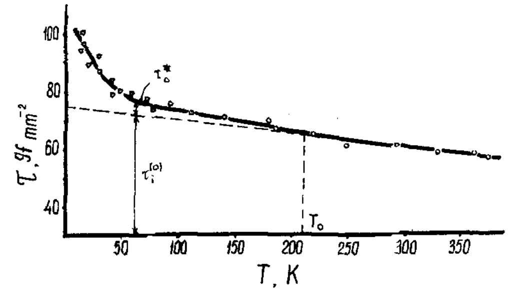

Figure 5 Temperature dependence of the critical shear stress in lead. The broken line shows the temperature dependence of the shear modulus.

Using the data of Osetski *et al.* [7], the $\alpha(T)$ can be extended to 1.5 K. However, Osetski *et al.* measurements were carried out for $\tau = 600$ gf mm^{-2} whilst the present results were obtained at $\tau = 260$ gf mm^{-2}. To make the data comparable, the temperature dependence of αV must be plotted (Fig. 6). As can be seen, the curves relating the two temperature ranges fit together. The departure from linearity observed at the lowest temperatures may be due to the influence of quantum effects [3, 7].

At temperatures above 200 K the creep alters sharply in character. The first peculiarity is inversion of the temperature dependence observed until about 240 to 250 K: the creep rate drops with increase in temperature (Fig. 7a).

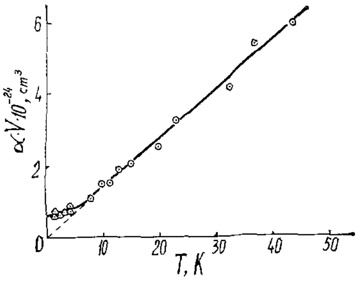

Figure 6 Temperature dependence of αV in the range 1.5 to 40 K. At temperatures above 20 K the curves of Figs. 4b and 6 coincide.

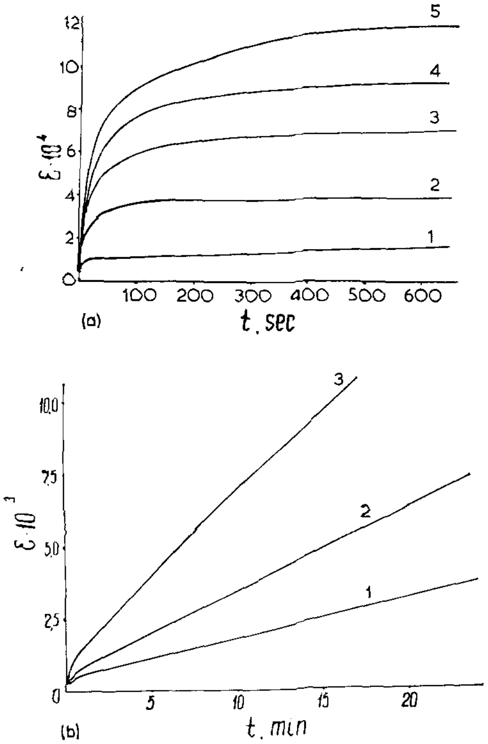

Figure 7 (a) The effect of temperature on the run of creep curves in the range 180 to 260 K. The curves were obtained under constant stress, $\tau = 260$ gf mm^{-2}. T = (1) 255 K, (2) 240 K, (3) 225 K, (4) 210 K and (5) 190 K. (b) Creep curves at $T = 296$ K. τ = (1) 250 gf mm^{-2}, (2) 280 gf mm^{-2}, (3) 340 gf mm^{-2}.

Another peculiar feature is the reduction of the transient creep stage that accompanies increase in temperature. At approximately 260 K the transient creep is not observed (Fig. 7a) and the steady-state creep rate is very low. Above 260 K the strain increment is almost exclusively due to secondary creep, the rate of which increases with temperature and stress (Fig. 7b). At first sight, these peculiarities cannot be interpreted in terms of a thermally activated mechanism of plastic strain. However, more detailed consideration allows the results to be explained within the framework of thermal activation.

According to theory based on the work-hardening concept [12], mobile dislocation segments in a creeping crystal are subject to an effective stress

$$\tau^* = \tau_0^* - \mathscr{H}\epsilon \qquad (4)$$

where τ_0^* is the stress at the initial moment and ϵ is the creep strain. Increase in strain during the creep process causes a continuous decrease in effective stress, which eventually becomes zero. From that moment on the creep should halt if recovery processes are absent. The development of the phenomenon is shown schematically in Fig. 8 (curve 1). τ_0^* decreases as the temperature is raised (see Fig. 5), hence the effective stresses disappear at lower values of creep strain (Fig. 8, curve 2). Curve 3, Fig. 8, corresponds to a still higher temperature. The diagram shows that an increase in temperature should bring about a reduction in duration of the transient stage, accompanied by a decrease in final creep strain. The latter completely vanishes at $T = T_0$. As can be seen from Fig. 7a, the suggested scheme agrees qualitatively with the dependences observed in the experiment. Initial sections of the experimental creep curves in Fig. 7a have merged. Apparently, for this reason, the characteristic intersections of the creep curves predicted for the idealized scheme (Fig. 7b) are not seen. Equation 4 gives, for the strain at which halts the transient creep:

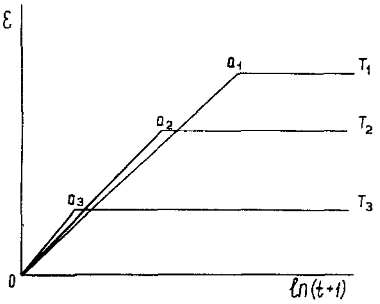

Figure 8 Schematic curves showing the effect of temperature upon deviations from the logarithmic creep law at $\tau^* \sim 0$.

$$\epsilon = \tau_0^*/\mathscr{H} . \qquad (5)$$

Assuming $\mathscr{H} = 10^4$ gf mm^{-2} and $\tau_0^* = 1$ to 2 gf mm^{-2} (which corresponds to $T = 200$ K) we obtain $\epsilon = 1$ to 2×10^{-4}. The time necessary to reach this strain is several seconds which cannot be resolved in Fig. 7a. Note that the intersections may not be observed if the activation volume increases sharply as the effective stress approaches zero (see Equation 3).

If the temperature is increased further, recovery processes begin to play an important role, increasing the steady-state creep rate. Fig. 7b shows some creep curves for $T = 296$ K where the recovery processes are quite developed.

The explanation suggested above for the observed behaviour of creep at $T > 200$ K seems rather probable. However, to prove that it is correct a more detailed study of the creep dependences over the temperature range studied is needed.

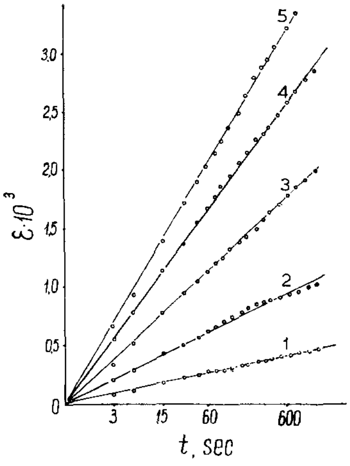

Figure 9 Stress dependence of the "logarithmic" creep at $T = 77$K. $\tau =$ (1) 180 gf mm^{-2}, (2) 310 gf mm^{-2}, (3) 450 gf mm^{-2}, (4) 600 gf mm^{-2} and (5) 720 gf mm^{-2}.

3.2. Stress dependence of creep

The stress dependence of transient low-temperature creep has not been studied until now. Therefore, it is of interest to investigate this dependence over the temperature range where a marked "logarithmic" creep is observed. Fig. 9 shows the logarithmic creep curves of lead single crystals obtained at $T = 77$ K for a variety of stresses (the "instantaneous" deformation stage is not shown). The range of the stresses studied corresponded to the linear hardening stage on the $\tau = f(\epsilon)$ curve (Fig. 1). In Fig. 10a stress

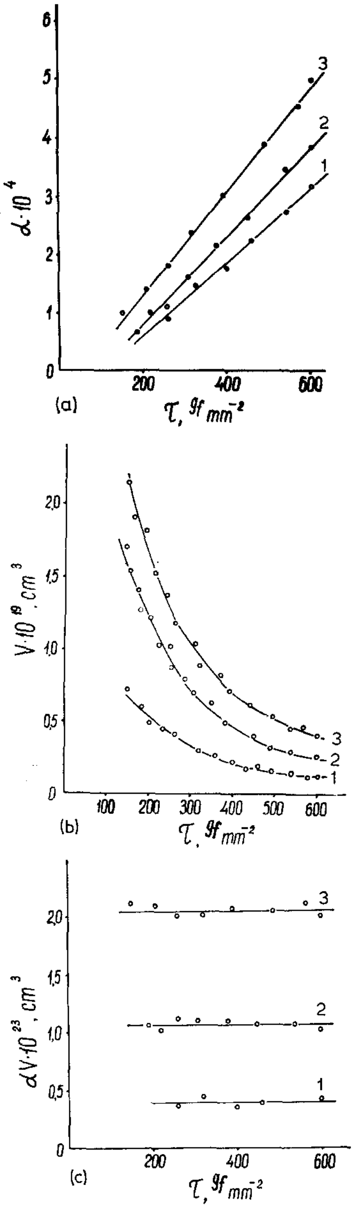

Figure 10 Stress dependences of the factor α (curve a), the activation volume V (curve b) and the product αV (curve c) at (1) $T = 33$ K (2) $T = 77$ K and (3) $T = 146$ K.

dependences of the coefficient α are given which were taken at $T = 33$, 77 and 146 K: these are linear. According to Equation 3, the stress dependence of α at the linear hardening stage is determined by that of the activation volume, V, because the hardening coefficient $\mathscr{H}$ is stress-independent in that range. Hence, the product αV should be independent of τ. To establish the validity of this prediction the stress dependence of the activation volume was investigated. The results obtained for several temperatures are shown in Fig. 10b. The $V(\tau)$ curves are of hyperbolic form typical for this dependence. Fig. 10c shows $\alpha V = f(\tau)$ for the same temperatures. The independence of αV of τ is quite obvious.

The value of the hardening coefficient $\mathscr{H}$ obtained from $\alpha V = f(T)$ equals 9.8×10^3 gf mm^{-2}. This is very close to the magnitude found from the $\tau(\epsilon)$ curve, namely 8×10^3 gf mm^{-2} at the linear hardening stage. This implies that Equation 3 not only provides a correct qualitative description of thermally activated transient creep but can also be used for quantitative calculations of the temperature and stress dependence of creep. Therefore, the results obtained confirm that creep in the temperature range 20 to 300 K can be explained in terms of a thermally activated dislocation motion.

Acknowledgements

The authors wish to express their gratitude to Yu. G. Kazarov and V. V. Demirski for growing the single crystals.

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Received 18 June and accepted 2 October 1974.