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Incubation periods and indentation creep in lead

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The experimental variation of the microhardness of lead with dwell time and temperature is reported. At homologous temperatures close to $0.5\,T_m$, and using low loads (i.e. 0.1 N), a period of zero indentation creep of the order of 300 sec is observed. This phenomenon, called the incubation period, and the subsequent indentation creep behaviour, are explained in terms of restructuring and recovery of the dislocation network beneath the indenter.

1. Introduction

The micro-indentation hardness test provides a simple and virtually non-destructive method of investigating the mechanical properties of solids. However, the results obtained may be influenced by a number of experimental variables including the shape of the indenter; anisotropy in the material properties; the normal load; the duration of the indentation cycle; etc. Changes of hardness number with increasing load time, i.e. the dwell time, have been noted in a variety of materials [1] and have been interpreted in terms of recovery or creep related phenomena. In particular, the analysis of Atkins *et al.* [2] based on the Mott model of primary creep has been used to interpret the decrease in hardness as a function of dwell time for homologous temperatures above $0.5\,T_m$. Subsequently, this method has been used in conjunction with a number of crystalline solids to determine the activation energy for the indentation creep process – see Table I; and those results are consistent with a controlling mechanism based on volume diffusion enabling dislocation climb.

In extending indentation and scratch hardness measurements for a wide range of solids and experimental conditions, we have observed a phenomenon in the behaviour of lead at room temperature which has not been reported previously but which, on further preliminary investigation, appears to be common to the behaviour of other crystalline solids indented at temperatures $\geqslant 0.5\,T_m$.

2. Experimental details

Plane specimens for hardness testing were initially prepared by spark machining from a large ⟨1 0 0⟩ orientation single crystal of lead at 99.99% purity grown by the Bridgman technique (supplied by Metal Crystals Ltd). The damage introduced by this procedure was removed by extensive and repeated etching and polishing with molybdic and nitric acids. Indentation hardness measurements were made on the polished low index faces using a Leitz Miniload hardness machine with various diamond indenters; dwell time; and a range of normal loads from 0.1 to 10 N. A small hot stage enabled the effect of increasing temperature on the hardness behaviour to be investigated in the range 300 to 465 K.

3. Results and discussion

A typical example of a hardness against log time curve, in this case at 300 K and using a 0.1 N load with the Knoop indenter aligned in a [1 1 0] direction on a (0 0 1) plane, is shown in Fig. 1. There was some scatter in the readings and each point, for dwell times up to 1000 sec, represents the mean of at least five indentations. The most significant feature of these data was the incubation period – approximately 300 sec in this particular case – before the onset of indentation creep. Some evidence of steps in the subsequent creep portion of the curve was observed, but this effect was eliminated by the use of a suitable lubricant applied to the surface before indentation. The

TABLE I Activation energies for indentation creep [2, 6] in kJ mol^{-1} and as nRT_m where T_m is the melting point and R the gas constant

Al	172	$22RT_m$
Sn	73	$18RT_m$
MgO	460	$19RT_m$
SiC	527	$21RT_m$
WC	460	$18RT_m$

length of the incubation period was found to vary slightly with indenter geometry (Knoop, Vickers, conical, etc.) and with indenter or crystal orientation. However, the effect of changes in the load or temperature was much more pronounced. For example, the incubation time was reduced to approximately 20 sec for a load increase from 0.1 to 10 N at room temperature – or by increasing the temperature to 400 K ($0.67\,T_m$) using a constant load of 0.1 N. Similar time dependent behaviour was found whilst indenting a polycrystalline specimen.

The reproducibility of the effect and the depth of penetration of the indenter in a material as soft as lead tend to mitigate against a surface or oxidation related phenomenon and suggest that the shape of the H_k–log t curve is a consequence of recovery effects. This is reinforced by the results obtained at higher homologous temperatures. Atkins *et al.* [2] have shown that the hardness behaviour above homologous temperatures of about $0.5T_m$ can be interpreted using the equation:

$$p^{-3} - p_0^{-3} \propto \exp\left(\frac{-Q}{3RT}\right)(t^{1/3} - t_0^{1/3}) \qquad (1)$$

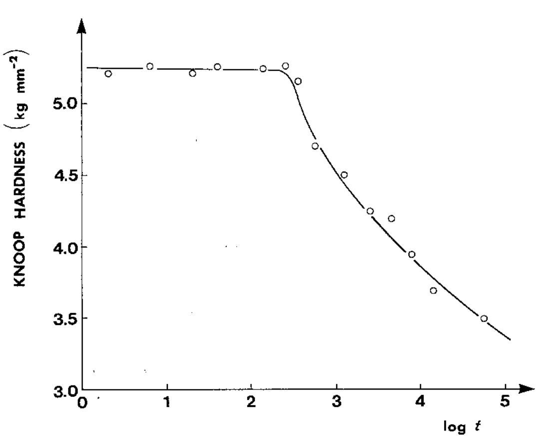

Figure 1 Variation of Knoop hardness with dwell time ("t" in sec) for single crystal lead.

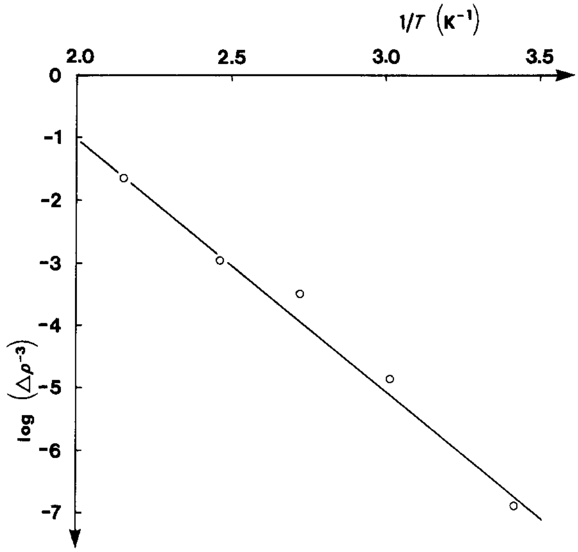

Figure 2 Indentation creep data, for single crystal lead, plotted in the Atkins *et al.* [2] form as log (Δp^{-3}) – [log ($p^{-3} - p_0^{-3}$) at constant $t^{1/3} - t_0^{1/3}$] against $1/T$ (K^{-1}).

where p and p_0 are the hardness values at time "t" and "t_0", "T" the absolute temperature, "Q" the activation energy of the creep process and "R" the gas constant. Hence a plot of the logarithm of ($p^{-3} - p_0^{-3}$), for a constant ($t^{1/3} - t_0^{1/3}$), against $1/T$ may be used to determine "Q".

Indentation creep measurements were carried out at five temperatures between 300 ($0.5\,T_m$) and 465 K ($0.78\,T_m$) and the results obtained after the incubation period plotted in this way (Fig. 2). The linear relationship is good and the apparent activation energy obtained from the slope of this line is 101 kJ mol^{-1} ($20RT_m$). Compensation for the temperature dependence of the elastic modulus ("E") [3], should reduce this by approximately 3 kJ mol^{-1} ($0.6RT_m$) to 6 kJ mol^{-1} ($1.3RT_m$) over the temperature range from 300 to 450 K. In any event, the activation energy is in close agreement with those obtained from conventional creep and self-diffusion measurements. Consequently, we conclude that indentation creep, beyond the incubation period, is controlled by a mechanism of bulk vacancy diffusion.

Incubation periods, i.e. intervals of zero change of strain following a stress reduction in conventional creep tests, have been reported for various metals (e.g. aluminium, nickel and copper). The phenomenon has been analysed by a number of authors [4, 5] in terms of the time required for the three dimensional network of dislocations

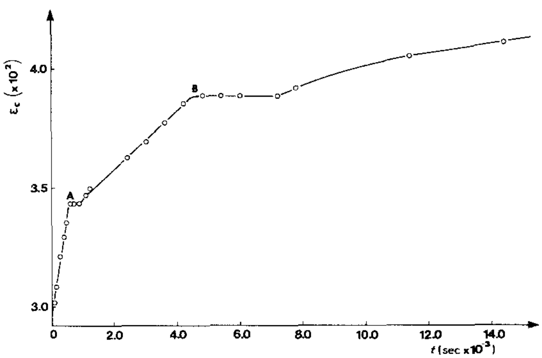

Figure 3 Compressive creep data, for single crystal lead, plotted as compressive strain (ϵ_c) against time "t" (sec).

established during secondary creep processes to be restructured following a stress reduction. Dislocation network analysis [5] describes the incubation time, Δt, in terms of the relative stress reduction ($\Delta\sigma/\sigma$) using the equation:

$$\Delta t = \frac{1}{2} \cdot \frac{G}{M_c b \sigma^2} \left[\frac{(\Delta\sigma/\sigma)(2 - \Delta\sigma/\sigma)}{\left(1 - \frac{\Delta\sigma}{\sigma}\right)^2} \right] \qquad (2)$$

where "G" is the shear modulus, "b" the Burgers vector and M_c the climb mobility of dislocations.

A similar effect during the primary creep phase in lead has been established in a simple compression test at room temperature on small blocks of the lead crystal used in these hardness measurements. The results are shown in Fig. 3. At "A" the stress was reduced slightly from 6.57 to 6.231 MN m^{-2} and subsequently, at "B", to 5.88 MN m^{-2} – new creep rates were established after an interval of zero creep in each case. Thus, although originally applied to linear secondary creep, the salient features of dislocation restructuring might be applicable to the primary regime. However, it should be borne in mind that the climb mobility is related to the diffusion coefficient "D" by:

$$M_c = D/kT \qquad (3)$$

where kT is the thermal energy and "D" itself varies exponentially with temperature. Also, the non-linear creep in this regime will introduce further variables. Consequently, the incubation period in the primary regime will be reduced in a more complex fashion than in secondary creep as the temperature is increased.

Now let us consider the incubation period in the indentation creep of lead. The conditions for the rigid–plastic mechanism of indentation are well established [6]. First, the solid should be isotropic. Although this would not generally be so for single crystals indented at low homologous temperatures [7], the turbulent nature of dislocation motion and plastic deformation in solids at homologous temperatures $> 0.5\,T_m$ justifies this assumption for the behaviour of lead crystals in these experiments. Secondly, work hardening should not occur during the indentation process. Again, at temperatures above $0.5\,T_m$, this should hold since the density of dislocations should remain constant as thermal softening and annealing offsets dislocation interactions and work hardening [8]. Finally, the value of $E/Y \tan\theta$ should be greater than 100. In the case of Knoop indentations in lead at room temperature, and using 72.12° for the semi-angle of the Knoop indenter [9] with a measured 15 MN m^{-2} for the flow stress ("Y"), this value is 345. Then we can determine the ratio of the radius of the elastic–plastic boundary ("c") to that of the hydrostatic core ("a") from:

$$\frac{\bar{p}}{Y} = \tfrac{2}{3} + 2\ln(c/a) \qquad (4)$$

where $\bar{p}$ is the mean indentation pressure acting over the boundary at "a". The resultant value of $c/a \simeq 3.8$ implies that elastic yielding of the hinterland, i.e. the material outside the deformed (or dislocated) volume, should not affect the plastic flow of the specimen. These conditions then correspond to the classical theory of a rigid–plastic solid. The indentation process produces localized stress reductions during the initial penetration of the indenter and the formation of the hydrostatic core. With the load still applied, a quasi-static elastic–plastic boundary is formed at "c". It is then reasonble to consider that, between the boundaries at "a" and "c", there are concentric shells of material subjected to a stress gradient ranging from the mean indentation pressure ($\bar{p}$) at "a" to the critical resolved shear stress (τ_c) at "c". Although it is generally supposed that indentation creep occurs by the expansion of the boundary at "c" into the elastic hinterland, the analogy with conventional creep is more convincingly drawn by reference to the expansion of the hydrostatic core, increasing the size of the indentation whilst decreasing the mean pressure $\bar{p}$. Thus the boundary at "a" moves, at a lower stress, through previously dislocated material. This process will be repeated by the concentric shells of material along the stress gradient between "a" and "c" –

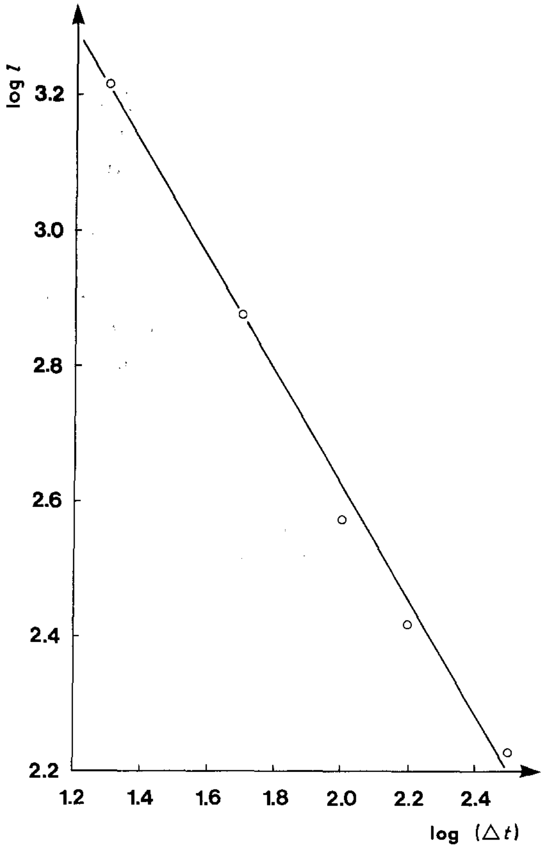

Figure 4 Logarithmic plot of incubation time and indentation size at a dwell time of 12 sec.

ultimately expanding "c" to maintain a constant c/a ratio. From the earlier considerations we might suppose that the incubation period is a function of the stress gradient from "a" to "c", i.e.:

$$\Delta t \propto \frac{(\bar{p} - \tau_c)}{(c - a)} \qquad (5)$$

Thus Δt will be proportional to a^{-1} and the stress gradient will be continuously ameliorated with increasing dwell time. A logarithmic plot of Δt against the indentation length "l" (at 12 sec dwell time), which is closely related to "a", is shown in Fig. 4. The linear relationship is good and the exponent predicted from the slope is 0.9, in reasonble agreement with the above analysis. We may therefore conclude that the duration of the incubation period will be dependent on such experimental variables as normal load, temperature, and crystallographic orientation of the indenter on a given crystal plane.

4. Summary

In summary, indentation creep in lead at homologous temperatures $> 0.5\,T_m$ can be explained in terms of a primary creep process with an activation energy close to that of self-diffusion and it is therefore concluded that this process is controlled by a mechanism of vacancy climb. At low loads and in the region of $0.5\,T_m$, an incubation period is observed in indentation creep which is thought to be due to a redistribution of the dislocation networks below the indenter analogous to that observed in conventional creep tests.

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