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Investigation of the superplasticity of tin–lead eutectic by impression creep tests

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The superplasticity of the Pb–Sn eutectic was investigated in the temperature range of 283 to 340 K. The strain-rate sensitivity, the activation energy and the activation volume were determined by impression creep measurements at various strain rates and temperatures. The results obtained are in good agreement with the results of conventional unidirectional tensile tests, which show that the simple and economic impression creep test can lead to equivalent results with the conventional creep test.

1. Introduction

Conventional creep testing requires many specimens to examine stress and temperature effects, therefore indentation hot hardness is used sometimes as a substitute. The utility of these measurements can be enhanced by changing the spherical or conical indenter to a flat cylindrical punch. This new measuring technique was developed by Chu and Li [1] and Murty [2].

Chu and Li [1] proved that impression creep curves can be converted to conventional tensile creep curves. To do this the impression velocity and the impressing load in an impression creep test have to be converted to equivalent strain rate and stress in a tensile creep test. An exact determination of the strain rate in impression creep is not possible since the strains around the punch are not known. However, Yu and Li [3] have shown that the impression creep velocity is proportional to the size (i.e. the diameter) of the punch. By means of finite element analysis they also showed that the equivalent steady-state strain rate, $\dot{\varepsilon}_s$, can be given by

$$\dot{\varepsilon}_s = v/d \tag{1}$$

where v is the steady-state impression velocity and d is the diameter of the punch [3].

The equivalent stress, σ, can be determined by the formula

$$\sigma = p/3 \tag{2}$$

where p is the pressure just below the punch. This equation was developed by Hill [4]. He applied slip-field theory to the deformation of an ideal plastic metal pressed by a flat punch.

Recently Yu *et al.* [5] found a close agreement between the stress–strain curves of the compression and impression tests of some homogeneous materials (annealed mild steel, aluminium, nickel and copper).

The superplastic behaviour of Pb–Sn eutectic alloy has been reported by many workers [6–8]. Ahmed and Langdon [9] found an exceptional tensile ductility of 4850% for this alloy. The superplastic behaviour of Pb–Sn eutectic appears in general in the temperature range of $273 < T < 413$ K. It has been demonstrated that this alloy shows a sigmoidal relationship between the steady-state strain rate and the applied stress. This relationship can be divided into three distinct regions. The maximum superplastic ductility is achieved in Region II where the activation energy is low and the strain-rate sensitivity is high [10]. According to literature data the activation energy in this region is between 42 and 57 kJ mol^{-1} [11–14].

It can be seen that the superplastic properties of lead–tin eutectic are very well documented, so it can be used as a model material for testing new measuring techniques. In an earlier investigation [15] we have studied the superplastic indentation creep of lead–tin eutectic by a conventional microhardness tester. It was found that the strain rate sensitivity and the activation energy of this process are in good agreement with previous results obtained by tensile tests. In this paper the same material is used for testing the impression creep technique to study superplastic behaviour.

2. Experimental procedure

The impression creep tests were carried out in a self-made apparatus designed according to Chu and Li [1]. A schematic picture of the equipment is shown in Fig. 1. The sample (1) and the punch (2) are immersed in a water bath, the temperature of which is controlled to $\pm 0.25^\circ$ C by a thermostat. The cylindrical flat punch (2) of 1 mm diameter and the pull rod (3) are made of stainless steel. The pull rod can be loaded by different weights at its end (4). The impression depth is measured with an accuracy of $\pm 2 \times 10^{-4}$ mm by an indicator dial (5).

Fig. 2 shows the microstructure of a typical sample. The dark zones are lead, the light zones are tin crystallites. The square samples with edges of 5 mm were cut from a sheet with thickness of 3 mm.

3. Results

Fig. 3 shows the impression depth, h, of the punch as a function of time, t, at 303 K for different loads. In Fig. 4 the impression depth–time relations can be seen which were obtained at different temperatures and at a constant load.

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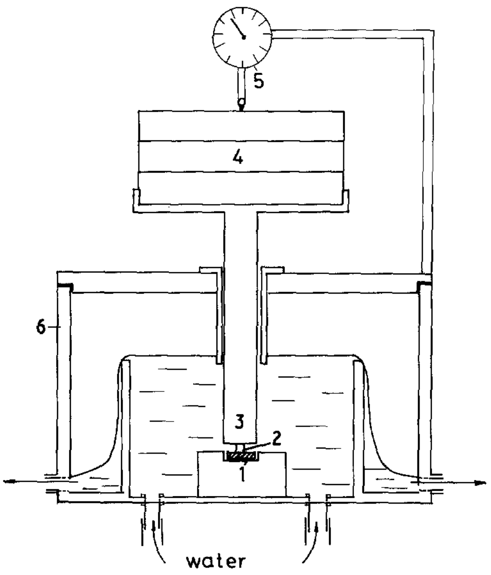

Figure 1 Schematic picture of impression creep apparatus: (1) specimen, (2) indenter made from stainless steel, (3) pull rod, (4) weights, (5) indicator dial, (6) bakelite vessel for thermostating the sample.

Both figures show typical impression depth against time curves which resemble conventional creep curves with both transient and steady-state stages. From the secondary part of the curves and the impression velocity of the punch and in the sense of Equation 1 the equivalent steady-state strain rate can be determined.

The validity of Equation 1 was proved by special measurements. Four punches of various diameters were loaded in such a way that the pressure below the punch was the same in each case. Fig. 5 shows the steady-state impression velocity of the punch at 40° C as a function of the diameter. The straight line obtained crosses the origin, so the v/d ratio due to a given press is constant.

Fig. 6 shows the log σ–log $\dot{\varepsilon}$ plot for different temperatures. The points measured at a given temperature lie typically along two different straight lines. At lower strain rates the slope, m, of the lines is higher and its value is about 0.4, while at higher strain rates $m \cong 0.2$. The higher the temperature, the larger is the extension of the part of the line with a higher slope.

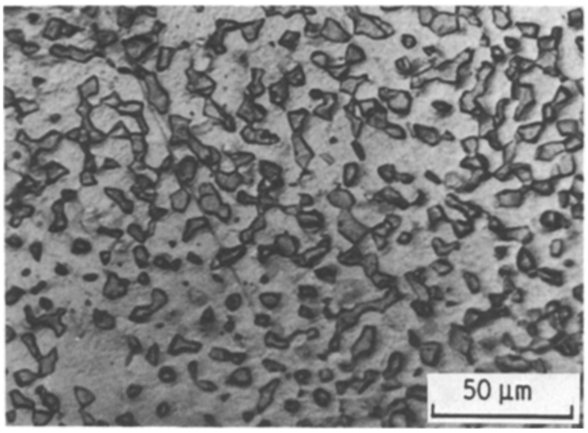

Figure 2 Optical micrograph of the lead–tin alloy.

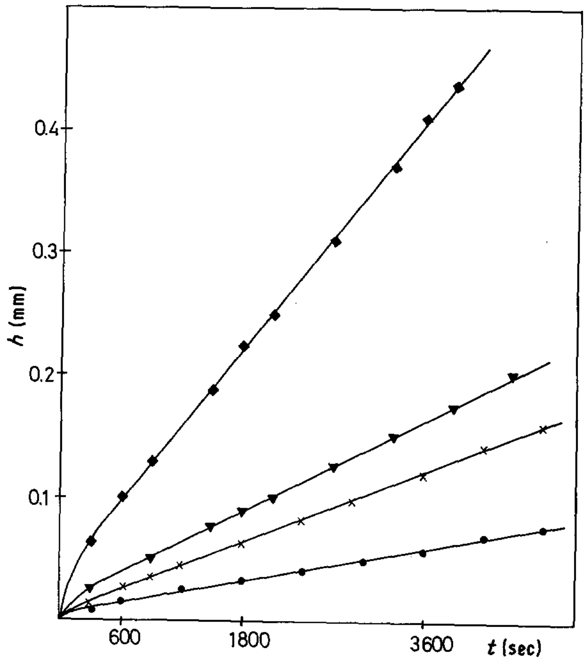

Figure 3 Impression creep curves at different stresses. $T = 303$ K. (◆) $\sigma = 50.5\,\text{N mm}^{-2}$, steady-state $V = 10.1 \times 10^{-5}\,\text{mm sec}^{-1}$; (▼) $\sigma = 38.3\,\text{N mm}^{-2}$, $V = 4.13 \times 10^{-5}\,\text{mm sec}^{-1}$; (x) $\sigma = 32.2\,\text{N mm}^{-2}$, $V = 3.15 \times 10^{-5}\,\text{mm sec}^{-1}$; (●) $\sigma = 26.1\,\text{N mm}^{-2}$, $V = 1.68 \times 10^{-5}\,\text{mm sec}^{-1}$.

The deformation can be regarded as a superplastic one if $m > 0.3$. In Fig. 6 the dotted line borders the region of superplastic deformation.

The equivalent strain rate, $\dot{\varepsilon}$, and equivalent stress, σ, determined from the present measurements can be described by the equation

$$\dot{\varepsilon} = C\sigma^{1/m}\,e^{-\Delta H/kT} \tag{3}$$

where C is a constant, which generally characterizes the steady-state creep. Here m is the strain-rate sensitivity parameter and ΔH is the activation enthalpy. To show the validity of this equation the data in Fig. 6

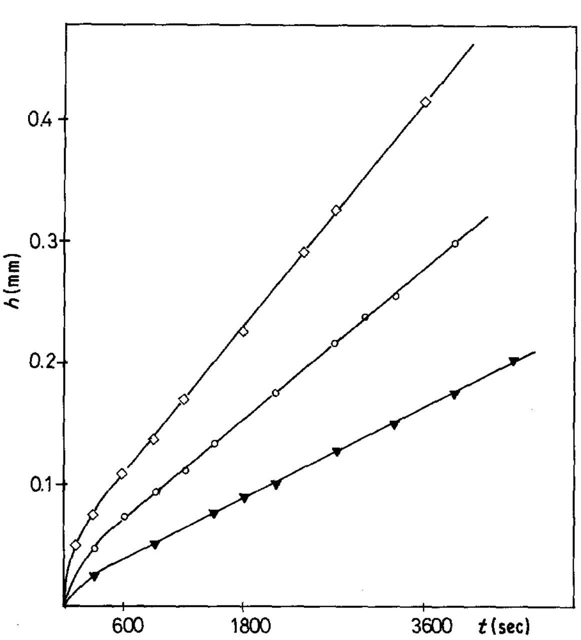

Figure 4 Impression creep curves at different temperatures. $\sigma = 38.3\,\text{N mm}^{-2}$. (◇) $T = 325.5$ K, steady-state $V = 10.31 \times 10^{-5}\,\text{mm sec}^{-1}$; (○) $T = 313$ K, $V = 6.86 \times 10^{-5}\,\text{mm sec}^{-1}$; (▼) $T = 303$ K, $V = 4.13 \times 10^{-5}\,\text{mm sec}^{-1}$.

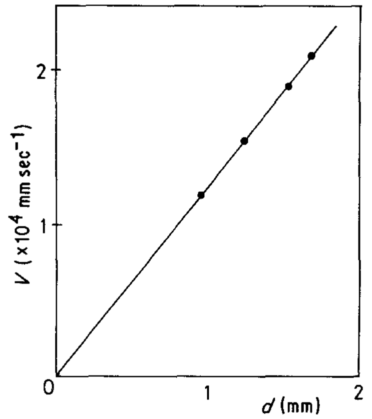

Figure 5 Impression velocity as a function of the diameter of the indenter. $T = 313\,\mathrm{K}$, $\sigma = 43.2\,\mathrm{N\,mm^{-2}}$.

are replotted in Fig. 7 to obtain the activation enthalpy, which as a function of the punching stress is shown in Fig. 8. The value of ΔH first decreases with increasing σ and after going through a minimum it increases. The value of ΔH extrapolated to zero stress is about $60\,\mathrm{kJ\,mol^{-1}}$.

The deformation process can also be characterized by the activation volume, V_a, given by

$$V_a = kT\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \tau_i}\right)_T \qquad (4)$$

where τ_i is the resolved shear stress. Supposing that τ_i is equal to the stress, τ, due to external forces and using the Taylor factor, τ can be approximated by

$$\tau = \sigma/3 \qquad (5)$$

In Fig. 9 the activation volume is plotted as a function of τ. The activation volume was determined from the slope of the ln $\dot{\varepsilon}$–τ curves.

The variable defined by Equations 4 and 6 is an apparent activation volume. The exact activation volume can be given by

$$V = kT\left[\left(\frac{\partial \ln \dot{\varepsilon}}{\partial \tau}\right)_T - \frac{1}{m\tau}\right] \qquad (6)$$

Fig. 10 shows this activation volume as a function of τ.

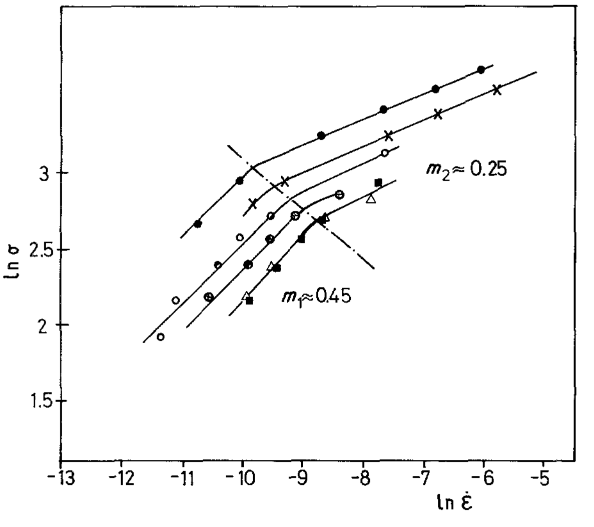

Figure 6 Double logarithmic plot of the σ–$\dot{\varepsilon}$ function. (●) 283 K, (x) 293 K, (○) 303 K, (⊕) 313 K, (△) 323 K, (■) 325.5 K.

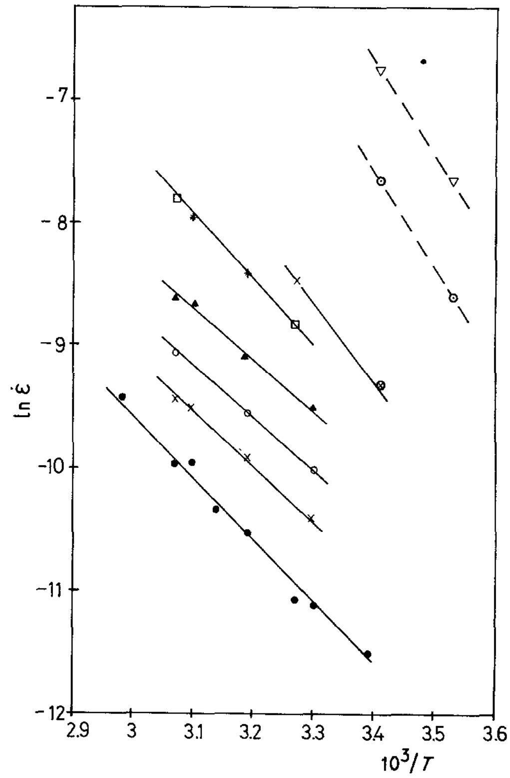

Figure 7 ln $\dot{\varepsilon}$ against ln $(1/T)$ curves for determination of the activation enthalpy. Values of ΔH (kJ mol^{-1}): (●) 42, (x) 37.4, (○) 35, (▲) 35, (□) 43, (⊕) 53, (⊙) ≈64, (▽) ≈61.

4. Discussion

The present results show that the equivalent strain-rate velocity defined by $\dot{\varepsilon} = v/d$ in the impression creep of Pb–Sn eutectic alloy can be described by Equation 3, namely

$$\dot{\varepsilon} = C\sigma^{1/m}\,\mathrm{e}^{-\Delta H/kT}$$

The deformation is regarded as superplastic if the strain-rate sensitivity $m > 0.3$. The m values obtained from impression creep measurements are in good agreement with the results determined on the basis of tensile creep measurements by Stüwe [16] and Kutsey *et al.* [14]. According to these results, Pb–Sn eutectic alloy is superplastic in the temperature range 10 to 65° C if $\dot{\varepsilon} < 10^{-5}\,\mathrm{sec^{-1}}$.

It is interesting to analyse the variation of the

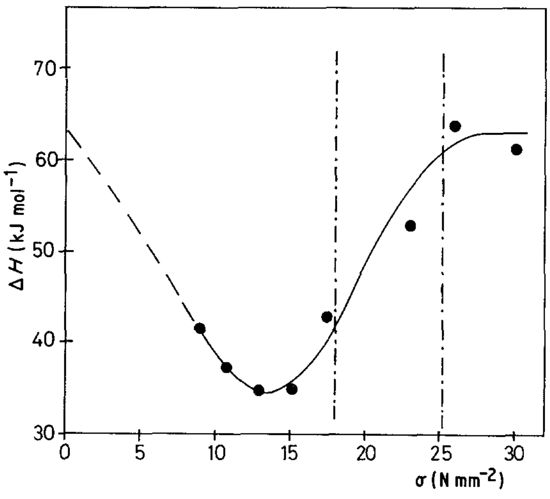

Figure 8 Activation enthalpy as a function of stress.

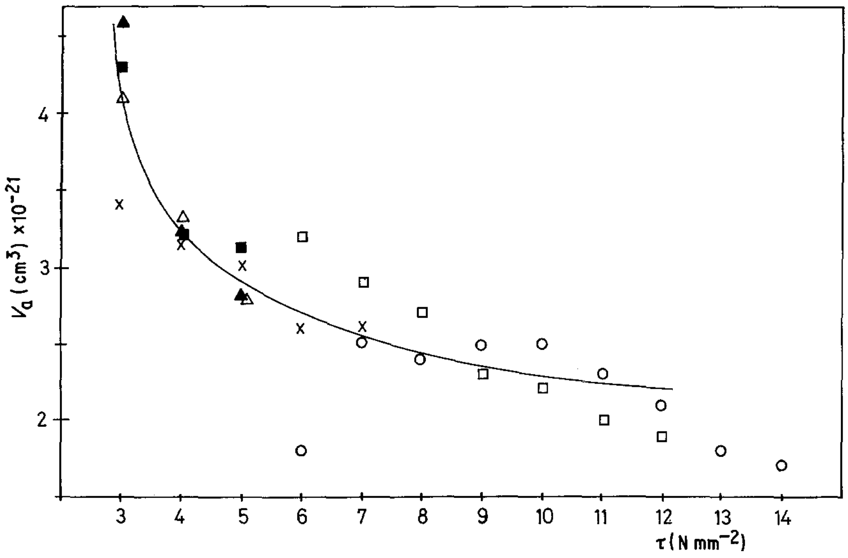

Figure 9 Apparent activation volume as a function of the resolved shear stress. Values of T(K): (○) 283, (□) 293, (x) 303, (△) 313, (■) 323, (▲) 325.5.

activation enthalpy and the strain-rate sensitivity as a function of the equivalent stress. Fig. 8 is divided into three parts by broken lines. In the high-stress region (A), $m < 0.3$ so the deformation is not superplastic. In the low-stress region (B) the deformation is superplastic because $m > 0.3$. Between these two characteristic regions there is a transitional zone.

It is useful to compare the ΔH data with the self-diffusion energy of pure lead (100 kJ mol^{-1}) and pure tin (82 kJ mol^{-1}). The activation energy of the normal deformation process obtained in the present work is larger by about 20 to 25% than the self-diffusion energy of tin, while the activation energy obtained in the superplastic region is much less, 40 to 45 kJ mol^{-1}, in good agreement with the data determined by tensile measurements [14]. The activation energy in the superplastic region agrees well with the grain-boundary diffusion energy. It shows that the controlling mechanism of superplastic flow in Pb–Sn alloys is grain-boundary sliding. ΔH changes strongly as a function of stress in the superplastic region. With decreasing σ the contribution of thermal fluctuations to the work necessary to overcome the velocity-controlling obstacles is increasing, while the contribution of the mechanical work is decreasing. The ΔH value extrapolated to zero stress gives therefore the energy necessary to overcome the obstacles.

The activation volumes determined by the impression creep technique, similarly to the activation enthalpy, are in good agreement with earlier measurements [14]. The apparent activation volume decreases monotonically with increasing stress. In the superplastic region the activation volume derived by Equation 6 is positive and also decreases with increasing stress. In the transition zone, negative activation volumes were obtained. The negative activation volume values show that the real activation volume cannot be determined by Equation 4.

5. Conclusion

The investigation of the superplasticity of Pb–Sn eutectic alloy by the impression creep technique leads to results equivalent to those from conventional unidirectional creep measurements. In impression creep tests small amounts of testing material can be used, and stress and temperature effects can be investigated on the same sample.

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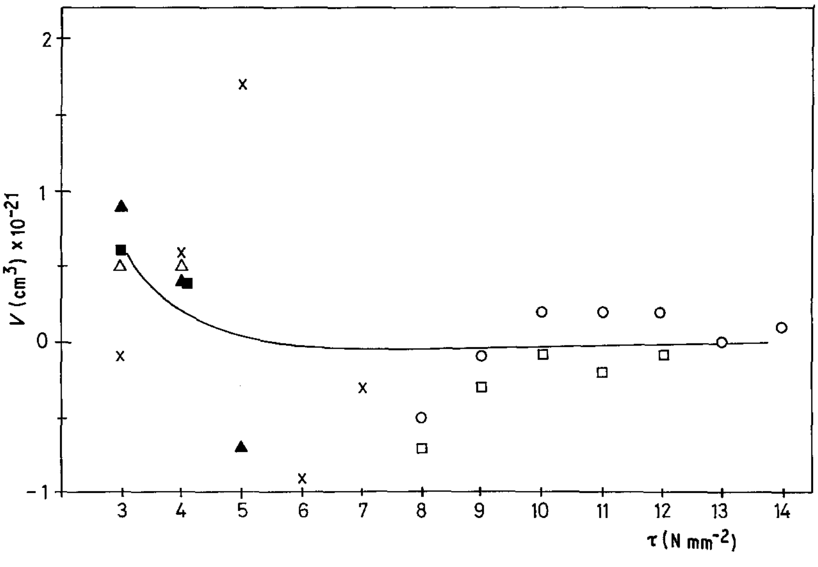

Figure 10 Real activation volume as a function of the resolved shear stress. Values of T(K): (○) 283, (□) 293, (x) 303, (△) 313, (■) 323, (▲) 325.5.

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