JOURNAL OF MATERIALS SCIENCE 25 (1990) 753–755

Investigation of superplasticity by the impression creep technique

N. KUMAR, K. S. RAMAN, D. H. SASTRY, E. A. LITTLE*
Department of Metallurgy, Indian Institute of Science, Bangalore 560 012, India

Impression creep tests have been carried out on a Zn–22% Al eutectoid alloy in two heat-treatment conditions, as a function of applied stress and at temperatures of ~25 to 270° C. At small grain sizes (~0.9 μm) and in the temperature range ~180 to 270° C, the observed deformation response conforms to Regions I, II and III typical of superplastic behaviour. Furthermore, strain-rate index values determined for Region II fall in the range 0.44 to 0.51, consistent with superplasticity. It is concluded that the impression creep technique offers considerable potential for characterization of superplastic alloy systems on small specimen volumes.

1. Introduction

Following the original suggestion of Larsen-Badse [1], Li and co-workers [2–5] and Murthy and Sastry [6–8] have employed an innovative experimental procedure, known as the impression creep test, to evaluate the creep behaviour of a range of metallic and non-metallic materials. In this test, a small diameter cylindrical indentor (or punch) with a flat base is allowed to form a shallow impression on the specimen surface under the action of an applied stress. The depth of penetration as a function of time then gives the impression creep curve. The form of such curves closely resembles that of conventional creep tests; in addition, the data exhibit the same stress, temperature dependence and stress exponent of steady state creep rate as in tensile creep. The rate-controlling deformation mechanisms in both impression and conventional creep have also been confirmed as equivalent [7, 8]. The validity of parameters obtained in impression creep tests is thus well established.

The technique offers significant advantages over conventional testing procedures, namely small specimen volume, constant stress at constant load, temperature and stress dependence of creep rate obtainable from a single sample, etc. New areas of application therefore merit investigation. Recently, the technique has been extended to the measurement of spatial variations in mechanical properties of weldments [9, 10], whilst consideration is being given to tests on highly irradiated materials, where only small quantities are available [11].

The present studies were initiated in order to examine whether the impression creep technique could usefully be applied to characterize the phenomenon of superplasticity.

2. Experimental procedure

A Zn–22 wt % Al eutectoid alloy was used in the present investigation; superplasticity in this alloy system is well documented for fine-grained conditions [12–14]. The alloy was prepared by melting high-purity constituent metals in a graphite crucible with a eutectic zinc chloride/ammonium chloride flux mixture. The ingot was processed to bar by rolling at room temperature to 15% reduction. Specimens of size 20 × 10 × 8 mm^3, with accurately parallel faces suitable for the creep tests, were cut from the bar. The specimens were examined in two heat-treatment conditions: (i) after homogenization at 370° C for 6 h, which resulted in a mean grain size of 145 μm; and (ii) after subsequent grain refinement, achieved by isothermal transformation at room temperature to complete the eutectoid reaction, using the time–temperature–transformation curves of Garwood and Hopkins [15]. This produced a mean grain size of 0.9 μm, as determined by scanning electron microscope examination. Samples were polished metallographically prior to testing.

The impression creep apparatus and test procedure have been described in detail elsewhere [7, 8]. A tungsten carbide punch, 2 mm diameter, was used to indent the sample; its displacement, and hence the impression depth, was continuously monitored to an accuracy of ± 2 μm using an LVDT transducer, and plotted as a function of time on a strip chart recorder, thereby giving the impression creep curve. Tests were carried out at various stresses and at temperatures in the range 25 to 272° C (i.e. below the eutectoid temperature of 275° C) using an oil bath controlled to ± 1° C.

3. Results and discussion

During elevated temperature tensile deformation of superplastic materials, the steady state flow stress (σ) exhibits strain rate sensitivity (m) defined by a power-law relationship

$$\sigma = B\dot{\varepsilon}^m \qquad (1)$$

where $\dot{\varepsilon}$ is the imposed strain rate. Values of σ and $\dot{\varepsilon}$

*Permanent address: Materials Development Division, Harwell Laboratory, Didcot, Oxfordshire, OX11 0RA, UK.

0022–2461/90 $03.00 + .12

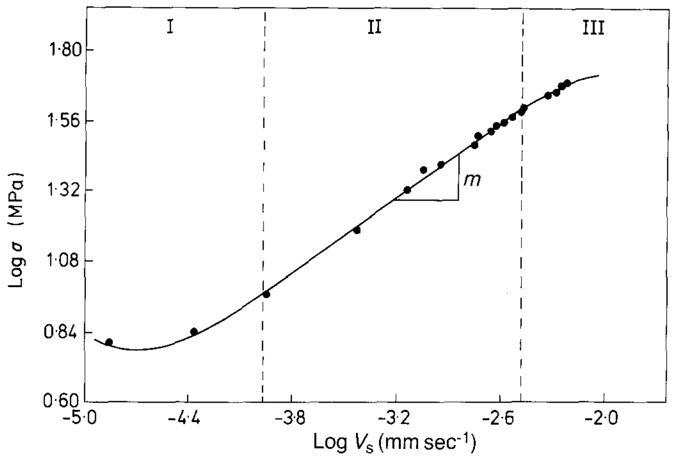

Figure 1 Stress dependence of the steady state impression creep rate at 180° C for fine-grained Zn–22% Al alloy, showing Regions I, II and III.

are generally plotted on a double-logarithmic scale which is typically sigmoidal in shape and divides into Regions I, II and III. The intermediate strain rate region with maximum strain rate sensitivity ($m \simeq 0.5$) is the superplastic regime (Region II) which exhibits the exceptionally high total elongations to failure, for which superplastic materials are noted. The regimes where m decreases ($\simeq 0.2$) at lower and high straiin rates are termed Regions I and III, respectively.

In impression creep testing, the punch achieves a steady state velocity (V_s) following an initial transient period where the punch velocity decreases with time, i.e. a response directly analogous to conventional tensile creep. Thus, the strain-rate sensitivity can be evaluated in these tests as

$$m = \frac{\Delta \log \sigma}{\Delta \log V_s} = \frac{\Delta \log \sigma}{\Delta \log \dot{\varepsilon}} \quad (2)$$

where σ is now the impressing or punching stress.

Within the above framework, the variation of V_s with test temperature and punching stress can now be examined for the Zn–22% Al alloy in the fine- and coarse-grained conditions for evidence of superplasticity under this mode of deformation.

Fig. 1 illustrates a double-logarithmic plot of the stress dependence of steady state impression creep rate (i.e. steady state punch velocity) for the fine-grained alloy at a test temperature of 180° C. The plot is sigmoidal and appears to exhibit three regions of strain-rate response, namely I, II and III as in conventional tensile superplastic behaviour. Furthermore, in Region II, a value for m of $\simeq 0.44$ is obtained, indicative of a superplastic deformation mode. Fig. 2 gives the stress–creep rate data for a range of test temperatures in Region II for the fine-grained material, whilst the derived strain-rate sensitivity index values are listed in Table I.

The data clearly indicate that the fine-grained alloy with grain diameter 0.9 μm exhibits superplastic behaviour in the temperature range 180 to 272° C, with m values in the range 0.44 to 0.51. These strain-rate sensitivity values are in good agreement with those cited in the literature for Zn–22% Al and other alloys and obtained by conventional test techniques [16–20].

The above behaviour can be compared with that for the Zn–22% Al alloy in the coarse-grained condition, with grain diameter 145 μm. Fig. 3 gives the stress dependence of the steady state impression creep rate as a function of test temperature over the range 25 to 255° C, and the derived values of strain-rate sensitivity are listed in Table II. It is clear that the coarse-grained condition is characterized by low values of m, typically in the range 0.09 to 0.17, indicating, as expected, conventional deformation behaviour.

In summary, the present results demonstrate that the impression creep test provides a convenient method for measuring strain-rate sensitivity and thereby assessing the ability of a material to undergo superplastic deformation. The temperature and strain-rate ranges for superplastic behaviour can be rapidly established, and the technique also offers the potential to determine thermodynamic parameters (namely activation enthalpy and area) for the deformation process [8]. In contrast, conventional procedures which utilize tensile tests to identify high levels of per cent elongation

TABLE I Strain rate sensitivity index as a function of test temperature for Zn–22% Al alloy in a fine-grained condition (grain diameter = 0.9 μm)

Test temperature (° C)	Strain-rate sensitivity index (m)
78	0.27
125	0.35
180	0.44
245	0.44
272	0.51

TABLE II Strain rate sensitivity index as a function of test temperature for Zn–22% Al alloy in a coarse-grained condition (grain diameter = 145 μm)

Test temperature (° C)	Strain-rate sensitivity index (m)
25	0.09
80	0.12
120	0.13
170	0.17
220	0.15
255	0.15

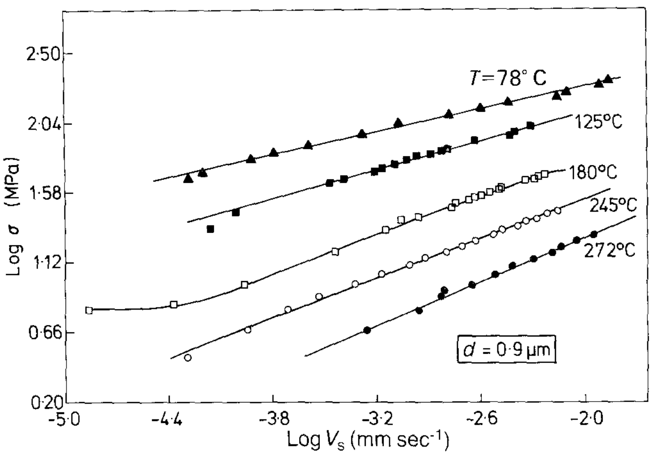

Figure 2 Effect of applied stress and test temperature on the steady state impression creep rate of fine-grained Zn–22% Al alloy.

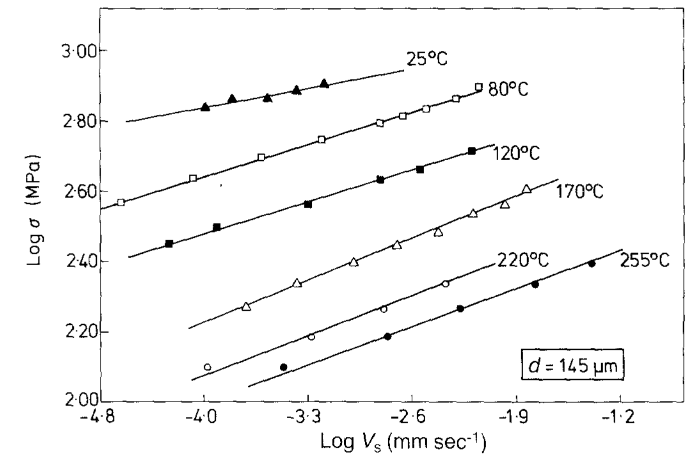

Figure 3 Effect of applied stress and test temperature on the steady state impression creep rate of coarse-grained Zn–22% Al.

associated with superplasticity suffer from the problem of changes in strain rate with strain. Impression tests, as opposed to unidirectional tensile or creep tests, also more accurately reflect the deformation mode operative during superplastic forming.

Additional studies are in progress on other superplastic alloy systems in order to identify the operative deformation mechanisms.

Acknowledgements

One of the authors (E.A.L.) thanks the Royal Society and the Indian National Science Academy for provision of financial support.

References

1. J. LARSEN-BADSE, Report ORNL-TM-1862, Oak Ridge, USA (1967).
2. S. N. G. CHU and J. C. M. LI, *J. Mater. Sci.* **12** (1977) 2200.
3. E. C. YU and J. C. M. LI, *Philos. Mag.* **36** (1977) 811.
4. H. Y. YU and J. C. M. LI, *J. Mater. Sci.* **12** (1977) 2214.
5. S. N. G. CHU and J. C. M. LI, *Mater. Sci. Engng* **39** (1979) 1.
6. G. S. MURTHY and D. H. SASTRY, *Trans. Ind. Inst. Met.* **34** (1981) 195.
7. G. S. MURTHY, PhD thesis, Indian Institute of Science (1981).
8. G. S. MURTHY and D. H. SASTRY, *Phys. Status Solidi* (*a*) **70** (1982) 63.
9. J. N. BUZRUK, MSc (Eng.) thesis, Indian Institute of Science (1988).
10. W. S. GIBBS, S. H. WANG, D. K. MATLOCK and D. L. OLSON, *Welding J. Res. Suppl.* **64** (1985) 53-s.
11. E. A. LITTLE, Internal Document, Harwell Laboratory (1987).
12. I. I. NOVIKOV, V. K. PORTNOY and T. E. TERENTIEVA, *Acta Metall.* **25** (1977) 1139.
13. P. SHARIAT, R. B. VASTAVA and T. G. LANGDON, *ibid.* **30** (1982) 285.
14. Z. R. LIN, A. H. CHOKSHI and T. G. LANGDON, *J. Mater. Sci.* **23** (1988) 2712.
15. R. D. GARWOOD and A. D. HOPKINS, *J. Inst. Metals* **81** (1952–53) 407.
16. H. ISHIKAWA, F. A. MOHAMED and T. G. LANGDON, *Met. Trans.* **8A** (1977) 9331.
17. D. A. HOLT, *Trans. Metall. Soc. AIME* **242** (1968) 25.
18. A. BALL and M. M. HUTCHINSON, *Metal Sci. J.* **3** (1969) 1.
19. O. A. KAIBYSHEV, B. V. RADIONOV and R. Z. VALIEV, *Acta. Metall.* **26** (1978) 1877.
20. D. M. LIVERSEY and N. RIDLEY, *Scripta Metall.* **1** (1982) 165.

Received 5 December 1988
and accepted 10 April 1989