Study of superplasticity in Pb-Sn eutectic alloy using impression creep technique

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Impression creep method has been used to study the creep behaviour of Pb-Sn eutectic alloy of two different grain sizes, viz., 3.4 and 7.5 µm in the temperature range of 30-125°C. The deformation response of the fine grained material conformed to regions II and III of a typical sigmoidal behaviour of a superplastic material. The strain rate sensitivity index values determined for region II, 0.43-0.55, were also indicative of superplastic behaviour. In coarse grained material, superplastic behaviour was seen only at higher test temperatures. These showed lower values of strain rate sensitivity index, namely 0.21-0.45. It is concluded that the impression creep technique is attractive for characterising superplasticity when only a small volume of material is available.

The progressive deformation of a material at a constant stress is called creep. To determine the engineering creep curve of a material, constant load is applied to a tensile specimen, maintained at a constant temperature and the strain undergone by the specimen is determined as a function of time. The measurement of creep resistance by conventional tests requires costly laboratory equipments and it may also take several months.

In order to overcome such difficulties, Li and coworkers and Murthy and Sastry introduced a new creep test called the impression creep test. In this test, a cylindrical indenter with a flat end is allowed under the action of a stress to make a shallow impression on the surface of the specimen. The depth of penetration of the indenter at a given stress is measured as a function of time to get an impression creep curve.

The form of such curves closely resembles that of conventional creep curves. 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 being equivalent. The validity of the parameters obtained in impression creep tests is thus well established.

This test offers several advantages over the conventional creep test, viz., shorter duration of tests, smaller quantity of testing materials, possibility of obtaining temperature and stress dependences of creep rate on a single sample, constant stress at constant load and absence of tertiary creep.

This technique has been extended for the measurement of spatial variations in the mechanical properties of weldments, localised stress relaxation and also to characterise superplasticity in Zn-22% Al eutectoid alloy.

The purpose of the present work is to examine the superplastic behaviour of Pb-Sn eutectic alloy of two different grain sizes using the impression creep method.

Experimental Procedure

A Pb-Sn eutectic alloy was used for the investigation. The alloy was prepared by high purity constituent metals in a graphite crucible and cast into ingots of size 100 x 30 x 15 mm³. The ingots were rolled to 50% reduction at room temperature. Specimens of size 13 x 10 x 6 mm³ were cut for impression creep testing. The alloy was tested under two conditions: (i) Samples were rolled to 50% reduction at room temperature which produced a grain size of 3.4 ± 0.3 µm and (ii) samples rolled to 50% reduction at room temperature and then annealed for an hour at 150°C, resulting in an average grain size of 7.5 ± 1.5 µm. (Grain size was determined by Heyn's linear intercept method).

The impression creep apparatus designed and fabricated is basically similar to the one described by Yu and Li. A schematic line diagram of the unit is shown in Fig. 1. Tests were carried out at various stresses and temperatures in the range of...
Fig. 1—Schematic diagram of impression creep apparatus

1. Results and Discussion

Figs 2 and 3 show the variation in creep behaviour at 100°C of fine and coarse grained materials, respectively, for various impression stresses. Similarly, Figs 4 and 5 show the creep curves for the two materials at a constant stress over a range of temperatures. After an initial transient period, steady state creep (steady impressing velocity) is observed in all cases. As in conventional creep tests, here also it is observed that the steady state creep rate increases with increasing stress at a given temperature or with increasing temperature at a given stress. Further, it is found that the creep rate is higher for the fine grained material than for the coarse grained variety. This is brought out well in Table 1, which lists the steady state creep values at different temperatures and stresses.
During elevated temperature tensile deformation of superplastic materials, the steady state flow stress ($\sigma$) obeys a power law, viz.,

$$\sigma = b\epsilon^m$$  \hspace{1cm} (1)

where $\dot{\epsilon}$ is the imposed strain rate, $m$ is the strain rate sensitivity index and $b$ is a structure and temperature dependent material constant.

When the values of $\sigma$ and $\dot{\epsilon}$ are plotted on a log scale, they exhibit a typical sigmoidal shape. This can be divided into regions I, II and III. The intermediate strain rate region with maximum strain rate sensitivity index ($m > 0.4$) is the superplastic region, in which exceptionally high total elongations to failure, are obtained. In regions I and III, the value of $m$ is lower.

In impression creep testing the punch achieves a steady state velocity ($v$) following an initial transient period, where the punch velocity decreases with time, a response directly analogous to conventional tensile creep. Thus, the strain rate sensitivity is estimated as,

$$m = \frac{\Delta \ln \sigma}{\Delta \ln \dot{\epsilon}}$$  \hspace{1cm} (2)

where $\sigma$ is the impressing stress.

Fig. 6 gives the stress-creep rate data for test temperatures ranging from 30 to 125°C for the fine grained material. Two stages could be seen in these plots. Stage I was not seen because the experiments were not carried out at sufficiently low strain rates. Only regions II and III were observed.

The strain rate sensitivity index ($m$) values obtained by the method of least squares for region II at the four test temperatures are listed in Table 2.

These data clearly indicate that the fine grained alloy with an average grain diameter of 3.4 $\mu$m exhibits superplasticity above the room temperature, which is in good agreement with the results given in the literature.

Fig. 7 gives a plot of the stress dependence of steady state impression creep rate for the coarse grained alloy at the same four test temperatures. The plots appear as straight lines even though the tests were carried out with very small increments in the applied stress. The values of $m$ for this case also are tabulated in Table 2.

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### Table 1—Grain size dependence of steady state creep rate for Pb-Sn eutectic alloy

<table>
<thead>
<tr>
<th>Test temperature $^\circ$C</th>
<th>Stress MPa</th>
<th>Steady state creep rate mm/s ($\times 10^3$)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fine grained material</td>
<td>Coarse grained material</td>
</tr>
<tr>
<td>30</td>
<td>77.8</td>
<td>21.74</td>
</tr>
<tr>
<td></td>
<td>83.8</td>
<td>52.94</td>
</tr>
<tr>
<td>70</td>
<td>27.8</td>
<td>16.70</td>
</tr>
<tr>
<td></td>
<td>70.40</td>
<td>15.06</td>
</tr>
<tr>
<td>100</td>
<td>27.8</td>
<td>16.70</td>
</tr>
<tr>
<td></td>
<td>70.40</td>
<td>15.06</td>
</tr>
</tbody>
</table>

### Table 2—Strain rate sensitivity index as a function of test temperature

<table>
<thead>
<tr>
<th>Test temperature $^\circ$C</th>
<th>Fine grained material</th>
<th>Coarse grained material</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>0.43 ± 0.06</td>
<td>0.21 ± 0.06</td>
</tr>
<tr>
<td>70</td>
<td>0.49 ± 0.11</td>
<td>0.31 ± 0.06</td>
</tr>
<tr>
<td>100</td>
<td>0.53 ± 0.08</td>
<td>0.35 ± 0.09</td>
</tr>
<tr>
<td>125</td>
<td>0.55 ± 0.08</td>
<td>0.45 ± 0.07</td>
</tr>
</tbody>
</table>
It is clear from the Table that the coarse grained condition is characterised by low values of \( m \) as in conventional deformation. It also shows that this material may exhibit superplasticity at test temperatures greater than 125°C, since its strain rate sensitivity index approaches values typical of superplastic condition beyond this temperature.

The present results indicate that the impression creep test provides a convenient method for measuring strain rate sensitivity of flow and thereby assessing the ability of a material to undergo superplastic deformation.

**Conclusions**

1. A logarithmic plot of stress and strain rate for fine grained Pb-Sn eutectic alloy showed the presence of regions II and III of superplastic flow. The slope in region II (the strain rate sensitivity index) has values greater than 0.4 at all test temperatures, which indicates that material may be behaving in a superplastic manner in this condition.

2. A logarithmic plot of stress versus strain rate for the coarse grained Pb-Sn eutectic alloy did not show sigmoidal behaviour at test temperatures lower than 125°C. The strain rate sensitivity index was less than 0.4. So the material in this condition is likely to be superplastic only at temperatures greater than 125°C (where \( m > 0.4 \)).

3. Pb-Sn eutectic alloy showed a decreasing creep rate with increasing grain size, which was in conformity with earlier results.

**References**