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A comparison of constant strain rate and creep testing procedures in superplasticity

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Tests were conducted on the Zn–22 wt% Al eutectoid alloy to compare experimental conditions of true constant strain rate and true constant stress when there is relatively minor grain growth. The stress–strain curves show the presence of significant strain hardening which precedes steady-state flow. In the steady-state condition, similar stresses are obtained when the strain rate is incrementally cycled on a single specimen. There is an extensive primary stage of creep in both Regions I and II, although in Region II the time involved is often very short. It is re-affirmed that, in the absence of significant grain growth, steady-state flow data may be obtained using either constant strain rate or creep testing procedures.

1. Introduction

The mechanical properties of superplastic materials are generally determined using one of two different, but complementary, procedures.

First, specimens may be deformed using a testing machine which imposes a true constant strain rate or a constant rate of cross-head displacement. In this case, the steady-state flow stress, σ, is recorded for an imposed strain rate, $\dot{\epsilon}$, and the data are plotted logarithmically as σ against $\dot{\epsilon}$. In some experiments, the strain rate is cycled periodically during the test, in the manner first suggested by Backofen *et al.* [1], so that a number of steady-state flow stresses are obtained from a (singular) specimen.

Second, specimens may be deformed under creep conditions of true constant stress or constant load. In this case, the steady-state strain rate, $\dot{\epsilon}$, is recorded for the imposed stress, σ, and the data are plotted logarithmically as $\dot{\epsilon}$ against σ. Again, it is possible to perform cycling tests in which the steady-state strain rates are measured at several different stresses on a single specimen.

Most investigations of superplasticity employ only one of these two procedures; but in those situations where both procedures have been used, the two sets of data obtained under steady-state conditions appear to be in excellent agreement in the strain rate range of about 10^{-5} to $10^{-3}\,\text{sec}^{-1}$ where the two techniques tend to overlap [2–7].

It was noted in early experiments on the Al–33 wt% Cu eutectic alloy that the presence of significant grain growth during the test (by up to, and in excess of, one order of magnitude) may markedly influence the apparent shape of the σ–$\dot{\epsilon}$ curve [8], especially at low strain rates where there is a tendency to overestimate the flow stress. The problems associated with an unstable microstructure were subsequently examined in detail by Suéry and Baudelet [9], and Arieli and Mukherjee [10] pointed out that difficulties may arise in the interpretation of superplastic data for the Zn–22 wt% Al alloy due to concurrent grain growth.

It has been demonstrated that the latter criticism is not generally important in Zn–22 wt% Al due to the very limited grain growth [11]. However, as a result of the experimental observations on Al–33 wt% Cu, it is not clear whether, in superplastic metals tested under conditions where there is rather minor grain growth, the two standard testing procedures provide a consistent rep-

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resentation of the mechanical characteristics of the material in the region preceding steady-state flow.

Accordingly, the present work was undertaken with three objectives. First, to check the nature of the stress–strain curves and the effect of strain-rate cycling in tests conducted under conditions where there is very limited grain growth. Second, to compare the shapes of the stress–strain curves with the corresponding creep curves obtained under comparable testing conditions. Third, to directly compare the steady–state strain rates obtained by these two different procedures.

2. Experimental material and procedure

The tests were conducted on a Zn–22 wt% Al eutectoid alloy. This material was selected because it was established earlier that grain growth is relatively minor in this alloy at testing temperatures of up to at least 503 K [12].*

The alloy was prepared from 99.9% purity Al using the procedure described earlier [2], and the final material contained 78.0 wt% Zn, 21.9 wt% Al, and the following minor impurities in ppm: Ag < 10, B < 10, Cr < 10, Cu < 5, Fe 20, Ga < 10, Mg < 10, Mn < 10, Ni 10, Si < 10, Ti < 10 and V < 10. Double-shear specimens were machined from this material, solution-heated in argon at 633 K for 15 h, quenched to 273 K in an ice-water bath, and then annealed for 1 h at 533 K to give an average equiaxed spatial grain size, $d (= 1.74 \times \bar{L}$, where $\bar{L}$ is the mean linear intercept), of 2.3 μm.

Two different procedures were used to deform specimens on an Instron machine. Initially, each specimen was tested at a single constant displacement rate, equivalent to a true constant strain rate for a double-shear configuration, and the shear stress, τ, was recorded as a function of the shear strain, γ, up to a total strain of 125%. Having obtained a number of standard stress–strain curves, a single specimen was then tested through a range of increasing strain rates to give a stress–strain curve for cyclic conditions.

Some specimens were tested under creep conditions at constant load, which is equivalent to a true constant stress for a double-shear configuration. For these tests, the strain was continuously monitored using a linear variable differential transformer, and recorded using an amplifier and strip-chart recorder.

Each test was performed by immersing the specimen in a bath of silicone oil which was heated electrically and stirred with bubbling argon. The bath temperature was measured with chromel–alumel thermocouples and maintained constant throughout each test at 484 ± 1 K.

3. Experimental results

The results obtained from the tests conducted at true constant strain rates are shown in Fig. 1, plotted as τ against γ up to shear strains of 125%. The solid lines show the individual stress–strain curves for the various specimens tested at different constant strain rates: the shear strain rates, $\dot{\gamma}$, are indicated for each curve. The broken line shows the composite stress–strain curve obtained by cycling a single specimen through a number of constant strain rates. For this test, the specimen was initially loaded at $\dot{\gamma} = 2.67 \times 10^{-3}\,\text{sec}^{-1}$, the shear strain rate was reduced by two orders of magnitude to $2.67 \times 10^{-5}\,\text{sec}^{-1}$ at $\gamma = 15\%$ and, thereafter, the strain rate was incrementally increased through various strain rates to a maximum of $1.33 \times 10^{-2}\,\text{sec}^{-1}$ at $\gamma = 100\%$. With the exception only of the increment at $\dot{\gamma} = 2.67 \times 10^{-4}\,\text{sec}^{-1}$, the shear strain rates selected for this cyclic test were identical to the shear strain rates used in the single tests on individual specimens.

Examples of two creep curves are shown in Figs 2 and 3 for true constant shear stresses of 4.75 and 1.07 MPa, respectively. These curves plot shear strain, γ, against time, t, and they indicate the regions of steady-state flow and the estimated values for the steady-state shear strain rates, $\dot{\gamma}_s$.

4. Discussion

4.1. Tests at constant strain rate

An examination of the stress–strain curves shown in Fig. 1 reveals three important trends.

First, the superplastic Zn–22 wt% Al alloy does not exhibit an immediate steady-state flow when tested at a true constant strain rate, but there is a region of significant strain hardening which precedes the steady-state behaviour. This strain-hardening region extends to shear strains of about 50% at the faster strain rates ($\dot{\gamma} \simeq 10^{-3}$ to 10^{-2}

*Experiments on Zn–22 wt% Al show an increase in the spatial grain size from 2.5 μm initially to 5.0 μm after testing to failure for 85 h at a strain rate of $1.33 \times 10^{-5}\,\text{sec}^{-1}$ and a temperature of 503 K [12]. There is less grain growth at faster strain rates and/or lower temperatures.

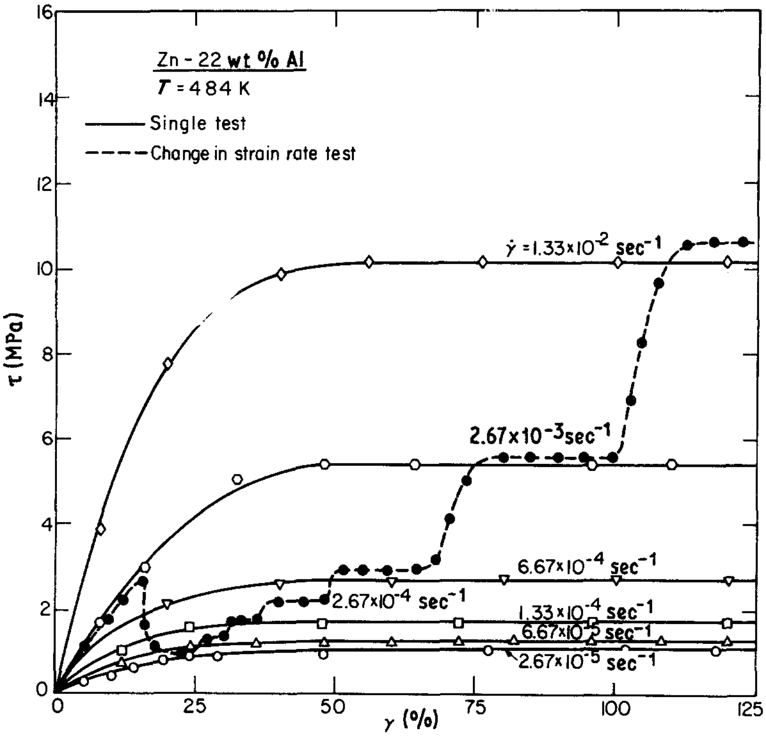

Figure 1 Shear stress plotted against shear strain for specimens of Zn–22 wt% Al at a temperature of 484 K, obtained either by testing each specimen at a single constant strain rate (solid lines) or by cycling a single specimen through a number of constant strain rates (broken line).

$\sec^{-1}$) and to shear strains of about 25 to 30% at the slower strain rates ($\dot{\gamma} \simeq 10^{-5}\sec^{-1}$).

Second, in the steady-state condition, typically at $\gamma > 50\%$, the shear stress remains constant up to at least $\gamma \simeq 125\%$, thereby giving a well-defined plateau in the stress–strain curves. In agreement with earlier observations [12], the absence of significant grain growth in the present experiments may be inferred indirectly from the insensitivity of the steady-state stresses to time and strain.

Third, within the steady-state region, the stresses obtained from the strain-rate cycling test are in very good agreement with those attained in the tests conducted at single strain rates. The latter result confirms that strain-rate cycling is a valid and acceptable procedure for those superplastic materials, such as Zn–22 wt% Al tested at a true constant strain-rate, where there is little or no concurrent grain growth and an obvious and well-defined steady-state condition.

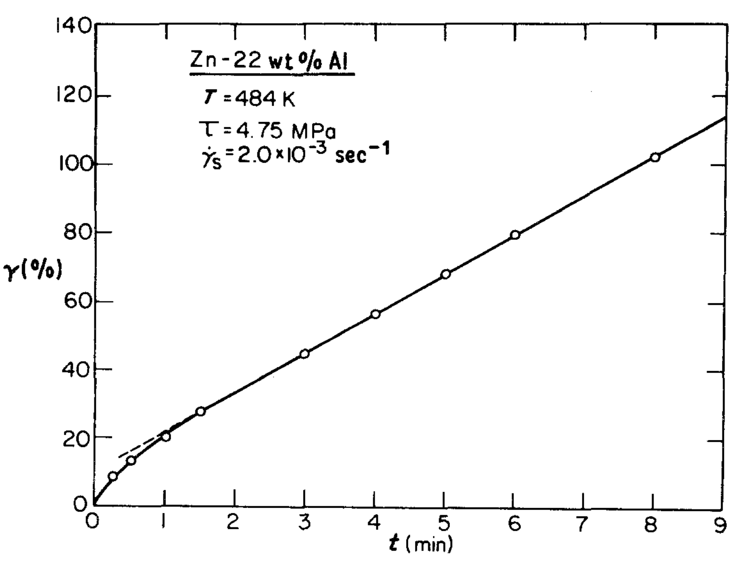

Figure 2 Creep curve of shear strain plotted against time for Zn–22 wt% Al tested at a temperature of 484 K at a shear stress of 4.75 MPa.

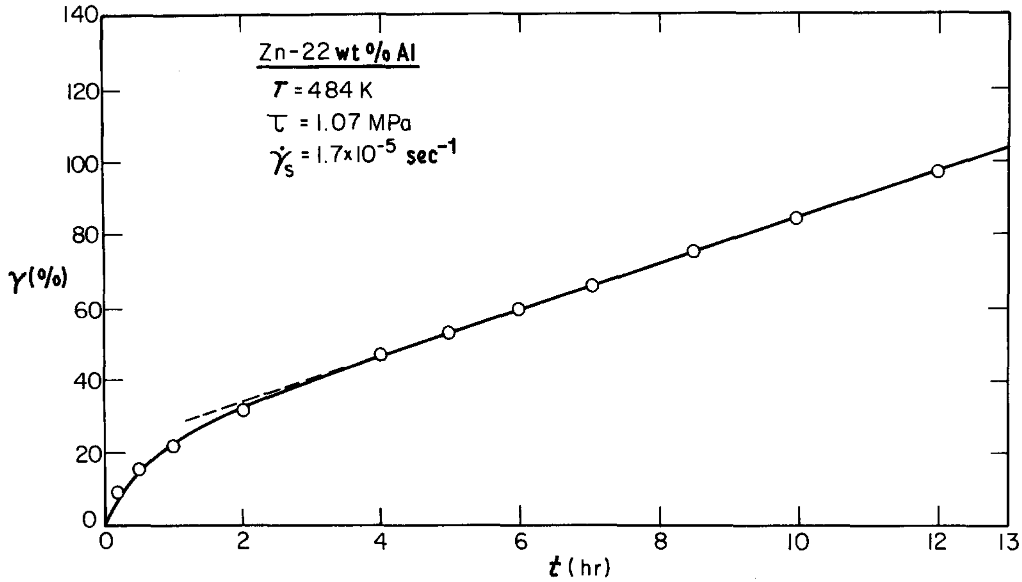

Figure 3 Creep curve of shear strain plotted against time for Zn–22 wt % Al tested at a temperature of 484 K at a shear stress of 1.07 MPa.

4.2. Tests at constant stress

A comparison of Figs 1 to 3 shows three significant similarities between the experimental procedures of constant strain rate and constant stress.

First, the creep curves in Figs 2 and 3 exhibit an extensive and normal primary stage which is analogous to the strain-hardening region visible in the stress–strain curves in Fig. 1.

Second, steady-state creep is established at shear strains of the order of about 20 to 40%, which is comparable to the onset of steady-state flow in Fig. 1. Thereafter, the strain rate remains constant up to $\gamma > 100\%$.

Third, it is clear by interpolation that the values of $\dot{\gamma}_s$ obtained in the creep tests in Figs 2 and 3 are consistent with the constant strain-rate data in Fig. 1. Thus, consistent information on steady-state flow may be obtained using either constant strain rate or creep testing procedures.

Superplastic materials generally exhibit a sigmoidal relationship between stress and strain rate so that the behaviour is divisible into three distinct regions: a low-stress region, Region I, where the elongations to failure are rather low; an intermediate-stress region, Region II, where the elongations are high and the behaviour is superplastic; and a high-stress region, Region III, where the elongations are again reduced [13]. A comparison with earlier work on the Zn–22 wt % Al alloy in a double-shear configuration [3] shows that the creep curve in Fig. 2 corresponds to the superplastic Region II, whereas the creep curve in Fig. 3 represents behaviour which is near to the upper limit of the low stress Region I. Thus, it is concluded that there is a primary stage of creep in both Regions I and II of superplastic deformation.

Earlier results showed the presence of an extensive primary stage of creep in Zn–22 wt % Al in Region I [11], and this is consistent with the creep curve in Fig. 3. However, there has been some disagreement on the shape of the creep curves in the superplastic Region II. On the one hand, Vaidya *et al.* [14] and Arieli *et al.* [7] testing Zn–22 wt % Al in double-shear, and Misro and Mukherjee [15] testing Zn–22wt % Al in tension, reported either a very brief primary stage or an absence of primary creep in Region II. On the other hand, Grivas [16] reported a short but measurable primary stage in Pb–62 wt % Sn tested in Region II and Shei and Langdon [17] observed a very short primary stage in Region II in a superplastic Cu alloy*. The present results indicate the occurrence of a short but measurable primary stage in Region II, which extends over a brief time interval (less than 2 min in Fig. 2) but, nevertheless, corresponds to a significant shear strain of $\gamma \simeq 20$ to 30%. By contrast, the primary stage in

*The primary stages in Region II extended for < 100 sec in Pb–62 wt % Sn [16] and < 1 min in the Cu alloy [17].

Region I extends over a much longer period of time ($t \simeq 2$ to 4h in Fig. 3), and the available evidence indicates that even longer time intervals occur at lower steady-state strain rates [11].

5. Summary and conclusions

Tests were performed on the superplastic Zn–22 wt% Al alloy under conditions where there is relatively minor grain growth. The results show that:

(a) Under conditions of constant shear strain rate, there is a region of significant strain hardening which precedes steady-state flow, the shear stress remains constant in the steady-state condition up to shear strains of at least 125%, and similar stresses are obtained when the strain rate is incrementally cycled on a single specimen;

(b) Under conditions of constant shear stress, there is an extensive primary stage of creep which precedes steady-state flow in both Regions I and II, the steady-state strain rate remains constant up to shear strains of more than 100%, and the stress–strain rate data from creep experiments are consistent with the results obtained from constant strain rate tests; the time interval for the primary stage in Region II is often very short.

It is re-affirmed that, when there is relatively minor grain growth, steady-state flow data may be obtained using either constant strain rate or creep testing procedures.

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