JOURNAL OF MATERIALS SCIENCE 18 (1983) 781–784

Concurrent grain growth corrections during creep of fine-grained materials

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This investigation demonstrates the need to correct for concurrent grain growth in low-strain-rate studies of superplastic materials. It examines the validity of two common equations for expressing the grain-growth kinetics and applies these equations to low-strain-rate work in the Zn–22 wt % Al alloy. After the correction was made, the stress exponent n changed from $n \simeq 4.1$ to $n \simeq 3.4$.

1. Introduction

The high-temperature mechanical behaviour of many fine-grained materials often gives a sigmoidal log stress against log strain rate curve (Fig. 1). At high strain rates, climb- or glide-controlled creep is the rate controlling mechanism in coarse-grained and fine-grained materials [1]. At intermediate strain rates, materials having very fine grains ($< 10\,\mu$m) often exhibit a much higher dependence of stress on strain rate, which results in superplastic behaviour [2, 3]. At still lower strain rates, a region of lower dependence of stress on strain rate is often observed.

Some time ago, Rai and Grant [4] realized that the behaviour at low strain rates may result from an artificial increase in stress caused by concurrent grain growth. The effect becomes more pronounced as the strain rate decreases and testing time to a fixed strain increases. Rai and Grant first used this reasoning to show that the true behaviour at low strain rates in their Al–Cu eutectic alloy was identical to that observed in the intermediate or superplastic range.

More recently, Arieli and Mukherjee [5] used the above argument to claim that the sigmoidal stress curve reported by Mohamed *et al.* [6–8] in superplastic Zn–Al eutectoid did not reflect the true behaviour of the alloy at constant grain size but instead was an artifact produced by concurrent grain growth. This claim sparked considerable controversy with regard to both the behaviour of Zn–22 wt % Al and the role of concurrent grain growth in general [9–12].

The present investigation has two objectives: first, to analyse the procedures for correcting concurrent grain growth used by Arieli *et al.* [5, 12] and Grivas *et al.* [11] and, second, to illustrate clearly that although concurrent grain growth was significant in the work of Mohamed *et al.* [6–8], a true sigmoidal shape remained even after correction was made.

2. Analysis

The high-temperature behaviour of most metals, regardless of the specific deformation mechanism, can usually be characterized by the well-known relation:

$$\frac{\dot{\epsilon}kT}{D_0Gb} = A_i\left(\frac{b}{d}\right)^{p_i}\left(\frac{\sigma}{G}\right)^{n_i}\exp\left(\frac{-Q_i}{RT}\right). \quad (1)$$

The values of the exponent of Burger's vector/grain size (p_i), the exponent of stress/shear modulus (n_i), the Arrhenius activiation energy (Q_i), and the dimensionless constant (A_i), depend on the rate-controlling mechanism. Specifically, in the superplastic "Region II" for Zn–22 wt % Al, it is generally agreed that $n = 2$, $p = 2$, and $Q = Q_{gb}$ (the activation energy for grain boundary diffusion). Mohamed *et al.* [7, 8] reported a Region I at lower strain rates having $n \simeq 4.1$. It is easily seen from Equation 1 that an increase in grain size d at constant strain rate results in an increased stress, giving rise to an artificially high n value in the low-strain-rate Region I.

Arieli and Mukherjee [5, 12] claimed that the $n = 4.1$ Region I of Mohamed *et al.* was actually part of Region II having $n \simeq 2.25$. They used an expression of the form

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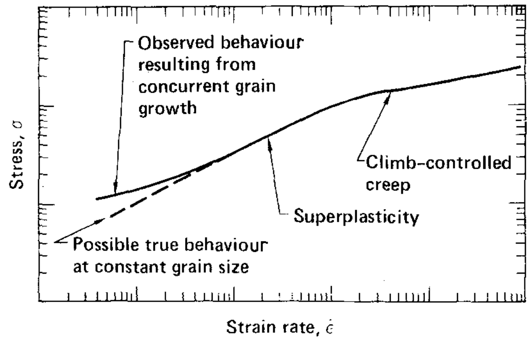

Figure 1 Plot of log stress against log strain rate.

$$d = d_0 + kt^c \quad (2)$$

to express grain size $d(\mu m)$ as a function of initial grain size d_0 and testing time t (sec). From the plot of d against t supplied by Grivas *et al.* (Fig. 2, [11]), the present author determined that $c \simeq 0.21$ and $k \simeq 0.164$ when Equation 2 is used. Taking any two points on the stress against strain-rate plot, a corrected n value may be calculated as follows:

$$n_{\text{apparent}} = \frac{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)}{\ln(\sigma_2/\sigma_1)}, \quad (3)$$

where $\dot{\epsilon}$ and σ are experimental strain rate and experimental stress. To correct for concurrent grain growth, $(\dot{\epsilon}^*)$ is then:

$$\dot{\epsilon}^* = \dot{\epsilon}\left(\frac{d_i}{d_0}\right)^p, \quad (4)$$

where d_0 is the initial grain size and d_i is the grain size at time t_i when strain rate $\dot{\epsilon}_i$ was measured. The corrected n is given by

$$n = \frac{\ln[(\dot{\epsilon}_2/\dot{\epsilon}_1)(d_2/d_1)^p]}{\ln(\sigma_2/\sigma_1)}$$

$$= n_{\text{app}}\left[\frac{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1) + \ln(d_2/d_1)^p}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)}\right]$$

$$= n_{\text{app}}\left\{1 + p\left[\frac{\ln(d_2/d_1)}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)}\right]\right\}. \quad (5)$$

Substituting $d = d_0 + kt^c$ for d_1 and d_2 yields:

$$n = n_{\text{app}}\left\{1 + \frac{p\left[\ln\dfrac{(d_0 + kt_2^c)}{(d_0 + kt_1^c)}\right]}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)}\right\}. \quad (6)$$

where p is the grain-size exponent, $\dot{\epsilon}_1$ and $\dot{\epsilon}_2$ are the two strain rates under consideration, and t_1 and t_2 are the corresponding testing times. In the following analysis, (t_1/t_2) has been substituted for $(\dot{\epsilon}_2/\dot{\epsilon}_1)$, which is valid except when considering transient creep.

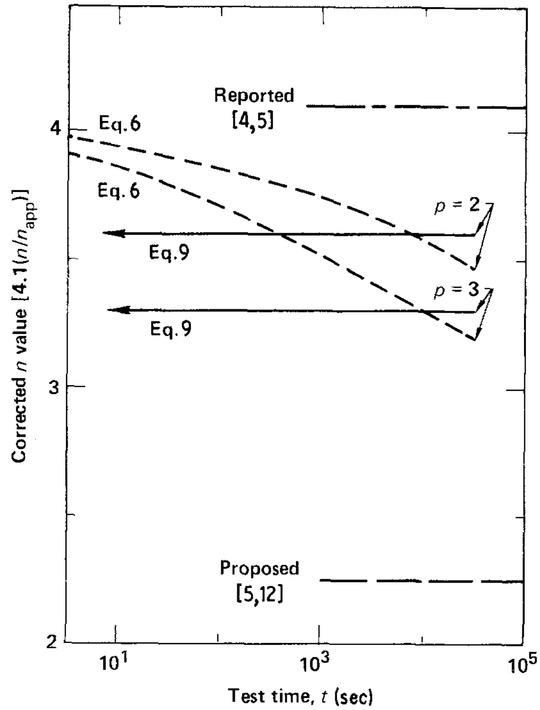

Figure 2 Plot of corrected n value against time.

Grivas *et al.* [11] argued that a better expression for concurrent grain growth is of the form

$$d = k't^{c'} \quad (7)$$

so that d_0 is ignored. From Fig. 2 of [11], the present author obtained $c' = 0.065$ in Equation 7, which differs slightly from the value $c' = 0.04$ reported in [6]. Using Equation 7 and the assumption that $(\dot{\epsilon}_2/\dot{\epsilon}_1) = (t_1/t_2)$,

$$n = n_{\text{app}}\left\{1 + \frac{p[\ln(t_2/t_1)^{c'}]}{\ln(t_1/t_2)}\right\} \quad (8)$$

or

$$n = n_{\text{app}}(1 - pc'). \quad (9)$$

In Equation 6, the correction to n_{app} is a function of the magnitude of t_1 and t_2, the ratio (t_2/t_1), the grain-size exponent (p), and the constants in Equation 2. In Equation 9, the correction depends only on p and the constant c' in Equation 7.

Using Equations 6 and 8, a plot of n against time was made using t_1 and t_2 as appropriate to $\epsilon = 0.10$ at different strain rates in Region I (see Fig. 2). The ordinate is given as $4.1(n/n_{\text{app}})$, which gives a corrected n value for the work of Mohamed *et al.* [7, 8]. Depending on the value of p (values of 2 or 3 were considered reasonable), the corrected n takes an average value of 3.3 to 3.6 for the values of $t > 10^3$ appropriate for Region I. At these values of t, essentially the same corrected n results whether grain growth is corrected using Equation 6 or Equation 9.

This value of $n \simeq 3.4$ agrees well with values found in the other high-n studies [9, 13]. It is much different from the value of $n \simeq 2.25$ proposed by Arieli and Mukherjee for the same data. The present author believes that the value of $n = 2.25$ as proposed in [5] is based on a data analysis that greatly overestimates the amount of concurrent grain growth in the work of Mohamed and Langdon [7].

The simplicity of Equation 9 is rather surprising, and indeed disturbing if considered carefully. The implication is that the correction to n_{app} is not a function of strain rate or testing time. Thus, according to Equation 9, although $n = 4.1$ in Region I is corrected to $n \simeq 3.6$, $n = 2.25$ in Region II must also be corrected to $n \simeq 2.0$. Indeed, the same correction must be made in Region III where testing time is but a few seconds. Previous works [13, 14] showed that significant grain growth does not occur in the higher-strain-rate Regions II and III, even though strains may be larger than those obtained in Region I. Therefore, although the amount of concurrent grain growth may be influenced by strain [15], it would appear that the most significant variable is the time during which plastic deformation occurred.

The obvious flaw in Equation 9 stems from the basic assumption of Equation 7 that d_0 is negligible in the grain growth expression. As previously pointed out [12], d_0 is negligible during recrystallization/grain growth experiments but not during studies where the final d is less than two or three times d_0. The net effect is, in general, that Equations 7 and 9 are not valid when correcting for concurrent grain growth. Equation 9 works reasonably well at long testing times, especially if the data for Equation 7 are obtained at similar times. However, Equation 9 continues to predict the same correction to n at higher strain rates (Region II and Region III) where negligible grain growth occurs.

3. Conclusions

In most fine-grained materials, a correction for concurrent grain growth during high-temperature deformation is important. In making such corrections, the relation $d = kt^c$ is only valid if the region of interest is such that $d \gg d_0$. However, if this condition exists, the correction is a very simple one (Equation 9). Otherwise, a variant of Equation 2 is preferred to express the grain growth kinetics.

The Zn–22 wt% Al work of Mohamed *et al.* [6–8] represents a special case; either method of correction lowers n from 4.1 to about 3.4 in the region of interest. However, this n value is still much higher than that proposed by Arieli *et al.* [5].

A more precise method for correcting concurrent grain growth is to examine the grain size at the strain at which the mechanical data are taken; this method should be used to obtain both p and n [13]. This concept of measuring the grain size at various stages of plastic deformation was used in another recent study [15]. However, although the amount of grain growth was not large in that work, a precise value for the grain size exponent p was not determined. Although such an omission certainly does not weaken the case that Livesey and Ridley [15] presented for the high-n Region I, it does leave their exact numerical values open to some question since the effect of grain growth, though small and perhaps even negligible, was not considered.

Acknowledgement

This work was performed under the auspices of the US Department of Energy by the Lawrence Livermore National Laboratory under contract No W-7405-Eng-48.

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Received 27 April
and accepted 19 July 1982