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LETTER

An evaluation of creep behavior in ultrafine-grained aluminum alloys processed by ECAP

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There is now a good understanding of the creep behavior in crystalline materials. Under steady-state conditions, the creep rate, $\dot{\varepsilon}$, varies with the applied stress, σ, the absolute temperature, T, and the grain size, d, through a relationship of the form [1, 2]

$$\dot{\varepsilon} = \frac{ADG\mathbf{b}}{kT}\left(\frac{\mathbf{b}}{d}\right)^{p}\left(\frac{\sigma}{G}\right)^{n} \quad (1)$$

where D is the appropriate diffusion coefficient [$=D_o \exp(-Q/RT)$ where D_o is a frequency factor, Q is the activation energy and R is the gas constant], G is the shear modulus, $\mathbf{b}$ is the Burgers vector, k is Boltzmann's constant, p and n are the exponents of the inverse grain size and the stress, respectively, and A is a dimensionless constant. Over a wide range of intermediate stresses the creep rate is controlled by intragranular processes so that $p = 0$ and there is no dependence on grain size, but at low stresses intergranular creep processes may become important, such as Nabarro–Herring [3, 4] and Coble [5] diffusion creep and/or grain boundary sliding [6], and this introduces a dependence on grain size with $p \geq 1$.

Most studies of creep involve the use of materials with grain sizes larger than ~ 5 μm. However, procedures were developed recently for the production of metals with grain sizes in the submicrometer or even the nanometer range and this has raised numerous speculations concerning the flow mechanisms occurring in these materials under creep conditions. An important question concerns the possibility of new and unidentified creep mechanisms appearing in these materials where the grain sizes are exceptionally small. These mechanisms may be due, for example, to the presence of non-equilibrium grain boundaries or very high dislocation densities. Recent reports have begun to examine the flow mechanisms occurring in ultrafine-grained materials processed by electrodeposition [7] and equal-channel angular pressing (ECAP) [8–10]. Nevertheless, it is important to recognize that it may be difficult to reveal new creep mechanisms in these ultrafine-grained materials because grain growth occurs easily at the elevated temperatures used in creep experiments: an example is the occurrence of significant grain growth in creep tests conducted on high-purity aluminum after processing by ECAP [11]. Accordingly, the present analysis was undertaken to examine the flow characteristics of different aluminum alloys where scandium was added to retain an extremely small grain size in high temperature testing.

Four sets of data were examined in this analysis and for all materials the measured linear intercept grain size, $\bar{L}$, was converted to the spatial grain size, d, as shown in Eq. 1, by multiplying by a factor of 1.74 [12]. In the first material, tests were conducted using a constant displacement rate and the data are presented as the measured flow stress versus the imposed strain rate. In the remaining three materials, tests were conducted under creep conditions and the results are presented as the steady-state strain rate versus stress.

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First, data were used for an Al–3% Mg–0.2% Sc alloy processed by ECAP at room temperature for eight passes to give $\bar{L} = 0.2$ μm and then tested in tension at a constant displacement rate at a temperature of 673 K [13]. The experimental results are shown in Fig. 1 where (a) summarizes the measured elongations to failure as a function of strain rate and (b) shows the corresponding flow stress plotted against the strain rate. It is important to note that this material exhibits remarkable superplasticity after processing by ECAP with a maximum elongation of more than 2000% under optimum conditions and elongations above 1000% in the strain rate range of $\sim 10^{-2}$ to 10^{-1} s^{-1}. The broken line in Fig. 1b shows the predicted theoretical strain rate developed for conventional superplastic flow, $\dot{\varepsilon}_{sp}$, given by Eq. 1 with $D = D_{gb}$ for grain boundary diffusion, $p = 2$, $n = 2$ and $A = 10$ [6]: this line was calculated using $D_{gb} = 1.86 \times 10^{-4}$ exp $(-86{,}000/RT)$ m^2 s^{-1} [14], $\mathbf{b} = 2.86 \times 10^{-10}$ m for pure aluminum and $G = \{3.022 \times 10^4\} - 16\ T$ MPa where the temperature is expressed in degrees Kelvin [14]. Inspection shows that the

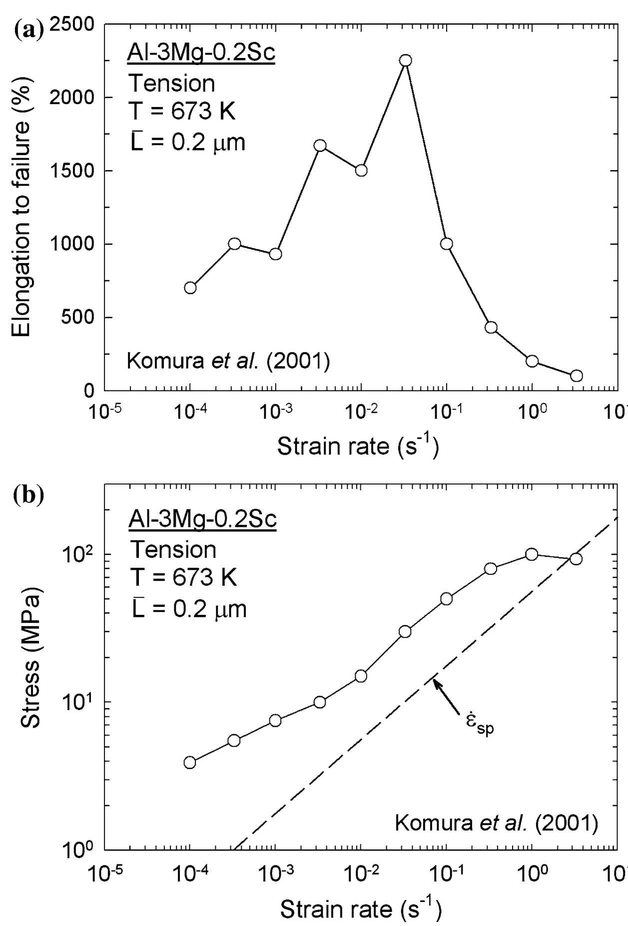

Fig. 1 **a** Elongation to failure versus strain rate and **b** measured flow stress versus strain rate for an Al–3% Mg–0.2% Sc alloy tested in tension at a constant rate of cross-head displacement [13]; the *broken line* denotes the prediction for conventional superplasticity [6]

predicted line for superplasticity is in good agreement, to within an order of magnitude, with the experimental data obtained over an intermediate range of strain rates and this is consistent with the very high superplastic elongations of >1000% obtained under these conditions [13]. This calculation confirms the general validity of the conventional relationship for superplastic flow even when testing materials with ultrafine grain sizes.

A second example is shown in Fig. 2a for a similar Al–Mg–Sc alloy having an initial grain size of $\sim$200 μm processed by ECAP at room temperature for eight passes to give a grain size of $\sim$1.5 μm [15, 16]. This material was tested under creep conditions in compression at 473 K and results are shown both for the unprocessed material and after processing by ECAP. The two broken lines in Fig. 2a denote the predictions for conventional superplasticity, $\dot{\varepsilon}_{sp}$, and Nabarro–Herring creep, $\dot{\varepsilon}_{NH}$, for a mean linear intercept grain size of 1.5 μm after ECAP where the latter mechanism was estimated using Eq. 1 with $A = 28$, $D = D_\ell$ for lattice diffusion, $p = 2$ and $n = 1$ where D_ℓ was taken as 1.86×10^{-4} exp $(-143{,}400/RT)$ m^2 s^{-1} [14]. The predictions show that Nabarro–Herring creep is too slow to account for the creep deformation in this ultrafine-grained alloy but again there is good agreement, to within an order of magnitude, with the predictions for superplastic flow except only at the lower stresses where the points deviate from linearity and there is evidence for the presence of a threshold stress. This threshold stress probably arises from the presence of coherent Al_3Sc precipitates.

The third example in Fig. 2b is for an Al–0.2% Sc alloy with an initial grain size of 10 mm which was also processed by ECAP for eight passes at room temperature to give a grain size of $\sim$0.9 μm and then tested in compressive creep at 473 K [16]. Again, experimental points are shown for the unprocessed alloy and for the alloy after ECAP and the two predictions are given for superplasticity and Nabarro–Herring creep for the material processed by ECAP with a grain size of 0.9 μm. For this material, the predictions for Nabarro–Herring creep are again too slow but there is excellent agreement between the experimental datum points and the predicted behavior in superplastic flow. The slightly higher stress exponent visible for the experimental datum points after ECAP is not understood at the present time but may reflect an inhibition in grain boundary sliding at the lowest strain rates due to the presence of intergranular Al_3Sc precipitates.

Finally, the fourth example in Fig. 2c shows an Al–0.2% Sc alloy with an initial grain size of 5–10 mm processed by ECAP for eight passes at room temperature to give a grain size of 0.55 μm and then tested in creep under tensile conditions at 473 K [17]. Again the predictions shown by the broken lines are for a grain size of 0.55 μm after ECAP and the predicted behavior for Nabarro–Herring creep is

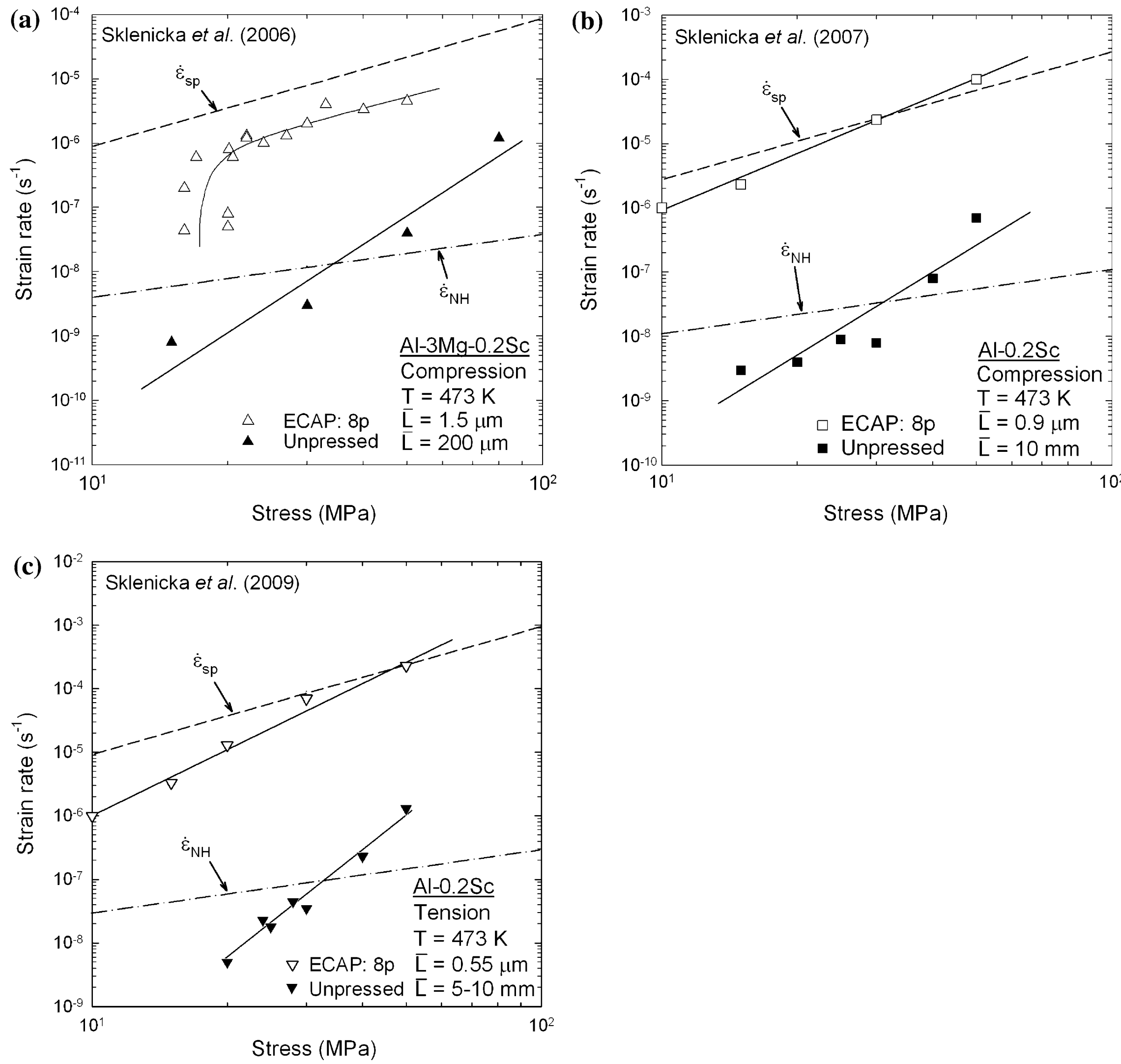

Fig. 2 Strain rate versus stress for **a** an Al–3% Mg–0.2% Sc alloy tested in compressive creep [15], **b** an Al–0.2% Sc alloy tested in compressive creep [16] and **c** an Al–0.2% Sc alloy tested in tensile creep [17]: the *broken lines* denote the predictions for superplasticity [6] and Nabarro–Herring creep [3, 4]

too slow but there is reasonable agreement with the model for superplastic flow.

Since the results from all four sets of experiments, shown in Figs. 1 and 2, exhibit a general consistency with the predicted behavior for conventional superplasticity, it should be feasible to plot all of the experimental datum points on a single diagram where the temperature and grain size compensated creep rate, $\left(\dot{\varepsilon}kT/D_{\mathrm{gb}}G\mathbf{b}\right)\left(d/\mathbf{b}\right)^{2}$, is plotted against the normalized stress, σ/G. The result is shown in Fig. 3 where all datum points are now included and the solid line denotes the theoretical prediction for superplasticity [6]. These results show an excellent agreement for the Al–0.2% Sc alloy and a very good agreement, to within one order of magnitude, for many of the datum points from the Al–3% Mg–0.2% Sc alloy. This calculation provides, therefore, a clear demonstration that conventional creep mechanisms, already developed for coarse-grained materials, may be used to explain the flow characteristics of materials with ultrafine grain sizes. Furthermore, at least for the aluminum alloys examined in this report, it is not necessary to invoke any new and different creep mechanisms. This conclusion is important both because of the many results now available demonstrating the occurrence of superplastic elongations in materials processed by ECAP [18] and because of the new possibilities that have been opened up, described in a recent review [19], for achieving exceptional superplastic elongations in materials with ultrafine grain sizes. Finally, it also confirms the potential, described elsewhere for a Pb–62% Sn alloy [20], for displaying flow behavior in the form of deformation mechanism maps.

In summary, four separate sets of creep data were analyzed for aluminum alloys containing small amounts of

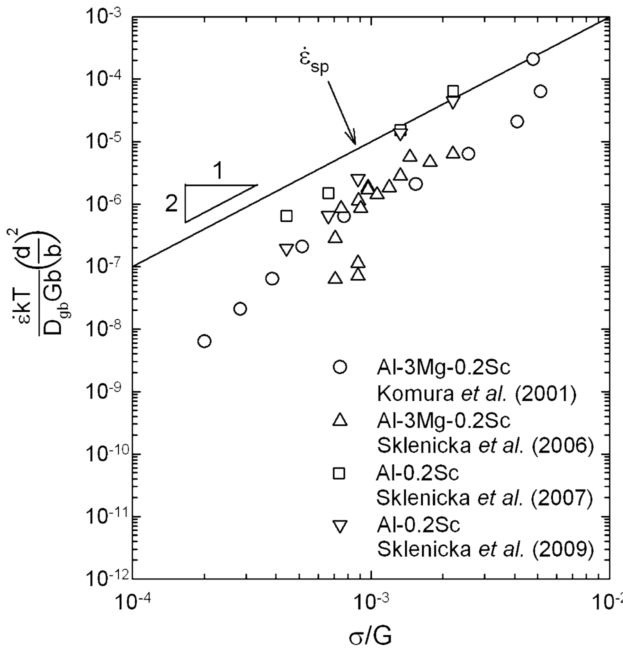

Fig. 3 Temperature and grain size compensated creep rate plotted against the normalized stress showing all experimental datum points [13, 15–17] and the predicted behavior for conventional superplasticity [6]

scandium to retain ultrafine grain sizes at elevated temperatures. It is shown that all results are mutually consistent and they are in reasonable agreement with a theoretical model for superplastic flow that was developed earlier for conventional superplastic alloys. This agreement confirms the possibility of using the theoretical model, combined with deformation mechanism maps [20], to predict the experimental conditions associated with optimum superplastic elongations in these ultrafine-grained materials.

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