Superplasticity in PM 6061 Al alloy and elimination of strengthening effect by reinforcement in superplastic PM aluminum composites

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Abstract

Plastic-flow behavior of a powder-metallurgy (PM) processed 6061 matrix alloy has been investigated in a wide range of elevated temperature between 430 and 620°C. It was found that the 6061 Al alloy exhibits superplasticity in a relatively wide range of temperature from 520 to 620°C at a high strain rate of $10^{-2}$ s$^{-1}$. Deformation behavior of the present alloy could be divided into three regions when the presence of threshold stress for plastic flow was assumed. They are $D_L$ controlled grain boundary sliding, $D_L$ controlled dislocation climb creep and powder-law breakdown, respectively. When temperature is as high as 590°C, however, the activation energy increases significantly higher than that for self-diffusion in aluminum and flow stress decreases further than normally expected. This phenomenon is likely attributed to the presence of liquid phase above 590°C. Comparison of the data in Region I below 610°C with those for a number of superplastic aluminum composites indicates that strengthening effect by reinforcement does not exist. Several speculations including diffusional relaxation in vicinity of reinforcements were made to explain this phenomenon. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

It has been demonstrated that a number of powder-metallurgy (PM) processed metal matrix composites, which have very fine grain size typically < 2 μm, can exhibit large tensile elongations at high strain rates over $10^{-2}$ s$^{-1}$ [1–11]. The 6061 Al composites are one of such composites and their superplastic characteristics have been extensively studied [9–11]. To date, however, there are few studies on the deformation of a superplastic PM 6061 Al matrix alloy, though there are some reports on superplastic behavior of ingot-processed 6xxx alloys such as 6013 alloys [12,13] where the maximum tensile elongations of 230% and 375% were attained at 520°C and $3 \times 10^{-4}$ s$^{-1}$ and at 540°C and $5 \times 10^{-4}$ s$^{-1}$, respectively, when their grain sizes were 12 and 10 μm respectively. In the present work, superplastic flow of a PM 6061 Al alloy was investigated in a wide range of temperature from 430 to 620°C. From the data, threshold stress and activation energy for plastic flow were analyzed and the deformation mechanisms were discussed. The current research is undoubtedly worthwhile to be pursued to understand the effect of reinforcement on superplastic flow and tensile elongation behavior of the PM 6061 Al composites. The effect of reinforcement on superplastic flow in aluminum composites was investigated based on the current data.

2. Experimental method

The present alloy was fabricated through powder-metallurgy route. The 6061 Al powders with an average diameter of 20 μm, which were supplied by Toyo
Aluminum Ltd, were mechanically stirred, ultrasonically mixed in an alcoholic solvent and then dried in air. The mixed powders were consolidated at 570°C in a vacuum hot press with a pressure of 50 MPa for 0.6 ks. The consolidated billet was heated to 500°C and then extruded by 85:1 at 350°C. After extrusion, the material was held in a furnace preheated to 529°C for 10.8 ks. and aged at 180°C for 28.8 ks. Tensile test samples with a gage length of 4 mm were machined from the heat-treated bar with the tensile axis parallel to the extrusion direction. Strain-rate-change (SRC) tests were carried out to establish the strain rate–stress relationship at various temperatures in a range between 430 and 620°C. Each SRC test covered the strain-rate range from $10^{-4}$ to $10^{-1}$ s$^{-1}$. The temperature of the sample was monitored using three separate thermocouples and controlled within $\pm 1$ K. Elongation-to-failure tests were conducted under a constant cross-head speed condition. Microstructures were characterized by transmission electron microscope (TEM). A differential scanning calorimetry (DSC) experiment was conducted on a 50 mg of the heat-treated material with a constant heating rate of 10 K/min.

3. Results

Fig. 1 is a typical transmission electron microscopy for the microstructure revealed after the heat treatment on the extruded alloy. The grains are seen to be equiaxed. The linear intercept grain size, $L$, measured based on 15 TEM micrographs was 3.5 μm (mean grain size, $d$, is 6.1 μm as $d = 1.74 l$). The result of DSC test for the heat-treated sample is shown in Fig. 2. The incipient melting point determined from the intercept of the two dotted lines on a continuous endothermic curve is 596°C. This temperature is close to the solidus temperature of a commercial 6061 Al alloy reported as 582°C [14]. A small endothermic peak reported to appear in the DSC curve of the 6061 Al composites [9,15] in front of a continuous endothermic peak cannot be detected in the present DSC result. This small peak has been interpreted to represent partial melting at the reinforcement/matrix interfaces and at Al grain boundaries where strong segregation of solute atoms such as Mg, Cu or Si took place [16].

Fig. 3 shows the effect of temperature on elongation-to-failure of the present material at different strain rates. Tensile elongations larger than 300% were achieved at $10^{-2}$ s$^{-1}$ in a relatively wide range of temperature between 520 and 600°C. The maximum tensile elongation of 430% was attained at 590°C and at $10^{-2}$ s$^{-1}$. Relatively small tensile elongations of 150–180% were obtained, however, at a higher strain-rate of $10^{-1}$ s$^{-1}$. This relatively low tensile elongations at $10^{-1}$ s$^{-1}$ should be attributed to the high values of stress exponent associated with this strain rate, which will be shown in the next figure. The rapid decrease in tensile ductility at 610°C, which is higher than the incipient melting temperature, on the other hand, is likely due to excessive formation of liquid phase or/and rapid grain growth.
Fig. 4. Strain rate–stress relationship established based on the data from the SRC tests at various temperatures.

The variation of strain rate as a function of flow stress at various temperatures in the range between 430 and 620°C is shown in Fig. 4. The relationship between the logarithmic strain rate and the logarithmic flow stress shows a sigmoidal shape. It is convenient to divide the data in Fig. 4 into four regions denoted by I, II, III and IV. The region I is for temperature range from 520 to 620°C and strain-rate range from \(10^{-4}\) to \(\sim 10^{-2}\) s\(^{-1}\), showing the characteristic curvature associated with the presence of a threshold stress. At the highest temperature of 620°C, high strain-rate data over \(10^{-3}\) s\(^{-1}\) could not be obtained because of premature failure in course of SRC testing. The region II is for the high strain rates beyond the range defined for Region I, showing the presence of another deformation mechanism, most probably, dislocation climb creep \((n = 5)\) [17]. The region III is characterized for the lower temperature range between 430 and 490°C, having curvatures similar to those shown in Region I but with steeper slopes. The region IV is associated with the high strain rate region in the temperature range for region III where stress exponents are significantly higher than 5.

4. Discussion

4.1. Plastic flow behavior of 6061 Al matrix alloy

In our analysis, we assumed that a gradual change of stress exponent with strain rate observed in Region I and III is attributed to the presence of threshold stress for plastic flow. In this case, the constitutive equation for the plastic flow is given as follows [18]

\[
\dot{\varepsilon} = KD \left( \frac{b}{d} \right)^p \left( \frac{\sigma - \sigma_{th}}{E} \right)^n
\]

(1)

where \(b\) is the burgers vector, \(E\) the Young’s modulus, \(p\) the grain size exponent, \(\sigma_{th}\) the threshold stress, \(D\) the relevant diffusivity, and \(K\) a material constant. The individual set of data for each temperature was plotted as \(\dot{\varepsilon}^{1/n}\) versus \(\sigma\) on linear scales to determine a value of threshold stress. If a linear relationship holds in this plot, then the assumed stress exponent will be validated. Fig. 5 shows the plot of \(\dot{\varepsilon}^{1/n}\) versus \(\sigma\) using the datum points in Region I. Inspection reveals that an excellent linearity is attainable for \(n = 2\). This value is most likely to be associated with grain-boundary-sliding deformation mechanism according to the theoretical models developed for description of deformation behavior of fine-grained materials at elevated temperatures [19,20]. The data in Region III, on the other hand, have been analyzed for \(n = 5\) and \(8\). These two values represent dislocation climb creep [17] and a constant structure creep [21], respectively. The results are shown in Fig. 6(a–b). Both values give reasonable linear-fit to the datum points for any given temperature. In the case of \(n = 8\), however, threshold stresses measured at all the three temperatures by extrapolating the data to zero strain rate are \(\geq 0\). This result has no physical sense. For this reason, \(n = 5\) was chosen as the true stress exponent representing the plastic flow in Region III. In Region II and IV, where too limited number of data is available to provide guiding information regarding the value of true stress exponent, an attempt for threshold-stress analysis has not been made.

The variation of strain rate as a function of the inverse temperature is shown in Fig. 7, where the strain rate is determined at \((\sigma - \sigma_{th})/E = 3 \times 10^{-4}\) for \(n = 5\) and \((\sigma - \sigma_{th})/E = 10^{-4}\) for \(n = 2\). The activation energy for plastic flow after threshold stress compensation can be computed using the following relation:

\[
Q_t = -R \int \frac{d\ln \dot{\varepsilon}}{d(1/T)} \frac{\sigma - \sigma_{th}}{E}
\]

(2)

From Fig. 7(a), it was found that the value of true activation energy, \(Q_t\), for Region III is 110 kJ/mole. This value is reasonably close to that anticipated from self-diffusion in pure aluminum, \(Q_L\) \((= 142\) kJ/mol [22]). In Region I, the activation energies measured in the temperature range from 520 to 590°C is 110 kJ/mole, being close to \(Q_t\), but those measured
between 590 and 620°C are abnormally higher than $Q_L$. A similar trend in the activation-energy behavior, increasing from $Q_t = Q_L$ to $Q_t \gg Q_L$, has been reported on the PM 20% Si$_3$N$_4$/6061 Al composites [23] in the similar temperature range defined for Region I. Such jumps in $Q_t$ value can be attributed to the creation of a liquid phase upon melting and its volume increasing with temperature, since the temperatures for the transition in activation energy almost corresponded to their incipient melting temperatures determined by their DSC testing. There is, however, a dissimilarity in the activation-energy behavior between the unreinforced and reinforced materials. That is, the transition temperatures for the PM 20% Si$_3$N$_4$/6061 Al composites are near 560°C, which is $\sim$ 30°C lower than that for the present PM 6061 matrix alloy. This difference is readily explainable if solute concentration at reinforcement/Al interfaces is much higher than at Al grain boundaries such that partial melting takes place in the composites before matrix melting. TEM studies on a Si$_3$N$_4$/5052 Al composite by Koike et al. [16] revealed in fact that Mg concentration was significantly higher at reinforcement/Al interfaces than at Al grain boundaries.

The variation in $\dot{\varepsilon}/D_L$ as a function of modulus compensated effective stress, $(\sigma - \sigma_0)/E$, is shown in Fig. 8. As can be seen, the data for Region I are well superimposed well onto a single line whose slope represents $n = 2$. The equation depicting the strain rate–stress relationship for Region I is represented in the following form:

$$\frac{\dot{\varepsilon}}{D_L} = 3.5 \times 10^{26} \left(\frac{\sigma - \sigma_0}{E}\right)^2 \left(\frac{h}{d}\right)^2$$

(3)

In this relation, the grain size exponent, $p$, was assumed to be 2 based on the phenomenological relation developed for a number of lattice-diffusion controlled metallic alloys [24]. It can be noted from the same figure that the data in Region II, III and IV are also almost overlapped on another single curve. In this plotting, the values of $(\sigma - \sigma_0)/E$ for Region II and IV were assumed to be equal to $\sigma/E$. This is not an unreasonable treatment since even though a threshold stress exists, apparent stress in these high strain rate regimes is anticipated to be much higher than the threshold stress. It is worthwhile to note that the linear relationship for $n = 5$ broke down from above the normalized strain rate of $\sim 3 \times 10^{12} \text{ m}^{-2}$. Sherby and Burke [21] demonstrated that power-law breakdown occurs in face-centered cubic metals at a normalized strain rate of the order of

Fig. 6. The plot of $\dot{\varepsilon}^{1/n}$ versus $\sigma$ for Region III, where: (a) $n = 5$; and (b) $n = 8$ is assumed.

Fig. 7. Activation energy measurement at given values of $(\sigma - \sigma_0)/E$ in: (a) Region I and III.

Fig. 8. $\dot{\varepsilon}/D_L$ versus $(\sigma - \sigma_0)/E$ relation for the present alloy.
\( \dot{\varepsilon}/D_L \sim 10^{13} \text{ m}^{-2} \). Therefore, it is concluded that the current data above \( \sim 3 \times 10^{12} \text{ m}^{-2} \) resides in the power law break-down region.

4.2. Reinforcement effect on superplastic flow of PM aluminum composites

Recently, Mabuchi and Higashi [25] analyzed the \( \dot{\varepsilon}/D_L(d/b)^2 \sim (\sigma - \sigma_{th})/E \) relation for a number of superplastic aluminum matrix composites including Si₃N₄p, w/6061 Al composites (p and w denotes particulate and whisker, respectively) in the temperature range below the incipient melting temperature determined by DSC testing, and demonstrated that composites are stronger than unreinforced matrix alloys. The high strength in composites was attributed to strengthening by ceramic reinforcement constraining the macroscopic plastic flow whose effect depends on its volume fraction and size. It should be pointed out, however, that their constitutive equation for the unreinforced alloys used to evaluate the strengthening effect by reinforcement was not based on the experimental data of the unreinforced superplastic alloys fabricated using the similar processing technique but on the phenomenological relation [24] developed for a number of superplastic metals mostly processed by ingot processing (IM). Fig. 9 is a plot directly comparing the relation of \( \dot{\varepsilon}/D_L(d/b)^2 \sim (\sigma - \sigma_{th})/E \) between the superplastic PM aluminum composites and PM 6061 Al matrix alloy studied in the present. The list of superplastic aluminum composites in Table 1 is from Table 2 in [25] except for the 20% SiCp/6061 Al composite studied very recently [26]. In the same figure, Sherby and Wadsworth’s phenomenological creep relation for superplastic metals [24] and a relation for a superplastic PM 2124 Al alloy [27] established based on the experimental result are also plotted for comparison. Two interesting results are found. First, the superplastic PM 2124 and PM 6061 Al alloys both having very similar strength are stronger than conventional superplastic metals. Second, the strength of PM aluminum matrix alloys is slightly higher or similar to those of PM aluminum composites, which contradicts with Mabuchi and Higashi’s conclusion on the strengthening effect by reinforcement in high-strain-rate superplastic composites. A possible origin of strength of superplastic PM alloys higher than that of superplastic IM alloys may be related to contribution from the fine oxide dispersoids that form as a result of processing the alloy by powder metallurgy, since it is well known that dispersion-strengthened aluminum al-

Fig. 9. \( \dot{\varepsilon}/D_L(d/b)^2 \) versus \((\sigma - \sigma_{th})/E\) for various aluminum composites and unreinforced Al matrix alloy.
The critical strain rate below which the reinforcement becomes ineffective is given as follows when a power low matrix is fully relaxed such that strengthening is eliminated. This occurs when the volume rate of matter contained, Eq. (5) is rewritten as follows [31]:

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \left( \frac{4\sigma}{(R/L)^2\Omega D_b} \right) \frac{\delta D_b}{\dot{\varepsilon}_0 kT R^3} n(n-1)$$

(5)

where \( L/R \) is the aspect ratio of reinforcement, \( 2R \) the width of reinforcement, \( 2L \) the length of reinforcement, \( \Omega \) the atomic volume, \( \delta D_b \) the diffusion parameter for interface diffusion along reinforcement/AI phase. When lattice contribution to lattice diffusion flux is included, Eq. (5) is rewritten as follows [31]:

$$\dot{\varepsilon}_c = \dot{\varepsilon}_0 \left( \frac{4\sigma}{\dot{\varepsilon}_0 kT \Omega (L/R)^2 R^3} + \frac{D_L}{(L/R) R^2} \right) n(n-1)$$

(6)

By letting \( \sigma = \sigma_{\text{th}} \) and \( n = 2 \), \( \sigma_0 = G \) where \( G \) is the shear modulus, \( \delta D_b = \delta D_{gb} \) (\( D_{gb} \) denotes grain boundary diffusion in pure aluminum), \( d = 2 \mu m \) and \( \dot{\varepsilon}_0 = 3.5 \cdot 10^{26}/2.6^3 \cdot D_L(b/d)^2 \), the values of \( \dot{\varepsilon}_c \) for the superplastic composites could be computed. The \( \dot{\varepsilon}_c \) calculated by Eq. (6) for different values of \( R \) and \( L/R \) is presented in Fig. 10 as a function of temperature. The result shows that \( \dot{\varepsilon}_c \) increases as temperature increases and \( R \) decreases. The \( \dot{\varepsilon}_c \) curve defines the regimes above which strengthening is retained and below which strengthening is eliminated. A shaded box superimposed in Fig. 10 represents the strain rate–temperature range where the experimental data for the composites in Fig. 9 are contained. Comparison between theory and experiment indicates that the model predicts that the material with \( R = 0.1 \mu m \) is embedded into the non-strengthening regime, which is well consistent with the experimental result that strengthening is eliminated in the tested temperatures and strain rates. In case of the material with \( R = 0.5 \mu m \) and whisker with \( L/R = 4/0.25 \), high strain-rate data over \( 10^{-2} \) or \( 10^{-3} \ s^{-1} \) resides in the strengthening regime while lower strain-rate data is in the non-strengthening regime. The predictions for the materials with \( R = 2.5 \) and \( 4 \mu m \), on the other hand, are completely inconsistent with the experimental finding since all the creep data are within the strengthening regime. Therefore, there remain observations for the large \( R \) sizes to be in conflict with Rösler model.

If continuous film of liquid exists between AI and reinforcement such that a higher diffusivity path is provided, then \( \dot{\varepsilon}_c \) can be higher than that estimated by Eq. (6). This speculation, however, is against the experimental result that the testing temperature range of the composites in Fig. 9 is below the incipient melting temperatures determined by the DSC testing, unless the amount of liquid phase created in this temperature range is too small to be detected by a DSC accuracy.
Damage accumulation around the reinforcements expected to occur during plastic flow can also reduce the strengthening effect by reinforcement [32]. Such damage, however, appears to be insignificant, at least, at the strain level (typically, \( \varepsilon = 0.1 \)) at which the stress values used to establish the strain rate–stress relations of the composites in Fig. 10 are read in the stress–strain curves. Recent cavitation study on a superplastic PM Si\(_3\)N\(_4\)/6061 Al composite [33] in which the volume of cavities at a small strain of \( \varepsilon = 0.2 \) was determined to be as low as 0.3% when tested at 833 K and 8 MPa supports this speculation. Another evidence for minimal damage accumulation within small strain in superplastic composites could be indirectly provided by investigating reproducibility of strain rate–stress curves of a superplastic 20% SiC\(_{\text{p}}\)/6061 Al composite obtained from the strain rate change (SRC) test repeated uninterruptedly [26]. Overlapping of the curves was observed, suggesting that little microstructural change including damage accumulation occurred during SRC testing.

5. Summary

1. Elevated temperature mechanical properties of a high-strain-rate superplastic PM 6061 matrix alloy have been studied in a wide range of temperature from 430 to 620°C and strain-rate range from \( 10^{-4} \) and \( 10^{-1} \) s\(^{-1} \).

2. Tensile elongations over 300% were obtained in the temperature range between 520 and 590°C at the strain rate of \( 10^{-2} \) s\(^{-1} \), indicating that the PM 6061 matrix alloy is superplastic at relatively high strain rates.

3. The whole testing range could be divided into three regions based upon the deformation mechanisms determined by the true n value evaluation: Region I is for the high temperature range at low and intermediate strain rates, where \( D_L \) controlled grain boundary sliding \((n = 2)\) governs the plastic flow. Region II and III are for the high temperature range at high strain rates and for the low temperature range at low and intermediate strain-rate range, respectively, where \( D_L \) controlled dislocation climb creep \((n = 5)\) dominates the plastic flow. Region IV is for the low temperature range at high strain rates, where powder-law relationship is broken down.

4. Comparison of the data in Region I with those for superplastic 6061 Al and other aluminum composites reveals that strengthening effect by reinforcement vanishes when superplastic flow controls the deformation of composites or testing temperature is quite high.
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References