[文章编号] 1004- 0609(2001)S1- 0143- 04

Effect of partial melting on superplasticity of AlNp/6061Al composite①

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[Abstract] AlN particulate reinforced 6061 aluminum alloy composite was fabricated by powder metallurgy method and hot-rolled after extrusion. Tensile strength and elongation at elevated temperature were measured by tensile test at initial strain rates between 10^{-2} s^{-1} and 10^{0} s^{-1}. The AlNp/6061Al composite exhibits an m-value of 0.42 and a maximum elongation of 450% at 863 K. Differential scanning calorimeter was used to ascertain the possibility of any partial melting in the vicinity of optimum superplastic temperature. Partial melting resulting from solute segregation at interfaces has much influence on superplasticity of the composite. It is postulated that AlNp/matrix interface sliding occurs along with grain boundary in superplastic deformation.

[Key words] superplasticity; aluminum matrix composite; AlN particulate
[CLC number] TG 115.5 **[Document code]** A

1 INTRODUCTION

Metal matrix composites are attractive for many structural applications because of their high specific strength and their modulus of elasticity. For metal matrix composites containing ceramic reinforcements, stress concentration during tensile deformation occurs at interfaces between the matrix and ceramic, so that, cavitation is excessively developed at interfaces, resulting in premature fracture. In a previous paper[1], it was suggested that the main deformation mechanism of high strain rate superplasticity (HSRS) in ceramic whisker or particulate reinforced aluminum alloy composites is grain boundary sliding (GBS), and that liquid phases in HSRS serve both to relax the stress concentration and to limit the appearance of internal cavitation and subsequent failure. Although the role of liquid phases on the HSRS is not yet fully understood, many experimental results[2~5] reveal that the development of cavitation during tensile deformations limited by the presence of liquid phases. However, the presence of liquid phases does not always lead to HSRS, because too much liquid phases at grain boundaries cause interfacial decohesion. It has been found that a large elongation is obtained at the temperature, which is close to or slightly above the solidus temperature of the matrix. This shows that there is an optimum amount of liquid phase for HSRS in these composites.

This paper aims to study the nature of interfaces and surfaces of deformed specimens in AlNp/6061Al composites to understand GBS related with liquid phases at interfaces during superplastic flow.

2 EXPERIMENTAL

AlN particles with an average particle size of 1.78 μm, fabricated by a carbothermal nitridation method were used as reinforcement material, whose chemical compositions are w(Fe) = 50×10^{-6}, w(Si) = 100×10^{-6}, w(O_2) = 0.01, w(N) = 0.338, w(C) = 0.02 and Al the balanced. 6061 aluminum alloy powder with a particulate size less than 45 μm was mixed with the AlN particulates in an alcoholic solvent for 24 h by a blender machine to produce composites with AlN volume fraction of 0.30. The mixed powders were compressed at 773 K in vacuum under the pressure of 200 MPa for 20 min, and then forged at 773 K in air under the pressure of 495 MPa for 20 min.

The distribution of Al_2O_3 in the materials is shown in Fig. 1 by SEM. The composite was hot extruded at 773 K with an extrusion ratio of 44. After heating in temperature range form 673 K up to 823 K, the extrusion billets were hot-rolled at 803 K. The strain in each pass was less than 0.1 and the reheating-holding time between rolling passes was about 5 min. The final thickness of the hot-rolled composite was less than 0.8 mm. Tensile specimens with a gauge length of 5 mm and a gauge width of 4 mm were deformed in the temperature range of 823 K to 893 K in the strain rate range from 10^{-2} to 10^{0} s^{-1}. The microstructural characterization after superplastic tensile was observed by SEM.

3 RESULTS AND DISCUSSION

Flow stress (σ) and true strain rate ($\dot{\varepsilon}$) in a

① [Foundation item] Project (597810004) supported by the National Natural Science Foundation of China
[Received date] 2000- 07- 18; [Accepted date] 2000- 10- 20

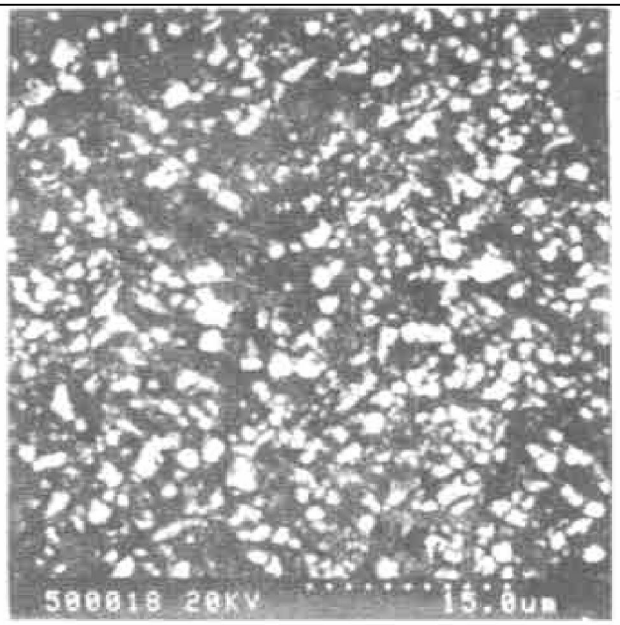

Fig. 1 Distribution of AlN particulate reinforcement in matrix of 6061Al alloy

superplastic material are related via the equation $\sigma = k\dot{\varepsilon}^m$, where K is a constant and m is the strain rate sensitivity index. The m-value of a superplastic material is normally greater than 0.3 because a high m-value is expected to suppress neck formation and lead to high elongation. The relationships between elongation-to-failure and the strain rate at different test temperatures are shown in Fig. 2(a). The maximum elongation of 450% was attained at a temperature of 863 K and at a strain rate of $1.67 \times 10^{-1}\ s^{-1}$, i.e. under the conditions where the maximum m value was found. DSC curve shows that the composite with $\varphi_f = 0.30$ has a solidus temperature of 845 K and a melting temperature of 904 K. The optimum temperature at which a maximum elongation is obtained lies in 853~873 K, which is above the solidus temperature (845 K) of the composite. This temperature is the same as the previously reported optimum superplastic temperatures for 6061Al alloy matrix composites[6,7].

A series of tests were conducted over a range of strain rates, and flow stresses at a true strain of 0.2 were measured. Fig. 2(b) shows a lg-lg plot of stress vs strain rate for various temperatures.

As shown in Fig. 2(b), these curves have a characteristic sigmoidal shape typically associated with superplastic behavior. The strain rate sensitivity index, m, was determined from the slope of these curves. The variation of m-value, as a function of strain rate, is also shown in Fig. 2(b). The highest strain rate sensitivity of $m = 0.42$ was achieved at a temperature of 863 K. At higher temperatures the maximum m-value decreased slightly.

Fig. 3 shows a DSC result for the as-rolled sample. The sharp endothermic peak at a temperature range of 845~904 K is attributed to the partial melting of reaction phases, which are probably related to solute segregation at interfaces. The onset temperature for partial melting is estimated to be 845 K, which is determined from an intercept of two solid lines shown in Fig. 3. In general the solidus temperature depends on the volume of solute atoms. Therefore, the difference in thermal stability between the composite and the rapidly solidified powder of the matrix alloy is probably related to solute segregation, because solute segregation and reaction phases resulting from solute segregation at the interfaces was previously reported in metal matrix composites[8].

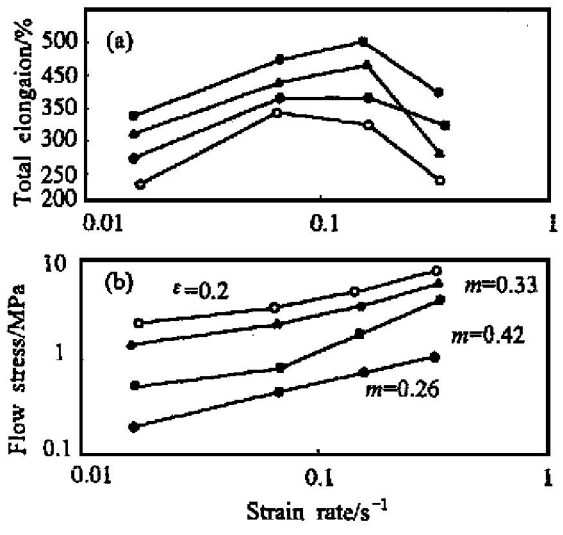

Fig. 2 Superplastic characteristics of AlNp/6061 Al composite extruded and hot-rolled
○—823 K; ▲—843 K;
■—863 K; ●—893 K

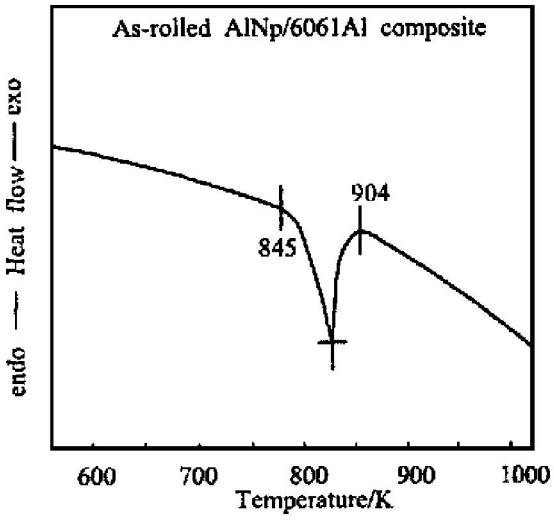

Fig. 3 DTA result for the as-rolled sample

Fig. 4 shows the fracture surfaces of the composite tested at 863 K at an initial strain rate of $1.67 \times 10^{-1}\ s^{-1}$. The deep dimple-patterns in the specimen tested show occurrence of ductile fracture. The long

thread-like filaments on the fracture surfaces at both testing temperatures suggest occurrence of sliding of viscous layer and occurrence of partial melting at grain boundaries.

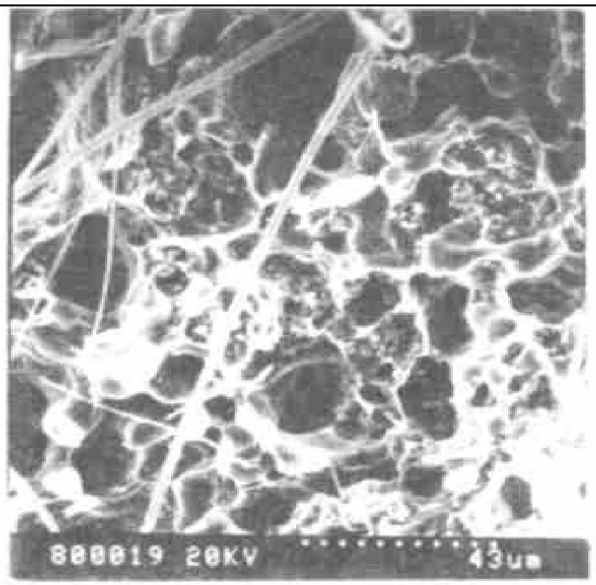

Fig. 4 SEM of fracture surface of compositetested at 863 K

Superplasticity is a thermally activated process, therefore the activation energy value is believed to be a good indicator of the deformation mechanism involved. The activation energy is normally determined by an Arrhenius plot of $\ln\varepsilon$ vs the reciprocal of temperature at constant stress. As aluminum matrix composites have been shown to exhibit a higher stress exponent with lowering strain rate, the concept of a threshold stress is commonly invoked[9]. In order to calculate the threshold stress the following power law equation has been used[10]:

$$\dot{\varepsilon} = \frac{ADGb}{kT}\left[\frac{b}{d}\right]^{p}\left[\frac{\sigma - \sigma_0}{E}\right]^{n} \quad (1)$$

where $\dot{\varepsilon}$ is the strain rate, D the appropriate diffusivity, G the shear modulus, b the Burgers vector, k the Boltzmann constant, T the test temperature, d the grain size, p the grain size exponent, σ the applied stress, σ_0 the threshold stress, E the Young's modulus, $n(=1/m)$ the stress exponent and A a dimensionless constant. According to the above equation, the threshold stress can be determined by plotting $\dot{\varepsilon}^{1/n}$ against flow stress σ and extrapolating the plot to zero strain rate. The stress exponent of 2, commonly found in conventional superplastic alloys, has been used in this study to calculate the threshold stress. The threshold stress values obtained by extrapolating the best straight line through the superplastic data are listed in Table 1.

Since the shear modulus G is equal to $E/2(1+\upsilon)$, where the Poisson's ratio, υ is assumed to be 0.34, and also assuming that the shear modulus of the Al_2O_{3p}/6061Al composite exhibits the same dependence on temperature, as aluminum, the relationship between the shear modulus and the temperature for the composite in this study may be expressed as:

Table 1 Threshold stress obtained through extrapolation

T/K	823	843	863	893
σ_0/MPa	1.34	0.85	0.316	0.148

$$G = 4.2535\times10^{4} - 14T \ (\text{MPa}) \quad (2)$$

In order to determine the activation energy for superplastic deformation of Al-MMCs, the following relationship at constant strain rate is derived from Equation (1)

$$Q = R\frac{\partial\ln((\sigma-\sigma_0)^{n}/G^{n-1}T)}{\partial(1/T)} \quad (3)$$

Fig. 5 shows a plot of $R\ln((\sigma-\sigma_0)^{n}/G^{n-1}T)$ vs $1/T$ at a strain rate of $1.67\times10^{-1}\ s^{-1}$ with $n=2$. In this figure the slope of the line which represents the activation energy for superplastic deformation is determined to be 330.53 kJ/mol, which was apparently higher than the activation energy for lattice self-diffusion in aluminum alloys (142 kJ/mol). Other researchers[11,12] also reported similar values of the activation energy of several other superplastic aluminum composites. Two explanations of the high activation energy have been proposed. Mabuchi et al[11] believe that high activation energies are due to the presence of some liquid phase at the testing temperature. Results from DSC support this point of view in the cases of 5052/Si_3N_{4p}, 6061/Si_3N_{4p}, and 7064/Si_3N_{4p} composites, but 2124/Si_3N_{4p} (particles of 0.2 μm) and 2124/Si_3N_{4w} also showed high activation energies in spite of the fact that DSC did not reveal partial melting in the temperature range studied. However, it is argued that there might have been such a small amount of liquid present that DSC could not detect it. The DSC results in the present study again suggest that there is liquid phase present at deformation temperature, despite the fact that the activation energy is high. The possibility of the presence of a liquid phase even when DSC can't detect it needs to be investigated by some other method such as in situ TEM

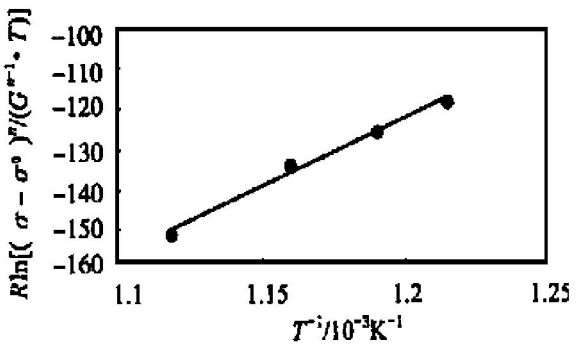

Fig. 5 Variation of $R\ln[(\sigma-\sigma_0)^{n}/(G^{n-1}T)]$ with $1/T$ at a constant strain rate of $1.67\times10^{-1}\ s^{-1}$ of a AlNp/6061Al composite with $n=2$

examination.

4 CONCLUSIONS

1) The AlNp/6061Al composite shows the maximum superplastic elongation of 450% at testing temperature of 863 K at the initial strain rate of $1.67 \times 10^{-1}\ s^{-1}$.

2) The fracture surface of the composite has filaments and partial liquid phase, which shows that interface sliding at the liquid phase has much influence on the HSRS in addition to grain boundary sliding in the composite.

3) The activation energy values in composites are much higher than that of aluminum alloy and it is thought that the superplastic deformation mechanism of the composite has changed greatly because of mixing the reinforcement into the matrix.

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(Edited by ZHU Zhong-guo)