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Production of superplastic aluminium composites reinforced with Si_3N_4 by powder metallurgy

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Superplastic aluminium composites were processed from these fine aluminium powders of less than 20 μm and reinforcements of either Si_3N_4 whiskers or Si_3N_4 particulates by hot extrusion at temperatures between 733 and 793 K with a reduction ratio of 100:1. The dispersion of the reinforcements was homogeneous, and the size of the grains of this matrix alloy after extrusion was fine, at less than 3 μm for the composites reinforced with either Si_3N_{4w} or Si_3N_{4p}. All composites showed large superplastic elongations of more than 300% in a relatively high strain-rate range from $4 \times 10^{-2} \sim 2\ s^{-1}$ at testing temperatures between 788 and 833 K. These superplastic composites also exhibited excellent mechanical properties at room temperature, which are supposedly attributable to both the homogeneous dispersion in reinforcements and the fine-grained structures.

1. Introduction

It has been demonstrated that ceramic whisker- or particulate-reinforced aluminium composites exhibit a unique combination of high specific room-temperature strength and modulus. Aluminium composites, however, have lower ductility than the matrix alloys, which leads to high costs in the final forming for composites. Therefore, an improvement of ductility in composites has been desirable for many structural applications.

Recently, there has been a growing interest in the development of fine-grained microstructures for superplastic forming of aluminium alloy matrix composites. By using this approach, the possibility of superplastic forming has been demonstrated in some aluminium alloy composites [1–5]. These superplastic composites were processed by foils, casting, or the powder metallurgy method. In particular, a SiC_w/2124 aluminium composite [2], which was processed with thermo-mechanical treatment after fabricating by the powder metallurgy method, showed a large elongation of about 300% at a high strain rate of $3.3 \times 10^{-1}\ s^{-1}$.

More recently, it was reported that some aluminium alloy composites reinforced with either Si_3N_{4w} [6–8] or Si_3N_{4p} [9–11] fabricated by the powder metallurgy method also showed superplastic behaviour at high strain rates of more than $10^{-1}\ s^{-1}$. It is noted that the superplastic aluminium alloy composites were processed with hot extrusion [12], without any special thermomechanical treatment for grain refinement. In this paper, the fabrication procedure of the superplastic aluminium composites reinforced with either Si_3N_{4w} or Si_3N_{4p} and these mechanical properties are presented.

2. Experimental procedure

Commercial powders of aluminium alloys, whose chemical composites are shown in Table I, were supplied by Toyo Aluminum Ltd. The characteristics of both Si_3N_4 whiskers and Si_3N_4 particulates (UBE Industries Ltd) are shown in Table II. The procedure used to prepare the composites was a powder metallurgy method; powders of both aluminium alloys and the reinforcements of either Si_3N_{4w} or Si_3N_{4p} were ultrasonically mixed for more than 3.6 ks in an alcoholic solvent prior to drying. The volume fraction of the reinforcements of either Si_3N_4 whiskers or Si_3N_4 particulates was 20 vol % each. The mixed powders were sintered at 873 K with a pressure of 390 MPa for 1.2 ks by a hot-pressing machine. Sintering was performed in vacuum, because a degassing treatment is necessary in order to suppress harmful effects of hydrogen [13, 14]. The sintered billet was then extruded (100:1 reduction) at various optimum temperatures between 733 and 793 K for each aluminium matrix alloy.

TABLE I Chemical compositions of commercial aluminium alloy powders

		Cu	Mg	Zn	Mn	Ti	Cr	Zr	Co	Si	Fe	Al
Al–Cu–Mg	(2124)	4.20	1.48	0.12	0.64	0.06	0.06			0.12	0.09	Bal.
Al–Mg	(5052)	0.04	2.46				0.25			0.16	0.16	Bal.
Al–Mg–Si	(6061)	0.39	1.03				0.15			0.75	0.47	Bal.
Al–Zn–Mg	(7064)	0.24	2.38	6.40	0.01		0.07	0.09	0.15	0.03	0.03	Bal.

TABLE II The characteristics of Si_3N_4 whiskers and particulates

Reinforcement		Size (μm)	
		Diameter	Length
Si_3N_4 whiskers		0.1–1.5	10–30
Si_3N_4 particulates	Coarse	< 1.0	
	Fine	< 0.2	

Tensile specimens were machined from the final extruded bar which was not processed with any additional thermomechanical treatment for the refinement of grain size. The tensile specimens had a gauge length of 5 mm and a gauge diameter of 2.5 mm. In order to investigate mechanical properties, tensile tests were carried out at high temperatures between 788 and 833 K and at room temperature in air. The tensile axis was selected to be parallel to the extrusion direction. The flow stress for each strain rate was determined at a constant small strain, $\varepsilon = 0.1$. Microstructures of the as-extruded composites were investigated by scanning electron microscopy, using the polished cross-sections of specimens. Grain morphology was observed by transmission electron microscopy, for which samples were taken parallel to the extrusion direction.

3. Results and discussion

3.1. Processing and microstructures

A schematic illustration of the typical fabrication procedure for a number of superplastic aluminium composites is shown in Fig. 1. The features of the atomized Al–Mg–Si (6061) aluminium alloy powder, which was used as a matrix alloy of Al–Mg–Si composites reinforced with either Si_3N_{4w} or Si_3N_{4p}, are shown in Fig. 2. The fraction of diameter of the matrix Al–Mg–Si powders is shown in Fig. 3. It was found that the sizes of most powders were finer than 22 μm. Si_3N_4 whiskers, whose mean lengths and diameters were 20 and 0.7 μm, respectively, are shown in Fig. 4. The Si_3N_4 particulates used in this Al–Mg–Si composite, whose mean size is about 1 μm, are shown in Fig. 5.

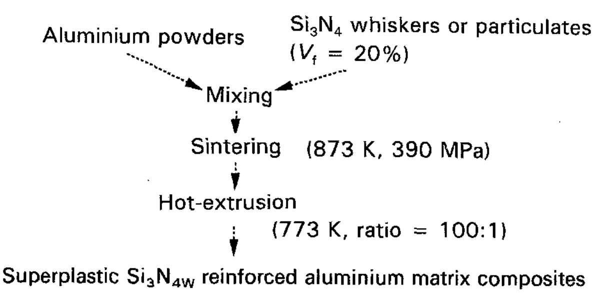

Figure 1 A schematic illustration of the typical fabrication procedure for a number of superplastic aluminium composites.

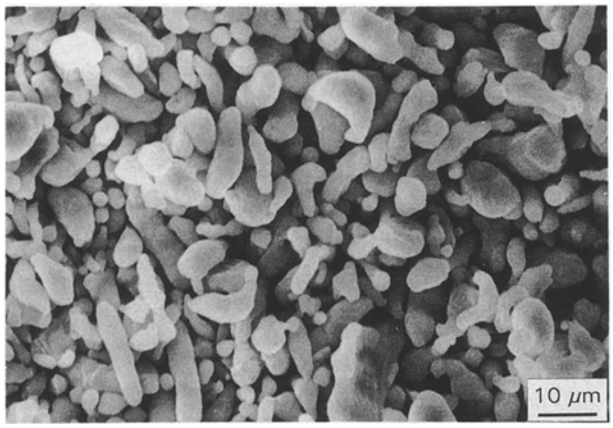

Figure 2 Features of the atomized Al–Mg–Si (6061) aluminium alloy powders used as a matrix alloy of Al–Mg–Si composites reinforced with either Si_3N_{4w} or Si_3N_{4p}.

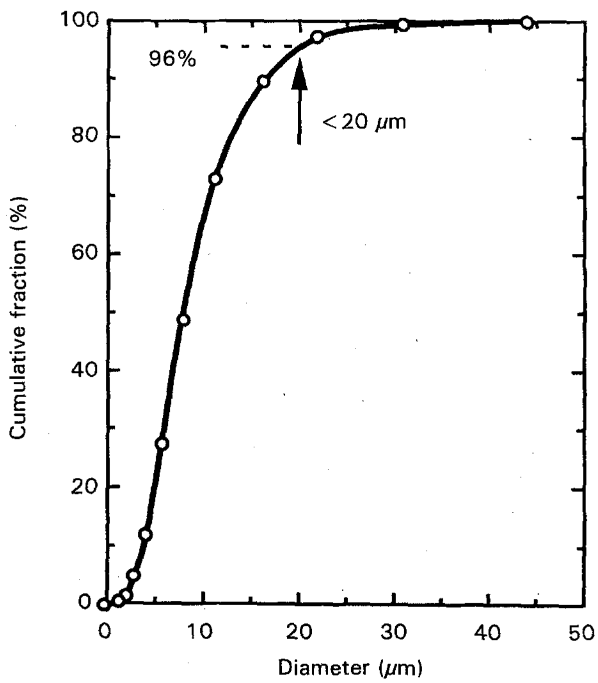

Figure 3 The fraction diameter of the matrix Al–Mg–Si powders (air atomized 6061 aluminium powder).

Typical scanning electron micrographs of the powders of Al–Mg–Si aluminium and either Si_3N_4 whiskers or particulates mixed under supersonic waves are shown in Figs 6 and 7, respectively. The micrographs show that dispersion of the aluminium

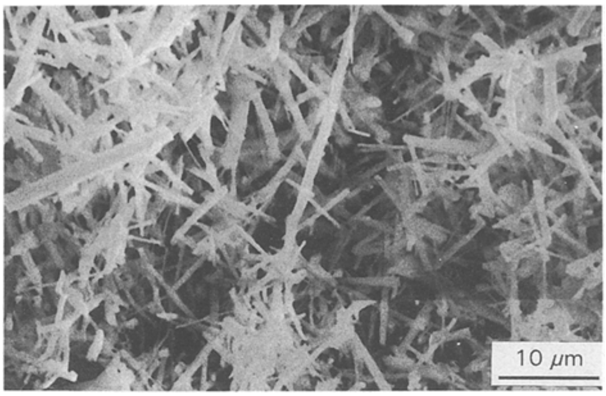

Figure 4 Typical features of Si_3N_4 whiskers.

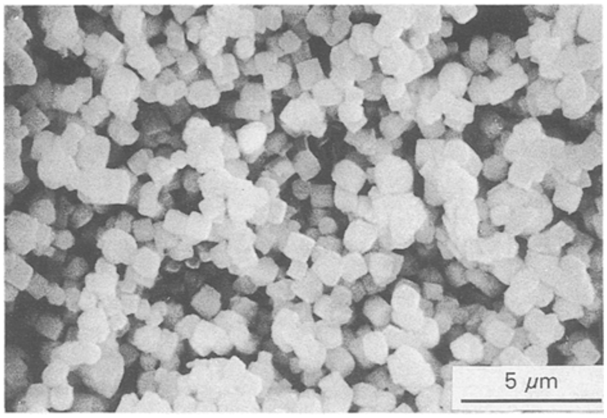

Figure 5 Typical features of Si_3N_4 particulates.

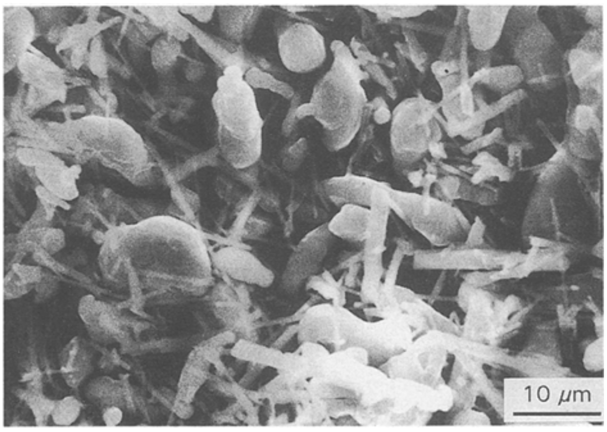

Figure 6 Typical scanning electron micrograph of the supersonically mixed powders of Al–Mg–Si aluminium and Si_3N_4 whiskers.

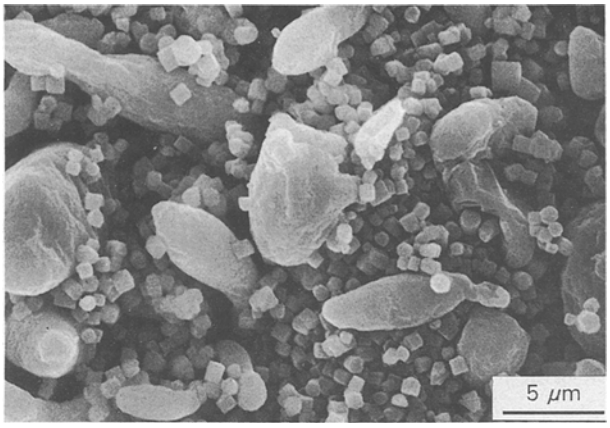

Figure 7 Typical scanning electron micrograph of the supersonically mixed powders of Al–Mg–Si aluminium and Si_3N_4 particulates.

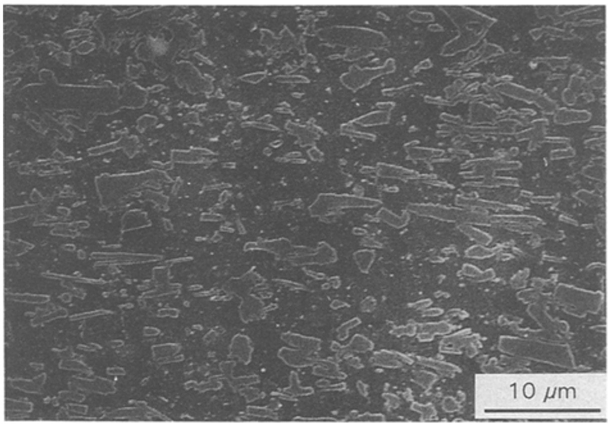

Figure 8 Typical microstructures of the Si_3N_{4w}/Al–Mg–Si aluminium composites after extrusion at a high reduction ratio of 100:1.

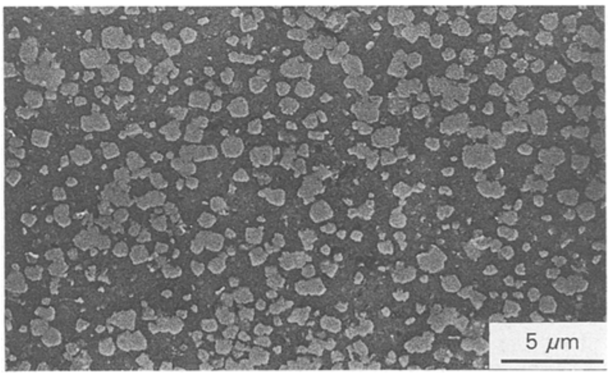

Figure 9 Typical microstructures of the Si_3N_{4p}/Al–Mg–Si aluminium composites after extrusion at a high reduction ratio of 100:1.

powders and the reinforcements of either whiskers or particulates were relatively homogeneous. In general, it is not easy for the whisker- or particulate-reinforced aluminium composites to achieve the homogeneous dispersion of the whiskers or the fine particulates, because some of them become a hard aggregation. It is probable that the relatively homogeneous dispersion of the reinforcements is attributable to both fine aluminium powders and the separation of them individually by supersonic waves.

Typical microstructures of both Si_3N_{4w}/Al–Mg–Si and Si_3N_{4p}/Al–Mg–Si aluminium composites after extruding at 773 K at a high reduction ratio of 100:1 are shown in Figs 8 and 9, respectively. It was found that both reinforcements of whiskers and particulates were dispersed more homogeneously. Dutta *et al.* [15] showed that the dispersion of particulates became more homogeneous following hot working, for an SiC_p/5083 aluminium composite. It is probable that the hot extrusion at a high reduction ratio of 100:1, in addition to the factors mentioned above, had a significant effect on the homogeneous dispersion of the fine reinforcements. In general, it is necessary for achievement of good consolidation in aluminium powders to break the oxide coating of the aluminium powders [16, 17]. Hot extrusion is one effective process for the compaction of aluminium powders, because the higher shear stress, which is caused during hot extrusion with a high reduction ratio, produces clean surfaces in the matrix powders and thus allows them to come into contact with each other. In addition, there were few pores present at the interfaces between reinforcements and the matrix alloy, as shown in Figs 8 and 9. Hot extrusion at a high reduction ratio of 100:1 in this work was also supposedly effective for the consolidation in both aluminium powders/powders and aluminium powders/ceramics reinforcements.

Typical grain morphologies of both Si_3N_{4w}/Al–Mg–Si and Si_3N_{4p}/Al–Mg–Si aluminium composites after extruding at 773 K at a high reduction ratio of 100:1 are shown in Figs 10 and 11, respectively. The grains of both composites were equiaxed and very small at about 2–3 μm. It is seen that hot extrusion in this work was effective for grain refinement [12] of the composites, as well as for good consolidation of both powders and reinforcements in the composites.

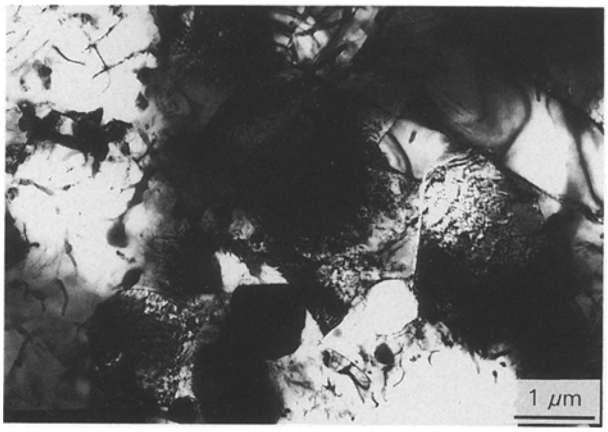

Figure 10 Typical grain morphologies of the Si_3N_{4w}/Al–Mg–Si aluminium composites after extrusion at a high reduction ratio of 100:1.

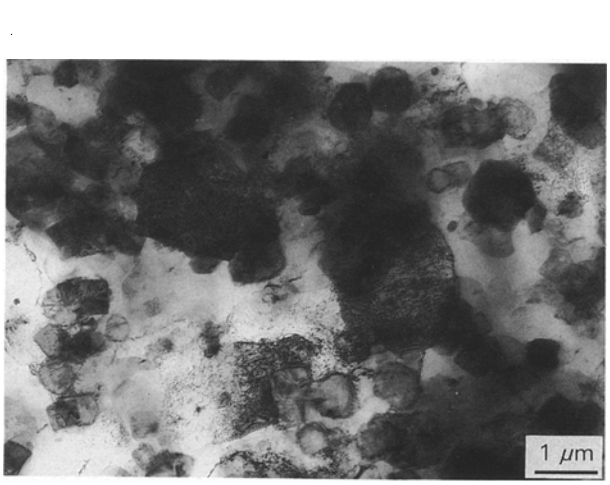

Figure 11 Typical grain morphologies of the Si_3N_{4p}/Al–Mg–Si aluminium composites after extrusion at a high reduction ratio of 100:1.

3.2. Superplastic properties

An example of the fractured specimen after superplastic deformation to failure (620%) at a high strain rate of $2\ s^{-1}$ at 833 K is shown in Fig. 12 for the as-extruded Si_3N_{4p}/Al–Mg–Si (6061) aluminium composite [11]. It is clear by comparison with an untested specimen, also shown in Fig. 12, that the specimen deformed at 833 K is fairly uniform with no visible necking. Such high superplastic potential is supposedly attributable to both homogeneous dispersions of the ceramic reinforcements and to fine grains of the matrix in the composite.

A summary of the mechanical properties in the superplastic P/M composites is given in Table III. All composites showed large superplastic elongations of more than about 300% in a relatively high strain-rate range from $4 \times 10^{-2} \sim 2\ s^{-1}$ at testing temperatures between 788 and 833 K. These composites showed high values of strain-rate sensitivity exponent of more than 0.3 at relatively high strain rates, where large superplastic elongations were obtained.

3.3. Room-temperature properties

Mechanical properties at room temperature for the as-extruded Si_3N_{4w}/Al–Mg–Si and Si_3N_{4p}/Al–Mg–Si (6061) aluminium composites are shown in Table IV, where σ_{uts} is the ultimate tensile strength, $\sigma_{0.2\%}$ is the yield strength and E(%) is the elongation

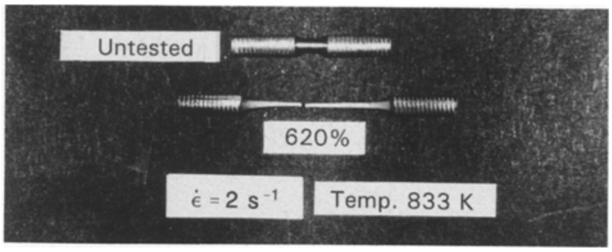

Figure 12 An example of the fractured specimen of the Si_3N_{4p}/Al–Mg–Si aluminium composite after superplastic deformation to failure (620%) at a high strain rate of $2\ s^{-1}$ at 833 K.

TABLE III Summary of superplastic properties in aluminium composites reinforced with whiskers and particulates

Composites	Temperature (K)	Strain rate (s^{-1})	m	Maximum elongation (%)
Si_3N_{4w}/2124	818	4×10^{-2}	> 0.3	280
Si_3N_{4p}/2124[a]	788	4×10^{-2}	> 0.3	830
Si_3N_{4p}/5052[b]	818	1	> 0.3	700
Si_3N_{4w}/6061	833	10^{-1}	> 0.3	285
Si_3N_{4p}/6061[a]	818	2×10^{-1}	> 0.3	615
Si_3N_{4p}/6061[b]	833	2	> 0.3	620
Si_3N_{4w}/7064	833	10^{-1}	> 0.3	380
Si_3N_{4p}/7064[a]	818	1	> 0.3	330

[a] Particulate sizes are less than 1.0 μm.
[b] Particulate sizes are less than 0.2 μm.

TABLE IV Mechanical properties at room tempeature for the as-extruded Si_3N_{4w}/Al–Mg–Si and Si_3N_{4p}/Al–Mg–Si (6061) aluminium composites

Matrix	Reinforcement	σ_{uts} (MPa)	$\sigma_{0.2\%}$ (MPa)	E(%)
6061 Al	Whisker	377.7	302.5	7.1
6061 Al	Particulate (1 μm)	307.0	225.7	7.7
6061 Al	None	199.5	114.7	20.4

to failure. Paton *et al.* [18] showed that a slight increase in both yield strength and the ultimate tensile strength, and a modest increase in the reduction of area, occurred with decrease of grain size from 60 μm to approximately 10 μm for 7075 Al–Zn–Mg–Cu alloy. The ultimate tensile strength of the Al–Mg–Si (6061) aluminium alloy without any reinforcements was about 200 MPa in the as-extruded condition. An increment of the ultimate tensile strength of both composites is supposedly attributable to reinforcement by whiskers or particulates (the homogeneous dispersion of the reinforcements) and to fine grains in the matrix of both composites.

4. Conclusions

The homogeneous dispersion of the reinforcements and the fine-grained ($1 \sim 3$ μm) structure in the matrix were obtained by the powder metallurgy method and hot extrusion for the aluminium matrix composites reinforced with either Si_3N_{4w} or Si_3N_{4p}, respectively. The composites showed excellent superplastic potential; large elongations of more than 300% at high strain rates from $4 \times 10^{-2} \sim 2\ s^{-1}$ at 788–833 K. In addition, the composites showed high strength at room temperature. These excellent mechanical properties are attributable to homogeneous dispersion of the reinforcements and the fine grain sizes in the matrix of the composites.

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