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Squeeze-casting conditions of Al/Al_2O_3 metal matrix composites with variations of the preform drying process

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The characteristics of the preform play a role in determining the final properties of MMCs. Effects of organic binder and microwave drying on preform microstructure have been examined by SEM. In the preform with organic binder, flocking processes are observed during drying. The preform has a uniform distribution of binder and dries quickly with microwave drying owing to its internal and volumetric heating patterns. The fundamental manufacturing process and controlling parameters of squeeze casting, including preform temperature, mould temperature, applied pressure and molten metal temperature, have been studied in Al/Al_2O_3 composites. MMCs have poor mechanical properties with too high temperatures of preform and molten metal due to thermal shocking of the preform, oxidation of the matrix and thermal damage to the fibers. Mould temperature barely affects the tensile strength of MMCs. High applied pressure reduces voids and solidifies the matrix faster. Conditions for squeeze casting to achieve optimal processing, are suggested. The tensile strength of MMCs can be improved by up to about 20% compared with the unreinforced matrix alloy.

1. Introduction

Recently, automotive industries have taken a growing interest in lightweight materials for saving fuel. Various research on the application of light weight materials, such as aluminium, titanium and magnesium alloys, plastics, and ceramics to the automotive engine has been carried out. Of these materials, aluminium alloys are well known to be very suitable for applications which require light weight and energy savings because of their low density. Their applications have often been limited due to their poor mechanical properties at elevated temperatures. However, aluminium alloys can meet various demands of many applications by the addition of reinforcements which have superior mechanical properties, wear resistance and good thermal stability. Because there are many advantages provided by metal matrix composites (MMCs) compared to the unreinforced aluminium alloys, they are, today, widely used in automotive engine components. These improved properties include strength, modulus, fatigue life, wear resistance, corrosion resistance, high-temperature properties, thermal conductivity, and acoustic insulation [1, 2]. Examples of automotive engine components which can be developed from MMCs are engine block, piston head, connecting rod, rocker arm, and cylinder liner.

The squeeze-casting process, because it uses a conventional casting process and because of its possibility of mass production, requires a porous preform that is a mixture of fibres and binder which is held in the casting mould by a fixture. Then, the molten metal is forced into the mould under a preset pressure for a brief period. As a result, a fibrous preform is quickly and totally infiltrated by the molten metal [3–12]. The fibrous preform is an important step in the fabrication of MMCs by squeeze-casting method, but it is difficult to make a green preform that satisfies requirements such as its shape, strength, and machinability. Several manufacturing techniques of fibrous preform which are currently known are fibre mixing by an ultrasonic cleaner to avoid a tangle of fibres [13], vacuum-assisted extraction for uniform distribution of fibres [13, 14], addition of inorganic binder to improve machinability [15], and change of strength and interfacial bonding according to the quantity of binder [16]. Because squeeze-casting conditions closely affect the MMC microstructure and mechanical properties, it is very important to determine the optimal processing conditions. Several investigations into these conditions, such as temperature and fibre distribution of the preform, magnitude of applied pressure, melt temperature, pressure holding time, and mould temperature, were performed experimentally [5, 6, 9] or numerically [12].

This study describes a processing route which is capable of fabricating preforms using various methods and experimentally determines optimal squeeze-casting conditions of Al_2O_3 short-fibre-reinforced MMCs.

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2. Variables of the squeeze-casting route

In the squeeze-casting process, complete infiltration of a base metal into the preform is very important. Therefore, determination of optimal processing conditions is needed to achieve uniform microstructure and good bonding between fibres and matrix. The major processing variables of squeeze casting are preform and melt temperature, infiltration velocity, applied pressure, and mould design [5–12].

Theoretically, the pressure difference necessary for infiltrating the molten melt into a preform can be evaluated by Hagen–Poisiulle's equation for a uniform cross-section of flow gaps, as follows in Darcy's law for a non-uniform cross-section of flow gaps, Equations 1 or 2, respectively [5]

$$\frac{\partial p}{\partial x} = -\mu u d_e^2 \quad (1)$$

$$\frac{\partial p}{\partial x} = \frac{-\mu U}{K} \quad (2)$$

where, μ, u, d_e, U, K are coefficient of viscosity, mean velocity of the fluid, diameter of the flow channel, velocity of flow in a non-fibrous preform, and infiltration coefficient, respectively. Equations 1 and 2 show that more pressure is needed to increase the infiltration velocity. Its calculated value is about 0.5 MPa. Although the pressure needed to overcome capillary pressure is about 0.45 MPa, the total required pressure is below 1 MPa for aluminium metal matrix composites reinforced with 38 vol % continuous SiC [6]. In the squeeze-casting process, the minimum required pressure to infiltrate the preform with aligned fibres is 5–10 MPa ignoring the gases (air) existing in the preform [7]. But when considering gases, the required pressure is increased because of the back pressure from these gases [8]. Therefore, a sufficiently high pressure is needed to prevent gases and voids in MMCs. For aluminium alloys, about 100 MPa applied pressure is needed to avoid shrinkage defects which occur during the phase change, i.e. from liquid state to solid state. But it is reported that MMCs can successfully be fabricated at about 30 MPa [9]. Generally, the applied pressure is about 50–80 MPa in the squeeze-casting process. It is possible to fabricate MMCs at about 150 MPa without damage to fibres because the fibres in the melt are subjected to hydraulic pressure [9].

Melt with a high latent heat has a greater increased infiltration distance than a melt with a low latent heat and the infiltration process should be stopped when molten metal reaches its melting point. The minimum superheating temperature which is required to avoid solidification prior to complete infiltration can be described as follows, Equation 3 [12]

$$\Delta T = (T_m - T_f)(C_f/C_m)[V_f/(1 - V_f)] \quad (3)$$

where, T_m, T_f, C_f, C_m and V_f are melting point of the metal, initial preform temperature, specific heat of the preform, specific heat of the melt, and fibre volume fraction, respectively.

A low melt temperature can obtain a fine microstructure due to fast solidification. But if the melt temperature is too low, the melt solidifies before complete infiltration. With high melt temperature (about 900 °C), SiO_2 in the binder reacts with the aluminium alloy as described by Equation 4, and the fibrous preform may be damaged by oxidation [9]

$$4Al + 3SiO_2 \rightarrow 2Al_2O_3 + 3Si \quad (4)$$

Infiltration velocity can be described briefly as follows

$$U = DV/s(1 - V_f) \quad (5)$$

U, D, V, s and V_f are infiltration velocity, diameter of the plunger, plunger velocity, cross-sectional area of the melt, and fibre volume fraction in the preform, respectively. Infiltration velocity, U, in Equation 5 is related to the pressure difference, Δp, in Equation 1. Because the melt is considered to be a laminar flow, for which the Reynold's number is below 100 in most cases, the interaction between melt and fibres is negligible. Hence the tensile strength of Al_{pure}/SiC composites differs little within the range 1–5 cm s^{-1} infiltration velocity, but over 20 cm s^{-1}, fibre strength is degraded in the case of 10 vol % composites [5].

TABLE I Chemical composition and mechanical properties of AC8A aluminium alloy

	Chemical composition (wt %)							Mechanical properties		
	Si	Cu	Mg	N	Fe	Mn	Al	TS (MPa)	E (GPa)	Elong. (%)
AC8A	12.7	1.1	0.9	1.6	0.8	0.1	Bal.	275	73	1

TABLE II Specifications of alumina short fibres

Material	Density (g cm^{-3})	Diameter (mm)	Length (mm)	Aspect ratio, (l/d)	TS (GPa)	Elastic modulus (GPa)
Al_2O_3 (Saffil)	3.3	4	150	38	2.0	310

3. Experimental procedure

3.1. Materials

The materials used in this investigation were Al–12Si–2Cu (JIS Type AC8A) alloy and Al_2O_3 short fibres. AC8A is mainly used as piston parts of an automotive engine because it has good mechanical properties and thermal stabilities, for example, thermal resistance, wear resistance, and low thermal expansion coefficient. The chemical composition of this alloy is given in Table I [17]. In the table, copper and magnesium contribute to the effect of heat treatment and nickel contributes to the increase of thermal resistance and wear resistance. The reinforcement was alumina short fibres (Saffil, RF grade, from ICI) containing 3%–4% silica. Specifications of the alumina fibres are shown in Table II [18].

3.2. Fabrication by the squeeze-infiltration method

3.2.1. Preform process

The preform was fabricated by means of vacuum-assisted extraction. Two drying methods were used to investigate distribution of binder, i.e. conventional and microwave heating. Fibres were mixed with only inorganic binder or inorganic and organic binders. The equipment for fabricating preforms was as follows. The preform mould was made of acryl. A 10 ton capacity hand press was used to control volume fraction of fibres easily. A vacuum pump and water tank for extracting water were installed. In the case of microwave heating, 2.45 GHz microwave was used. The inorganic and organic binders were silica colloid and glucose, respectively.

The preform was fabricated according to the following procedures. (1) A 5% aqueous medium containing inorganic binder was made. Two-thirds inorganic binder to one-third organic binder was used. (2) The reinforcement, alumina fibre, was dispersed into the aqueous medium using agitation. The level of agitation was carefully controlled to avoid agglomeration of fibres without damage. (3) Then the aqueous medium containing fibres was put into the mould and water was extracted by the vacuum pump as mentioned previously. Pressure was maintained at 30–40 mm Hg during extraction by a gate valve. (4) Finally, volume fractions of the preform were controlled by hydraulic press. These preforms have dimensions of 110 mm diameter and 20 mm thick. In one case of conventional drying, preforms were dried at 100 °C for 4 h and fired at 1000 °C for 2 h. In another case of microwave drying, 20–60 min drying times were selected to confirm effectiveness of drying times.

3.2.2. Squeeze infiltration method

The processing variables and conditions of squeeze-casting for this study are listed in Table III. For each case, composites were fabricated according to the following procedures. Aluminium matrix alloy was melted in a nitrogen environment furnace, and preform and mould were preheated at the predetermined temperature. The preheated preform and mould were set on the hydraulic press. The bottom part of the mould, had tapered holes for extracting gases. After pouring melt into the mould, the preform was infiltrated with 0.85 cm s^{-1} plunger velocity sustained under a preset pressure for 60 s. The dimensions of the ingot were 11 cm diameter and 3–4 cm thick. Aluminium matrix alloy and composite systems were T6 heat treated (solution heat treatment: 4 h at 510 °C → water quenched → ageing: 6 h at 170 °C → cooled in air).

TABLE III Processing variables of the direct squeeze-infiltration method

Variables	Conditions
Molten aluminium (°C)	750, 800, 850
Upper mould temperature (°C)	350, 500
Low cap temperature (°C)	350, 500
Preform temperature (°C)	500, 800
Ram speed (cm s^{-1})	0.85
Applied pressure (MPa)	15, 25, 40
Holding time (s)	60

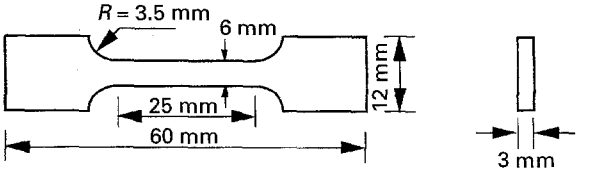


Figure 1 Configuration and dimensions of tensile specimen.

Microstructure and fibre distribution of preforms fabricated by different processing conditions were investigated by SEM. MMCs were then fabricated with those preforms under conditions of 25 MPa applied pressure, 800 °C melt temperature, and 500 °C preform and mould temperatures. Mechanical properties of the composites according to the fabrication methods of the preform and the processing conditions of MMCs, were examined by static tensile tests. The configuration and dimensions of the specimens according to ASTM E8 are shown in Fig. 1.

4. Results and discussion

4.1. Fabrication of the preform

Precise control of the volume fraction and uniform distribution of fibres can be achieved by means of vacuum-assisted extraction. The extraction pressure was maintained at 30–40 mm Hg during the process. Under a pressure of 20 mm Hg, a difference in density existed along the preform thickness because it took time to remove the aqueous medium out of the 46 μm mesh size filter. Fig. 2 illustrates difference between these two heating methods. That is, in conventional heating, the preform is dried by conduction and radiation from the external heat sources but heat is generated internally within the preform in the microwave heating. As a result of this internal and volumetric heating, the thermal gradients and the flow

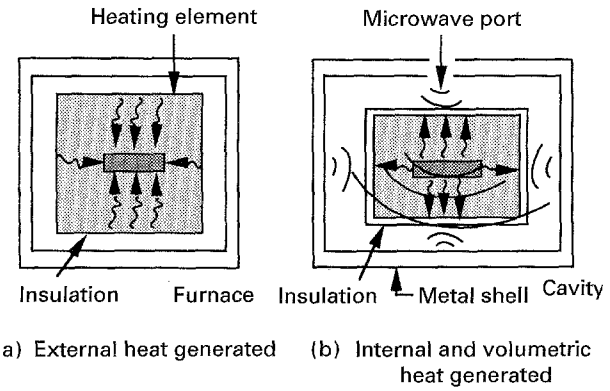


Figure 2 Heating patterns in (a) conventional and (b) microwave furnaces [19].

of heat in microwave-processed preforms are the reverse of those in preforms processed by conventional heating. Consequently, microwave drying makes it possible to heat both small and large shapes very rapidly and uniformly and to reduce thermal stresses that cause cracking during processing [19]. In this study, it took over 5 h to dry preforms by conventional heating, but it took less than 30 min by microwave heating.

Fig. 3 shows the microstructure of preforms according to different drying methods. As shown in the figure, the distribution of binder was affected by the drying methods. In Fig. 2a (conventional furnace-dried), excessive binders exist outside the preform and almost none inside, because binders were moved to the outside as evaporation of water occurred [20]. However, in Fig. 2b (microwave-dried), binders are distributed uniformly on both the inside and outside of the preform. These facts are a result of the difference between conventional and microwave processing.

Fig. 4 shows binder distribution on the outside of the preform according to drying time in microwave heating. Drying speed also affects the distribution of binder in microwave heating. In the case of 1 h drying time, binders existed excessively on the outside of the preform, similar to conventional heating.

Fig. 5 illustrates the action of organic binder. As discussed by Kruger and Kainer [15], organic binder produces inorganic binder which is added to retain the formation and to strengthen the preform for distribution on the intersecting point of fibres during firing at 1000 °C.

Fig. 6a and b show scanning electron micrographs of the inside of microwave-dried preforms with only

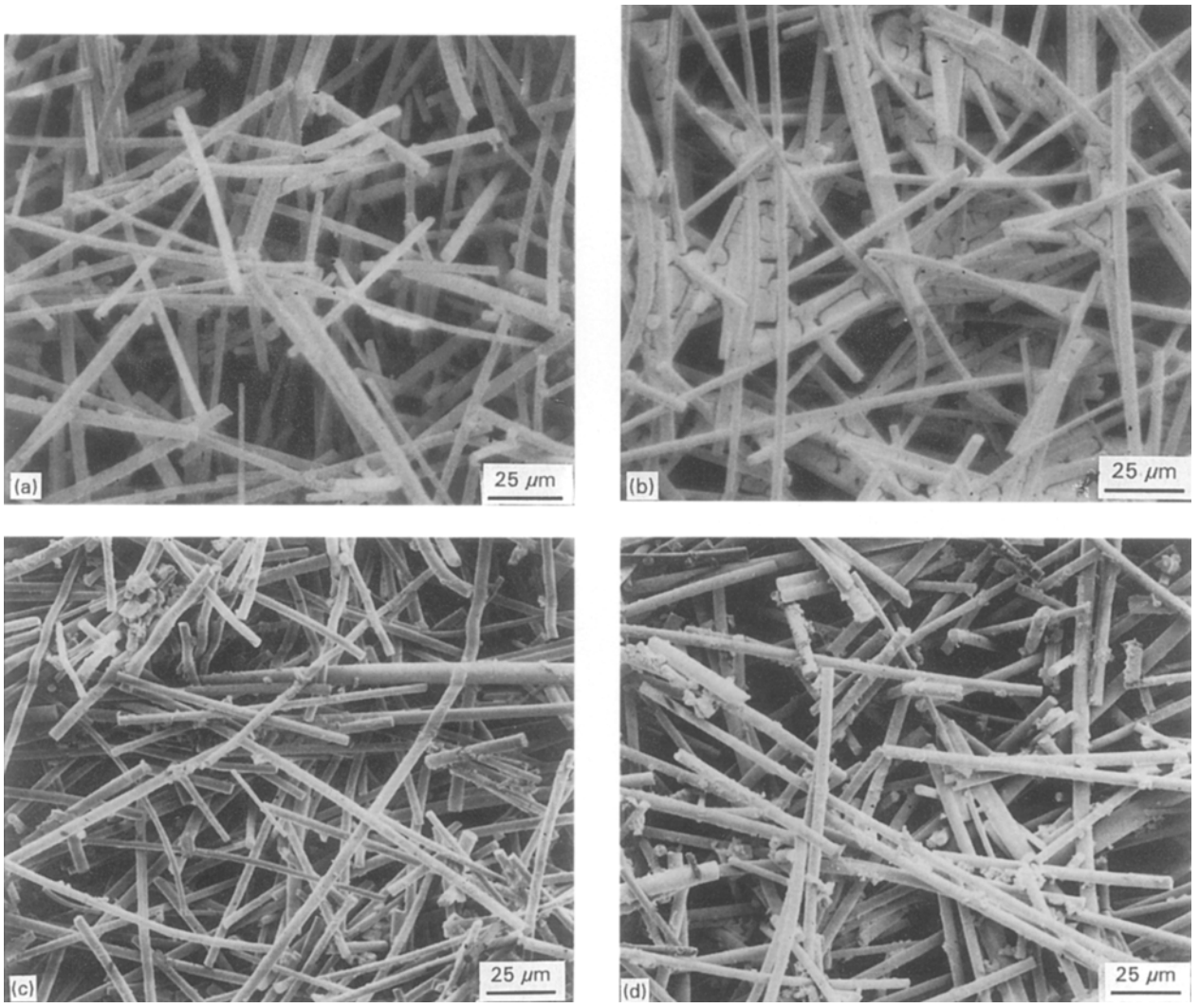
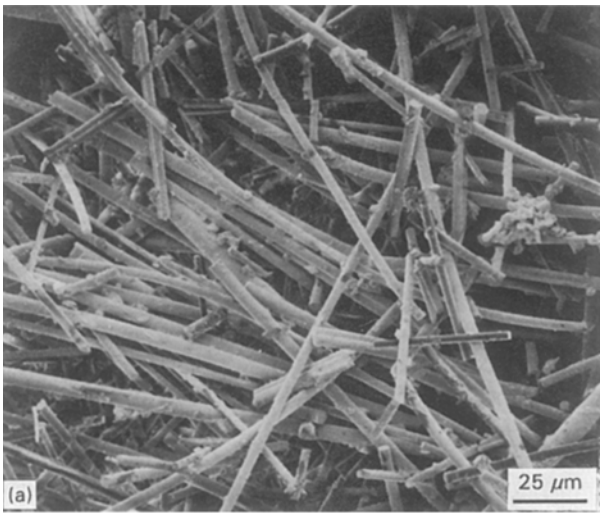


Figure 3 Scanning electron micrographs of preform (a, c) inner and (b, d) outer side, (a, b) conventionally dried, (c, d) microwave dried.



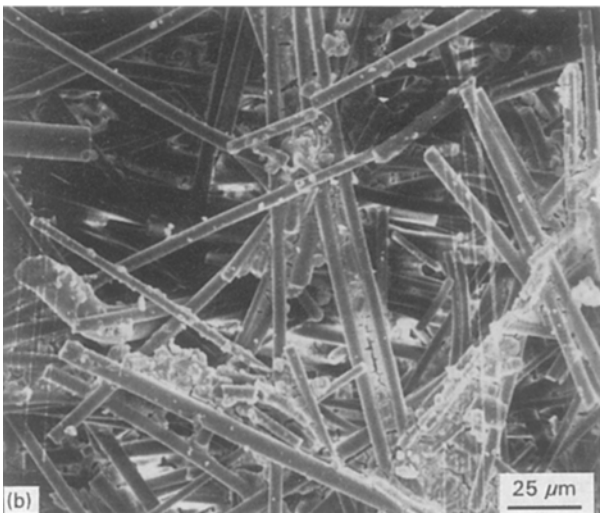


Figure 4 Scanning electron micrographs of a preform dried by microwave for (a) 20 min, (b) 60 min.

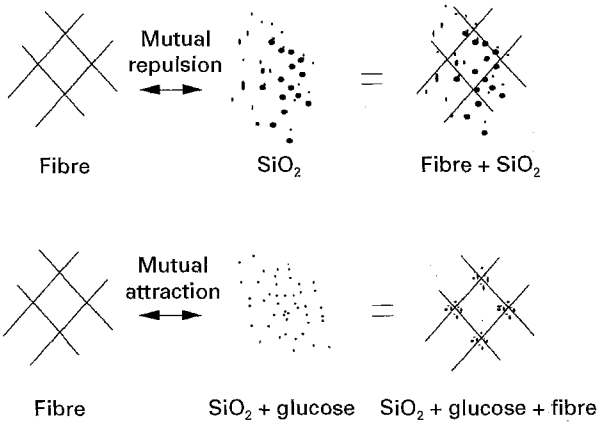
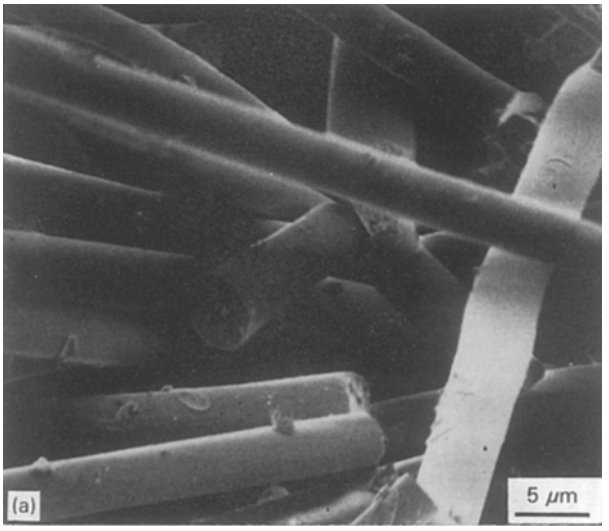


Figure 5 Schematic illustration of the flocking process [15].



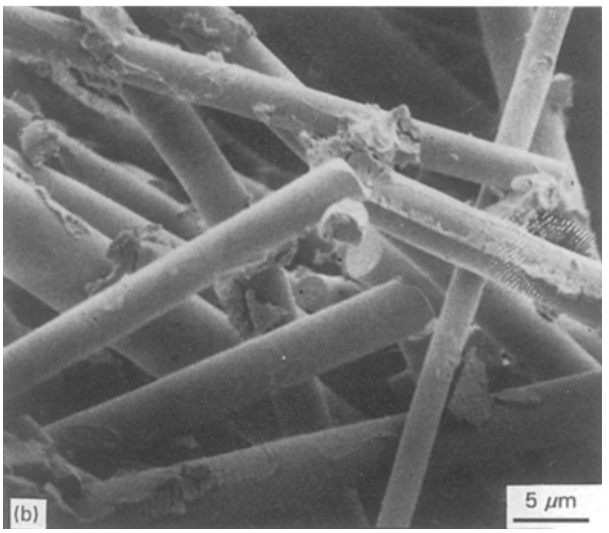


Figure 6 Scanning electron micrographs of the preform: (a) using only inorganic binder, (b) using mixed inorganic binder with organic binder.

TABLE IV Tensile strength Al/Al_2O_3 composities according to the drying method of the preform

	Conventional drying (inorganic binder)	Microwave drying	
		Inorganic binder	Inorganic + organic binder
Tensile strength (MPa)	307	317	290

inorganic binder (a) and a mixture of inorganic and organic binder (b). In Fig. 6b, binder is on the intersecting point of the fibres as mentioned above. These conditions maintain the structure of the preform itself, but act adversely on mechanical properties of MMCs, as shown in Table IV. MMCs made from a microwave-dried preform show slightly increased tensile strength, i.e. 307 MPa to 317 MPa, over conventionally dried ones, and have a lower tensile strength of 290 MPa when an organic binder was used. Therefore, considering mass production, microwave heating has several merits compared with conventional heating, i.e. improvement of machinability due to elevation of bonding strength, uniform distribution of fibres and reduction of drying time. A preform with organic binder is expected to increase machinability of the preform owing to a bridging effect between fibres and binder, but it was not conducive to improvement of tensile strength of MMCs.

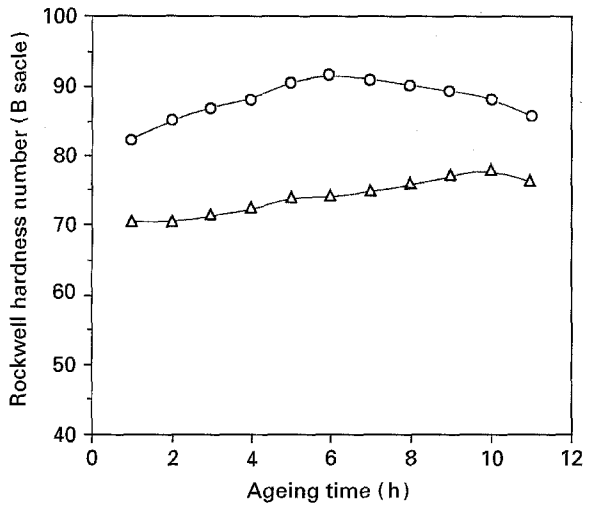


Figure 7 Effect of ageing time on hardness of Al/Al_2O_3 (15%) composite. (△) AC8A, (○) Al/Al_2O_3.

4.2. Processing conditions of squeeze casting

The effect of ageing time on hardness of Al/Al_2O_3–15 vol % composite is illustrated in Fig. 7. Hardness of Al/Al_2O_3 composite is improved up to 20% by the addition of reinforcements compared with that of matrix alloy. In the metal/ceramic composite materials, there is usually a difference in coefficients of thermal expansion between the reinforcement and the matrix alloy [21]. This difference causes strain incoherencies or a high density of dislocations near the interface between the reinforcement and the matrix alloy. Precipitation reactions are accelerated because incoherencies and high densities of dislocations act as heterogeneous nucleation sites for precipitates. Therefore, the ageing rate of MMCs is much faster than that of the unreinforced matrix alloy [17]. In this study, the optimal ageing rate of MMCs was 6 h, which was 4 h shorter than that of the unreinforced matrix alloy.

Fig. 8 summarizes effects of fabrication conditions on the tensile strength of MMCs. Fabrication conditions are applied pressure, melt temperature, preform temperature, and mould temperature. As well as these conditions, plunger speed, which affects infiltration speed, can be selected as one of the fabrication conditions. However, as discussed by Fukunaga [5], infiltration speed is about 1.13 $cm\,s^{-1}$ when the plunger speed is 0.85 $cm\,s^{-1}$ and it is estimated not to cause fibre breakage and degradation at such a magnitude of speed. Thus, the plunger speed was fixed at 0.85 $cm\,s^{-1}$ in this study.

In Fig. 8a, tensile strengths of MMCs according to melt temperature, have almost the same values in the range 750–800 °C, and a decreased value at 850 °C. These results are caused by oxidation of the base metal and thermal damage to fibres at the high temperature. In Equation 3, the superheating temperature of squeeze casting, which is needed to infiltrate melt into the preform, is a function of the melting point of the base metal, initial preform temperature, specific heat of each material, and fibre volume fraction, respectively. On substituting $T_m = 565\,°C$, $T_f = 500\,°C$, $C_m = 0.9$, $C_f = 0.6$ and $V_f = 0.15$ [22, 23] into Equation 3, the superheating temperature is calculated to be about 7.65 °C. However, in the actual squeeze-casting process, there exists a delaying time of about 30 s from the end of preheating to pressure

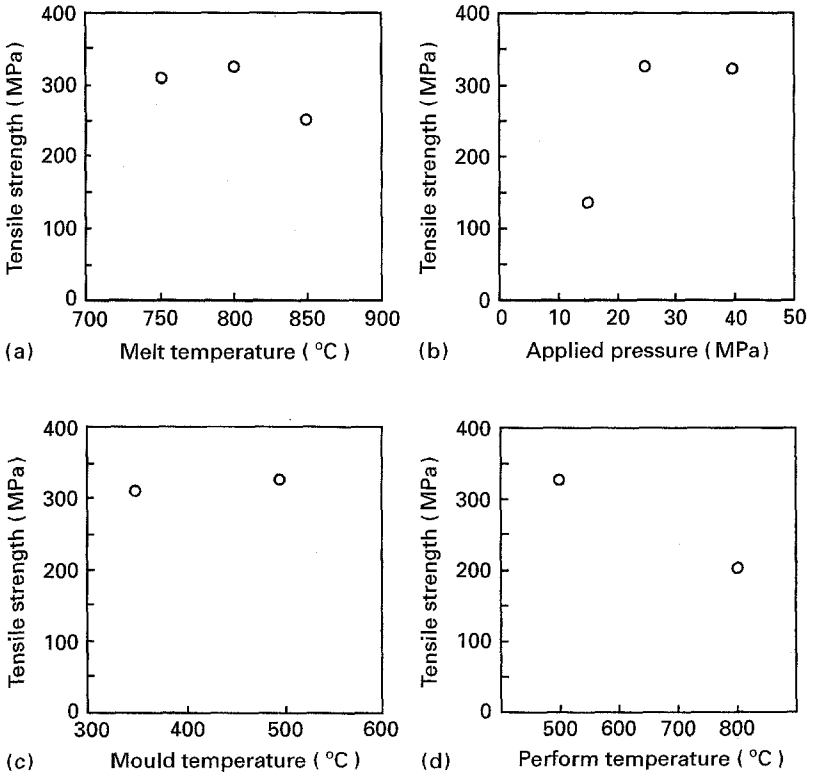


Figure 8 Effect of fabrication conditions on tensile strength: (a) melt temperature, (b) applied pressure, (c) mould temperature, (d) preform temperature. Applied pressure, 25 MPa; mould temperature, 500 °C; preform temperature, 500 °C; melt temperature, 800 °C.

application, thus a higher superheating temperature, which is over the calculated value, is needed. For complete infiltration and to prevent matrix oxidation and fibre damage, the melt should be superheated 150–200 °C from the melting point of the matrix.

In Fig. 8b, tensile strength according to applied pressure is increased at 15–25 MPa and is almost the same at 40–25 MPa. In the low range of applied pressure, tensile strength is increased as mentioned above, because the porosities of MMCs are reduced and the solidification speed is increased according to pressure rise. Although having a fine microstructure, the tensile property of MMCs with too high an applied pressure was not increased significantly due to fibre damage. Consequently, it is desirable that the optimum applied pressure is applied at 25 MPa for production efficiency.

Fig. 8c shows the tensile strength of MMCs at 350 and 500 °C mould temperature. There was no significant effect of mould temperature. In Fig. 8d, MMCs which had 800 °C preform temperature have 60% tensile strength compared with those at 500 °C preform temperature. This is because of preform damage due to thermal shocking caused by removing the MMCs from the furnace and exposing the material to a high processing temperature in air. Thus, it is necessary to determine the minimum preform temperature for preventing thermal shock and solidification of the melt. A 500 °C preform temperature is suitable, because tensile strength is increased up to 20% over the base metal.

5. Conclusions

1. In the drying methods of a preform, microwave heating has merits compared with conventional heating. These are, increase of preform formation due to uniform distribution of binder inside the preform and reduction of drying time. MMCs fabricated with a microwave-dried preform have slightly increased tensile strength over those conventionally dried.

2. Organic binder distributes inorganic binder on the intersecting points of fibres. This fact is desirable for processing and strengthening the preform, but does not greatly effect improvement of the tensile property of MMCs.

3. At high temperature of preform and melt, the tensile strength of MMCs is decreased due to thermal shocking, matrix oxidation, and thermal damage of the fibres. According with increase of applied pressure within a definite range, the tensile strength is increased due to shrinkage of porosities and fast solidification speed. Mould temperature does not closely affect the tensile property of MMCs.

4. The optimal fabrication conditions of AC8A aluminium alloy reinforced with 15 vol % alumina short fibres are 500 °C mould and preform temperature, 25 MPa applied pressure, and 800 °C melt temperature, respectively. The tensile strength of MMCs made under these conditions was increased by up to 20% more than that of unreinforced matrix aluminium alloy.

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