Preparation of cast aluminium – silica particulate composites

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 α -quartz particles (average size 153 µm) were dispersed in the matrix of aluminium castings by mechanically stirring these particles in the melts, followed by casting of the melts in suitable permanent moulds. For obtaining good composite castings with more than 2 wt % SiO₂ dispersions, the silica particles should be preheated to 700° C and added while they are hot (>500° C) together with magnesium pieces (3.5 wt %) to the surface of the melts, kept between 720 to 790° C. Microscopy of castings indicated a reaction zone at the outer periphery of the dispersed SiO₂ particles, and the electron microprobe analysis confirmed that the magnesium added to the surface of the melt concentrates around the periphery of the silica particles. The hardness of AI–2.5 wt % SiO₂ particulate composite was found to be four times that of pure aluminium, and abrasion resistance was found to be three times that of pure aluminium under the conditions of the present investigation.

1. Introduction

In recent years attempts have been made to prepare cast metal-refractory particle composites by conventional foundry techniques [1-12], where the particles are introduced into alloy melts above their liquidus followed by casting the melts in suitable moulds. Graphite, mica and alumina particles have been dispersed in melts above their liquidus temperatures to produce antifriction aluminiumgraphite and aluminium-mica composites [1-9, 12], and anti-abrasion aluminium-alumina composites. [9] However, to date no work has been reported on dispersing uncoated silica particles in aluminium alloy melts above their liquidus temperatures. There has been some work on dispersing glass and silica particles in Al alloys using the compocasting technique requiring special equipment. where the particles are stirred between the liquidus and solidus temperatures [13].

In this paper optimum conditions for dispersion of silica particles (less than $53\,\mu\text{m}$ in size) in alu-

2. Experimental procedure

Materials of the following compositions (in wt %) were used in the present study: commercially pure aluminium; aluminium 99.6%, silicon 0.4%; quartz silica powder SiO₂ 98.00%, Al₂O₃ 0.90%, Fe₂O₃ 0.30%, CaO 0.20% (average particle size $53 \mu m$).

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minium alloy melts, including the pretreatment of the particles, the temperature of the melt, and the need for a reactive metal like magnesium, are described. The distribution of silica particles in static and centrifugal casting, and their effects on the hardness and abrasion resistance have been assessed. Silica particles have a density (2.6 g cm^{-3}) close to that of liquid aluminium (2.37 g cm^{-3}) , therefore they would not be expected to float excessively in the melt under normal conditions. The hard silica particles are likely to impart abrasion resistance to cast aluminium alloys, as has been reported for aluminium–silica composites produced by powder metallurgy techniques [14].

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In most of the experiments about 3 kg of commercially pure aluminium was melted in oilfired furnace or electrical resistance furnace and the melt temperature was increased to between 800 and 920° C. The molten metal was degassed and kept in a holding furnace which was maintained at about 760 to 780° C. The molten metal was stirred by a steel impeller (coated with china clay) rotating at about 450 r.p.m. and silica particles (5 wt %, and heated to a temperature 500° C) were added to the melt surface near the centre of the vortex [4]. In some experiments, magnesium was added either as 1 cm³ size pieces to the surface of the melt along with the silica powder, or it was present in the melt as a previously-added alloying element. After the additions of the silica powder the melt was stirred manually and cast in permanent moulds of diameter 5 cm and length 20 cm. In some cases where the effects of higher melt temperatures up to 1200°C were studied, the melts were made in a 10 kW induction furnace and the silica powder was added to the melt in the induction furnace itself.

The castings were examined (a) visually, after machining a 1 mm thick layer of the metal from the surface (Fig. 1), and (b) microscopically after sectioning longitudinally and transversely, and polishing to study the distribution of particles. Electron microprobe analysis was carried out on selected regions of the composites to find the concentration of different elements in the composites, especially around the particles. The dispersed silica particles were extracted from the composite castings by dissolving the matrix in dilute hydrochloric acid, and then examined by X-ray diffraction to find out whether the particles had undergone any crystallographic transformation. The crystal structure of silica particles, both before their addition to the melts and after their extraction from composite castings, was found to be α -quartz.

2.1. Effect of particle pretreatment on dispersion

Attempts to disperse (a) cold untreated silica particles and (b) cold SiO_2 particles which were heated to 700° C but then cooled to ambient temperature did not succeed, as most of the particles were visibly rejected by the melt (Table I). This could be due to (1) the adsorbed gases or water molecules covering the silica particles or (2) poor



Figure 1 One millimetre deep machine-cut surface of composite casting showing a uniform distribution of SiO_2 particles.

wettability between the particles and molten aluminium at these temperatures.

Up to 2 wt % of preheated but hot SiO_2 particles (500 to 700°C) could be successfully added to the melt with the retention of 1% SiO_2 particles in the melt. When attempts were made to add more than 2 wt % hot SiO_2 particles to the melt (Table I), the particles agglomerated, resulting in flotation and complete rejection from the melt.

2.2. Experiments with magnesium additions

The use of Mg addition was explored to obtain a retention of more than $2 \text{ wt } \% \text{ SiO}_2$ in the castings, since thermodynamically magnesium can reduce SiO₂ and change its surface characteristics in the following manner:

 $SiO_2(s) + 2 Mg(1) = Si(s) + 2 MgO(s).$ (1) In the first set of experiments Al-0.5 to 5 wt% Mg alloys were made and cast beforehand; these were then remelted and hot SiO_2 particles were added to the melt. The retention of silica particles increased from 0.1 to 1.45 wt%, with an increase in Mg content in the melt (Table II) from 0.5 to 4.51 wt%. When the magnesium content was increased above 5 wt%, excessive oxidation of Mg lead to the formation of dross at the surface of the melt, preventing the entry of particles in the molten metal. Flux could not be added to the surface of the melt to prevent oxidation since it interfered with particle additions.

| Trial no. | Pretreatment of particles prior to dispersion | | Weight | Weight |
|--------------|---|---|---|--|
| | Pretreatment of the particles | Temperature of the particles at the time of dispersion in the melt (° C) | percentage of SiO_2 particles added to the melt | percentage of SiO ₂ particles retained in the castings |
| 1 | Untreated | Room temperature | 5 | 0.05 |
| 2 | Heated to 700° C and cooled | Room temperature | 5 | 0.10 |
| 3 | Heated to 700° C | 700 | 5 | 0.70 |
| 4 | Heated to 700° C | 700 | 2.5 | 0.95 |
| 5 | Heated to 700° C | 700 | 1.5 | 1.00 |
| 6 | Heated to 700° C | 700 | 1.0 | 0.90 |
| 7 | Heated to 700° C | 600 | 1.5 | 1.00 |
| 8 | Heated to 700° C | 500 | 1.5 | 1.00 |
| 9 | Heated to 700° C | 400 | 1.5 | 0.50 |
| 10 | Heated to 700° C | 600 along with 3.5 wt % Mg added to the sur- face of the melt | | |

TABLE I Effect of pretreatment of SiO_2 particles on their retention in aluminium castings

TABLE II Effect of magnesium additions on recovery of SiO_2 particles in the castings when 5 wt% hot (500 to 600° C) SiO_2 particles were added to the melt. Magnesium present in the melt was as previously added alloying element

| Trial no. | Melt composition | Weight percentage SiO_2 retained in the castings |
|--------------|------------------|--|
| 1 | Pure aluminium | 0.05 |
| 2 | A1-0.5 wt % Mg | 0.10 |
| 3 | Al-1.45 wt % Mg | 0.62 |
| 4 | Al2.80 wt % Mg | 0.88 |
| 5 | Al-3.65 wt % Mg | 1.32 |
| 6 | Al-4.31 wt % Mg | 1.45 |
| 7 | Al-5.11 wt % Mg | 0.75 |

In the next set of experiments Mg pieces (about 1 cm^3 in size) were added to the surface of Al melts along with the hot silica powder to increase further the recovery of silica particles. Table III shows that retention of particles increased with increasing amounts of fresh Mg added to the melt surface, and a maximum of 2.85 wt% SiO₂ could be retained in the castings with 3.48 wt% Mg addition. Additions of more than 3.48 wt% Mg to the surface of the melt resulted in excessive oxidation of Mg.

The X-ray line scan (Fig. 2) and the electron image of the particles (Fig. 3) show preferential segregation of Mg in the outer periphery of SiO_2 particles dispersed in the castings. The surface of the SiO_2 particles appeared to have undergone a partial reduction resulting in improved wettability between the particle and the melt. Metallographic examination also suggested a reaction layer at the interface of particle and the matrix (Fig. 4).

2.3. Effect of melt temperature

When the melt temperature (Table IV) was below 720° C there was tendency for agglomeration of silica particles. When it was between 720 and 800° C, good dispersions of particles were obtained, while above 800° C the magnesium at the surface of the melt started to burn and a layer of reaction product was formed at the interface around the silica particles. The particles showed a tendency to agglomerate in the castings.

Limited experiments were done to disperse hot SiO_2 particles above 1000° C in an induction furnace while 3.5% Mg was added to the melt surface. While adding the Mg, hot spots and hard black scum were observed to form at the surface of the melt. The scum was found to be a complex

TABLE III Effect of magnesium additions on retention of SiO₂ particles in the castings when 5 wt % hot SiO₂ (500-600° C) particles were added to the melt. Magnesium was added in the form of 1 cm^3 pieces to the surface of the pure aluminium along with SiO₂ particles

| | · · · · · · · · · · · · · · · · · · · | |
|--------------|---------------------------------------|---|
| Trial no. | Magnesium added (wt %) | SiO ₂ retained in the castings (wt%) |
| 1 | 0.53 | 0.83 |
| 2 | 1.23 | 1.68 |
| 3 | 1.94 | 1.75 |
| 4 | 2.65 | 2.50 |
| 5 | 3.48 | 2.85 |
| 6 | 4.30 | 2.45 |
| 7 | 5.10 | 2.10 |
| | | |

alumino-silicate slag containing few entrapped SiO_2 particles. Microscopic examination of the cast composite revealed cuboids of silicon (similar to Si cuboids in hypereutectic Al–Si alloys) dispersed in an Al matrix. These were unexpected, since despite some increase in Si levels of the composite during these experiments, the final Si levels were far below the hypereutectic level where the cuboids of Si should normally be observed. Apparently the Si pick-up is due to the following reaction at the particle-melt interface:

 $3 \operatorname{SiO}_2(s) + 4 \operatorname{Al}(l) = 3 \operatorname{Si}(s) + 2 \operatorname{Al}_2 \operatorname{O}_3(s).$ (2)

This Si, which should normally dissolve in the melt



Figure 2 X-ray line scan of the particle showing the concentration of Si, Al and Mg at the interface, in the particles.



Figure 3 Electron image of a $50 \,\mu$ m SiO₂ particle showing: (a) particle; (b) electron image of aluminium; (c) electron image of silicon; (d) electron image of magnesium.



Figure 4 Typical cross-section of Al-2.65 wt % SiO₂ dispersed casting showing silica particles. The SiO₂ particles show a dark etching layer at the outer periphery (\times 360).

and reprecipitate as eutectic Si, remains in the form of cuboids, probably due to the coating of Al_2O_3 on the surface. The reduction of SiO_2 by molten Al, resulting in the formation of Al_2O_3 has also been reported when a SiO_2 rod was dipped in molten Al [15].

2.4. Movement of silica particles in the melts

In the melting experiments the silica particles (density 2.6 g cm^{-3}) did not enter and sink through even through they are heavier than the melt (density 2.34 g cm^{-3}), possibly due to the oxide layer which acts as a barrier and the convection currents in the melt. It was necessary to give proper stirring to break the oxide layer and give

| Trial no. | Melt temperature (°C) | Experimental observations | SiO ₂ retained in the castings (wt %) |
|--------------|-----------------------------|---|---|
| 1 | 690 | Particles agglomerated in the melt, uneven distribution in the castings | 0.10 |
| 2 | 700 | particles agglomerated in the melt | 0.10 |
| 3 | 720 | Good distribution | 2.52 |
| 4 | 760 | Good distribution | 2.85 |
| 5 | 780 | Good distribution | 2.65 |
| 6 . | 800 | Agglomerated and float-out | 1.85 |
| 7 | 850 | Excessive evaporation of magnesium and float-out | 1.85 |
| 8 | 900 | Excessive evaporation of magnesium and float-out | 0.1 |

TABLE IV Effect of melt temperature on retention of SiO_2 particles in the castings when 5 wt % hot SiO_2 particles were added to the surface of the melts along with 3.5 wt % Mg pieces

enough kinetic energy to facilitate the transfer of the SiO_2 particles into the melt.

Likewise the distribution of the silica particle in the castings indicates that under the present experimental conditions the silica particles do not sink through the melt in accordance with the predictions of Stoke's law. For instance the observed solidification time of the chill mould casting (5 cm diameter; 20 cm height) is about 70 sec. During this time the 53 μ m silica particle would be expected to sink about 5 cm, and the top portion of the castings should be free from silica. However, the castings (Fig. 1) show a uniform distribution of silica particles without a depleted zone near the top; if anything there is a very small zone at the bottom free from silica particles indicating flotation of these particles.

In fact during centrifugal casting of Al-3.2 wt% Mg-2.5 wt% SiO₂ melts (at 720° C), when they were poured into the rotating moulds (800 r.p.m.), the silica particles congregated at the inner periphery of the cast sleeves (Fig. 5). Under similar conditions [16] the graphite particles (which are much lighter than the melt) also congregated at the inner periphery of cylindrical castings, suggesting that the silica particles behave as if they are lighter than the melt. This may be due to adsorbed gases on the surface of the particles and lack of wettability, or porosity in the particles.

2.5. Macroscopic and microscopic examination of castings

Macroscopically the SiO_2 particles were uniformly distributed in the castings (Fig. 1). Microscopic 2282

examination indicated a fairly uniform distribution of silica particles (Fig. 4). However, there were locations in which agglomeration of SiO_2 particles was observed, especially when the melt temperatures were high.

2.6. Mechanical property studies

The tensile strength of cast Al–2.65 wt % SiO₂ particulate composite was found to be about 11.2 kg mm⁻², whereas pure aluminium cast under identical conditions gave a strength of 11.0 kg mm⁻². The hardness of the composite was found to be around 80 BHN, whereas the hardness of the matrix alloy was found to be around 20 BHN when cast under identical conditions.



Figure 5 Cross-section of a centrifugally cast SiO_2 dispersed composite showing concentration of SiO_2 particles towards the inner periphery of the castings.

The abrasion resistances of Al-2.65 wt % SiO₂ composite and the matrix alloy without SiO₂ particles were measured by abrading samples over a rotating steel disc covered with 600 grit silicon carbide emery paper at a pressure of about 0.1 kg mm^{-2} . The disc was rotated at 1440 r.p.m. for 10 min while a controlled quantity of water was supplied continuously. After the test, the samples were washed, dried and their weight loss was computed. The percentage weight loss for Al-2.65 wt % SiO₂ composite was found to be three times less than the weight loss with the pure aluminium alloy. Visual examination showed that in the case of Al-2.65 wt % SiO₂ composites, the aluminium matrix had deformed and had a tendency to cover the SiO₂ particles.

3. Conclusions

(1) SiO₂ particles (α -quartz, average size 53 μ m) can be dispersed in the melts of aluminium, which can be subsequently cast to get aluminium-silica particulate composites.

(2) To get good dispersion of over 2 wt \% SiO_2 particles in the composites, the particles should be added when they are hot (at a temperature greater than 500° C) and the temperature of the aluminum melt is around 720 to 790° C, and pieces of magnesium (3.5 wt%) added simultaneously to the surface of the melt.

(3) Electron microprobe analysis showed that the magnesium added to the melts concentrates preferentially around the surface of silica particles, possibly improving the wettability.

(4) The hardness of Al-2.5 wt % SiO₂ particu-

late composites is four times that of pure aluminium. The percentage weight loss of $Al-2.65 \text{ wt \% SiO}_2$ particulate composites under abrasion is three times less than that of aluminium without the dispersions under similar conditions.

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