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Preparation of cast aluminium alloy-mica particle composites

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The optimum conditions for producing cast aluminium alloy–mica particle composites, by stirring mica particles (40 to 120 μm) in molten aluminium alloys (above their liquidus temperatures), followed by casting in permanent moulds, are described. Addition of magnesium either as pieces along with mica particles on the surface of the melts or as a previously added alloying element was found to be necessary to disperse appreciable quantities (1.5 to 2 wt.%) of mica particles in the melts and retain them as uniform dispersions in castings under the conditions of present investigation. These castings can be remelted and degassed with nitrogen at least once with the retention of about 80% mica particles in the castings. Electron probe micro-analysis of these cast composites showed that magnesium added to the surface of the melt along with mica has a tendency to segregate around the mica particles, apparently improving the dispersability for mica particles in liquid aluminium alloys.

The mechanical properties of the aluminium alloy–mica particle composite decrease with an increase in mica content, however, even at 2.2% the composite has a tensile strength of 14.22 kg mm^{-2} with 1.1% elongation, a compression strength of 42.61 kg mm^{-2}, and an impact strength of 0.30 kgm cm^{-2}. The properties are adequate for certain bearing applications, and the aluminium–mica composite bearings were found to run under boundary lubrication, semi-dry and dry friction conditions whereas the matrix alloy (without mica) bearings seized or showed stick slip under the same conditions.

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1. Introduction

A self-lubricating composite with antifriction properties suitable for use under severe conditions can be produced by dispersing particles of solid lubricants in a metallic matrix. Under extreme conditions of temperatures (including cryogenic) and pressures, liquid lubricants fail to work and the need for solid lubricants becomes imperative due to their stability at these temperatures and pressures and under severe chemical environments. In addition, the solid lubricants do not require oil seals. In several conditions these advantages of solid lubricants outweigh some of their limitations like high coefficient of friction and poor heat dissipation. Mica has a layered structure which makes it a good solid lubricant. It has also been successfully used [1–4] as a solid lubricant dispersed in the matrices of copper, silver and nickel. To date, metal–mica composites having good antifriction properties have generally been made by the powder metallurgy techniques, wherein metal and mica powders were mixed together, pressed and sintered. Powder metallurgy is generally expensive and permits only the

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manufacture of parts of limited sizes and shapes. However, there is a report which describes an alternative method to powder metallurgy, namely, the compocasting technique which Sato and Mehrabian [5] have used to make aluminium alloy–mica particle composites. The compocasting technique involves stirring of mica particles in aluminium alloys while the temperature of the latter was maintained between the liquidus and solidus temperatures. The compocasting technique, however, requires specialized equipment.

In this paper we have described a relatively simple and conventional foundry technique for making aluminium–mica particle composites wherein the mica particles are stirred in molten aluminium alloys above their liquidus temperatures, followed by casting these composite melts in suitable permanent moulds. Aluminium–graphite particle composites have already been made by other workers using a similar technique [6–11]. However, no work is reported on the production of aluminium–mica composites using this technique.

Mica was explored as a dispersoid alternate to graphite since it has almost the same density as liquid aluminium alloys and, therefore, is less likely to float up in the molten alloys as compared to graphite. In addition, mica has a better resistance to oxidation [12] than graphite, and is abundantly available in India.

2. Experimental procedure

2.1. Materials

2.1.1. Alloy

Aluminium–copper alloy of the following chemical composition was used as the base alloy to make the composites: Cu 3.95 to 4.50%, Si 0.31 to 0.35%, Mg 0.42 to 0.45%, Fe 0.10 to 0.15%, and balance aluminium.

2.1.2. Mica powder

Ground mica powder was obtained from Mysore Micanite Industries, Bangalore which was produced by heating muscovite mica sheets to 150° C and grinding them in water. Mica powder was produced by milling muscovite mica sheets put together in a vice. The cleavage planes of mica sheets were kept vertical and the cutting edge of milling cutter was perpendicular to the edges of the mica cleavage planes. 90% of the powder produced by milling was found to be $< 70\,\mu$m in size.

2.2. Experimental apparatus and procedure used for making the composite

The experimental apparatus consisted of an electrical resistance holding furnace and a stirrer assembly as shown in Fig. 1. About 3 kg alloy was melted in a super salamander crucible using an oil-fired furnace. The metal was superheated to about 800° C and then degassed with nitrogen bubbled through concentrated sulphuric acid and anhydrous calcium chloride at a rate of 10 litre min^{-1} for 2 min. Degassing was carried out while the crucible containing the molten metal was kept in an electrical resistance holding furnace as shown in Fig. 1. The temperature of the holding furnace was kept at about 700° C.

A mechanical stirrer was then inserted into the molten metal and the speed of its rotation was adjusted to 1000 rpm with a variable voltage transformer to create the necessary vortex. The axis of the stirrer blade was at an angle 15° from the vertical. The diameter of the stirrer blade was 75 mm. Mica powder, preheated at 200° C for 12 h and then at 700° C for 30 min was added into the vortex at an approximate rate of 30 g min^{-1}. Magnesium pieces (about 6 mm cube size) were added to the surface of the melt along with the mica powder. Melt was stirred for at least $\frac{1}{2}$ min after the addition of powder was completed, and then degassed by bubbling nitrogen at a rate slower (about 2 to 3 litre min^{-1}) than that used for degassing before dispersion. The degassing was accompanied by further stirring at about 500 rpm for 1 min. The stirrer was then stopped and withdrawn. The molten metal was taken out of the holding furnace and hand stirred with graphite rod and poured into permanent moulds. The time interval between stirring and pouring should be minimum to avoid the floating of mica particles and also to ensure uniform dispersion in the melt and in the subsequent castings.

2.3. Remelting and degassing of aluminium alloys containing dispersed mica particles

About 2.5 kg mica-dispersed aluminium alloy castings were melted in an electrical resistance furnace. The temperature of the melt was kept between 700 and 750° C. The melt was simultaneously stirred and degassed for 1 min with nitrogen, in a holding furnace maintained at about 700° C. The metal was then taken out of the furnace. stirred and graphite rod and poured

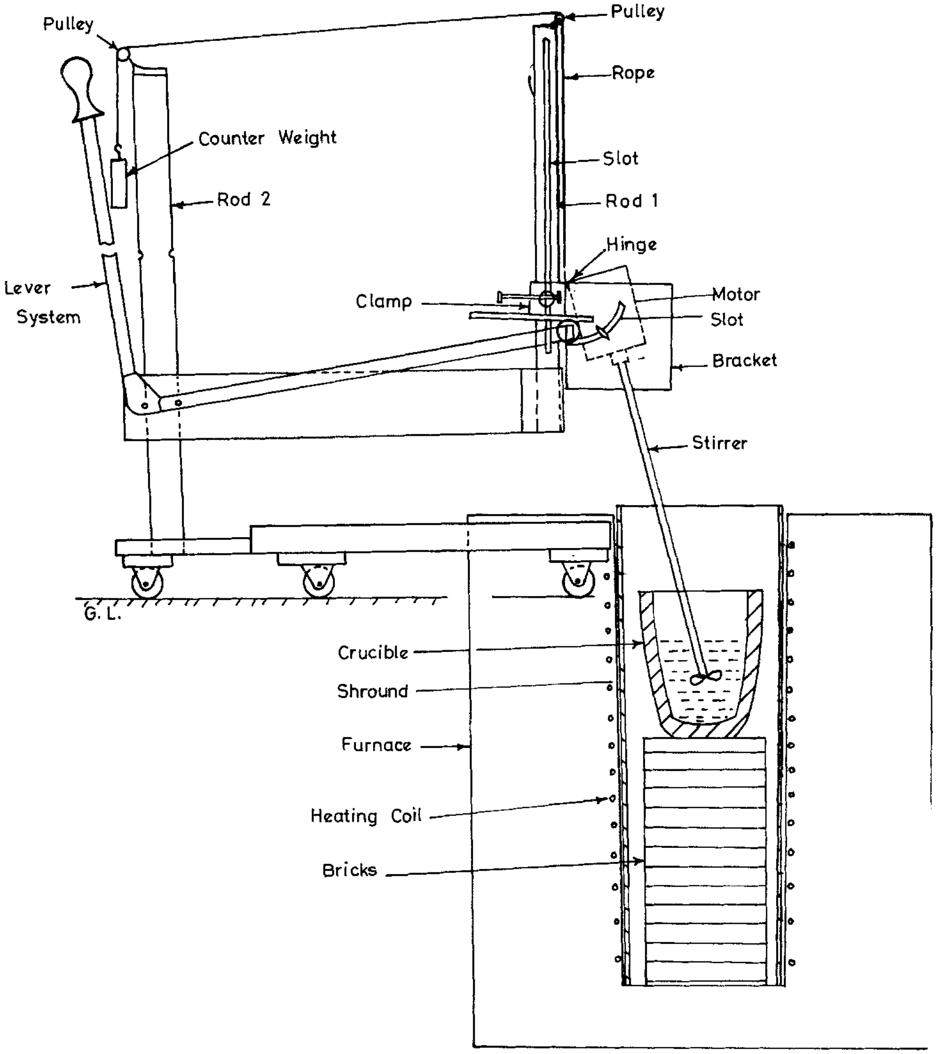

Figure 1 Schematic diagram of experimental apparatus used for dispersion of mica particles in aluminium alloy melts.

into permanent moulds. The above procedure was repeated three times. The castings after each remelt were analysed for their mica contents.

2.4. Microscopic and X-ray diffraction studies of as-received mica powder and the mica powder extracted from composites

Both the as-received mica powder, and the mica powder, extracted from composites (by dissolving the metallic matrix in acids) were studied using X-ray diffraction techniques and a mineralogical microscope to detect any structural changes that might have taken place during the process of making the composites.

3. Results and discussion

3.1. Dispersion of mica particles in liquid aluminium alloys

Mica particles were successfully dispersed in aluminium and Al–4% Cu–1.5% Mg alloy melts by the vortex method using simultaneous additions of magnesium cubes (about 6 mm) and mica particles into the melt. Using this technique, it was possible to disperse up to about 3 wt % mica particles in liquid aluminium alloys without much difficulty. When the mica powder added to the melt exceeded 3 wt %, the mica particles were rejected by the melt.

The mica particles were found to be uniformly dispersed in the matrix on a macroscopic scale, in suitable permanent mould castings of these com-

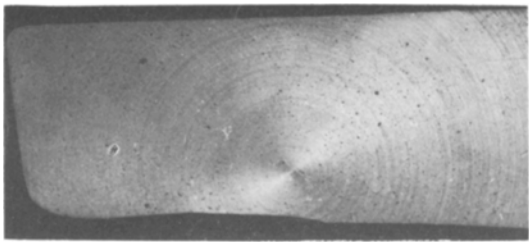

Figure 2 Macrophotograph of bottom portion of 12.5 mm × 125 mm × 150 mm, plate casting of Al–4% Cu–1.5% Mg–1.5% mica (40 μm), × 0.455.

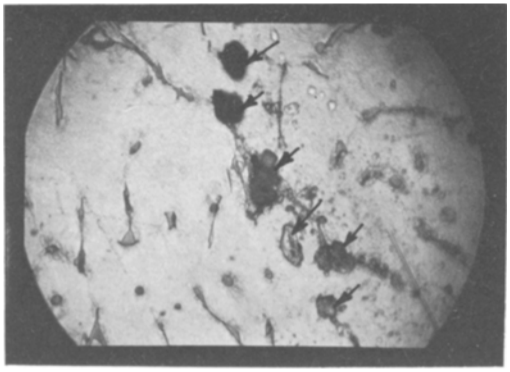

Figure 3 Microphotograph of Al–4% Cu–1.5% Mg–2% mica alloy, etchant 0.5% HF, × 210 (mica particles pointed by arrows).

posite alloys. Fig. 2 shows a uniform dispersion of 1.5% mica particles near the surface of the bottom portion of a 12.5 mm thick × 125 mm × 150 mm plate casting of Al–4% Cu–1.5% Mg alloy. A similar distribution of mica particles was also observed in the top portion of the castings. The black patches in Fig. 2 are due to the presence of mica particles. The patches are sometimes quite large owing to agglomeration of mica particles and to staining effects due to particles.

In the present investigation it was found that the dispersion of mica particles was easier, and their distribution in the casting was also better, when the melt size was larger.

A typical microstructure of the mica-dispersed aluminium alloy is shown in Fig. 3. This shows six mica particles in the matrix of Al–4% Cu–1.5% Mg alloy. The mica particles appear to be preferentially located near the second-phase particles (presumably $CuAl_2$ or $CuMgAl_2$) precipitated from the last freezing intercellular liquid as a result of segregation during solidification.

Mineralogical microscopic studies of the as-received mica powder and the powder extracted from the cast composite, showed that there is no measurable change in the characteristics of mica during the process of making the composite. The refractive index and the inclined extinction angle remained at less than 1.8534 and 22°, respectively.

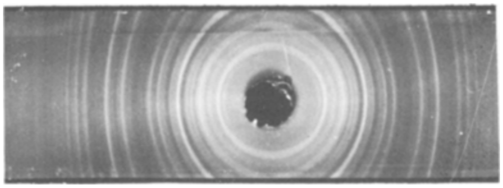

Figure 4 X-ray pattern of as-received mica powder.

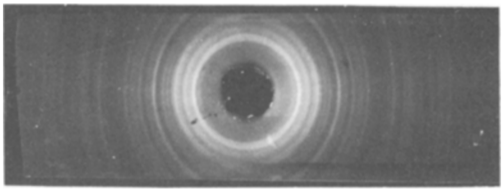

Figure 5 X-ray pattern of mica powder extracted from composite.

X-ray diffraction patterns of as-received mica powder and of the mica powder extracted from the composites are shown in Figs. 4 and 5, respectively. A comparison reveals that there is no significant change in the structure of mica during the process of making the composites.

3.2. Remelting of aluminium alloy mica particle composite

Fig. 6 shows the effect of remelting of cast composites on the recovery of mica particles in the casting. During remelting of 2.5 kg heats of mica-dispersed aluminium alloys, the mica particles come to the surface of the melt, but they can be stirred back and easily reintroduced in the melt, and the composite alloy can be again cast in permanent moulds with a slight decrease in mica content each time (Fig. 6). These alloys can be remelted at least once with the retention of about 80% mica particles in the casting. After remelting the mica-dispersed composite alloy three times, the recovery of mica comes down to even 50 or 60% of the mica content initially present in the casting. However, while remelting, the temperature of the melt should not exceed 800° C, otherwise almost all the mica particles are rejected by the melt in such a manner that they cannot be stirred back into the melt. The rejection of mica particles

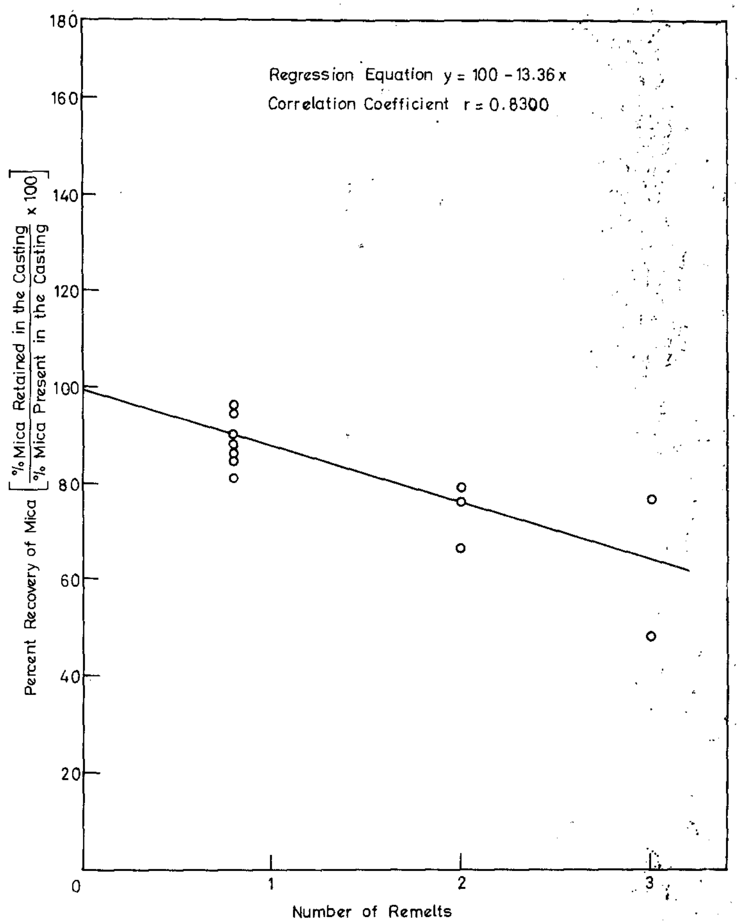

Figure 6 Effect of remelting on the recovery of mica in the composite castings.

at temperatures higher than 780° C may be due to dehydroxylation of mica at these temperatures, although no direct evidence for this has been collected. On the basis of DTA studies on muscovite mica, it is concluded that above 800° C an endothermic transformation associated [17] with dehydroxylation takes place in this mica. The DTA study of mica used in the present investigation also showed an endothermic change upon heating around 780 to 800° C.

3.3. Degassing

Degassing of aluminium alloy melts with nitrogen was carried out before and after dispersion of mica particles. In the initial experiments, when degassing was done only before dispersion, the castings showed a large degree of porosity. Degassing of the quiescent melts after dispersion was difficult, since during degassing mica particles had a tendency to float up and become rejected by the melt. However, slow bubbling of nitrogen through the melt was possible if the melt was stirred slowly during degassing. Limited degassing with chlorine-bearing hexachloroethane tablets can be done before dispersion without any deleterious effect. However, degassing with hexachloroethane after dispersion led to complete rejection of mica particles by the melt.

3.4. Factors affecting the recovery and distribution of mica particles in aluminium alloy castings

The following factors affect the recovery and distribution of mica particles in the castings.

3.4.1. Type of mica powder

In this work attempts were made to disperse two different types of mica powders, i.e. the ground mica and the cut mica. It was found difficult to disperse ground mica particles in aluminium alloy melts by the method described in this paper. The recoveries of ground mica particles in the castings were also very poor as shown in Fig. 7. The same figure shows that the recoveries of cut mica powders were several times the recoveries of ground mica powders under similar experimental

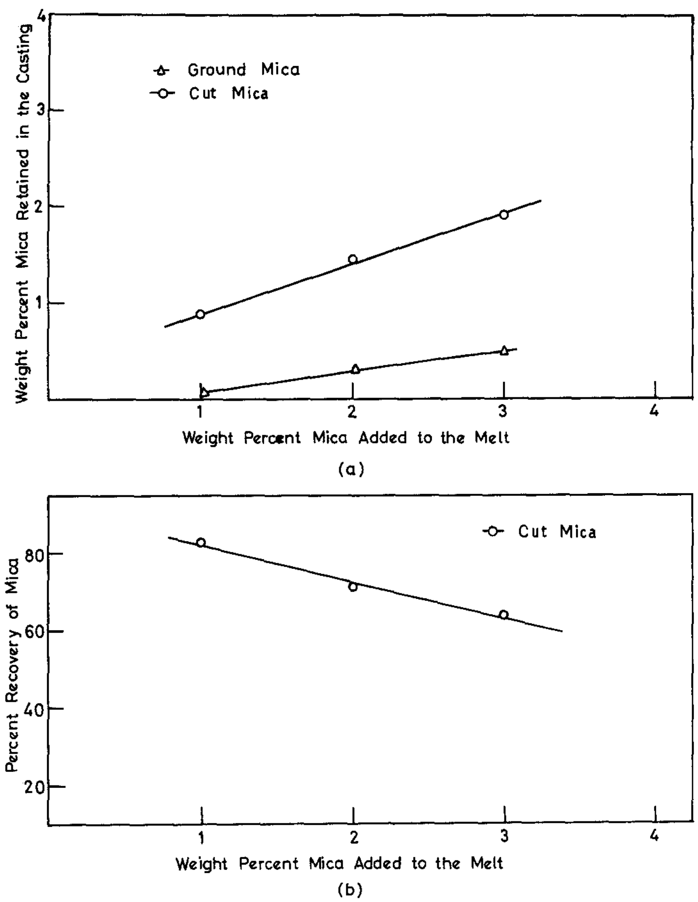

Figure 7 Effect of increasing mica content on the recovery of mica in aluminium alloy castings while magnesium added to the melt was kept constant at 1.5%. (a) wt % mica retained in the casting. (b) % recovery of mica.

conditions. It might have been possible to disperse cut mica powder more easily than the ground mica powder owing to the higher ratio of surface area of the non-basal plane to basal planes in cut mica particles than in the ground mica particles. In addition, the more granular geometry of cut mica particles may have played a helpful role in their dispersion in the melts.

3.4.2. Addition of magnesium

In the present investigation, magnesium pieces (about 6 mm cubes) were added to the surface of the melts along with the mica particles. The effect of increase in the amount of mica addition to the melt on the recovery of mica in the castings at 1.5% magnesium level is shown in Fig. 7a. It can be seen that at this magnesium level, the net mica content of the casting increases with an increase in the mica added to the melt but the percentage recovery of the mica powder decreases (Fig. 7b). Effects of increasing magnesium additions on the recovery of mica particles in the castings is shown in Fig. 8, when mica added in each melt was 3%. Fig. 8 shows that for the same amount of mica added to the melt, the recovery of mica in the casting increases linearly with an increase in magnesium content within the range investigated (i.e. up to 4% magnesium). Above 4% magnesium there was excessive vapourization of magnesium.

Results of the experiments on the dispersion of mica particles in aluminium alloys where 2% magnesium was present as a previously added alloying element (instead of being freshly added to the surface of the melt along with mica), are shown in Fig. 9. The general trend in the recoveries of mica were similar to the trends observed when magnesium pieces were added to the surface of the melt along with mica, as described above. However, for a given amount of magnesium, the recoveries of mica were comparable or at best slightly higher when fresh magnesium was added along with mica to the surface of the melt, as compared to the recoveries when the same amount of magnesium was present in the melt as previously added alloying element. While all the results in this paper (except Fig. 9) correspond to simultaneous additions of fresh magnesium to the surface of the melt, it should nonetheless be

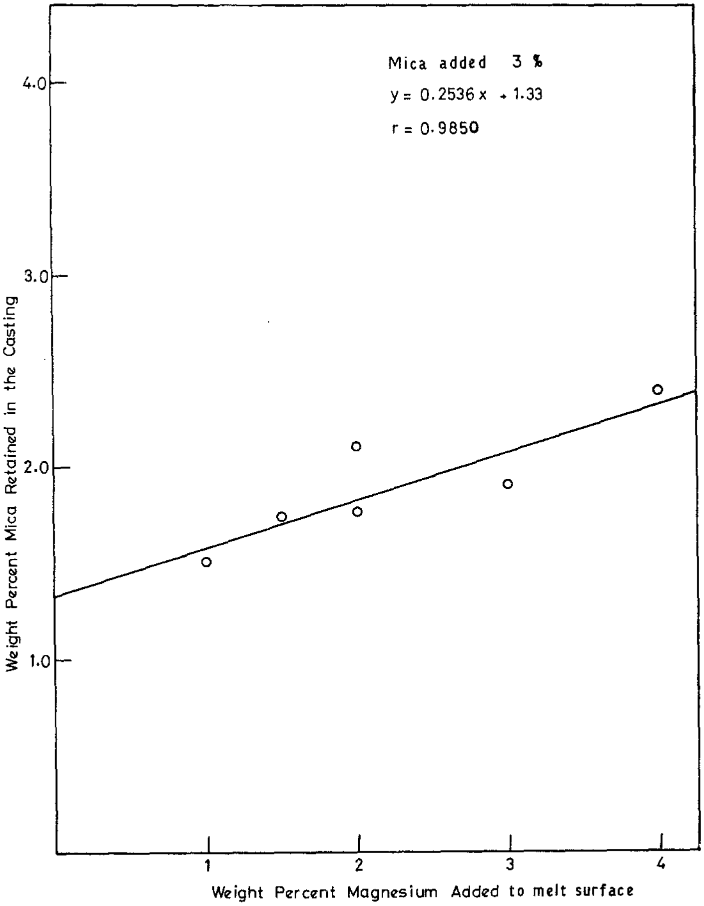

Figure 8 Effect of increasing magnesium content on the recovery of mica in aluminium alloy castings while mica particles added to the melt were 3% in all cases.

noted that composites containing up to 1.80% mica can be made even when magnesium is present as a previously added alloying element.

3.4.3. Temperature of pouring

The distribution of mica particles in mica-dispersed aluminium alloy castings was found to be very much dependent on the pouring temperature of the melt. Optimum pouring temperature was found to be in the range 690 to 710° C. When the pouring temperature was less than 690° C, pouring was difficult due to insufficient fluidity [13]. Pouring temperatures greater than 710° C resulted in excessive segregation of mica particles to the top portions of the castings.

3.5. Role of magnesium in aiding the dispersion of mica particles

Most of the ceramic particles are not wetted [14–15] by liquid metals, unless some reaction takes place at the interface. It is reported [16] in the literature that the reactive metals added to the metal melts (while dispersing ceramic particles in liquid metal) are preferentially adsorbed on the surface of the particles. This apparently improves the wetting of the particles and thereby improves their dispersability in the melt. Magnesium has been found to give the most satisfactory results in this respect [9]. In the present investigation, it was found that when mica was dispersed without additions of magnesium, the particles began to be rejected by the melt. On the other hand, if magnesium was added to the surface of the melt while the mica particles were being rejected, the rejection was prevented, and the rejected mica particles could be stirred back and again dispersed in the liquid alloys.

Electron probe micro-analysis of three separate regions from a mica-dispersed aluminium alloy casting indicates segregation of magnesium around mica particles (Fig. 10a to c). In these figures, the simultaneous electron probe microanalysis, using a double pen recorder, is given for potassium and magnesium. The morphology of the particles across which the analysis was done is shown in each chart run. In all three chart runs, the higher potassium concentrations corresponded to the presence of mica particles. In addition, there was

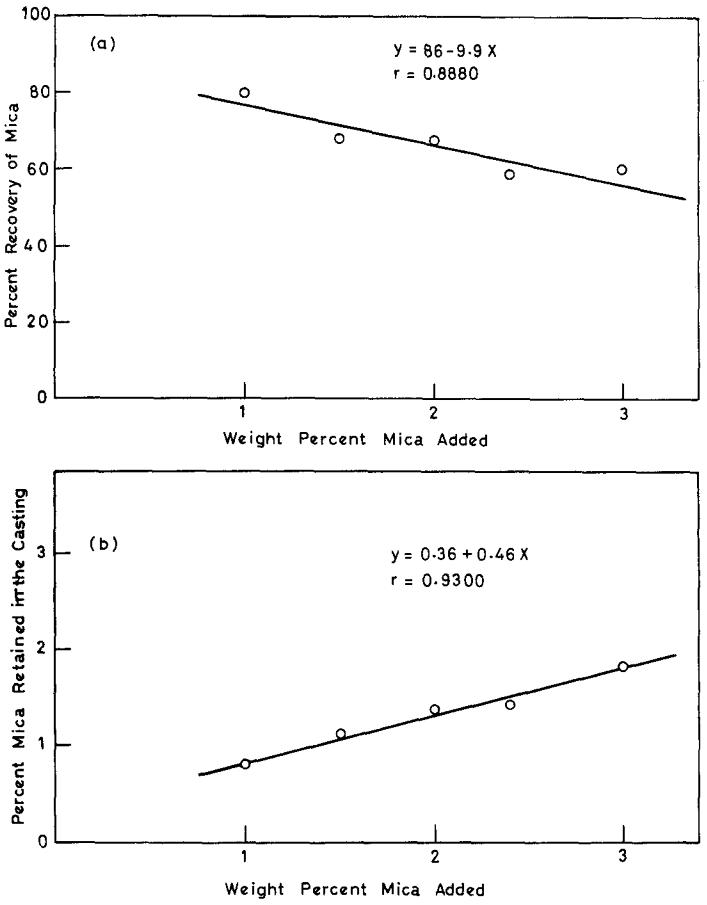

Figure 9 Recovery of mica in composite casting as a function of wt % mica added to the melt when 2% magnesium is present as previously added alloying element in the melt. (a) % recovery of mica. (b) wt % mica retained in the casting.

an increase in the magnesium concentration at the metal–mica interface, and in some cases across the entire mica particles. This suggests that magnesium added to the melt preferentially segregated around the mica particles. The magnesium peaks are generally seen near the metal–mica interfaces. In Fig. 10b where the EPMA was performed along a relatively rare mica–mica boundary, a continuous magnesium build up was observed. Fig. 10c shows that there is magnesium segregation all across the mica particles, but the segregation is more intense at the metal–mica interface.

These preliminary microprobe results suggest that magnesium is either preferentially adsorbed on to the mica particles or it reacted with the mica particles, thereby facilitating the dispersion of mica particles in aluminium alloy melts.

3.6. Mechanical properties of cast aluminium alloy mica particle composite

Cast aluminium alloy mica particle composite were tested for various mechanical properties (tensile strength, compression strength, hardness and impact strength) to characterize this material for bearing applications. All these mechanical properties of the composite were found to decrease with increasing weight or volume percent of mica particles. The percentage decrease in mechanical properties were much more than the volume percent of mica added. The regression equations for tensile strength, percent elongation, compression strength and proof strength under compression were found to be $y_{(\mathrm{TS})} = 31.85 - 7.8x$ (correlation coefficient $\gamma = 0.8917$), $y_{(\%\mathrm{El})} = 2.28 - 0.5x$ ($\gamma = 0.9944$); $y_{(\mathrm{CS})} = 64.20 - 10.64x$ ($\gamma = 0.8913$) and $y_{(\mathrm{PS})} = 28.83 - 3.72x$ ($\gamma = 0.9016$) respectively. In the above equations, y is the mechanical properties and x the percent mica. Tensile strength, percent elongation, compression strength, proof strength under compression, hardness and impact strength of the matrix alloy without mica were found to be 32.83 kg mm^{-2}, 2.16%, 67.80 kg mm^{-2}, 27.50 kg mm^{-2}, 130 BHN, 0.60 kgm cm^{-2}, respectively, and the above properties of the composite alloy

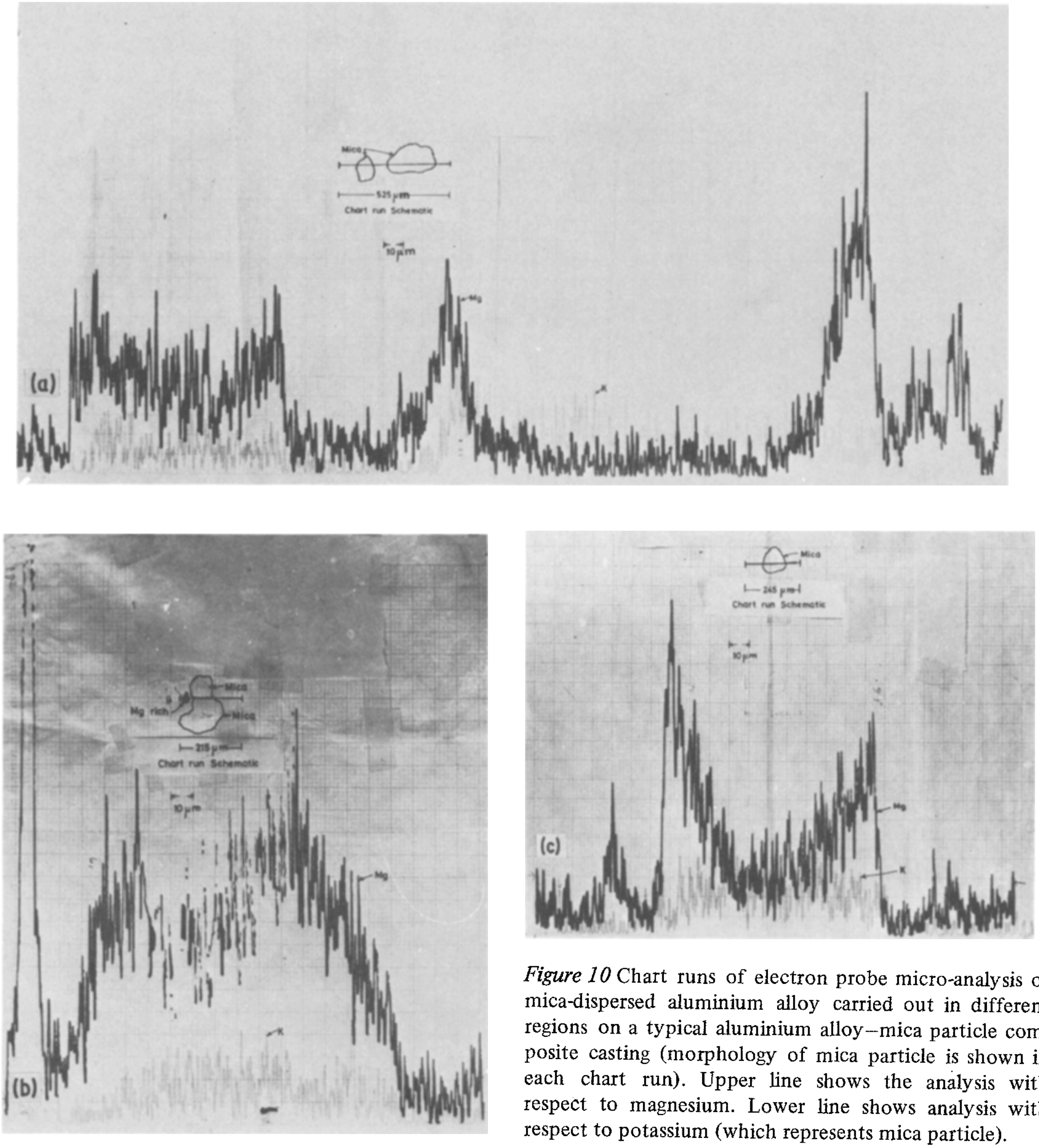

Figure 10 Chart runs of electron probe micro-analysis of mica-dispersed aluminium alloy carried out in different regions on a typical aluminium alloy–mica particle composite casting (morphology of mica particle is shown in each chart run). Upper line shows the analysis with respect to magnesium. Lower line shows analysis with respect to potassium (which represents mica particle).

with 2.2% mica were 14.22 kg cm^{-2}, 1.1%, 42.61 kg mm^{-2}, 19.69 kg mm^{-2}, 60 BHN, 0.30 kgm cm^{-2}, respectively. These properties of the composite alloy were adequate for several bearing applications as will be shown in the next section. It was also observed that the plastic deformation of the composite under compression was larger than the same under tension. Scanning electron microscopic studies of the fractured tensile specimen has indicated that the void formation at the particle matrix interface was the probable mechanism of fracture of the present composite material.

3.7. Wear resistance and bearing performance of cast aluminium alloy mica particle composite

The adhesive wear test results indicated that additions of up to 2% mica increase the wear rate of the matrix alloy at normal pressures above 2.11×10^{-2} kg mm^{-2}. The temperature rise of the matrix alloy during wear test was more than the temperature rise of the mica dispersed alloy, and this difference was increased with time when lubrication was stopped. Under dry test conditions at a pressure below 0.705×10^{-2} kg mm^{-2}, there was an increase in wear due to dispersion of mica particles.

The bearings made from the matrix alloy and composite materials were tested under lubrication, without lubrication (dry test) and semi-dry conditions (where the oil from the bearing tested under lubrication was cleaned off and the same bearings were tested again). The results have indicated that the composite bearings can run under boundary, semi-dry and dry friction conditions against a hardened steel shaft, whereas the matrix bearings either seize or show stick slip under the same conditions. The bearing surface of the matrix alloy tested under semi-dry conditions showed considerable wear and tear (observable with naked eye), but the composite bearings did not show any such appearance under the same testing conditions. This, plus the lower coefficient of friction of the composite bearing under semi-dry conditions than that under dry conditions, indicated that the composite bearing has an ability to absorb oil. The ability of the composite bearing to run under severe conditions has been shown to be related to the presence of mica particles at the bearing–shaft interface. The shaft run with composite bearings had a better surface finish (indicated by a mirror polish of the shaft and CLA measurement) than that shaft run with matrix alloy bearings under lubricated conditions. This gave indirect evidence of the presence of mica particles at the shaft–bearing interface, since the mica particles are known to act as a lapping compound.

4. Conclusions

(1) Al–4% Cu–1.5% Mg alloy mica particle composites can be made by stirring cut mica powder in liquid aluminium alloys followed by casting the melts into suitable permanent moulds. It was found necessary to add magnesium, either as an alloying element to the melt or in the form of pieces to the surface of the alloy melts, while mica was being added to the vortex of the melt, to aid the dispersion of up to 3 wt % mica powders in the melts, and to obtain high recoveries of mica in the castings.

(2) Percentage recovery of mica (as % mica added to the melt) in the castings increases with increase in the weight percent of magnesium added to the surface of the melt for a fixed quantity (3%) of mica powder added to the melt.

(3) For a fixed magnesium content, the amount of mica retained in the castings increases with increasing amounts of mica (up to 3 wt % investigated in this study) added to the melt during dispersion. However, the percentage recovery of mica particles decreases with an increase in the percentage of mica added to the melt.

(4) The presence of mica particles in the castings was confirmed by microscopic examination, chemical analysis, electron microprobe analysis, and X-ray diffraction of extracted particles from the composites. Magnesium added to the surface of the melts has been observed to segregate around the mica particle presumably facilitating the dispersion of mica particles in the melts.

(5) Aluminium–mica particle composite castings can be remelted, degassed with nitrogen and recast with mica recoveries of up to 80%.

(6) The mechanical properties of aluminium–alloy mica particle composite decrease with mica content. At 2.2% mica levels the aluminium–mica composite has a tensile strength of 14.22 kg mm^{-2} with 1.1% elongation, a compression strength of 42.61 kg mm^{-2} and an impact strength of 0.30 kgm cm^{-2}. The bearings of the composite at this mica level were found to run under boundry lubrication, semi-dry and dry friction conditions, whereas the matrix bearings without any mica seized or showed stick slip under the same conditions.

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