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Microstructure and mechanical properties of Al–Al_2O_3–MgO cast particulate composites

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Al–Al_2O_3–MgO cast particle composites prepared by an MgO coating technique were investigated for their microstructural and mechanical property features. In all, sixteen compositions of the composite were subjected to this study. Generally, a uniform distribution of Al_2O_3 particles was observed in the composites. But in the upper half portion of the cylindrical castings, Al_2O_3 particles were found to segregate along the grain boundaries and also within the grain-forming chain-like structures. The microhardness of the base matrix revealed that the retention of submicrometre MgO particles causes dispersion strengthening. In the case of maximum strengthening, the microhardness of the base matrix rises to $35\,kg\,mm^{-2}$ from $19\,kg\,mm^{-2}$ for as-cast pure aluminium. The composites also displayed excellent high-temperature tensile properties up to 250° C (523 K). At the level of 21% V_f retention of Al_2O_3, the composite displayed a UTS value of $110\,MN\,m^{-2}$ with corresponding 0.2% offset yield strength of $65\,MN\,m^{-2}$ and 12% elongation at ambient temperature. At 150° C (423 K) and 250° C (523 K), the composite retains 69% and 53%, respectively, of its room-temperature UTS value. This was the optimum retention of Al_2O_3 and the best composite obtained in the present work. The excellent high-temperature characteristics of the composite are thought to be due to the sum total effect of both the submicrometre MgO particles and the coarser Al_2O_3 particles retained in the aluminium base matrix.

1. Introduction

A number of techniques have been developed in the recent past to incorporate a wide variety of ceramic particles in aluminium- and zinc-based alloys via the route of liquid metallurgy techniques [1–7]. Two common problems, namely the poor wettability of dispersoid particles and their non-uniform distribution in the cast composite, have always been associated with this route.

The objective of incorporating these particles in the composite at specific locations or throughout the matrix is to develop specific properties in the cast material. High-temperature hardness and strength, creep resistance, improved tribological properties such as seizure resistance, anti-friction and adhesive wear resistance etc. are some of the important properties in view while fabricating these particle composites. Thus, all the metal–nonmetal particulate composites are intended to be designed for specific end-applications.

Al–Al_2O_3 and aluminium alloy–Al_2O_3 composites are known to possess excellent high-temperature hardness and strength up to 350° C [3, 6]. Their adhesive wear characteristics have also been found to be superior to those of aluminium [3, 6]. As such, these composites can be put to high-temperature and wear-resistant applications.

In recent studies [8–10], a new method termed the "MgO coating technique" was developed for the preparation of Al–Al_2O_3 cast particulate composites. A special feature of this technique is that both the submicrometre MgO particles (0.3 to 0.4 μm) as well as the coarse MgO-coated Al_2O_3 particles (8 to 80 μm) are retained simultaneously in the base matrix. This leads to the preparation of Al–Al_2O_3–MgO cast particulate composites. The retention and uniform distribution of submicrometre (0.3 to 0.4 μm) MgO particles in the composite is taken to be a welcome feature, since it would result in matrix hardening and thus positively help in improving the properties of the composite further.

In the present work, the mechanical properties of these composites were determined in tension up to 250° C (523 K). Further, the role of MgO retention and dispersion in influencing the hardness of the base matrix was examined through micro-hardness measurements. These results and also the morphological features of retained Al_2O_3 particles on a microscale are reported in this paper.

2. Experimental procedure

2.1. Materials used

Commercial purity aluminium (≥ 99.9% purity) containing a maximum of 0.1% total of iron and silicon as chief impurities was employed for the preparation of Al–Al_2O_3–MgO cast partuculate composites.

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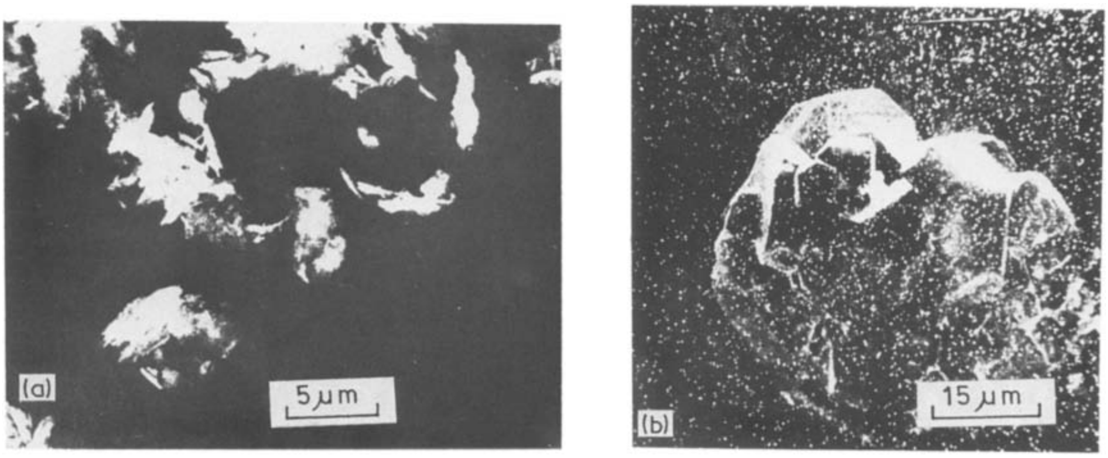

Figure 1 SEM pictures of (a) flakelike aggregates of submicrometre MgO particles and (b) MgO-coated Al_2O_3 particle.

Coarse lavigated Al_2O_3 particles (8 to 80 μm) and submicrometre MgO powder (0.3 to 0.4 μm) were employed for dispersion in the melt. Figs 1a and b show SEM pictures of a needle-like assemblage of MgO particles and an MgO-coated Al_2O_3 particle, respectively.

2.2. MgO coating technique

Chill-cast cylindrical castings of $Al-Al_2O_3-MgO$ cast particulate composites (25 mm diameter, 140 mm length) employed for the fabrication of metallographic and tensile test specimens were prepared using the MgO coating technique [8-10]. Details of this technique have been described previously [9, 10].

Briefly, a thin coating of MgO was obtained around the surface of coarse Al_2O_3 particles by mechanically mixing the two powders in a blender for 30 to 35 min. The two powders were taken in definite proportions in order to vary the MgO content of the total powder mixture (Al_2O_3 + MgO) systematically. The amount of total powder mixture added to the liquid aluminium melt was varied as 3, 5, 7 and 10 wt % of the melt. Also, the MgO content of each set of total powder mixture was varied as 5, 10, 15 and 20%. Thus in total sixteen powder compositions were employed for the preparation of the composites in the form of chill-cast cylindrical bars for onward metallographic and mechanical property investigations. In each case, 1.5 kg aluminium melt was well superheated to 910 ± 5° C before the commencement of stirring for powder dispersion.

2.3. Optical and SEM studies

Specimens drawn from the top and bottom sections of the castings were ground and polished in the usual manner employing diamond paste commencing from 9 to 0.25 μm. HF (0.5%) or a special reagent (10% HF + 25% HCl + 15% HNO_3 + 50% distilled water) was employed for etching the polished specimens. Freshly etched specimens were subjected to metallographic observations using a Reichert Universal Camera Microscope and a Hitachi Mini SEM. In a few instances, the total length of the casting was also slit through the centre to look for the distribution of Al_2O_3 particles all along the length.

2.4. Mechanical properties

2.4.1. Tensile properties

Tensile specimens of 30×10^{-3} m gauge length and 7.98×10^{-3} m diameter were pulled in tension at three test temperatures (ambient, 150° C and 250° C) using a 10-ton Instron Universal Testing Machine. A cross head speed of 2×10^{-4} m min^{-1}, yielding a constant strain rate of 10^{-4} sec^{-1} on 30×10^{-3} m gauge length, was employd in each case. A 2 kW furnace attachment of the Instron machine helped in the determination of the tensile properties of various composites at elevated temperatures. At least two specimens were run for each kind of composites and for each temperature of test. The cross-section of the tensile specimens close to their fracture surfaces were also examined optically to study the distribution of Al_2O_3 particles.

2.4.2. Microhardness

Microhardness measurements were carried out well within the grain and also in the region close to the grain boundaries using a table-type microhardness tester. The objective of this study was to obtain indirect evidence of the possible hardening effect caused by the dispersion of submicrometre MgO particles and their segregation tendency, if any, close to the grain boundaries.

3. Results and discussion

3.1. Microstructural features

Generally, a uniform distribution of retained Al_2O_3 particles was observed [9, 10]. But in many instances, where the volume fraction V_f of Al_2O_3 retained increased, the tendency of coagulation also increased. This happened mainly in the upper half-portion of the cast ingot. The tendency of coagulation resulted in the formation of a number of interesting morphologies. Some of the typical ones are shown in Figs 2 to 5. It can be seen that most segregation of Al_2O_3 has occurred along those regions which were the last to freeze. Some coagulation of Al_2O_3 particles was also observed to occur within the grains, mostly in the form of long chain-like structures. This morphology is clearly indicative of the possibility that clusters of Al_2O_3 particles already existed in the melt. Interaction of these

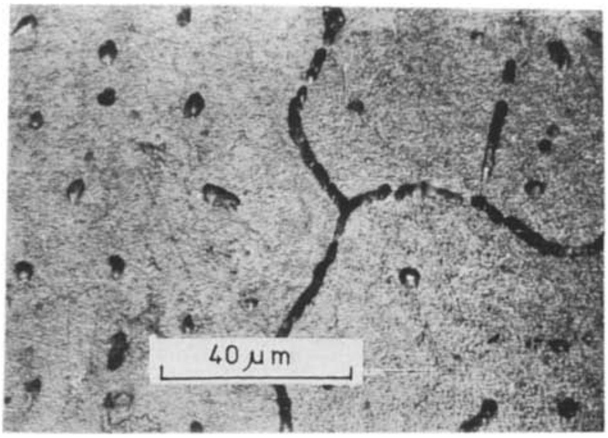

Figure 2 Optical picture showing the segregation of Al_2O_3 particles along the grain boundaries and some coagulation of Al_2O_3 within the grain.

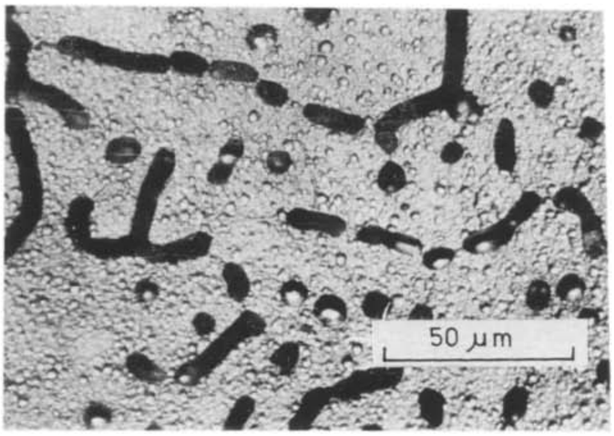

Figure 4 Optical picture showing typical chain-like structures of coagulates Al_2O_3 particles.

clusters with the advancing solidification interface would tend to produce the chain-like structures shown in Figs 2 to 5. Fig. 6 explains this mechanism schematically. Good fluidity of the liquid melt at ≈800° C, greater mobility of dispersoid particles in the slurry, the action of the stirrer and simple surface energy considerations, actively aid the process of coagulation of Al_2O_3 particles in the melt. Those clusters which are not trapped by the advancing solidification interface are pushed to the grain-boundary region, where clustered particles rearrange themselves in a single row all along the length of the grain boundary. These and other kinds of segregation patterns shown in Figs 2 to 5 are considered potentially harmful to the final mechanical properties of the composite.

3.2. Role of submicrometre MgO particles

Hardening of the base matrix caused by simultaneous dispersion of submicrometre MgO particles as revealed by microhardness measurements is shown in Fig. 7. It can be seen that the retention pattern of submicrometre MgO particles is similar to those of larger Al_2O_3 particles reported earlier [10]. Here again, optimum hardening of the base matrix results when the MgO content of any powder mixture is raised to 15 wt %. Also, the extent of hardening is seen to be a function of the amount of total powder added to the liquid aluminium. In the case of maximum hardening, the microhardness rises from $19\,\mathrm{kg\,mm^{-2}}$ for as-cast pure aluminium to nearly $35\,\mathrm{kg\,mm^{-2}}$ for MgO-stiffened base matrix (Fig. 7).

These hardening effects must be the result of dispersion strengthening of the base matrix by submicrometre MgO particles. Thus the series of $Al-Al_2O_3-MgO$ cast particle composites prepared in the present work are strengthened by a dual mechanism namely, (a) the dispersion strengthening by submicrometre MgO particles and (b) normal composite strengthening by comparatively large Al_2O_3 particles. In the present work, however, adequate data were not generated to isolate the effect of submicrometre MgO particles on the tensile behaviour of the composites. This work is currently in progress. The presented tensile behaviour of the $Al-Al_2O_3-MgO$ class of composites up to 250° C (523 K) therefore representes the sum total effect of both the submicrometre MgO particles plus the larger Al_2O_3 particles dispersed in the base matrix.

Results presented in Fig. 8 confirm that MgO particles tend to segregate in grain-boundary regions. All those reasons which are responsible for the segregation of Al_2O_3 particles are also responsible for the segregation of MgO particles.

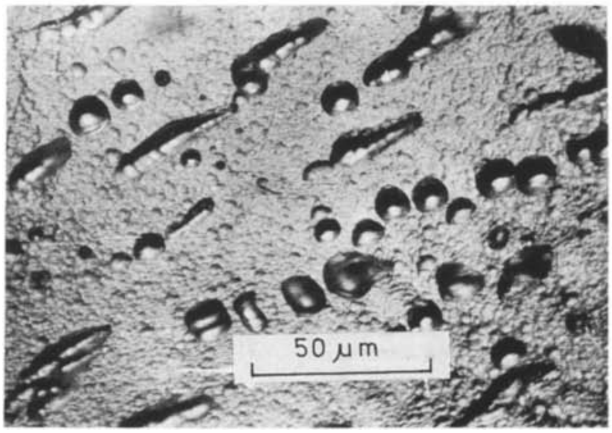

Figure 3 Optical picture showing chain-like morphology of coagulated Al_2O_3 particles in the matrix.

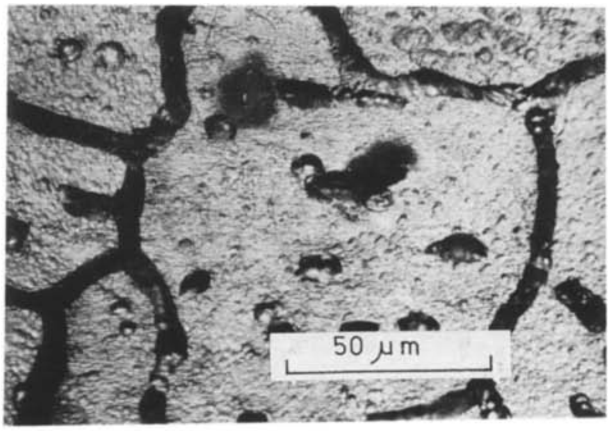

Figure 5 Optical picture showing a single row of Al_2O_3 particles segregated all along the grain boundaries. Some Al_2O_3 particles are also seen coagulated within the grain.

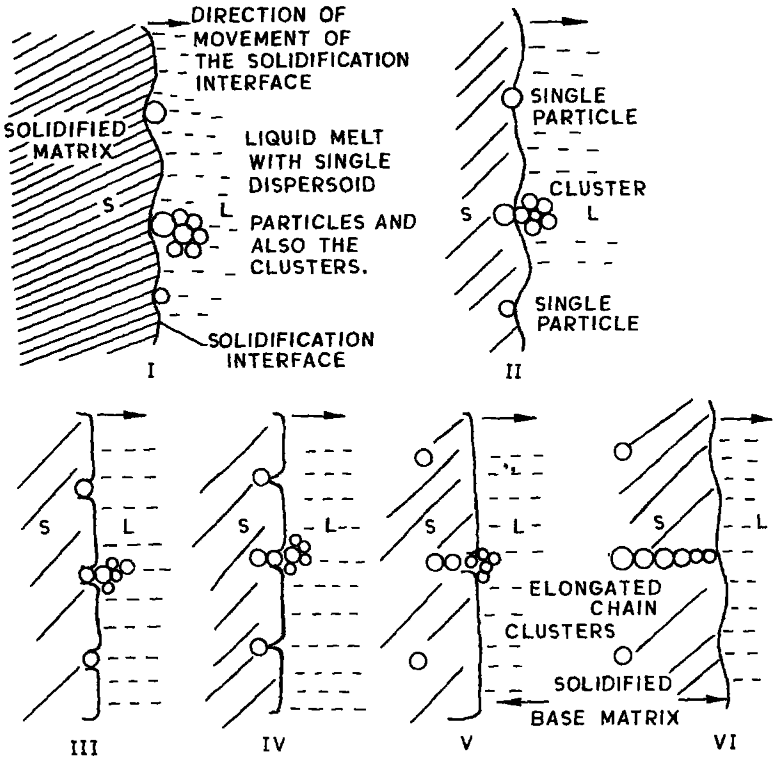

Figure 6 Schematic diagram showing the mechanism of formation of chain-like morphology of coagulated Al_2O_3 particles within the grain. S = solid, L = liquid.

3.3. Tensile behaviour

The combined effect of the simultaneous dispersion and retention of both the larger Al_2O_3 particles and the submicrometre MgO particles on the tensile behaviour of the composites is summarized in Table I. A striking correlation between the retention behaviour pattern of larger Al_2O_3 particles and the resulting tensile properties of the composites was observed at all levels of MgO content of various powder mixtures added to the melt. The tensile behaviour of various composites at 250° C (523 k) test temperature is shown in Figs 9, 10 and 11 to illustrate this point. As expected, the mechanical properties of the composites continue to improve with increasing retention of Al_2O_3 and submicrometre MgO particles, till overcrowding of particles beyond 15 wt % MgO content of any powder mixture sets in the downward trend.

Maximum strengthening of the composite is obtained when 10 wt % total powder mixture containing 15 wt % MgO is stirred in the melt (Table I). This corresponds with nearly 21% V_f retention of coarser Al_2O_3 particles in the matrix, which was also the maximum observed in the present series of experiments with Al–Al_2O_3–MgO cast particle composites [10]. Characteristics of only this composite are discussed in the following paragraphs. The UTS value of this composite at ambient temperature was found to be 110 MN m^{-2}, with corresponding 0.2% offset yield

TABLE I Tensile properties of Al–Al_2O_3–MgO cast particle composites at 25, 150 and 250° C test temperatures

Specimen No.	Total (Al_2O_3 + MgO) powder addition to 1.5 kg liquid aluminium (wt %)	MgO content in individual powder mixture (wt %)	V_f Al_2O_3 retained (%)	Mechanical properties: UTS (MN m^{-2})			0.2% offset yield strength (MN m^{-2})			Elongation on 3 × 10^{-2} m gauge length (%)		
				25° C	150° C	250° C	25° C	150° C	250° C	25° C	150° C	250° C
1	As-cast pure aluminium	–	–	81	44	29	32	26	15	42	55	70
2	3	5	0.304	84	44.5	30.0	33	27.5	18	36.5	42.5	51
		10	0.350	90	51.0	36.0	39	34.0	27	30.5	37.0	45.5
		15	0.468	95	54.0	40.5	44	37.5	32.5	27.5	31.5	35.0
		20	0.500	93	50.5	37.0	38.5	33.0	27.5	28.5	32.0	36.0
3	5	5	2.11	88	53.0	40.0	40.0	34.0	23	27.5	32	40
		10	2.20	92.5	57.0	45.0	48.0	38.0	30	24	28	33
		15	4.34	101.5	63.5	50.0	55.5	42.0	37	21	24	28.5
		20	4.14	99.0	59.0	45.0	51.0	39.0	32	22	25	30.0
4	7	5	7.9	91	56.0	45.0	45.0	36.5	25	22	24	30
		10	4.9	98.5	65.0	49.5	54.0	44.0	35	18.5	20	25
		15	8.9	105.0	69.0	54.0	59.0	48.0	40	15.5	16	20
		20	9.6	102.0	65.0	51.5	54.0	43.0	35	16.0	17.5	22.5
5	10	5	16.62	94	58.0	48.5	48.0	40.0	29	16	18.0	23.0
		10	18.4	105	72.0	54.0	60.0	48.0	37	12.5	14	19.0
		15	20.8	110	76.0	58.0	65.0	53.0	44	12.0	12	15.0
		20	16.9	106	70.0	55.5	60	48.0	37	12.0	13.5	16.5

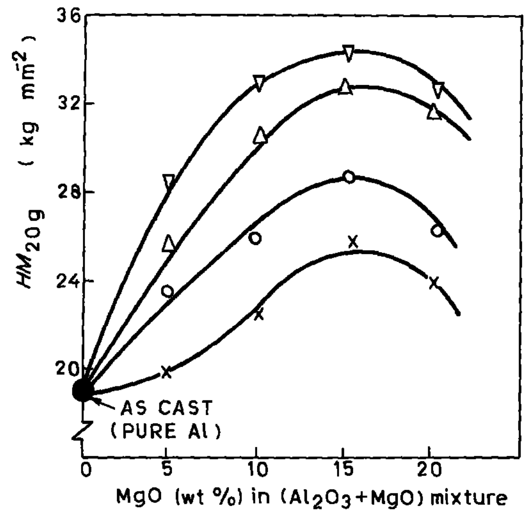

Figure 7 Microhardness of the base matrix of Al-Al_2O_3-MgO cast particle composites, shown as a function of the MgO content of the total powder mixtures stirred in liquid aluminium. Content of (Al_2O_3 + MgO) (×) 3%, (○) 5%, (△) 7%, (▽) 10%.

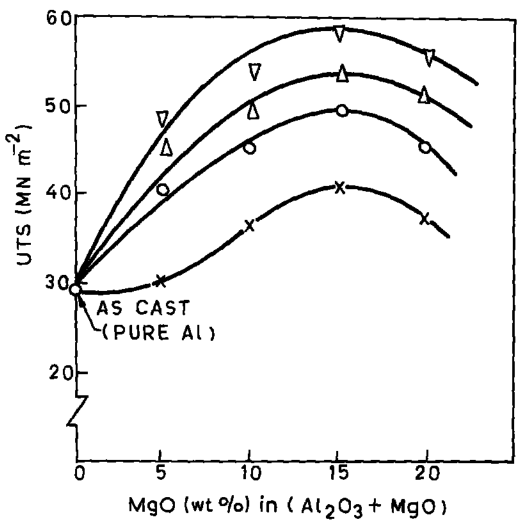

Figure 9 UTS of Al-Al_2O_3-MgO cast particle composites at 250° C (523 K) shown as a function of the MgO content of various powder mixtures stirred in liquid aluminium. Content of (Al_2O_3 + MgO) (×) 3%, (○) 5%, (△) 7%, (▽) 10%.

strength value of 65 MN m^{-2} and 12% elongation. This represents an improvement of 35.8% over the UTS value of as-cast pure aluminium. But what is more significant is the widening of this gap at elevated temperatures; the composite retains its strength better at elevated temperatures compared to as-cast pure aluminium. The gap between the UTS value of the composite and that of as-cast pure aluminium at 150° C was found to be 72.72%, and this value rises to 100% at 250° C.

The observed 0.2% offset yield strength values of the composite at various temperatures (which is the design criterion) are of rather greater significance. The yield strength of the composite represents an increase of 103% over the corresponding value of as-cast pure aluminium at ambient temperature. This difference remains the same at 150° C test temperature. But at 250° C, the difference is magnified to 193%, which is indicative of the high-temperature strength characteristics of the composite. Retention of 68% yield strength of the composite, as against 47% by as-cast pure aluminium at 250° C, is a clear index of the high-temperature strength of the composite. Thus an initial high strength of the composite, coupled with a relatively much slower rate of loss of strength when exposed to high temperature, results in a composite which has a yield strength nearly three times that of as-cast pure aluminium at 250° C. This can be a useful design criterion.

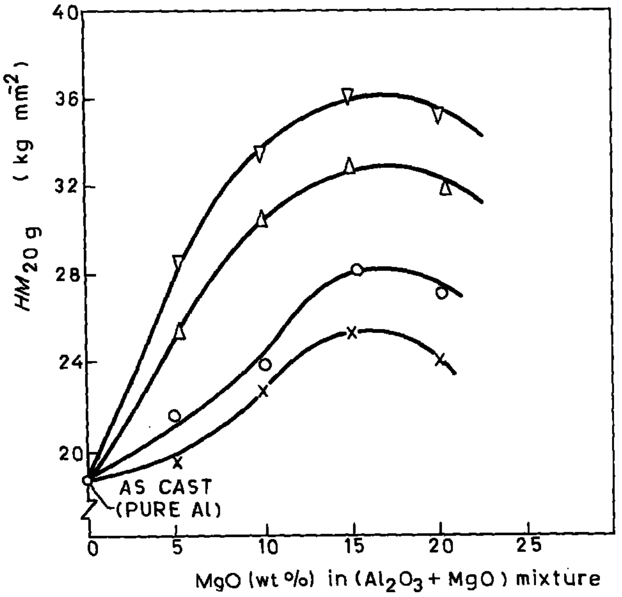

Figure 8 Microhardness observations showing higher hardness in grain-boundary regions compared to the body of the grain in Al-Al_2O_3-MgO cast particle composite with 3% and 7% total powder mixture (Al_2O_3 + MgO) additions. Values measured (×) at the matrix of aluminium with 3% mixture; (○) at the grain-boundary region of aluminium with 3% mixture; (△) at the matrix of aluminium with 7% mixture; (▽) at the grain-boundary region with 7% mixture.

The role of progressively increasing the V_f of Al_2O_3 particles on the peak UTS and 0.2% offset yield strength values of different composites at the three test temperatures is summarized in Fig. 12. It can be seen that the general trend of improvement in the

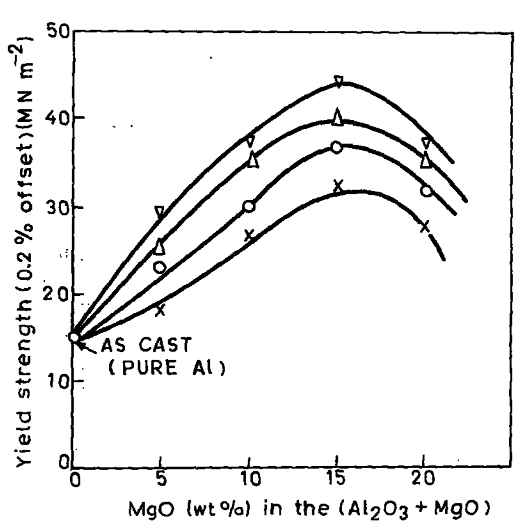

Figure 10 0.2% offset yield strength of Al-Al_2O_3-MgO cast particle composites at 250° C (523 K) shown as a function of the MgO content of various powder mixtures stirred in liquid aluminium. Content of (Al_2O_3 + MgO) (×) 3%, (○) 5%, (△) 7%, (▽) 10%.

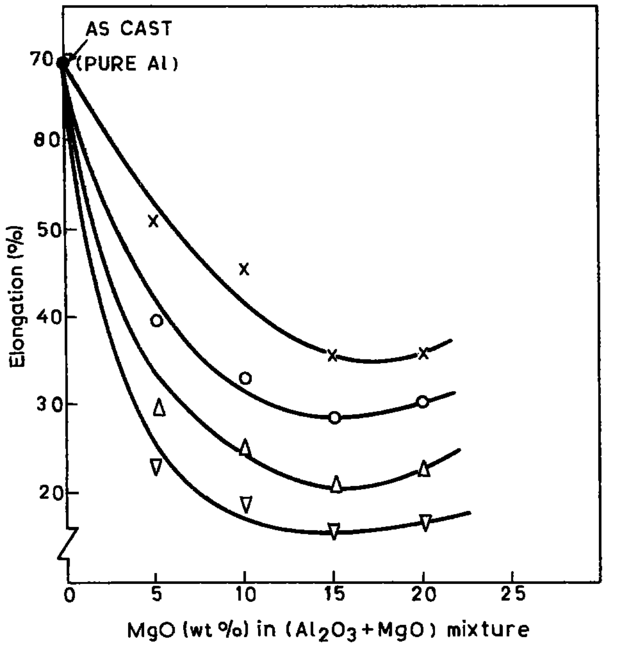

Figure 11 Percentage elongation of Al–Al_2O_3–MgO cast particle composites at 250° C (523 K) measured on 30×10^{-3}m gauge length, shown as a function of the MgO content of various powder mixtures stirred in liquid aluminium. Content of (Al_2O_3 + MgO) (×) 3%, (○) 5%, (△) 7%, (▽) 10%.

mechanical properties of the composites as a result of progressively increasing the retention of Al_2O_3 particles is roughly indentical at all three test temperatures. A considerable drop in the UTS values at 150° C compared to the values at ambient temperature may be noted. Such a large-scale shift is not observed in the values of 0.2% offset yield strength. Also a maximum improvement of the order of 5 MN m^{-2} is observed in both the UTS as well as 0.2% offset yield strength values beyond 9% V_f of Al_2O_3 retained at all the test temperatures. Fig. 13 shows that the retention of Al_2O_3 particles confers an overall embrittling effect on the composites.

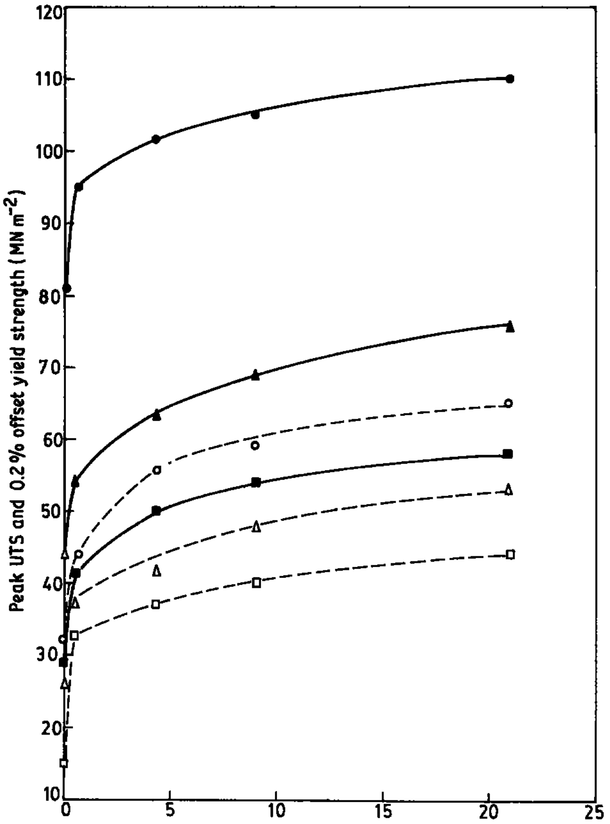

Figure 12 The effect of progressively increasing percentage V_f retention of Al_2O_3 particles on (——) peak UTS and (– – –) 0.2% offset yield strength values of different Al–Al_2O_3–MgO cast particle composites at the three test temperatures: (●, ○) 25° C; (▲, △) 150° C; (■, □) 250° C.

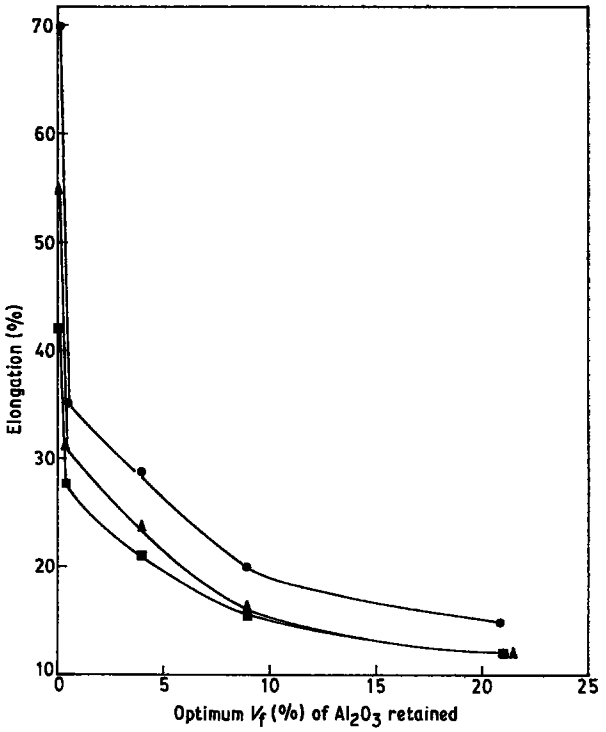

Figure 13 Influence of progressively increasing percentage V_f retention of Al_2O_3 particles on the percentage elongation values (30 mm gauge length) of the composites shown in Fig. 12, at the three test temperatures (●) 25° C, (▲) 150° C, (■) 250° C.

The characteristics of the composites summarized in Figs 12 and 13 may be attributed to the presence of hard and refractory oxide particles embedded in the comparatively softer aluminium matrix. The submicrometre MgO particles would cause dispersion strengthening of the base matrix as shown in Figs 7 and 8. The harder Al_2O_3 particles embedded in this matrix would further strengthen the composite – the larger the V_f of Al_2O_3 retained, the greater the extent of strengthening. These Al_2O_3 particles were also found nicely bonded with the base matrix [9, 10]. Under these conditions, part of the load applied to the composite will be shared by hard Al_2O_3 particles. In addition to these factors, Al_2O_3 particles also tend to restrict matrix deformation by applying mechanical restraint to any plastic deformation of the softer base matrix. Thus, in the present class of Al–Al_2O_3–MgO cast particle composites, both dispersion strengthening as well as the coarse Al_2O_3 particle strengthening seem to be operative. A comparatively larger upward shift in the UTS values at different test temperatures compared to that obtained for the yield strength is indicative of the role of Al_2O_3 particles as a load-bearing constituent. A relatively drastic fall in UTS at 150° C compared to that obtained at ambient temperature is attributed to the correspondingly large magnitude of matrix weakening as reflected in the

UTS values obtained for cast aluminium. The further weakening at a still higher temperature of 250° C is comparatively less, even in the case of cast aluminium, and is also observed in the composites. The slightly higher UTS and percentage elongation values of as-cast pure aluminium obtained in the present work at ambient temperature compared to the values reported by some other workers [3, 6] is thought to be due to extremely dry (low humidity) conditions during actual experiments. It is for this reason that even those composites which retained 21% V_f of Al_2O_3 exhibited nearly 12% elongation at ambient temperature (with a corresponding UTS value of $110\,MN\,m^{-2}$ and a yield strength of $65\,MN\,m^{-2}$).

4. Conclusions

1. With an increase in the retention of Al_2O_3 particles in the base matrix, the tendency of coagulation of particles increases. Generally these particles tend to segregate along the grain boundaries, forming a single row of particles. They were also found to segregate within the grain-forming chain-like structures.

2. Microhardness measurements of the base matrix indicated that submicrometre MgO particles cause dispersion strengthening of the base matrix. Maximum strengthening was observed to occur at 15 wt % MgO content of any powder mixture. These particles also tend to segregate along the grain boundaries. In the case of maximum strengthening, the microhardness rises from $19\,kg\,mm^{-2}$ for pure aluminium to nearly $35\,kg\,mm^{-2}$ for the MgO-stiffened base matrix.

3. $Al-Al_2O_3-MgO$ cast particle composites display excellent tensile characteristics up to 250° C (523 K). Composites prepared with the addition of 10 wt % total powder mixture containing 15% MgO exhibited maximum Al_2O_3 retention and also the best mechanical properties. This composite was found to possess $110\,MN\,m^{-2}$ UTS, $65\,MN\,m^{-2}$ 0.2% offset yield strength and 12% elongation at the ambient temperature corresponding to 21% V_f retention of Al_2O_3. The retention of nearly 69% and 53% of its room-temperature UTS value at 150 and 250° C, respectively, was a special feature. The 0.2% offset yield strength of this composite was found to be nearly three times that of as-cast pure aluminium at 250° C. The composite also retains ductile fracture features and a fair amount of ductility (indicated by 12% elongation) at ambient temperature, in spite of the retention of 21% V_f of Al_2O_3.

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