

Characterization and Microstructural Modifications of a Pressure Die Cast Eutectic Aluminium–Silicon Alloy-Graphite Composite

B. K. Prasad*, T. K. Dan** and P. K. Rohatgi***

*Regional Research Laboratory (CSIR), Bhopal-462 026 (M.P.), India

**Central Glass & Ceramic Research Institute (CSIR), Naroda Centre, Ahmedabad-382 330, India

***University of Wisconsin, Milwaukee, Wisconsin-53201, U.S.A.

The present investigation was carried out to see if intricate shaped castings can be made from a eutectic Al-Si (BS LM13) alloy dispersed with 5.5 mass% graphite particles by pressure die casting technique. A bushing spring guide (BSG) component used for electrical applications was selected for the purpose. The study involved the characterization of a few properties *e.g.* hardness, density and electrical resistivity of the pressure die cast (PDC) composite and their comparison with that of the gravity die cast (GDC) one. The influence of heat treatment on the microstructure of the PDC composite has also been studied.

Visual examinations revealed that the components were perfect in shape and pore free indicating that the technique could be a promising route to synthesize graphitic aluminium alloys into intricate shapes.

Machined sections of the PDC components indicated reasonably uniform distribution of graphite particles in various regions of the former. This was also confirmed by the quantitative analysis of the graphite content recovered from the dissolved specimens. The variation in hardness, density and electrical resistivity of the composite was quite less agreeing well with better uniformity of distribution of graphite particles in the matrix.

The matrix microstructure of the PDC composite was considerably refined over the one processed by GDC technique, although the morphology of the microstructural constituents remained unchanged. The higher rate of solidification in this case was found to be responsible for the improvement in the uniformity of graphite distribution in the matrix and the microstructural refinement. Reduced secondary dendritic arm spacing (DAS) further confirmed a higher rate of solidification as a result of applying the pressure.

Improvement in the graphite/matrix interfacial bonding was found to be one of the interesting features of pressure application. This was attributed to the increased solubility of the dissolved gases in the matrix and squeezing out of the entrapped gases from the latter under the conditions of applied pressure during solidification. The graphite particles were found to have fractured in this case probably due to the possible application of a combination of impact, shear and compressive stresses under the influence of the applied injection pressure.

Heat treatment of the PDC composite was found to have brought about significant and useful modifications in the matrix microstructure at the little loss in properties like hardness, density and electrical resistivity.

(Received August 28, 1992)

Keywords: metal matrix composite, graphitic aluminium alloy, pressure die casting, microstructural modifications, heat treatment, structure-property correlation

I. Introduction

Benefits associated with metal matrix composites (MMCs) are well known. The MMCs containing hard dispersoids offer high strength to weight ratio, high modulus, elevated temperature strength and good tribological properties⁽¹⁾⁻⁽⁶⁾. On the other hand, the ones reinforced with softer dispersoids have been demonstrated to be possessing good seizure and wear resistance⁽⁷⁾⁻⁽¹⁰⁾. Aluminium alloys consisting of graphite particles form an important series of engineering materials in the latter category of the MMCs. They have been established to be potential candidate materials for a variety of engineering applications⁽⁷⁾⁻⁽¹¹⁾ owing to their promising tribological properties such as good seizure and wear resistance⁽⁷⁾⁻⁽¹⁰⁾.

Quite poor wettability of graphite with the aluminium matrix resulting from the high contact angle between the two⁽¹²⁾, inspite of their comparable density values, gives rise to problems like segregation of the dispersoid in the upper regions of the casting and poor graphite/matrix interfacial bonding. In tribological applications, a poor interface between the graphite and the Al matrix facilitates complete or partial removal of the dispersoid in lumps leaving behind holes in the matrix. Such a phenomenon does not allow the graphite to smear on the mating surfaces and offers higher wear rate⁽¹³⁾. Thus, the whole purpose of graphite addition to the Al matrix gets defeated and rather a negative effect in terms of higher wear rate and premature seizure of the composite is experienced. On the other hand, although the good graphite/Al matrix interface does not help in load transfer since graphite is quite weaker than Al, it facilitates efficient

smearing of graphite on the mating surfaces. This phenomenon reduces the extent of metal-to-metal contact in operation ensuring smooth functioning of the components even if boundary lubrication conditions are encountered sometimes. Another important factor governing the efficient functioning of the composites is the matrix microstructure. It has been reported that the good graphite/Al matrix interface associated with spheroidal microstructural constituents in the matrix offer a reduced coefficient of friction and a low wear rate of the Al-graphite composites⁽¹⁴⁾.

Selection of heat treatable variety of Al–Si alloys for synthesizing graphitic aluminium composites has been suggested to be of great advantage as far as morphological modifications of microstructural phases in the matrix by heat treatment are concerned.

Application of pressure during solidification^{(15)–(19)} has been found to be one of the important measures towards minimizing the extent of segregation of the dispersed phase in the Al matrix and improving the dispersed/matrix bonding thereby reducing earlier mentioned problems with the composites.

It has been observed that very little information are available in literature on pressure die casting of graphitic aluminium alloys⁽²⁰⁾ and no work has been carried out regarding examining their response towards heat treatment. In view of this, an attempt has been made to pressure die cast a heat treatable variety of Al–Si (BS LM13) alloy containing dispersion of graphite particles and see the influence of applying pressure and heat treatment on its matrix microstructure and properties.

II. Experimental

1. Composite preparation

The composite material was prepared by the liquid metallurgy route using vortex method⁽²¹⁾. Essentially, the technique consists of dispersing the graphite particles on the vortex of the matrix alloy melt created with the help of a mechanical stirrer. In this investigation, 5.5 mass% uncoated, heat treated graphite particles (size: 45–150 μm) were dispersed in the melt of BS LM13 (Al–11.7Si–1.0Cu–1.5Ni–0.8Fe–0.5Mn–1.0Mg) alloy. The composite melt was transferred in the die cavity of a cold chamber pressure die casting machine. The injection pressure applied was 5.0 atmospheres (505 KPa).

2. Specimen preparation

Pieces from various parts of the castings were cut and machined for visual examination. Fine chips were also generated from the regions, dissolved chemically and the residue particles of graphite were weighed. An average of six observations has been reported in this study.

Specimens for metallographic observations and quantitative analyses were prepared by standard metallographic techniques and etched with Keller's reagent.

3. Microscopy and quantitative metallography

The secondary dendritic arm spacing (DAS) and the thickness of the αAl -Cell boundary of the matrix was measured in the fractured regions of and away from the dispersed graphite particles in the composite. The reported values of the DAS and the thickness of the αAl -cell boundaries are the average of twenty readings in each case.

The particle size and inter particle spacing of graphite was measured using the intercept method in the matrix. The observations are based on a large number of measurements in at least ten regions of the specimens in each case.

Microstructural observations were made using optical and scanning electron microscopes. Prior to SEM examination, the specimens were sputtered with gold.

4. Heat treatment

Typical specimens were also subjected to heat treatment (solutionizing followed by artificial ageing). The heat treatment parameters selected were solutionizing at 516°C for 3–8 h and ageing at 180°C for the same duration. The specimens were solutionized at 516°C for 5 h prior to ageing. The specimens were quenched in water at 40°C after heat treatment.

5. Property evaluation

Hardness, density and electrical resistivity of the as cast as well as heat treated composites were measured. Hardness measurements were made using a Brinell hardness tester. A Mettler microbalance was used for measuring the density by water displacement technique. The electrical conductivity measured with the help of a Technofour type 757 conductivity meter was converted into electrical resistivity.

Reported values of hardness, density and electrical resistivity represent an average of six observations.

III. Results

The PDC components were found to be visually sound and perfect in shape (Fig. 1(a)). Intricacy in the shape of the casting may also be revealed in the figure. A typical machined section of the casting further indicated the absence of porosity (Fig. 1(b)). Reasonably uniform distribution of graphite particles in various parts of the section on a macro level is evident in Fig. 1(b).

Table 1 shows the graphite content in the GDC and PDC composites recovered after chemically dissolving the matrix part of the specimens. Measured hardness, density and electrical resistivity along with the variation (maximum and minimum) in the properties are also shown in the Table. Improved hardness and density and reduced electrical resistivity of the PDC composite may be noted (Table 1). The extent of variation in the parameters in this case was less (Table 1) than the GDC composite.

Figure 2 shows the frequency distribution of the size

Table 1 Properties and graphite content of the pressure die cast (PDC) and gravity die cast (GDC) composites.

S. No.	Property/Parameter	Pressure die cast composite			Gravity die cast composite		
		Maximum	Minimum	Average	Maximum	Minimum	Average
1.	Graphite content, mass%	5.75	5.13	5.24	4.88	3.32	4.27
2.	Density, Mg/m ³	2.65	2.62	2.63	2.58	2.46	2.51
3.	Hardness, BHN	142	133	139	107	81	102
4.	Electrical resistivity, nΩm	93.2	83.2	85.8	95.7	70.3	88.3

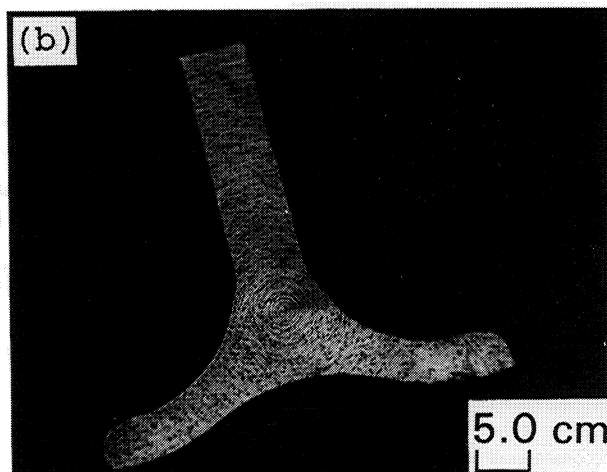
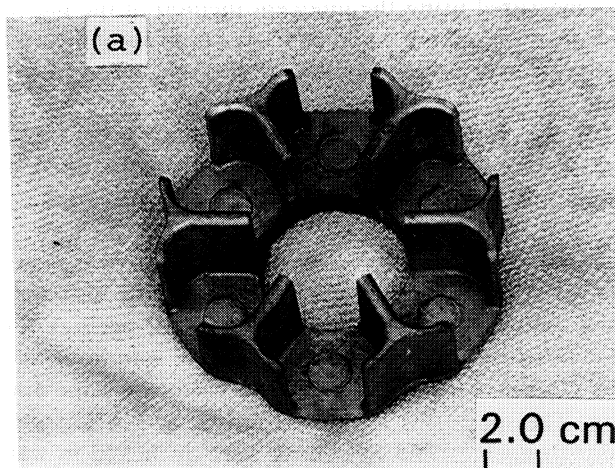


Fig. 1 (a) Pressure die cast (PDC) bushing spring guide component and (b) a typical machined section of the component. Perfect shape of the component free of porosity and the uniform distribution of graphite particles on a macro level may be noted in (a) and (b), respectively.

and interparticle spacing of graphite in the matrix. It may be seen that nearly 80% of the particles retained in the matrix were in the size range of 40–100 μm (Fig. 2(a)). Further, about 75% of the particles lied in the interparticle spacing range of 50–300 μm (Fig. 2(b)). The trend was similar in the GDC and PDC composites except that in the GDC specimens larger spacings were also observed (Fig. 2(b)).

The microstructures of the PDC and GDC composites are shown in Fig. 3. Microstructural refinement of the matrix in the PDC composite (Fig. 3(a)) over that of the

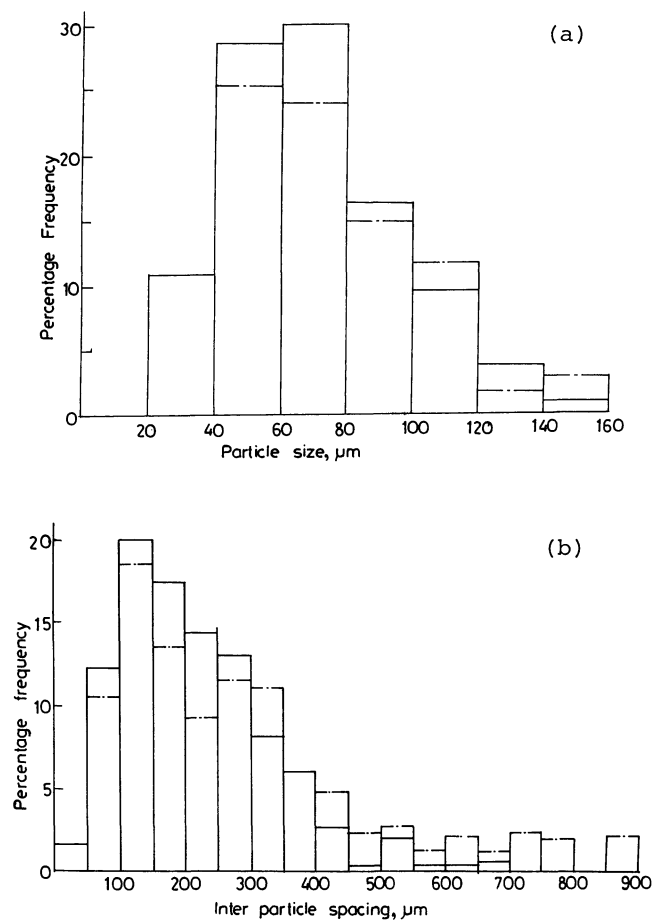


Fig. 2 Frequency distribution of (a) the particle size and (b) the interparticle spacing of graphite in the matrix of PDC (—) and GDC (---) composites.

GDC one (Fig. 3(b)) is evident. A magnified view (Fig. 3(c)) of the PDC specimen revealed the needle shaped eutectic silicon particles. GDC specimens also showed similar morphology of the phase. Fracture of a typical graphite particle in the PDC composite may also be seen in Fig. 3(c). Microstructural refinement and reduction in the thickness of the αAl -cell boundaries in the crevices of the fractured graphite particles is also evident in the figure. Interestingly, the fracture events in this case were noted even in the recovered graphite particles too (Fig. 4(a)). On the other hand, the graphite particles recovered from the GDC specimens did not show any such fracture (Fig. 4(b)).

Table 2 reveals the DAS values of the PDC and GDC

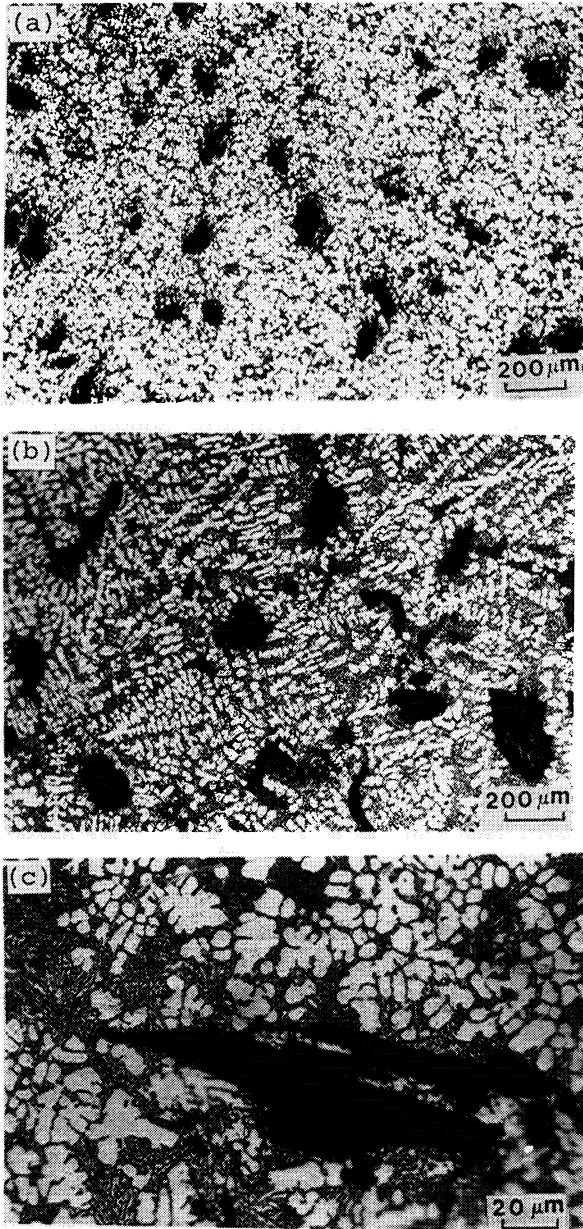


Fig. 3 Micrographs of (a) the PDC & (b) the GDC composites revealing microstructural refinement in case of the former and (c) a magnified view of the microstructure of the PDC composite showing the needle shaped eutectic silicon phase and fracture of a typical graphite particle.

composites and the corresponding cooling rates. The cooling rates were computed using the following equation suggested for Al–Si alloys⁽²²⁾:

$$\log(dT/dt) = -\frac{\log(DAS) - 1.66}{0.40} \quad (1)$$

where dT/dt is in K/s and DAS is in μm . Reduced DAS value of PDC over the GDC composite is evident in the Table. Significantly improved cooling rate of the PDC composite can also be noted in the table. The table also shows the thickness of αAl -cell boundaries in the GDC and PDC composites. Reduction in the thickness of the boundaries in PDC specimen may be noted in the table.

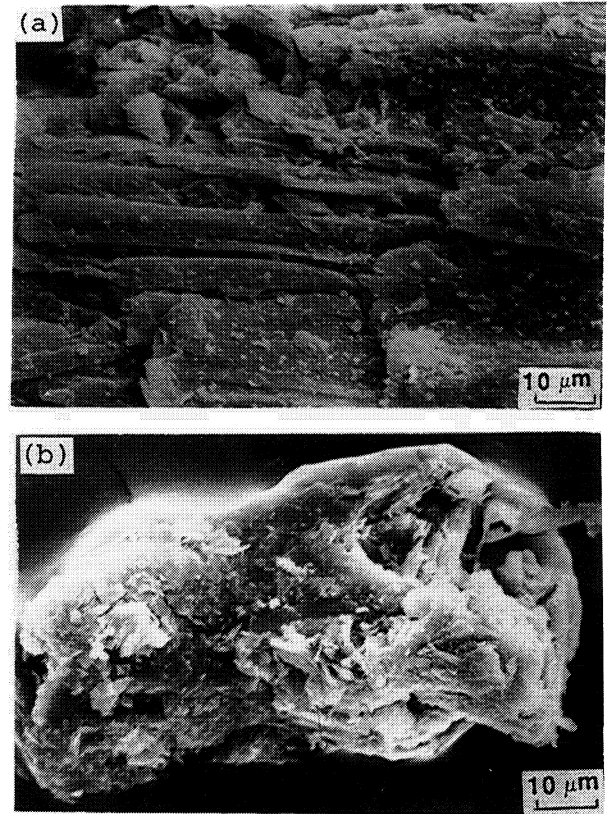


Fig. 4 SEM micrograph of the graphite (residue) particles obtained after chemical dissolution of the matrix revealing a typical (a) fracture of graphite and (b) unfractured graphite particle in the PDC and GDC composites respectively.

Table 2 Secondary dendritic arm spacing (DAS) values, corresponding rate of solidification (dT/dt) and the thickness of the αAl -cell boundaries (X) of the pressure die cast (PDC) & gravity die cast (GDC) composites.

S.No.	Region	PDC composite			GDC composite		
		DAS, μm	dT/dt K/s	$X/\mu\text{m}$	DAS, μm	dT/dt K/s	$X/\mu\text{m}$
1.	Inside the fractured regions of graphite particles	2.76	2226.03	0.37	—	—	—
2.	Away from the graphite particles	7.05	107.03	1.65	16.69	12.41	2.46

Improved dispersoid/matrix interfacial bonding of the PDC composite (Fig. 5(a)) over the GDC one (Fig. 5(b)) may be noted. Interfacial porosity indicating partial bonding in the latter is evident in Fig. 5(b).

Figure 6 indicates significant microstructural modifications (spheroidization) of the matrix brought about by the heat treatment (solutionizing and ageing) of the PDC specimens over that of the as-cast one (Fig. 3(c)). Further, no significant microstructural changes occurred with the soaking duration at the solutionizing and ageing temperatures adopted (Fig. 6(a)–(d)).

Figure 7 delineates the changes in the properties like hardness, density and electrical resistivity of the PDC

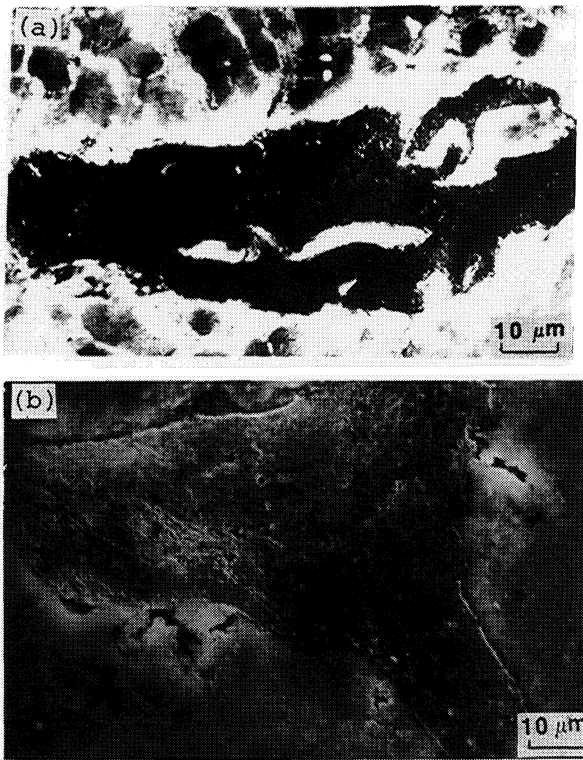


Fig. 5 SEM micrographs showing (a) good graphite/matrix interface and (b) the presence of interfacial porosity in the PDC and GDC composites respectively.

composite after solutionizing and ageing. It may be noted that all the properties of the composite have decreased after the heat treatment (Fig. 7). However, the decrease in the properties was marginal.

IV. Discussion

The perfect shape and soundness of the PDC composite (Fig. 1(a)) indicates the pressure die casting technique to be a promising route towards synthesizing graphitic aluminium alloys into intricate shapes. The intimate contact between the die surface and the melt maintained by the injection pressure during solidification seems to have offered good surface finish to the PDC component. The pressure also led to proper flow of the melt into the farthest and thinnest regions of the die cavity which was responsible for the perfect shape of the PDC castings. Another advantage of the intimate contact in this case was faster rate of heat dissipation from the solidifying melt to the surroundings via the die walls⁽¹⁶⁾ offering higher rate of solidification (Table 2).

The higher rate of solidification in the PDC composite (Table 2) was able to reduce the extent of flotation of the graphite particles and bring about microstructural refinement of the matrix (Fig. 3(a), Table 2). The improved uniformity in the dispersion of graphite in this case (Fig. 1(b), Table 1) was due to the reduction in the extent of flotation of graphite during solidification, the latter was obvious from the relatively less variation in properties of the composite from one region to the other (Table 1).

Better uniformity of the distribution of the dispersoid phase in PDC composites has also been reported in an earlier investigation⁽¹⁹⁾.

Higher hardness, density and lower electrical resistivity of the PDC composite (Table 1) was attributed to the microstructural refinement of the matrix (Fig. 3(a), Table 2) and reduced porosity (Fig. 5(a)). It may be noted that graphite being poorly wettable with molten aluminium contains an envelope of air/gas around it. The apparent density of the graphite particles enveloped in the gases also reduces thereby facilitating their flotation during solidification. Partial breaking of the envelopes is possible due to the stirring action while adding the dispersoid to the melt. As a result, even after solidification, the graphite particles are partially surrounded by the gaseous envelopes under the conditions of gravity die casting. This leads to partial bonding at the graphite/matrix interface in this case (Fig. 5(b)) and thereby allows easy nucleation of cracks during tensile fracture⁽²³⁾. Conversely, sound dispersoid/matrix interface in the PDC composite caused restricted nucleation of such cracks at the interface during tensile loading under identical conditions⁽²³⁾.

The gases entrapped at the interface which appear as porosity (Fig. 5(b)) get squeezed out in the case of PDC composites. At the same time, the solubility of the gases in the melt also increases due to pressure according to Sievert's law⁽²⁴⁾ in the PDC composite. This helped to reduce the interfacial porosity (Fig. 5(a)) making the graphite/matrix interface sound. Since the interface is the most favourable site for the nucleation of porosity due to the poor interaction of graphite with aluminium, a sound interface in the PDC composite directly suggests reduced porosity in this case.

The molten composite enters the die cavity through a narrow ingate at a high velocity under the influence of the injection pressure and strikes the die wall. The possibility of generation of impact, shear and compressive stresses on the graphite particles in the melt cannot be ruled out under the circumstances. These forces might be responsible for the fracture of graphite particles in the matrix (Fig. 3(c)). Microstructural refinement in the crevices of the fractured regions may be attributed to the non availability of space causing restricted growth of the dendrites there (Fig. 3(c)).

Reduction in the thickness of the α Al-cell boundaries of the PDC composite (Table 2) is in agreement with the observations made in an earlier investigation⁽²⁵⁾. Pressure during solidification increases the solid solubility of silicon in aluminium resulting into reduced quantity of eutectic silicon in the interdendritic regions. Further, the microstructural refinement would also have its effect on the silicon particles there. Both may be jointly responsible for the reduction in the thickness of the α Al-cell boundaries in the PDC composite (Table 2).

Significant morphological changes in the microstructural phases such as the spheroidization of eutectic silicon particles (Fig. 6) induced by heat treatment resulted from the dissolution of the constituents at sharp tips and edges due to higher concentration gradient

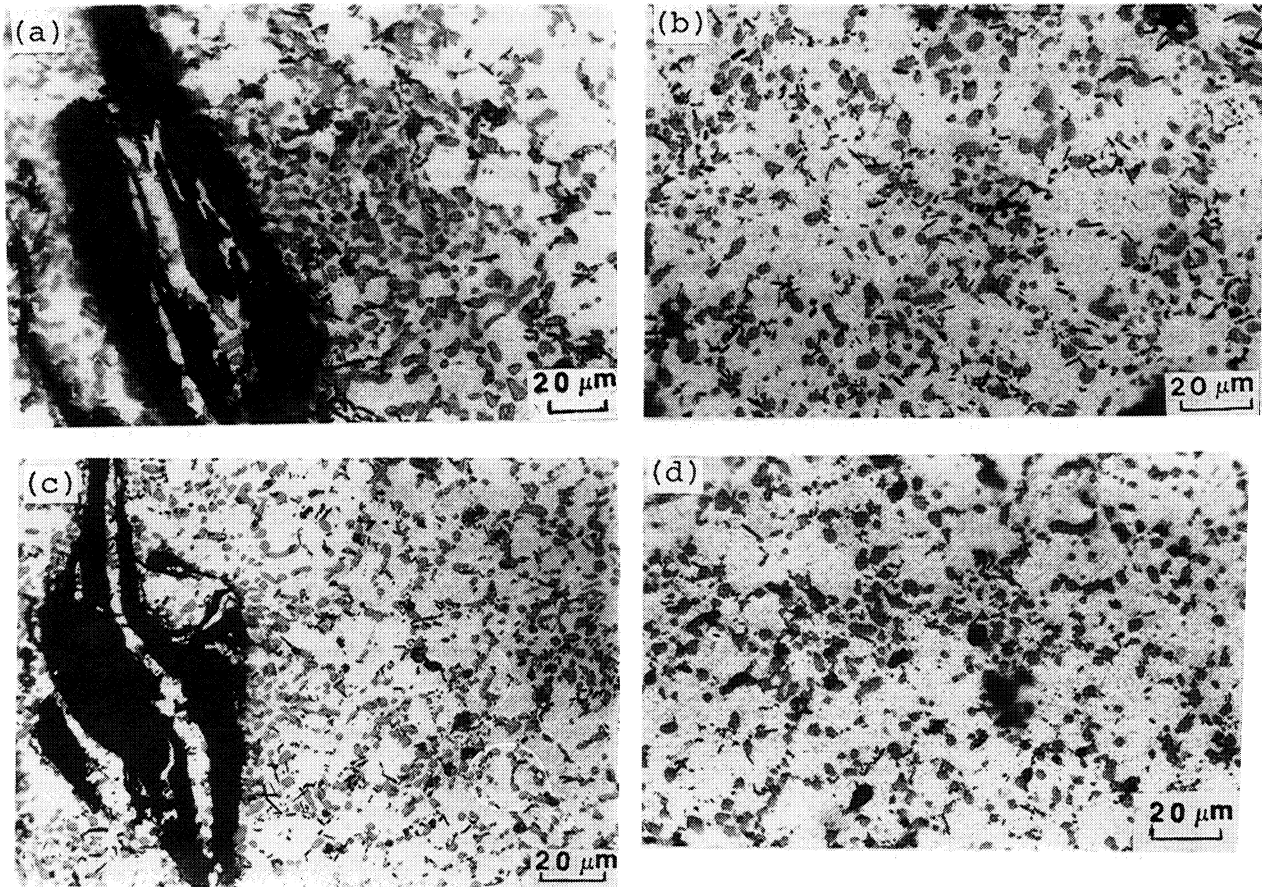


Fig. 6 Morphological modifications of the eutectic silicon particles in the PDC composite after solutionizing at 516°C for (a) 3 h (b) 8 h and ageing at 180°C for (c) 3 h and (d) 8 h.

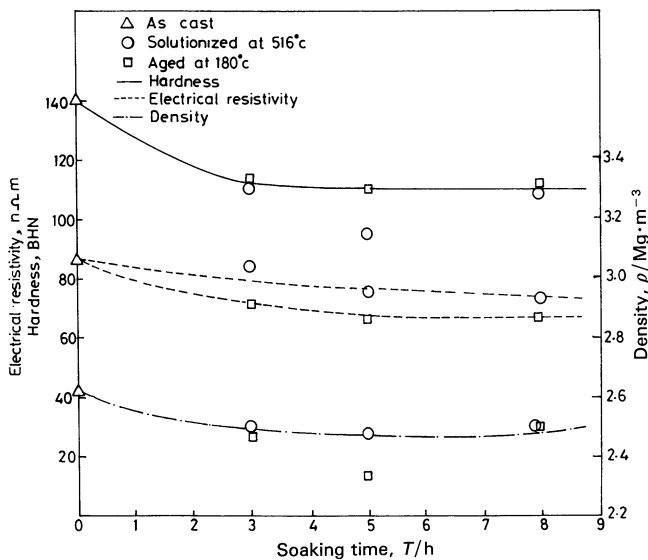


Fig. 7 Effects of soaking duration on the hardness electrical resistivity and density of PDC composite while solutionizing at 516°C and ageing at 180°C.

there⁽²⁶⁾. Such a matrix microstructure has been suggested to be offering good friction and wear characteristics⁽¹⁴⁾.

Reduction in the hardness of the heat treated PDC

composite (Fig. 7) could be attributed to the morphological changes of the microstructural constituents (Fig. 6). Comparable values of density and electrical resistivity of the composite after heat treatment indicate that practically no secondary porosity was generated during heating.

Observations made in this investigation indicate that pressure die casting technique could be a technically viable and promising route for synthesizing graphitic aluminium alloys into intricate shapes. Advantages offered by the technique such as improved physical and mechanical properties of the composite can compensate for the loss in the properties due to the addition of graphite in aluminium at least partially and hence make the process further attractive. Improved dispersoid/matrix interface in this case can help towards more efficient smearing of the graphite particles in tribological applications and allow the dispersoid to play its role of solid lubricant more efficiently⁽¹⁴⁾. Fracture of graphite in the PDC composite could make the process of solid lubrication more effective in view of the increased surface area of the dispersoid which can make contact with the mating surfaces during wear. Further, considerable morphological modifications of the microstructural phases at little loss in properties after heat treatment clearly indicates that there is a tremendous scope of obtaining

a desired matrix microstructure at comparable level of properties. Such a matrix microstructure has been found to be offering good friction and wear properties.

V. Conclusions

(1) Synthesis of the graphitic aluminium-silicon alloy by pressure die casting technique allowed to cast it into intricate shapes. The composite so processed offered a number of advantages such as uniform dispersion of graphite particles, improved wettability of graphite with aluminium in terms of better interface between the two, refined matrix microstructure and better properties.

(2) Heat treatment of the composite brought about significant morphological changes at the cost of insignificant loss in properties. Such a matrix microstructure has been found to offer improved friction and wear properties in an earlier study.

(3) A combination of processing steps such as the dispersion of graphite in a heat treatable variety of aluminium alloy melt, solidification of the composite melt by pressure die casting technique and suitable heat treatment of the castings can help towards improving their physical, mechanical and tribological properties.

REFERENCES

- (1) I. F. Richardson: *The Foundryman*, **82** (1989), 538.
- (2) F. Rana and D. M. Stefanescu: *Met. Trans.*, **20A** (1989), 1564.
- (3) A. Sato and R. Mehrabian: *Met. Trans.*, **7B** (1976), 443.
- (4) K. J. Bhansali and R. Mehrabian: *J. Metals*, **32** (1982), 30.
- (5) S. V. Prasad, P. K. Rohatgi and T. H. Kosel: *Mater. Sci. Eng.*, **80** (1986), 213.
- (6) S. V. Prasad and P. K. Rohatgi: *J. Metals*, **39** (1987), 22.
- (7) B. C. Pai, P. K. Rohatgi and S. Venkatesh: *Wear*, **30** (1974), 117.
- (8) L. Bruni and P. Iguera: *Automo. Eng.*, **3** (1978), 29.
- (9) P. K. Rohatgi and B. C. Pai: *Wear*, **59** (1980), 323.
- (10) F. A. Badia and P. K. Rohatgi: *Trans. Soc. Automo. Eng.*, **77** (1969), 700.
- (11) B. P. Krishnan, N. Raman, K. Narayanaswamy and P. K. Rohatgi: *Wear*, **20** (1980), 205.
- (12) Yu. V. Naidih and G. A. Kolesichenko: *Proshkovaya Met. Akud. Nauk. Ukr. SSR*, **1** (1961), 55 (Brutcher Translation 5940).
- (13) S. K. Biswas and B. N. Pramila Bai: *Wear*, **68** (1981), 347.
- (14) S. Das, S. V. Prasad and T. R. Ramachandran: *Wear*, **133** (1989), 173.
- (15) P. R. Gibson, A. J. Clegg and A. A. Das: *Mater. Sci. Technol.*, **1** (1985), 559.
- (16) A. A. Das and S. Chatterjee: *The Metall. Mater. Technol.*, (1981), 137.
- (17) D. G. Gelderloos and K. R. Karasik: *J. Mater. Sci. Lett.*, **3** (1984), 232.
- (18) S. Sujuki, H. Hino, Y. Miyashita, T. Miyata and Nissan Motor Company: UK Patent No. GB 2112813 A.
- (19) M. R. Madhav, S. Raman, P. K. Rohatgi and M. K. Surappa: *Scri. Met.*, **15** (1981), 1191.
- (20) P. K. Rohatgi, R. Asthana and S. Das: *Int. Met. Rev.*, **31** (1986), 115.
- (21) B. P. Krishnan, M. K. Surappa and P. K. Rohatgi: *J. Mater. Sci.*, **16** (1981), 209.
- (22) M. Droutzy and M. Richard: *Fonderie*, **285** (1969), 500.
- (23) B. K. Prasad, T. K. Dan and P. K. Rohatgi: *J. Mater. Sci. Lett.*, **6** (1987), 1076.
- (24) G. S. Upadhyaya and R. K. Dube: *Problems in Metallurgical, Thermodynamics and Kinetics*, Pergamon Press, New York, (1980), p. 144.
- (25) A. Ya, Shinyaev, A. I. Litvintsev and O. G. Pivkina: *Met. Sci. Heat Treat.*, **25** (1983), 129.
- (26) J. W. Christian: *The Theory of Transformation in Metals and Alloys (Part I): Equilibrium and General Kinetic Theory*, 2nd Edition, Pergamon Press, New York, (1981), p. 169.