Microstructures and properties of Al₂O₃/ Al-AlN composites by Pressureless Infiltration of Al-alloys

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ABSTRACT

Al alloys were infiltrated into Al₂O₃ preforms in N₂ and N₂- 2% H₂ gas mixture in the temperature regime of 900-1200°C. The kinetics of nitridation during infiltration were continuously monitored by recording weight gained during infiltration of the preform. The weight gains that are attributed to the formation of AlN in the matrix were observed to increase with processing temperature. In addition, higher weight gains were recorded in N₂-H₂ as compared to in N₂ atmosphere. Analysis of the composites indicates that controlled matrices of AlN/Al can be formed by selection of appropriate process parameters viz. temperature and atmosphere. Based on this study it has been demonstrated that net shaped metal matrix composites (MMCs), with varying amounts of AlN in the matrix can be fabricated by infiltrating alumina preforms at 900-1000°C in commercial nitrogen. By controlling the preform characteristics, it is possible to fabricate composites with significant variation in microstructural scale, and consequently matrix hardness and elastic modulus.

1 Introduction

Aluminium alloys reinforced with very high volume fractions of ceramic particulates or fibres (> 60%) are evincing much interest for structural applications that require high specific modulus, low thermal expansion coefficient and wear resistance. However, the development of these materials has been hindered by difficulties in fabrication, in particular shaping them for end use applications. The most favoured routes for fabrication of these composites have been through infiltration of molten metal into preformed and porous ceramic bodies. For example, Al-alloys have been successfully infiltrated with hydraulic [1-4] or gas [5-7] pressure into SiC, Al₂O₃ and AlN preforms. However, fabrication of composites through these routes is still limited to relatively simple shapes due to the expensive tooling required for infiltration or machining operations. On the other hand, pressureless infiltration techniques such as reactive melt penetration [8-10] and directed melt infiltration [11-13], though relatively slow processes, have shown much advantage in the ability to form near-net shape composites. Reactive melt penetration [8-10] requires a sacrificial preform, usually silica-based precursor or mullite, that reacts with the molten Al-alloy and acts to drive the infiltration front. The resulting composites show an interpenetrating microstructure of Al₂O₃/Al with volume fractions of Al₂O₃ in the range of 60-75 %. Wider ranges of microstructure have also been demonstrated by infiltrating Al-alloys into preforms of porous silica or SiC/Silica [14,15]. In directed melt infiltration [11-13], the molten Al-alloy reacts with the ambient atmosphere (air, oxygen or nitrogen) to form Al₂O₃/Al or AlN/Al composites with an interpenetrating microstructure. Al₂O₃/Al composites grown freely and into SiC, Al₂O₃ preforms by directed melt oxidation have been well investigated and documented [11-13,16-19]. However, similar studies on AlN/Al composites formed by nitridation of Al-Mg and Al-Sr based alloys are relatively fewer. Creber *et al.* [19] and Aghajanian *et al.* [20, 21] initially reported the pressureless infiltration of Al-Mg alloys into particles of Al₂O₃, SiC, TiB₂ etc. over a range of temperature. They assessed the critical

process parameters to be the alloy composition, specifically, the Mg content in the alloy, process temperature and the nitrogen content in the atmosphere.

Studies on the nitridation behaviour of Al and its alloys are rather limited and related to synthesis of AlN from the Al melt or fine Al particles [22-24]. Early work by Sthapattanonda *et al* [25] on the kinetics of nitridation of Mg and Al indicated that Al did not show any reaction with N₂ until the temperature is raised to 530°C. Lehuuy and Dallaire [26] studied the nitridation of Al-Mg alloys (without preforms) in the range of 900-1300°C. They concluded that critical amounts of Si and Mg in the alloy were essential to form dense AlN/Al composites at reduced process temperatures and time. Similar studies by Scholz and Greil [27], on nitridation of Al alloys and infiltration into fibre preforms at 1000°C, showed that with increasing Mg/Si ratio and decreasing oxygen content, the alloys wetted the fibre preform. With increasing Mg/Si ratio, initially, isolated AlN precipitated in the Al matrix by volume nitridation, followed by a formation of a three-dimensional interconnected AlN/Al composite microstructure and finally rapid nitridation to form a porous, friable AlN. It has been observed that although the AlN/Al composite microstructures mimic that of Al₂O₃/Al systems, they were an order of magnitude finer. Moreover, unlike in the Al₂O₃/Al system, the amounts of AlN formed could be controlled by selection of process temperature and gas composition [28]. Daniel [29] demonstrated that the same Al-Mg alloy could be used to infiltrate Al₂O₃ and SiC preforms and the AlN/Al ratio in the matrix could be varied by controlling the process temperature to yield metal matrices with isolated AlN particles (MMC), predominantly AlN matrix (CMC) and composites with microstructural gradients. Daniel and Murthy [30] studied the effect of temperature, alloy composition and filler material size and chemistry on the microstructure of SiC particulate and fiber reinforced with AlN/Al reaction product and reported the fracture toughness and flexural strength. The fracture toughness of the composites was seen to decrease with increase in process temperature whereas, flexural strength increased

with process temperature. In addition, an increase in particle size from 32-45 μm to 71-160 μm decreased the fracture toughness from 4.8 MPam^{1/2} to 2.9 MPam^{1/2} and, flexural strength decreased from 177 MPa to 125MPa. Aghajanian *et al.* [31] studied the effect of particulate loading on mechanical behaviour of Al₂O₃/Al metal matrix composites made by a pressureless infiltration technique. An increase in reinforcement content resulted in increased Young's modulus and the rate of increase was observed to be well within the bounds of the Hashin-Shtrikman model. The Young's modulus of the highly loaded (56 vol.%) composite was measured as 170 GPa and was nearly thrice the un-reinforced alloy (65 GPa). The measured yield strengths were nominally the same in both tension and compression although, greater ultimate strengths and strain-to-failure were observed in compression than in tension. The composites also displayed a nearly constant fracture toughness for all particulate loadings, with values approaching 20MPam^{1/2} compared to a value of 29MPam^{1/2} for the base alloy.

Systematic studies have so far not been made to study the effect of the amount of AlN formed in the matrices (processed at various temperatures/gas compositions), and its role in the final microstructure and on mechanical properties. The present work attempts to investigate the role of process parameters in fabrication of composites by infiltrating an Al-Mg-Si alloy into alumina preforms. The composites microstructures formed over a temperature range and gas composition have been characterised to further identify processing conditions for the bulk fabrication of composites. The composites fabricated under the optimised conditions have been characterised for their microstructure, matrix hardness and elastic modulus.

2 Experimental Procedure

Thermogravimetric studies in the temperature range of 900-1200°C were carried out to identify the range of microstructures that could be produced. Based on the observations, a more limited

range of temperature (900-1000°C) was employed to produce bulk composites for mechanical property evaluation.

Thermogravimetry

An Al-Mg-Si-Fe alloy (composition given in table 1) was melted in a resistively heated furnace and cast into cylindrical billets of 60 mm. diameter. The billets were homogenised at 550°C for 2 hours to minimise segregation and extruded into 10 mm. rods from which billets of 6.7 mm. diameter and 10 mm. height were machined and ground to 400 grit prior to infiltration experiment. Alumina preforms of 7 mm. diameter and 10 mm. height were made by cold pressing powders with PVA (poly vinyl alcohol) as binder. The green preforms were sintered at 1200°C for 2 hours to achieve a final density of 55% [table 1]. The alloy billet and the preform were placed in a recrystallised alumina tube whose one end was closed and the whole crucible assembly was hung in a *TGA (CAHN TG171, Cerritos, California, USA)* with a sensitivity of 1µg. The reaction chamber was purged with N₂ for 15 minutes and a heating rate of 50°C/min. up to 1000°C and 15°C/min. above 1000°C was employed to reach the processing temperature. The weight gains were recorded in commercially pure N₂ and in N₂-2% H₂ gas flowing at 0.046 lit. /min. After each experiment, the composite was sectioned longitudinally and measured for the infiltration height. The top transverse section of the composite was polished to analyse the matrix structure by optical metallography, X - ray diffraction and microhardness.

Bulk processing

An alloy (table.1) similar to that used for TGA was prepared by melting in an inductively heated furnace and sand cast into ingots. The ingots, after homogenisation at 480 °C for 24 hours, were cut into billets of 45x45x15 mm. and ground to 400 grit prior to infiltration experiments. Preforms of dimension 45 x 50 x 25 mm. were made by uniaxial dry pressing of commercially available alumina powders (Grindwell Norton Ltd., Bangalore and Ceramic Technology Institute, Bangalore) in a hardened steel die. The binder quantity (dextrin, PVA or base metal powder), applied pressure and the sintering temperature were adjusted to obtain preforms with the requisite density as given in table 2. The alloy billet and the preforms were placed in a graphite crucible and the whole assembly was kept in an alumina tube furnace. The tube was evacuated and purged with nitrogen before admitting the reaction gas(i.e. commercially pure N₂) at slightly excess of atmospheric pressure and a flow rate of 250 cc/min. To avoid back diffusion into the tube, the exhaust gas was passed through a column of paraffin oil before exhausting it to the open atmosphere. The furnace was ramped at 4°C/min. from 500°C to the processing temperature of 900°C and held for a period of 6-12 hours depending on the preforms. Preforms with lower particle size were kept for longer infiltration periods to achieve uniform infiltration and the requisite composite height (table 2). To study the effect of process temperature on the properties, composites (23µm, V_f-0.61) were also processed at 950 and 1000°C.

Bulk composite samples were cut randomly, homogenised at 500°C for three hours and quenched in air and their microstructure characterised by optical microscopy. Hardness was measured at loads of 5 gms. (Shimadzu 2000) and the average of ten values were taken as the representative value of the matrix. Young's modulus of the composite was measured on samples of dimensions 5 x 5 x 50 mm by Elastosonic technique. An average of 20 readings taken on three sets of the same composite is reported as the representative value of the composite.

3 Results

3.1 Process kinetics and microstructure evolution

The various stages of the pressureless infiltration and nitridation kinetics of the Al-alloy into Al_2O_3 preforms are shown for 900 and 1200°C in fig 1. All samples showed initial rapid weight gain followed by flattening of the curves at the latter stages of the infiltration process. Examination of preforms after experiment revealed that the infiltration had terminated within the preform in all cases. Interrupted experiments indicated the flattening of weight gain curve was concomitant with termination of infiltration before the billet was totally consumed. In addition, it was observed that reaction of Mg with residual oxygen contributed to a portion of the weight gain and therefore the net weight gains were not entirely due to N_2 pick up. Also, since both the amount of nitride and extent of infiltration were markedly independent variables, the rates of weight gain could not be related to linear infiltration speeds

The microstructures of the composites processed at various temperatures in N_2 are shown in fig.2 (a-d). At lower processing temperature of 900°C (fig.2 a), the microstructure showed alumina particles dispersed in a matrix that was predominantly Al-alloy. With increasing temperature, the AlN particles were observed to precipitate at the reinforcing particle/matrix interface and grow into the matrix. At 1200°C, the matrix was predominantly AlN with isolated aluminium alloy channels distributed in them (fig. 2 d). The XRD spectra revealed the formation of AlN in the composite (fig. 3) only above 1000°C. In addition, a small amount of MgAl_2O_4 (spinel $\leq 1\mu\text{m}$) was also evident in XRD data of composites processed above 1000°C. The spinel was observed to have formed at the interface between the particulate and the matrix and increased as the original interface of the preform with the billet is approached, thereby suggesting that it formed during the period in which the molten alloy matrix was in

contact with the preform. The matrix porosity varied in the composite from a negligible amount at 900°C to a slightly porous (<5%) matrix at 1200°C. The pores in the latter case were primarily associated with the AlN reaction product. The microstructures generated in N₂-2%H₂ atmosphere were similar to those described above. However, a finer distribution of AlN was observed and the formation of a predominantly AlN (> 50%) matrix was observed at lower processing temperatures of 1000°C as compared to 1100°C in N₂ atmosphere.

Hardness measurements were carried out on the matrix : a) very close to the particle and b) at the centre between two particles. The indentations were made so as to cover a very small area near the particle in (a), and an area approximating the inter-particle spacing in (b) (fig 4.). For composites processed in N₂, the matrix hardness at the centre increased from 1.4 GPa for samples processed at 900°C to ~11 GPa for samples processed at 1200°C. The rate of increase in hardness with infiltration temperature was a maximum near~1050°C and there was little change in hardness beyond 1150°C. The measured hardness was higher in matrices processed in N₂-2%H₂ at a given process temperature. In N₂-2%H₂, the matrix hardness at the centre showed a similar relationship with process temperature until 1100°C. However, unlike the N₂ - processed composites, further increase in process temperature resulted in a continued increase in the matrix hardness. At 1200°C, the hardness near the particle had approached the value of 13 GPa, that reported for pure AlN [32].

3.2 Bulk composite fabrication

3.2.1 Effect of particle size and aspect ratio

The microstructures of the composites are shown in fig. 5 (a-c). In coarse particle composites (23-180µm), the matrix structure was homogeneous with negligible matrix and bulk porosity. Also, dispersions of Mg₂Si and free Si were observed in the matrix. With decreasing particle

size ($< 23\mu\text{m}$), the bulk porosity increased and Si was observed to be segregated in the matrix. The microstructural parameters, matrix properties and Young's moduli are given in table 3. The measured Young's modulus indicated a maximum increase of 2.8 times (composite C₁) over that of the base alloy and did not show significant variation with particle size. The composite reinforced with platelets, however, showed a higher Young's modulus and higher scatter as compared to particulate reinforced composite. The matrix hardness values of the composites were much higher than those of the base alloy (63 VHN). In addition, the hardness of the composite with $23\mu\text{m}$ was significantly larger than those with coarse particulate.

3.2.2 Effect of particle volume fraction

The microstructure of the composites, shown in fig. 6(a-d), revealed alumina particles present predominantly as a dispersed phase with few inter particulate contacts in the Al-alloy matrix (fig. 6 a). However, with increased loading, the particles were observed to form increasingly interconnected structures (fig. 6 b-d). The bulk porosity in the composites was observed to vary with the particle loading. In composites with ~36 vol.% alumina, bulk porosity was high and associated with clusters formed due to settling of alumina particles within the molten metal filler. The porosity was negligible in composites with up to ~60% particle loading, but thereupon, isolated pores were found in composites with ~75% loading. The matrix hardness and the Young's modulus increased with particle loading(table 4).

3.2.3 Effect of process temperature

The microstructure (fig.7) showed increased formation of AlN and bulk porosity in the composite with increasing process temperature. The microstructure was slightly inhomogenous with respect to the AlN distribution in the matrix through the thickness due to

residence times at lower processing temperatures, which yield metal-rich matrices. The Young's modulus and matrix hardness of the composites was observed to be strongly dependent on the processing temperature. A 12% increase in Young's modulus was measured in composites processed at 1000°C as compared to those processed at 900°C. The matrix hardness also increased steeply, by up to 2.5 times, over the matrix hardness of composites processed at 900°C(table 5).

4 Discussion

4.1 Processing and Microstructure

The major issues that merit attention in this route of composite processing are : 1) change in nitride content with temperature and formation of spinel at the particle/matrix interface; 2) frequent termination of infiltration within the preform and, 3) porosity in the composites fabricated.

At low temperatures (900-1000°C), the reaction of Al with nitrogen to form AlN is controlled by the diffusion of nitrogen through the surface AlN layer. Nitridation in the absence of a preform has been reported to occur inward into the melt [27] by diffusion from the atmosphere across a surface layer. One must therefore conclude that during infiltration, the amount of nitride formed is governed, in part, by the speed with which the liquid/vapour interface moves into the preform : the greater the speed, the lower the nitrogen pick-up. As the temperature increases ($> 1000^{\circ}\text{C}$) , the influence on the nitridation reaction is expected to be greater than on factors such as viscosity and surface tension which dictate the capillarity induced flow of metal. Also, significant conversion of alloy into AlN has the additional effect of reducing the cross-section available for liquid infiltration thereby reducing the rate of infiltration and increasing the residence time of the interface.

The role of H_2 in the gas ambient and Mg in the alloy on the infiltration and nitridation characteristics of the alloy can be related to the need to getter oxygen in the system. All aluminium surfaces, whether metal or alloy, invariably have a few monolayers thick alumina at the room temperature. The alumina layer on the surface of the molten aluminium obstructs the actual contact between the molten Al and ceramic surface as indicated from the larger scatter in contact angles measured in sessile drop experiments ($90\text{-}167^{\circ}$ at 700°C in Al/ Al_2O_3 system) [33-37]. However, it has been shown that a contact angle of 90° can be obtained by placing the

molten aluminium droplet on sapphire at 700°C such that the liquid-solid interface is free of oxide layer [38]. In Al-Mg alloys, the alumina layer present at room temperature is replaced by porous MgO (above 350°C) that allows molten metal to wet the alumina particles. Furthermore in a nitrogen atmosphere, Mg can reduce the partial pressure of residual oxygen to a value where the alumina layer is no longer stable. In such conditions, the molten metal can wet the alumina particles and infiltrate the preform by capillary motion resulting in composite formation. In flowing nitrogen, as in the present experiments oxygen is constantly replenished in the system. The oxygen thus introduced needs to be continuously gettered to avoid the formation of passivating alumina layer in order to create conditions favourable for infiltration continuation. The observed continuation of infiltration and resultant composite growth thus implies that the high level of Mg in the alloy has been effective in getting the residual oxygen present in the flowing nitrogen atmosphere. This corroborates previous work on infiltration of binary Al-Mg alloys into alumina preforms which had established that the principal role of Mg lay in its ability to remove trace amounts of oxygen in the atmosphere by vapour phase reaction [39]. Addition of H₂ to nitrogen lowers the oxygen partial pressure in the in-coming gas to the value dictated by the H₂O/H₂ equilibrium thereby making the gettering process more effective. It also appears likely that reduction of the partial pressure of oxygen to the equilibrium defined by the dissociation of H₂O, can lead to higher nitride content in composites made in N₂-H₂ as compared to commercial purity N₂.

The reaction between the alloy and alumina to form spinel is thermodynamically favoured at low temperatures since the amount of Mg in equilibrium with Al₂O₃ increases with temperature [37, 40]. However, as shown by others, the reaction is slow below 1000°C [37]. The increasing thickness of the spinel layer on the particulate as one approaches the original melt - preform interface favours the hypothesis that the spinel forms due to prolonged contact between the alumina and the melt.

The termination of infiltration (fig.1) has been attributed to a surface passivation that arises from localised depletion of Mg in the alloy at the surface of the composite [39] and not to a bulk reduction in the Mg content of the alloy. It has been shown that this passivation may be avoided by providing an outlet for Mg vapourisation that is not constrained by the need to supply Mg by diffusion through the alloy channels of the composite. A convenient mechanism to carry out this modification is to use an alloy cross section which is larger than that of the preform and bulk processing was carried out in this manner. Unfortunately, this solution was not applicable for producing thick uniform composites from the present alloy at high temperatures since AlN/Al composite forms between the crucible wall and preform which drastically reduces the effective area available for Mg evaporation. Another problem of growing AlN-rich matrices at high temperatures ($>1000^{\circ}\text{C}$) using the present alloy is that a considerable portion of the preform gets infiltrated to form a metal matrix composite during the transient stage of heating. These features of the process are shown in fig. 8

Porosity in these composites can be distinguished as a) micro pores and b) bulk porosity

Micro porosity is observed in the matrices of composites processed at temperature above 1000°C and is probably associated with the mechanism of nitridation. This phenomenon is also observed in melt oxidation, where it is related to the rapid infiltration along particle surfaces and growth of alumina that can seal off the inter-particle pores before they are infiltrated. Similarly, in the present case, porosity was found largely nearer the AlN reaction product or particle surfaces.

Macro porosity was predominantly observed in bulk processing of composites. In coarse particle composites ($23\text{-}180\mu\text{m}$), residual porosity was negligible since the larger pores in the preform facilitated molten metal penetration. However, in fine particle composites ($\leq 3\mu\text{m}$), the ability of molten metal to penetrate into fine pores goes down due to the increase in viscous drag, thereby leading to un-infiltrated regions that are present as bulk porosity. Also, in

composites processed by infiltrating alumina preforms diluted with the base alloy binder, the melting of the alloy binder at the high processing temperature (900°C) resulted in settling and segregation of alumina particles. Increased bulk porosity in these composites was largely found associated with the segregated alumina particles.

4.2 Elastic modulus of the composites

The measured modulus of all the composites was observed to fall within the Hashin-Shtrikman bounds [41] for two-phase materials as shown in fig. 9. The bounds in the present case were calculated by assuming the elastic properties of the matrix and the reinforcement particles. The Young's modulus and Poisson's ratio of the matrix alloy was measured as 69 GPa and 0.3 respectively while, the elastic properties of the reinforcement phase was estimated from the elastic properties of porous sintered Al_2O_3 as 363 GPa [42]. The assumption of lower modulus for the alumina particles was necessary due to the presence of micro porosity within the fused and calcined particles. With the present assumption of particle elastic properties, the elastic modulus of the composites could be predicted with a higher degree of accuracy.

The elastic moduli of the composites did not show any apparent relationship with the reinforcement particle size. At a given particle size, the elastic moduli are observed to be strongly affected by the reinforcement volume fraction in the matrix. Whenever the modulus approaches the computed lower bounds, it can be observed that there is an increase in porosity as seen from the microstructures of composites with particles size $> 75\mu\text{m}$ or in the volume fraction of 36 %. The moduli of the composites increases with the reinforcement volume fraction such that at volume fractions of $\sim 60\%$ and above, the moduli are higher than the computed average of the two bounds of the model. The sharp increase in modulus can be attributed to the reinforcement phase becoming interconnected in the more compliant alloy

matrix. In composites C₄, with the highest measured modulus of 241 GPa, the microstructure indeed revealed that the alumina particles were largely interconnected. With decrease in reinforcement volume fraction, the moduli begin to fall closer to the lower bound since the continuous phase is now observed to be the compliant matrix alloy. The reduction in modulus is in agreement with the trend observed in aluminium-based metal matrix composites [43]. The above observations also suggest that further increase in modulus could result if greater contiguity and neck formation could be brought about in the preform.

The measured moduli are in good agreement with the values reported by Aghajanian *et. al* in composites processed by similar route [21, 31]. In the present work, it has been shown that in composites with a fixed particle size and volume fraction (23µm, 60%), the modulus increases with processing temperature due to the increased formation of AlN (E~320 GPa) in the matrix (fig. 7). The measured moduli values in that case are observed to lie closer to or above the upper bounds of the calculated modulus. However, the assumed model is not wholly applicable since significant quantities of a third phase (AlN) are present.

4.2 Matrix hardness

The additions of ceramic reinforcements to an aluminium matrix alloy leads to modifications in the matrix properties. The changes in matrix characteristics come about due to the changes in composition of the matrix resulting from interfacial reactions between the matrix and the reinforcement during processing or thermal treatment [44-48]. Also the difference in thermal expansion coefficient between the matrix ($\alpha_{Al \text{ -alloy}} \sim 25 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) and reinforcements ($\alpha_{Alumina} \sim 8 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$) introduces thermal residual stresses and dislocations when any thermal treatment is applied to these materials [40, 41]. The microhardness of the matrix processed at 900°C, was nearly twice that of the matrix alloy and in addition, varied with the reinforcement

particle size (D_i), aspect ratio (D_i/d_i), size distribution, and volume fraction (V). The decrease in interparticle (λ_i) spacing between alumina particles by changing any of the above variables, suggests an enhanced dislocation density in the matrix and thus higher measured hardness. In the present study, the matrix hardness was measured from indents, which approximately covered 60% of the interparticle spacing and thus are more likely to be affected by the stress field surrounding the particles. On the other hand, composites D ($75\mu\text{m}$, $V_f \sim 0.63$), E ($180\mu\text{m}$, $V_f \sim 0.64$) and C₁ ($23\mu\text{m}$, $V_f \sim .37$) with a much larger interparticle spacing indicated slightly lower matrix hardness which probably represents the true matrix hardness of composites processed by this route. The increased matrix hardness can also be attributed to the precipitation of fine isolated AlN ceramic particles alongside intermetallic Mg₂Si and free Si in the matrix. With increased processing temperature, the enhanced formation of AlN in the matrix results in increased matrix hardness. An abrupt increase in hardness (of the order 2) was observed in matrices of composites processed at 1050°C as compared to those processed at 1000°C. Thereupon the increase was more predominant in composites processed in N₂ -2%H₂ than in N₂ and at 1200°C was approximately the hardness of AlN. The abrupt change in hardness at 1050°C coincided with similar changes in the process kinetics (fig. 10.). This change can be attributed to the reported bulk nitridation characteristics of an Al-Mg-Si alloy [27].

Net shape fabrication

The major objective of the present work has been in establishing process conditions for fabrication of bulk metal matrix composites (MMCs) and ceramic matrix composites (CMCs). The understanding gained on the role of process parameters has been utilised to fabricate net shaped MMCs (fig. 11) with a very wide microstructure scale for mechanical property characterisation viz. tensile and bend strength and compact tension (C-T) specimens for fracture toughness studies [49].

CONCLUSIONS

1. Processing of Al_2O_3 / (Al-AlN) composites involves controlling the interplay of infiltration and nitridation characteristics of Al alloys into alumina preforms. At lower temperatures (900-1000°C) composite growth is singularly governed by capillarity, provided sufficient Mg is available at the infiltration front. On the other hand, at higher temperatures (1000-1200°C) composite growth is governed by capillarity and nitridation of the Al-alloy. The micro and macro structures of the composites are also dictated by the process temperature. At lower temperature, the composite was predominantly metal matrix with no significant presence of micro or macro pores. However, processing above 1000°C resulted in composite with $\sim 5\%$ porosity that was to be found associated with AlN rich regions in the matrix.
2. The relation between the process parameters and the final composite microstructure has been determined using an optimised process, bulk metal matrix composites were made to near net shapes for evaluation of mechanical properties.
3. The elastic moduli of the composites are observed to be relatively independent of the reinforcement particle size but strongly dependent on the reinforcement volume fraction and process temperature. Pronounced increases in moduli from 134 GPa to

241 GPa can be gained by increasing the contiguity of the preform or by increasing the AlN content of the matrix.

4. Matrix hardness increases from 1.06 to 1.32 GPa when the particle size is decreased from 180 μ m to 23 μ m owing to greater constraint exerted on plastic deformation with diminishing inter particle spacing. Similar reasons lead to an increase in matrix hardness from 0.9 - 1.75 GPa due to increase in reinforcement volume fraction from 36% to 76%. A much larger increase of up to 2 GPa in the matrix hardness may be effected by increasing the AlN content by increasing the process temperature to 1000°C.

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Figures

Fig 1. a)

Fig. 1 TGA curves indicating composite growth_in a) N_2 and b) N_2 - 2% H_2

Fig 1. b)

Fig. 1 TGA curves indicating composite growth in a) N_2 and b) N_2 - 2% H_2

a)

b)

c)

d)

Fig.2. Microstructure of the composites processed at various temperatures : a) 900°C b)1000°C c) 1100°C and d)1200°C. The matrix has been etched to reveal the presence of AlN. The various phases are A) Alumina reinforcement particles, B) Al alloy matrix and C) AlN

Fig. 3. X-Ray analysis of the composites processed at different temperatures.
The phases present are a) Al_2O_3 , b) Al, c) AlN and d) MgAl_2O_4 .

Fig. 4. Matrix hardness as a function of process temperature in N_2 and $N_2 - 2\%H_2$

a)

b)

c)

Fig. 5. Microstructure of the composites at nominally constant volume fraction (~ 0.6) with variation in particle size : a) $<1\mu\text{m}$ b) $3.7\mu\text{m}$ and c) $180\mu\text{m}$ processed at 900°C

Fig. 6. Microstructure of the composites reinforced with a) 36%, b) 47%, c) 60 and d) 75% calcined alumina (avg. particle size 23 μm) processed at 900°C

Fig. 7. Microstructure of the composites ($23\ \mu\text{m}$, $V_f \sim 0.61$) processed at 1000°C . The matrix shows the increased formation of AlN [in the matrix] upon processing at higher temperatures

a)

b)

c)

Fig. 8) (a) AIN/Al composite formed between the crucible wall and preform, (b) Microstructure of the composite formed during heating to the set-point temperature (1100°C) and, (c) Microstructure of the AIN rich matrix composite formed at the set-point temperature.

Fig. 9 a)

Fig. 9 b)

Fig. 9) Composite modulus as a function of : a) reinforcement particle size and b) volume fraction

Fig. 10. Infiltration and nitridation kinetics of Al-alloy in N_2 and $\text{N}_2-2\% \text{H}_2$ at 1050°C .

Fig. 11 Near net shaped composites fabricated by Pressureless Infiltration for evaluation of mechanical properties

Tables

Table 1 **Composition of the base alloy, gas and alumina powders used for fabricating the composites**

	Composition
Alloy :	
For TGA studies	Mg - 6.1%, Si - 6.5%, Fe - 0.27%, Al - balance
For Bulk fabrication	Mg - 5.5%, Si - 6.6%, Fe - 0.35%, Al - balance
Alumina powder	Al ₂ O ₃ - 99.6%, SiO ₂ - 0.07%, Fe ₂ O ₃ - 0.07%, Na ₂ O - 0.19%, TiO ₂ - trace, Avg. particle size : 37µm
Commercial N₂ Gas	99.5% purity, 4500 ppm O ₂
Forming Gas N₂ - 2% H₂	10 ppm O ₂

Table 2 **Preforms to fabricate Al₂O₃/Al composites**

Preform	Avg. Particle Size, µm	Binder	Sintering Temperature °C/ Time	Density g/cc	Infiltration Temperature °C/ Time
A [‡]	1	dextrin, PVA	1000, 2 hrs.	2.25	900, 14 hrs.
B ₁ [*]	3.79	dextrin, PVA	1000, 2 hrs	2.31	900, 14 hrs.
B ₂ [‡]	3/0.3	dextrin, PVA	1000, 2 hrs.	2.32	900, 14 hrs.
C ₁ [*]	23	base alloy powder			900, 6 hrs.
C ₂ [*]	23	dextrin, PVA	1200, 2 hrs	1.83	900, 10 hrs.
C ₃ [*]	23	dextrin, PVA	1300, 3 hrs.	2.41	900, 12 hrs.
C ₄ [*]	23	dextrin, PVA	1300, 3 hrs.	2.39	950, 12 hrs.
C ₅ [*]	23	dextrin, PVA	1300, 3 hrs.	2.43	1000, 12 hrs.
C ₆ [*]	23	dextrin, PVA	1500, 2 hrs.	2.96	900, 14 hrs.
C ₇ [*]	23	dextrin, PVA	1500, 2 hrs	2.98	900, 14 hrs.
D [*]	75	dextrin, PVA	1550, 3 hrs.	2.47	900, 6 hrs.
E [*]	180	dextrin, PVA	1550, 3 hrs.	2.51	900, 6 hrs.

‡ From CTI, Bangalore, calcined alumina * and fused alumina★ from Grindwell Norton Ltd., Bangalore, India

Table 3 Microstructural parameters and properties as a function of particle size and aspect ratio

Composite	Avg. particle size, μm (D_i)	Volume Fraction (V_i)	Young's Modulus, GPa (E)	Interparticle spacing, μm (λ_i)	Matrix Hardness, at 5 gms. (VHN)
A	< 1	0.56	158	0.80	-
B₁	3.79	0.58	166	1.10	-
B₂	3/0.3	58	163,174	1.27	-
C₃	23	0.61	195	8.9	132
D	75	0.63	193	37.5	103
E	180	0.64	187	95.3	106

Table 4 Microstructural parameters and properties as a function of particle volume fraction

Composite	Avg. particle size, μm (D_i)	Volume Fraction (V_i)	Young's Modulus, GPa (E)	Interparticle spacing, μm (λ_i)	Matrix Hardness at 5 gms. (VHN)
C₁	23	0.36	134	13	90
C₂	23	0.47	147	11.5	114
C₃	23	0.61	195	8.9	132
C₆	23	0.75	241	8.5	160
C₇	23	0.76	233	7.2	174

Table 5 Microstructural parameters and properties as function of processing temperature.

Composite	Avg. particle size, μm (D_i)	Volume Fraction (V_i)	Young's Modulus, GPa (E)	Interparticle spacing, μm (λ_i)	Matrix Hardness at 5 gms. (VHN)
C₃ (900°C)	23	0.61	195	8.9	132
C₄ (950°C)	23	0.61	206	8.6	206
C₅ (1000°C)	23	0.62	219	8.7	330

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