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Comparative Study of Tensile Properties of Cast and Hot Forged Alumina Nanoparticle Reinforced Composites

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***Abstract*—**Particle reinforced Metal Matrix Composite (MMC) succeeds in synergizing the metallic matrix with ceramic particle reinforcements to result in improved strength, particularly at elevated temperatures, but adversely it affects the ductility of the matrix because of agglomeration and porosity. The present study investigates the outcome of tensile properties in a cast and hot forged composite reinforced simultaneously with coarse and fine particles. Nano-sized alumina particles have been generated by milling mixture of aluminum and manganese dioxide powders. Milled particles after drying are added to molten metal and the resulting slurry is cast. The microstructure of the composites shows good distribution of both the size categories of particles without significant clustering. The presence of nanoparticles along with coarser particles in a composite improves both strength and ductility considerably. Delay in debonding of coarser particles to higher stress is due to reduced mismatch in extension caused by increased strain hardening in presence of the nanoparticles. However, higher addition of powder mix beyond a limit results in deterioration of mechanical properties, possibly due to clustering of nanoparticles. The porosity in cast composite generally increases with the increasing addition of powder mix as observed during process and on forging it has got reduced. The base alloy and nanocomposites show improvement in flow stress which could be attributed to lowering of porosity and grain refinement as a consequence of forging.

***Keywords*—**Aluminum, alumina, nanoparticle reinforced composites, porosity.

I. Introduction

METAL matrix with nano-sized ceramic particles reinforced composites has become a major innovation in the field of advanced structural materials. Increasing application of these composites in engineering components has helped to achieve better fuel economy in automobile, aero-space, and space industries. Traditionally, particle-reinforced MMCs have been fabricated by using several processing routes such as powder metallurgy [1], [2] deformation processing and various solidification processing techniques including spray deposition [3]. The cheapest of these composites was prepared by adding reinforcing particles into aluminum alloy melt while stirring, generally containing surface active element magnesium to facilitate entry of poorly wetting particles into the melt [4]. The resulting slurry was cast to the desired shape of engineering components. The development of composite based on the magnesium alloy is weight-saving, and application of magnesium alloys and composites steadily increased in engineering components, particularly in automobiles [5].

The problems normally which are encountered in cast MMCs reinforced by hard ceramic coarser particles, is limiting both strength and ductility [6], due to the difference in deformation characteristics of hard ceramic particles and the surrounding metal matrix. That results in progressively increasing mismatch in extension with application of increasing load during tensile testing, which often leads to early debonding for coarser particles. During the early cast composite developed by using coarse size (~ 80 μm) ceramic particles for the ease of processing and so, ductility used to be very poor. However, the debonding could be delayed by (i) reducing the size of reinforcement to decrease mismatch in extension for a given stress and (ii) increasing the interface shear strength between the particle and the matrix [7], [8].

The incorporation of finer size ceramic particles into molten alloy becomes more difficult due to adverse surface tension of ceramic particles dominating over gravity, since most of the ceramic particles are poorly wetted by the molten matrix alloy [9]. Continuing efforts to find a processing route to fabricate a composite with lower reinforcement size led to emergence of in-situ composites, where particles are not added externally but generated inside the matrix alloy. These particles are generally of sizes less than a micron and the strength of in-situ composites improved significantly but ductility is not always improved [10], but there are some instances of improved ductility as well [11].

The reinforcing particles may be added from outside or generated in-situ. Both these routes have been followed in the context of stir-casting but the resulting nanocomposites are often plagued with large scale clustering, which deteriorates mechanical properties. The nanoparticles have larger surface area and their agglomeration is thermodynamically favorable. There has been intensive effort to break these clusters particularly by application of ultrasonic stirring. The cluster size could be reduced by ultrasonic stirring but clustering could not be totally eliminated [12]-[15]. Forging improves ductility by decreasing porosity and by eliminating cast

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structure by removing segregation, and this effect is counteracted by the debonding of particles on forging and development of voids in between clustered particles [16], [17].

The present study involves solidification synthesis of cast composite based on aluminum alloy reinforced by two size classes of particles: (i) Nanoparticles of alumina are generated by chemical reaction of manganese dioxide with aluminum during high energy milling and also, inside molten alloy and (ii) Coarser alumina particles of submicron size are generated in the melt by internal oxidation of molten aluminum alloy. The objective of developing nanocomposites in this study by stir-casting is to compare the cast and their hot forging effect on microstructure of the composites including particle distribution, porosity and tensile properties.

II. Experiments

In this study, aluminum alloy {(wt.%): 5 Mg and Al (balance)} was used as the matrix material while the mixture of Nano particles may be divided into three broad size classes of 1-10 nm, 10-200 nm and above 200 nm were used as the x wt.% (x = 1, 2, 3 and 4) reinforcements. Nano-sized alumina particles have been generated by milling mixture of aluminum and manganese dioxide powders mixed in ratio of 1:7 by weight, and the particle size has been varied by milling in a vibratory disc mill (RS200, Retsch, Germany) for 450 minutes at 700 rpm and toluene was used as the process control agent. The resultant product has been characterized by Transmission Electron Microscope (TEM) and X-ray for size and chemical nature of particles.

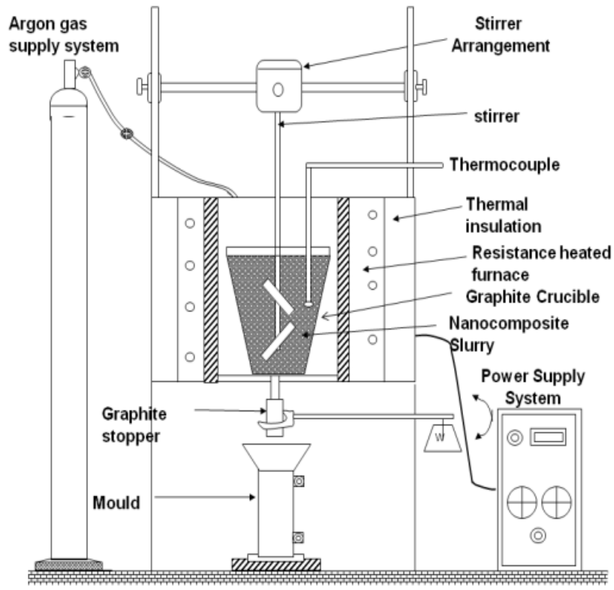

Fig. 1 Schematic diagram showing experimental set-up for stir casting used for solidification processing of cast in-situ nanocomposites and unreinforced base alloys

An argon supply to the graphite crucible inside the muffle, Fig. 1 schematically illustrates the experimental set-up for this casting method. About 750 g of commercially pure aluminum was melted and weighed amounts of milled particles were added and the particles are dispersed in the melt by using coated pitched blade stirrer. Magnesium lump of 5 wt.% was plunged into the melt-particle slurry to improve the wettability of the melt. When the desired time of the stirring elapsed, the speed of stirrer is reduced. After completion of the process steps, the graphite stopper at the bottom of the crucible is removed using the lever to pour the melt-particle slurry into split type graphite coated and preheated permanent steel mould, kept right below the graphite stopper. The mould containing that cast ingot is quenched immediately in water. In order to investigate the microstructure and mechanical properties, the height of the cast ingot was cut into to two parts. First part was for the study of cast properties and the second part was used for forging. Second part of cast ingots was heated in furnace up to a temperature of 500 °C for one hour to homogenize the sample temperature throughout the volume. Thereafter they were forged; the extent of forging in different composites is about 25% in terms of reduction of height and then cooled in air before sampling for various tests. The polished specimen of different cast and forged composites microscopy was conducted using scanning electron microscope (SEM) and TEM. The tensile tests were carried out at ambient temperature for cast and forged nanocomposites and unreinforced alloy.

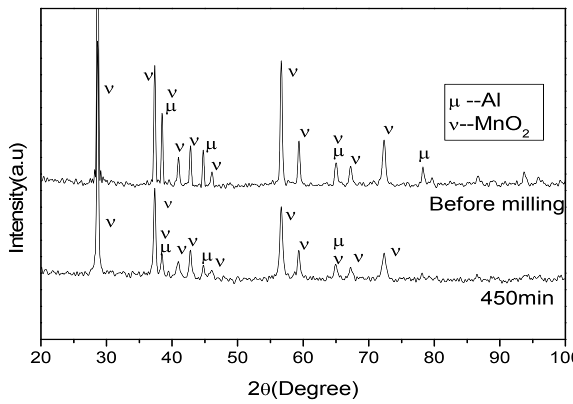

Fig. 2 Comparison of XRD pattern of powder mix before milling and after milling for 450 minute

III. Results and Discussion

The powder mixtures of Al and MnO_2 in the ratio of 1:7 before milling and the particles have a wide range of sizes changing from 0.5 μm to 20 μm. During milling, the oxide particles have broken into smaller sizes but aluminum particles appear fairly flattened by continued beating by the rings inside the milling chamber. The larger particles are possibly aluminum deformed highly during milling but did not easily fragment into lower sizes similar to brittle oxides. During milling there are two distinct roles of the powders as perceived – (i) to generate nano-sized alumina by oxidation at the high energy impact points due to collision between aluminum and manganese dioxide particles and (ii) to keep nano-sized alumina particles physically separate by the presence of aluminum and manganese dioxide powders. The reaction between manganese dioxide and molten alloy releases manganese for alloying with the molten Al-Mg alloy. The choice of manganese dioxide is based on excellent tensile properties obtained in composite developed by addition of this oxide to molten Al-Mg alloy, particularly in respect of ductility [18].

The X-ray diffraction (XRD) patterns, before milling and after milling for 450 minutes, are compared in Fig. 2. Progressively with milling, the peaks of aluminum are reducing in height, but the extent of broadening increases. The height of some of the weak peaks has decreased so much as to disappear by merging with the noise of the XRD pattern, Al_2O_3 has been generated but the amount is so small that it has not been detected by XRD. There may be progressive reduction by aluminum, may be at nano-size level of contact during impact and so, the peaks get weaker.

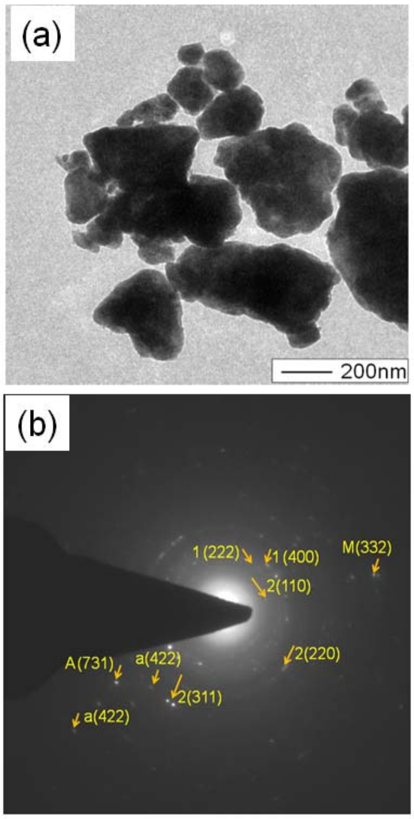

Fig. 3 (a) TEM micrograph of powder mix after 450 min of milling and (b) Selected area electron diffraction patterns showing the sharp ring-spot pattern; 1, 2 and 3 and also the spots A, M and a. is resulting from the presence of γ-Al_2O_3, MnO_2 and aluminum respectively

The bright field transmission electron micrograph of the particles in the powder mix after 450 min of milling has revealed sizes from 6 to 260 nm, as shown in Fig. 3 (a). Selected area electron diffraction (SAD) patterns shown in Fig. 3 (b), shows sharp ring-spot pattern that is the characteristic of the presence of polycrystalline particles resulting in the ring 1, 2 and 3 and also the spots A, M and a. resulting from the presence of γ-Al_2O_3, MnO_2 and aluminum respectively.

The porosity of the cast and forged composites has been determined from density. The results for increasing porosity in cast composites with increasing addition of powder mix after milling for 450 min and at higher particle addition, the porosity in the composite is more are shown in Fig. 4. Since aluminum powder in the powder mix will melt on addition to molten Al-Mg alloy, wt.% of MnO_2 will give the actual measure of reinforcement addition.

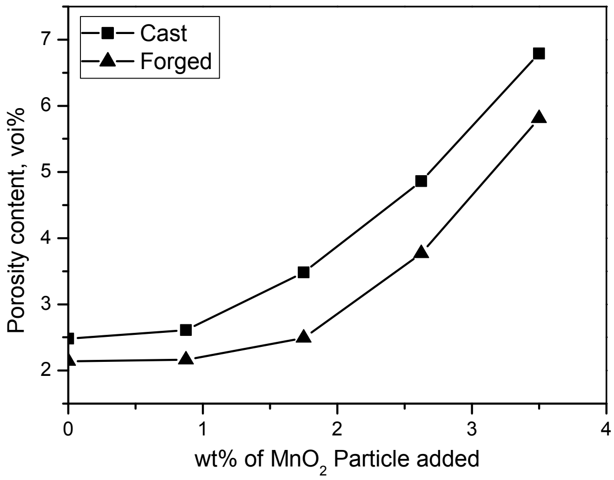

Fig. 4 The variation of porosity content with amount of powder mix in terms of wt.% MnO_2 added from forged composites and their comparison with corresponding cast composites after milling for 450 min

The extent of forging in different composites has been is about 25% in terms of reduction of height. Forging has led to significant reduction in porosity over that in corresponding cast composite particularly at higher particle content is as shown in Fig. 4. When particles are stirred into melt, there is vortex formation at the centre resulting in suction, which helps in transferring particles often with bubbles [19], [20]. It may also happen during solidification as the dissolved gases start nucleating on the heterogeneous surfaces of particles. Often these bubbles are not able to float out rapidly due to increased density because of attached particles and they get entrapped during solidification, enhancing the porosity in cast composite.

A. Cast and Forged Nanocomposites

The powder mixes milled for 450 minutes have been used to develop cast particle reinforced aluminum MMCs, by solidification of slurry obtained by dispersion of externally added milled powder into molten aluminum. The powder mix contains nanoparticles of alumina and MnO_2 apart from particles of aluminum. Aluminum particles may melt on addition to molten aluminum. MnO_2 particles may be reduced to the elemental manganese by aluminum, which gets oxidised to alumina. The elemental manganese is released for alloying the remaining molten aluminum.

To observe the dispersed particles of nanometer sizes the microstructure of the cast and forged composites developed by addition of 2 wt.% powder mix milled for 450 minutes at higher magnifications are shown in Figs. 5 (a) and (b) respectively. One may observe fine particles of different sizes of around 10-40 nm. Fig. 5 (b) shows that individual nanoparticles do not appear to be affected by forging.

The distribution of nano-sized particles could be more clearly observed under TEM and so the thinned specimens of the composites have been examined. Fig. 6 shows typical microstructures of (a) cast and (b) forged composites developed by addition of 2 wt.% of the powder mix milled for 450 minutes. It is clearly observed that the particles are occurring individually although there are some clusters of two or three particles observed sometimes.

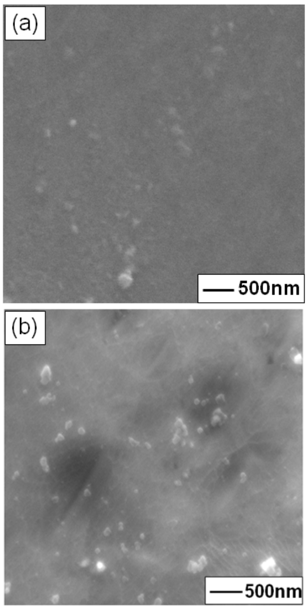

Fig. 5 FESEM micrographs of (a) cast and (b) forged composites developed by addition of 2 wt.% powder mix milled for 450 minute at higher magnifications

Mula et al. [14] and Padhi et al. [15] have synthesized cast nanocomposite of Al with 2 wt.% nano-sized Al_2O_3 (average size ~10 nm) by ultrasonic stirring and the microstructure shows non-uniform distribution of particles. The clustering of nano-alumina has been attributed to pushing of nano-sized Al_2O_3 particles by the solidification front to the regions near the grain boundaries. But particle pushing may push particles to dendrite boundaries and not grain boundaries. After carrying out ultrasonic dispersion of nano-sized SiC particles in molten A356 aluminum alloy, Yang et al [12] have observed that for 2 wt.% addition of nanoparticles in the resulting cast nanocomposite, there extensive clustering. The particle distribution in the cast and forged composites, developed by addition of powder mix, shows individual particles and no significant clustering as shown in Fig. 6. Thus, it is evident that the presence of aluminum and manganese oxides during milling succeeded in keeping the nanoparticles formed during milling separate from each other. Fig. 6 (b) shows that individual nanoparticles do not appear to be affected by forging. But, if these are in clusters, the interface could be damaged as indicated by arrows.

B. Tensile Properties in Cast and Forged Composites

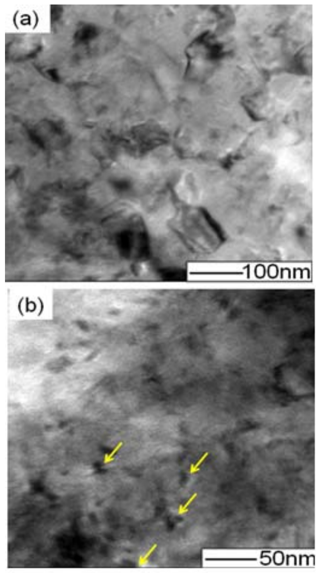

Fig. 6 TEM micrograph of (a) cast and (b) Forged composites developed by addition of 2 wt.% powder mix milled for 450 min

The stress-strain behaviour obtained in the best of the specimens tested for the cast and forged composites developed by addition of different amounts of powder mix as determined by tensile tests are shown in Fig. 7. The stress-strain behaviour obtained in the forged Al-Mg alloy (AM) of composition similar to that of the matrix of the composites is shown in Fig. 7 (a), which indicated improvement of both strength and ductility on forging. The stress-strain behaviour of the composite synthesized by addition of 2 wt.% of powder mix without milling, AM0P2, has been shown in Fig. 7 (b), and it is observed that there is improvement in both strength and ductility but the extent is less than that in the alloy. The stress-strain curve for the composites developed by addition of 1, 2, 3 and 4 wt.% powder mix milled for 450 min, designated as AM4P1, AM4P2, AM4P3, and AM4P4, are shown in Figs. 7 (c), (d), (e), and (f) respectively and the improvement in the strength by forging is more with increasing addition of powder mix till 3 wt.% of powder addition and beyond this level of addition, the extent of improvement is less. But the ductility improves less with increasing addition of powder mix in the composite till 3 wt.% but for the composite with 4 wt.% addition of powder mix there is significant improvement in ductility. But these trends are on the basis of the specimens showing the best tensile properties. However, the tensile strength in composite improves significantly on forging particularly for 3 and 4 wt.% addition of powder while developing the composites. The ductility of the forged composites as measured by percent elongation to fracture indicates an interesting behaviour. Although ductility of the alloy improves on forging but in the composites developed by addition of 1 wt.% powder mix there is a slight decrease in the ductility on forging as shown by Fig. 8 (b). But for composites developed by addition of 2, 3 and 4 wt.% powder mix, there is significant improvement of ductility on forging. However, the ductility of forged composite increases with increasing addition of powder mix till 3 wt.% and thereafter it decreases. Thus, it appears the finer particles are able to overcome the damaging effects of coarser particles in respect of tensile strength. But, between addition of 3 and 4 wt.% something wonderful happens to synergize both the coarser and finer particles to increase both the yield and tensile strengths.

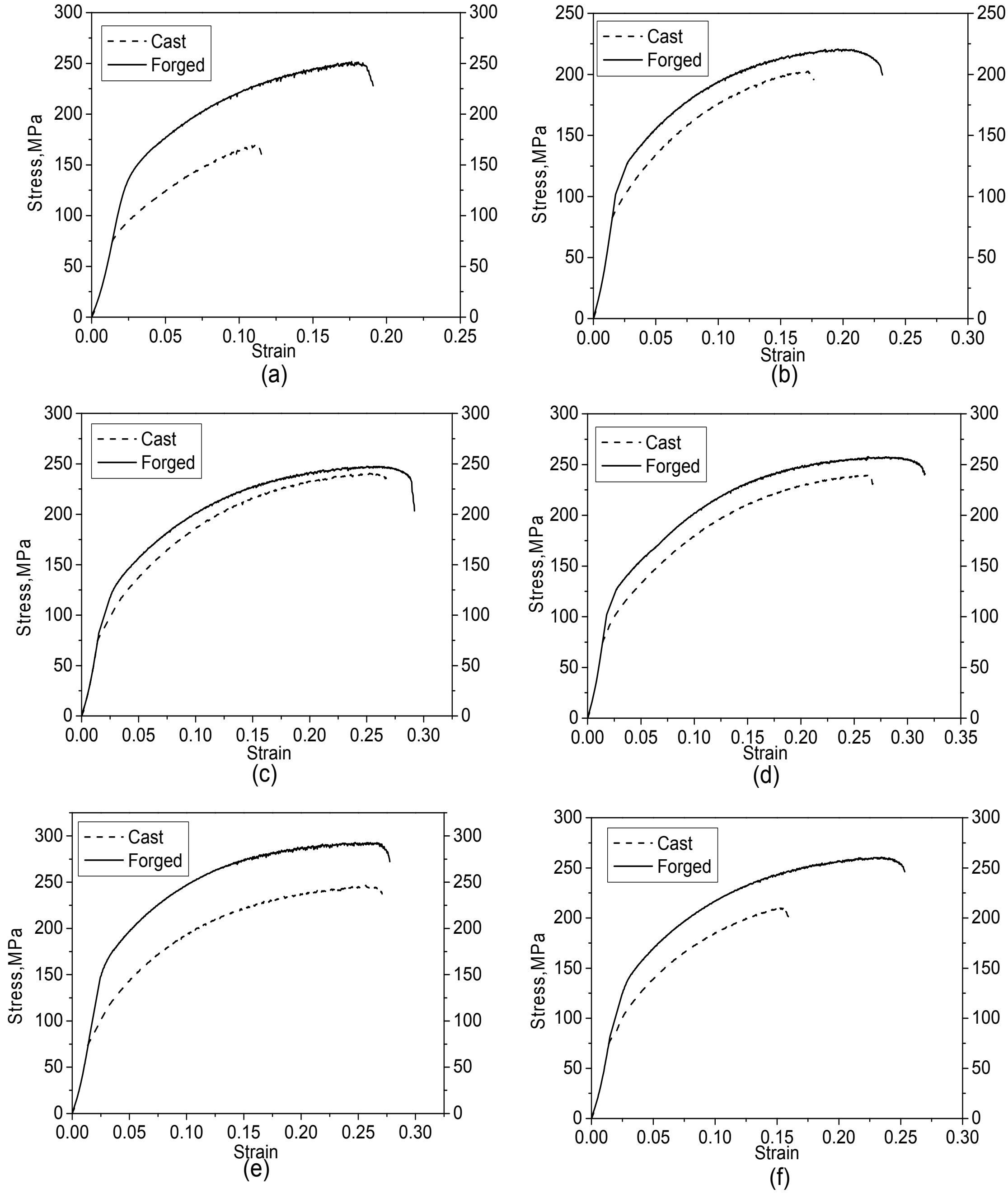

Fig. 7 Comparison of Tensile stress-strain behaviour of cast and forged composites developed by addition of (a) 0 wt.% (AM alloy), (b) 2 wt.% of un-milled powder mix, (c) 1 wt.% of milled powder mix, (d) 2 wt.% of milled powder mix, (e) 3 wt.% of milled powder mix and (f) 4 wt.% of milled powder mix designated as AM, AM0P2, AM4P1, AM4P2, AM4P3 and AM4P4 respectively

Generally, in a composite, loss of strength and ductility is caused by debonding of the particles due to shear stress generated by difference in flow behaviour across the interface between the matrix and the particle. The debonding has been delayed in the composite due to release of manganese in the matrix alloy restricting its flow and so, there is improvement in strength as well as ductility.

The variation of yield strength, tensile strength and percent elongation with increasing addition of powder milled for 450 min have been determined in forged composites from stress-strain behaviour as shown in Figs. 8 (a) and (b) respectively and compared with those observed in their cast counterpart. The yield strength of the forged composite has increased from that of the alloy with 1, 2 and 3 wt.% addition of powder and thereafter, it decreases as shown in Fig. 8 (a). The tensile strength in the forged composite improves over that observed in the forged alloy a little with 1 and 2 wt.% addition of powders but there is significantly more improvement with 3 wt.% addition of powder as shown in Fig. 8 (a). Beyond 3 wt.% addition of powder, the tensile strength decreases similarly as that observed for yield strength.

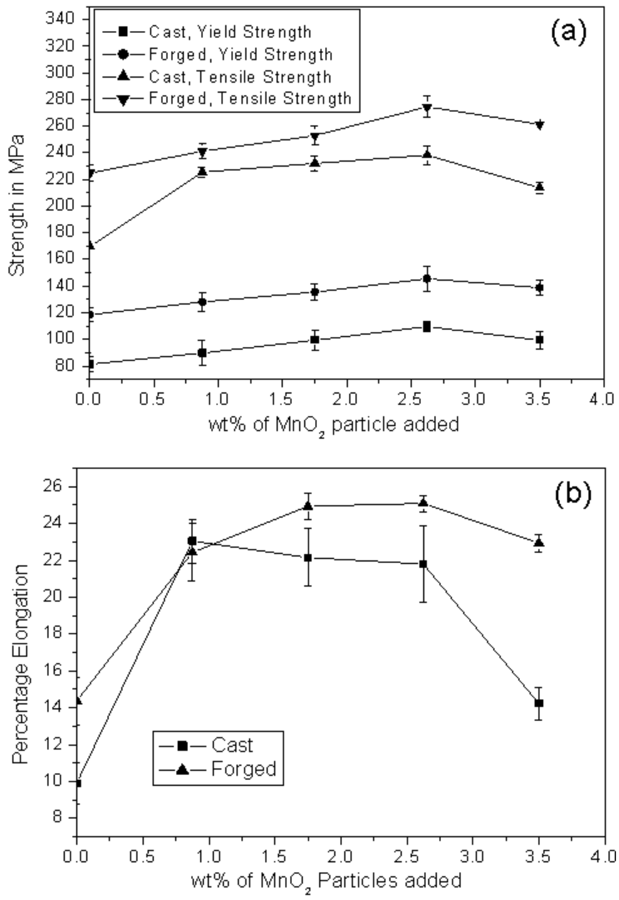

Fig. 8 Comparison of (a) yield strength and tensile strength, and (b) percent elongation in cast and forged composites respectively with increasing addition of powder mix milled for 450 min

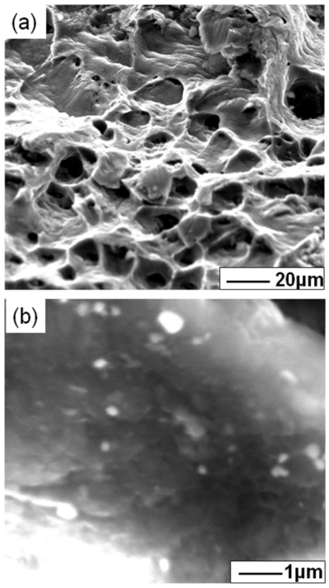

Fig. 9 SEM micrograph of the fractured surfaces of tensile specimens (a) composites developed by addition of 1wt.% powder milled for 450 min and (b) highlighting position of small particles in reference to dimples at higher magnification

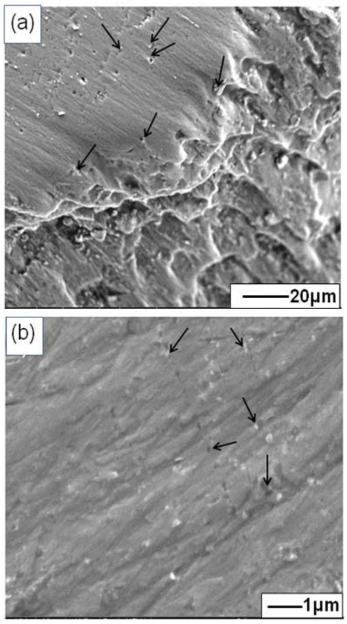

Fig. 10 SEM micrograph of polished longitudinal section of failed tensile specimen of forged composite AM4P2, just below the fracture surface at (a) lower and (b) higher magnifications

The SEM micrographs of the fracture surfaces of the composites shown in Fig. 9 (a) reveal dimpled ductile fracture which is consistent with high ductility observed. At higher magnification under SEM, it is further observed that a number of small sized particles are sticking within the same dimple as shown in Fig. 9 (b). The dimples in Fig. 9 (a) are the voids generated by debonding of particles and grown subsequently with the increasing strain. Possibly the voids are generated mostly by the coarser alumina particles generated during melt processing and are independent of the number of nano-size particles, which are not initiating voids.

One of the broken half of the failed tensile specimen has been sectioned longitudinally along the loading axis to examine the state of void nucleation and particle distribution below the fractured surface and a typical SEM micrograph is shown in Fig. 10. It is observed that the voids have nucleated at submicron sized particles and at large particles. Nano-sized particles appear to have remained well embedded within the matrix.

IV. Conclusion

1) Nano-sized γ-alumina particles have been generated possibly by oxidation at the high energy impact points of aluminum and manganese dioxide particles.
2) Porosity in cast nanocomposites increases with increasing addition of milled powder mix and on forging it has reduced by about 1 to 2%, more in the composite with higher cast porosity. The clusters of particles resulting at higher addition of powder mix often open up in voids during forging, contributing to relatively higher porosity in forged composites rich with powder addition.
3) The microstructure of the composites under TEM shows that the nano-sized particles are mostly occurring individually although there are some clusters of two or three particles observed sometimes.
4) The engineering stress-strain behaviour as observed during tensile tests show that the composites have improved strain hardening compared to the base alloy as indicated by higher load for the same plastic strain in the nanocomposites.
5) There is increasing yield strength and tensile strength in cast and forged nanocomposites developed with increasing addition of powder mix but addition beyond 3 wt.% impairs both yield and tensile strength.
6) Forging improves ductility by decreasing porosity and by eliminating cast structure by removing segregation and this effect is counteracted by debonding of particles on forging and development of voids in between clustered particles. Balance or domination of one or the other is responsible for the observed trends in ductility.
7) The finer nanoparticles are able to overcome the damaging effects of debonding of coarser particles to tensile strength and between 3 and 4 wt.% addition of powders, there is synergy between both the coarser and finer particles to increase both the yield and tensile strengths.
8) The fracture in nanocomposites is preceded by relatively small localized strain possibly indicating that the voids have nucleated by debonding of relatively coarser particles during uniform deformation, leading finally to dimpled fracture surface. Nano-sized particles do not appear to nucleate voids.

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He is main author in many published papers (i) "Processing and Mechanical Properties of Cast Al (Mg, Mn) - Al_2O_3 (MnO_2) Composites Containing Nanoparticles and Larger particles" International Journal of Metallurgical and Materials Transactions A (The Minerals, Metals & Materials society and ASM International 2014), July -2014, Vol:45, Issue:8, PP:3467-3480, (ii) Material Science Journals Elsevier Publications the title of the paper "Development and Mechanical Properties of SiC Reinforced Cast and Extruded Al Based Metal Matrix Composite" has been published in Procedia Materials Science-5 on 2014, PP: 934-943,(iii) Material Science Journals Elsevier Publications the title of the paper "Synthesis and Characterization of γ-Al_2O_3 Nano Powder by Disc Milling of Al and MnO_2 Powder" has been published in Procedia Materials Science-5 on 2014, PP: 416 – 425. The author is a Fellow (No: F-118345-8) of Institution Engineers (India) (2014) and a senior Member (No: LM-39227) of Indian Society for Technical Education (2004).