

FABRICATION AND CHARACTERIZATION OF AL-AL₂O₃ NANO COMPOSITE

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Abstract-Aluminium composite with nano-Al₂O₃ was fabricated using a three step mixing - stir casting process. Its dispersion and mechanical properties such as tensile strength and hardness were investigated and compared with the characteristics of the nano-composite fabricated using conventional method as well as the base matrix alloy. The results had confirmed the successful fabrication of the nano composite with uniform distribution of particles resulting in improved mechanical properties. More than 50 % improvement in the yield and ultimate tensile strength values and more than 60 % improvement in hardness had been observed for the nano composite fabricated using the three step mixing process compared to the base matrix alloy. Index Terms - Al-Al₂O₃ nano composite, Stir casting, Microstructure, Tensile strength, **Hardness**

I. INTRODUCTION

Aluminum matrix based composites reinforced with nano-sized Al_2O_3 particles are group of advanced materials, which are widely used for high performance applications such as automotive, military,

aerospace industries. The composites possess improved physical and mechanical properties compared to the matrix material, such as superior strength to weight ratio, ductility, strength and modulus. They also exhibits low thermal expansion coefficient, high wear resistance and high corrosion resistance [1]. The methods used to fabricate the composites can be broadly classified into (i) solid state methods, (ii) semi-solid state methods and (iii) liquid state methods. The state method includes metallurgy, mechanical alloying and diffusion bonding. These methods generally involve the fabrication of the metal matrix composites (MMC) from blended elemental powders [2]. This allows a wide range of materials to be used as the matrix and reinforcement. Also, the methods are less prone to separation effects and intermetallic phase formations in the fabricated composites . However, solid state methods are relatively more complex, involves more number of steps, expensive, and energy consuming [3]. The matrix and the reinforcement containing both solid and liquid phases are mixed in the semi-solid process (SSP). Examples for SSP compocasting and thixoforging. Semi-solid process allows very large sized components to be fabricated yielding high productivity rates. However, the process is restricted to alloys with longer freezing range and also has the same limitations as the fully liquid mixing methods [4]. Liquid metallurgy technique is the most economical of all the three methods for the production of MMC and can be classified into four categories: pressure infiltration, stir casting, spray deposition and in situ processing. Among these four methods, stir casting process has some important advantages. The advantages are wide selection of materials, better matrixparticle bonding, easier control of matrix structure, simple and inexpensive processing, flexibility and applicability to large quantity production and excellent productivity for near-net shaped components [5]. However, achieving good wettability and homogeneous distribution of the particles are the challenging issues in this method, particularly when nano-sized particles are used. Since, nano particles possess larger surface area and surface energy, the tendency agglomeration will be more compared to the micro-reinforcements. Poor wettability of reinforcement in the melt leads to poor bonding. This may happen due to many reasons such as the surface tension, very large surface area and high interfacial energy of reinforcement particles, presence of oxide on the melt surface and presence of a gas layer on the surface of the ceramic particle.

Mechanical stirring is usually applied in order to mix the particles into the melt, but the particles tend to return to the surface once stirring is stopped. Methods such as heat treatment of the particles before dispersion into the melt to improve the wettability of the reinforcement particles were used. Heat treating the reinforcement particles before mixing within the molten matrix alloy removes the adsorbed gases from the particle surface and hence leads to better wettability [4]. Another problem is achieving uniform distribution of reinforcement particles in the

molten matrix. The particles tend to sink or float on the surface of the molten melt due to the density differences between the reinforcement particles and the matrix alloy. Hence, the dispersion of the ceramic particles will not be uniform and the particles have high tendency for agglomeration and clustering. In addition to mechanical stirring, other techniques are used for introducing particles into the matrix. One of them is injection of the particles into the melt with an inert carrier gas. It has been reported that this technique is helpful in improving the distribution of the reinforcement particles within the melt [6]. In this study, a three step mixing method was used to fabricate aluminium an nanocomposite with Al₂O₃ particles. The dispersion characteristic and mechanical properties of the fabricated nano-composite are analysed and compared with the composite of same material composition fabricated using conventional mixing process. The next section presents the material and experimental procedures used. In section III, the results are presented and discussed. Section IV concludes the paper.

II. MATERIALS AND EXPERIMENTAL PROCEDURES

Aluminium alloy matrix used in this work has the chemical composition of 0.03% Ti, 0.07% Mn, 0.2% Cr, 0.09% Si, 0.2% Fe, 1.4% Cu, 2.3% Mg, 5.7% Zn and 90% Al by weight. 1.5% Al₂O₃ nano powder (99% purity, 20nm average particle size), purchased from US Research Nanomaterials, Inc, Houston, USA is used as reinforcement. The stir casting setup with bottom pouring arrangement which is used for the fabrication of nano-composite is shown in Fig. 1. Melting of the aluminium alloy matrix was carried out in a graphite crucible placed in a resistance furnace.



Fig. 1 Experimental setup used for the fabrication of nanocomposite

The setup consists of the graphite crucible fixed in the middle of the furnace with a hole in the bottom of the crucible for bottom pouring. It also consists of a lever arrangement for opening and closing of the bottom hole. K-type thermocouple and a high frequency stainless steel stirrer system were positioned on the top of the furnace. Injection of the reinforcement particles into the melt was carried out using a stainless steel injection tube and argon gas is used as the inert carrier. The particles were heat treated in the inert atmosphere before mixing into the melt. Provision for this is made in the setup by means of a pre-heating chamber as part of the injection system.

The crucible was charged with the calculated amount of the aluminium alloy and heated up to 700 °C for complete melting of the alloy. The nano-reinforcement was added to the melt following the three-step mixing method given below [7]-[8].

(i) Heat treatment of reinforcement particles at 1100 °C for 10 min in an inert atmosphere.

- (ii) Injection of heat-treated particles within the melt by inert argon gas.
- (iii) Stirring of the melt 10 min. before and after incorporation of particles at the speed of 300 rpm.

Then, the stirrer was turned off and the molten mixture was rested for 5 min at 700 °C. Finally, the lever was swilled for opening the bottom hole and the composite slurry was poured into a preheated cylindrical mould located below the furnace. The samples thus fabricated are used in this investigation.

Transmission Electron Microscopy (TEM) is used to study the distribution of Al₂O₃ nanoparticles in the matrix. The composition of the fabricated nano-composites is verified using Energy Dispersive Spectroscopy (EDS). To investigate the mechanical behaviour of the composites tensile tests were carried out using TUE-C-1000 testing machine according to IS1608. The crosshead speed was set at 0.05s⁻¹ on the round specimens. Each test was repeated four times and mean value was taken. Also, the Brinell hardness tests were conducted on the nano composite fabricated by conventional mixing method and new mixing method and were determined using a ball with 2.5 mm diameter at a load of 10 kg. The average of six measurements is taken as the hardness of the samples.

III RESULT AND DISCUSSION

A. Analysis of Microstructure

Fig. 2 shows TEM image of as-received nano-Al₂O₃ powder. From the figure, the approximate size of the nano particles can be observed as 20 nm. The clusters observed in the figure indicates tendency of the nano powder particles to agglomerate. In conventional method, the reinforcement is preheated and then mixed into the molten metal matrix gradually by using mechanical stirring. It is extremely difficult to break the reinforcement clusters by mechanical stirring alone. Consequently, it is very difficult to eliminate agglomeration and clustering when

the nanocomposite is fabricated using conventional stir casting process.

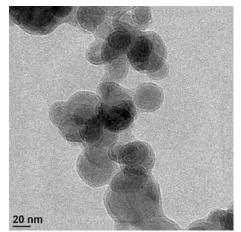


Fig. 2 TEM image of the as-received nano-Al₂O₃ powder

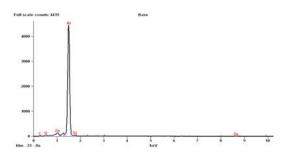


Fig. 3 EDS of the fabricated nano composite

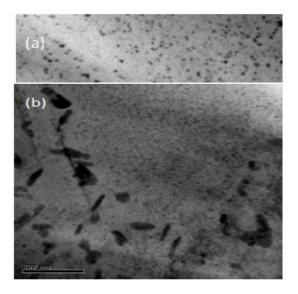


Fig. 4 TEM image of the aluminium nanocomposite fabricated using (a) Three-step mixing method and (b) Conventional method

The EDS plot and TEM micrographs of the composite sample with 1.5 wt.% of nano Al₂O₃, fabricated under the optimized production conditions using three-step mixing method, are shown in Fig. 3 and Fig. 4(a) respectively. The TEM image of the nano composite fabricated using conventional method is shown in Fig. 4(b). In Fig. 4(a), the presence and uniform distribution of the nano particles can be observed which indicates successful fabrication of the nano-composite, whereas, in Fig. 4(b) the distribution of the particles is inhomogeneous. Moreover, the particles are also agglomerated.

B. Analysis of Mechanical Properties

Photograph of the tensile tested specimens are shown in Fig. 5. The flow curves of the matrix material and the nano composites fabricated using the three step mixing method and conventional method are shown in Fig. 6. The flow curve of composites would be greatly influenced by the dislocation density and resistance to plastic flow due to the reinforcement particles. The elongation in the flow curve of nano composite fabricated using the three step method can be attributed to the uniform dispersion of Al₂O₃ particles. In the case of nano composite fabricated using conventional method, the elongation is not significant. Due to the thermal mismatch stress, there is a possibility of increased dislocation density within the matrix which leads to increased local stress.



Fig. 5 Tensile tested specimens

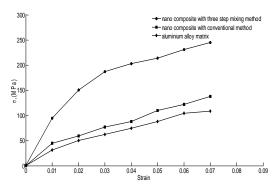


Fig. 6 Flow curve comparison of the matrix alloy and nano-composites

In addition to the strong multidirectional thermal stress at the Al/ Al_2O_3 interface, nano particle size and homogeneous distribution of the Al_2O_3 particles also aids in effective transferring the applied tensile load to the strong Al_2O_3 particulates [9].

Thus increased strength is observed for the composite fabricated using the three step mixing method. With conventional fabrication method it is not possible to achieve homogeneous distribution of particles and proper wetting of the particles in the melt. This leads to more porosity and hence only marginal improvement in strength is observed compared to the matrix alloy.

Hardness tests were performed on a Brinell hardness machine. The hardness values obtained for the base alloy as well as the composites are tabulated in Table 1 along with yield strength and tensile strength. It is

evident from the table that the hardness of the composites is higher than that of the nonreinforced alloy. The hardness increases for nano composite fabricated using conventional as well as the three step mixing method. However, the improvement is significant in the case of nano-composite fabricated using the three step mixing method. The increase in the hardness value may be attributed to the fact that Al₂O₃ particles act as obstacles to the movement of dislocations. The advantage of the nano sized reinforcement is effectively utilized in the case of three-step mixing method, which ensured uniform distribution of the nano particles without agglomeration. Due to inadequate bonding and clustering of nano particles only a marginal improvement is observed in the case of conventional method.

Table I Mechanical properties of the base alloy and nano composites

SI. n o	Materials	Yield streng th (MPa)	Ultimat e tensile strengt h (MPa)	Hardn ess (BHN)
1	Al alloy matrix	71	89	37
2	Al- nano composite with conventio nal mixing method	87	98	46
3	Aluminium nano composite with conventio nal mixing method	112	140	62

IV CONCLUSION

Aluminium composite with nano- Al_2O_3 was fabricated using a three step mixing - stir casting process and its mechanical properties such as tensile strength and hardness were investigated. The characteristics of the nanocomposite thus fabricated, is compared with the nano composite fabricated using conventional method as well as the base matrix alloy. The results had confirmed the successful fabrication of the nano composite with uniform distribution of particles. Further conclusions derived from the analysis are as follows.

- The microstructure obtained using TEM revealed homogeneous distribution of the nano-particles in the case of the three step mixing method, whereas poor incorporation of the particles and particle clustering were observed in the case of nano composite fabricated using conventional stir casting method.
- The nano composites exhibited better yield and ultimate tensile strength compared to the base alloy. The improvement is not significant in the case of conventionally fabricated nano composite whereas, more than 50 % improvement had been observed in the yield and ultimate tensile strength values for the nano composite fabricated using the three step mixing process.
- Due to the homogeneous distribution of the particles without agglomeration, the nano composite fabricated using the three step mixing process exhibited more than 60 % improvement in hardness compared to the base matrix alloy.

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