



# Role of nano-size reinforcement and milling on the synthesis of nano-crystalline aluminium alloy composites by mechanical alloying

Hafeez Ahamed, V. Senthilkumar\*

Department of Production Engineering, National Institute of Technology, Thiruchirappalli-620015, India

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## ABSTRACT

High-energy wet ball milling was successfully employed to synthesize nano-crystalline Al 6063 alloy powders reinforced with 1.3 vol.%Al<sub>2</sub>O<sub>3</sub>, 1.3 vol.%Y<sub>2</sub>O<sub>3</sub> and 0.65 vol.%Al<sub>2</sub>O<sub>3</sub>/0.65 vol.%Y<sub>2</sub>O<sub>3</sub> at nano-size level. In the present study, the crystallite size of the matrix powder particle was affected by the addition of different types of nanoceramic particles separately and in combination keeping total volume percentages of such addition as constant. The nano-composite powders were characterized by SEM, FESEM, HRTEM, XRD, particle size analyzer, EDAX and DTA. Using Williamson–Hall equation, crystallite size and lattice strain of various aluminium composite powders were estimated with broadening of XRD peaks. XRD results showed that the crystallite size of aluminium reached 53 and 37 nm, respectively, after 40 h milling in case of pure Al 6063 and Al 6063/0.65 vol.%Al<sub>2</sub>O<sub>3</sub>/0.65 vol.%Y<sub>2</sub>O<sub>3</sub> nano-composite powder with uniform particle size distribution. HRTEM observation confirmed the nano-crystalline nature of Al 6063/0.65 vol.%Al<sub>2</sub>O<sub>3</sub>/0.65 vol.%Y<sub>2</sub>O<sub>3</sub> milled powder.

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## 1. Introduction

Interest on powder metallurgy (P/M) aluminium metal matrix composites (MMCs) is increasing, since there is a potential field of applications in aerospace, chemical, transportation, structural and automotive industries. P/M aluminium MMCs have improved strength, high elastic modulus, increased wear resistance, low density, and high stiffness over conventional base alloys [1,2]. Reinforcing aluminium matrix with much smaller particles, sub-micron or nano-sized range, is one of the key factor in producing high-performance composites, which yields improved mechanical properties. P/M route employing mechanical alloying (MA) is found to be the most economical method for manufacturing aluminium MMCs [3]. Aluminium MMCs are generally manufactured by means of gas-atomized powders, mixing with reinforcement particles by MA process, die compaction, sintering, hot extrusion and heat treatments, to achieve full densification. Final properties of the MMCs depend on matrix and reinforcement particle properties, bonding between reinforcement and matrix, size and distribution of the reinforcement particles into the aluminium matrix. By MA one can avoid the segregation and agglomeration of the reinforcement particles and also, it provides better homogeneous distribution of

the reinforcement particles into the aluminium or metallic alloy powders [4,5].

Owing to low density, low melting point, high specific strength and thermal conductivity of aluminium, a wide variety of reinforcement particulates such as SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, AlN, Si<sub>3</sub>N<sub>4</sub> TiC, TiO<sub>2</sub>, TiB<sub>2</sub> and graphite have been reinforced into it. Among these particulates, SiC, B<sub>4</sub>C, Al<sub>2</sub>O<sub>3</sub>, TiB<sub>2</sub> additions improved the wear behavior of aluminium MMCs. On the other hand, it has been shown that, nano-crystalline matrices strengthened by nano-sized reinforcement are expected to have much better micro-structural stabilities and performance than nano-crystalline materials [6] because of the concurrence of strengthening by both grain-boundary and nanoparticle reinforcements [7–9]. Generally, smaller particles are known to be less susceptible to fracture than long and elongated particles [10]. Previous studies made by the researchers [11–14] show that several composites having a nanometric grain and reinforcing particles of finer/nano size with a more uniform distribution in the matrix can be synthesized by high-energy ball milling process [15]. The high-energy ball milling offers grain size refinement, making the crystals less susceptible to fracture, and hence nanocrystallization process of aluminium MMCs has been the subject of intensive research in recent years.

During high-energy ball milling, whenever two balls collide with each other or when the balls collide with the inner surface of the vial, some amount of starting powder particles are trapped in between them which cause repeated deformation, rewelding and fragmentation of premixed powders resulting in the forma-

\* Corresponding author. Tel.: +91 431 250 3519; fax: +91 431 250 0133.

E-mail addresses: [hafeez\\_ahamed@rediffmail.com](mailto:hafeez_ahamed@rediffmail.com) (H. Ahamed), [vskumar@nitt.edu](mailto:vskumar@nitt.edu) (V. Senthilkumar).

tion of fine dispersed hard ceramic reinforcement particles in the grain-refined soft matrix. During the milling operation the two essential processes affect the particle characteristics. First, the cold-welding process leads to an increase in average particle size of the composite, and secondly the fragmentation process causes breaking up of composite particles. Steady-state equilibrium is attained when a balance is achieved between these processes after a certain period of milling. During high-energy ball milling for synthesizing of Al matrix reinforced with nano SiC particles, showed reduced crystallite size significantly rather than that of micron size [16]. Al7075 aluminium alloy doped with graphite nano particles [17] and Al-based nano-composites reinforced with multi-walled carbon nanotubes [18] prepared by MA in a high-energy ball milling showed significant increase in the maximum tensile strength and hardness values. Abdoli et al. showed high strength Al–AlN composite compacts synthesized from high-energy ball milling can be cold compacted and sintered for high densities [19]. For high-energy ball milled powders Nayak et al. showed fully dense Al-based composites could be obtained when cold compaction and sintering is followed by forging [20]. Usually high-energy ball milling is carried out under inert gas, liquid or solid process control agent or in dry air atmosphere. Usage of toluene (sulphur free) as a process control agent in high-energy wet ball milling has negligible amount of iron contamination as reported in the earlier work [21].

The main contribution to the strengthening of MMCs, is not only the grain size refinement and reinforcing particle size, but also the particle addition. Several particle parameters which affect the mechanical properties of MMCs, include the volume fraction ( $v_f$ ), size, shape, and distribution of reinforced particles within the metal matrix. The most influential among these parameters is volume fraction [22]. Earlier studies made by the researchers [23–26] showed that the maximum yield strength, ultimate tensile strength and ductility of nano-sized reinforced matrix composites started decreasing when the volume fraction of nano reinforcement addition exceeds 1.5%.

In this paper, an attempt was made to synthesize Al 6063, Al 6063/1.3 vol.%Al<sub>2</sub>O<sub>3</sub>, Al 6063/1.3 vol.%Y<sub>2</sub>O<sub>3</sub> and Al 6063/0.65 vol.%Al<sub>2</sub>O<sub>3</sub>/0.65 vol.%Y<sub>2</sub>O<sub>3</sub> nano composite powders by

**Table 1**

Purity, nominal chemical composition and mesh size used to make 6063 aluminium alloy powder.

Sl. No.	Name of the elements	Purity %	wt.%	Mesh size
1	Chromium	99.9	0.1	–200 to –325
2	Copper	99.7	0.1	–325
3	Iron	99.5	0.35 max	–325
4	Silicon	98.5	0.2–0.6	–325
5	Zinc	99.9	0.1	–325
6	Magnesium	99	0.45–0.9	–200 to –325
7	Manganese	99.8	0.1	–200 to –325
8	Titanium	99	0.1	–200 to –325
9	Aluminium	99.7	Balance	–325

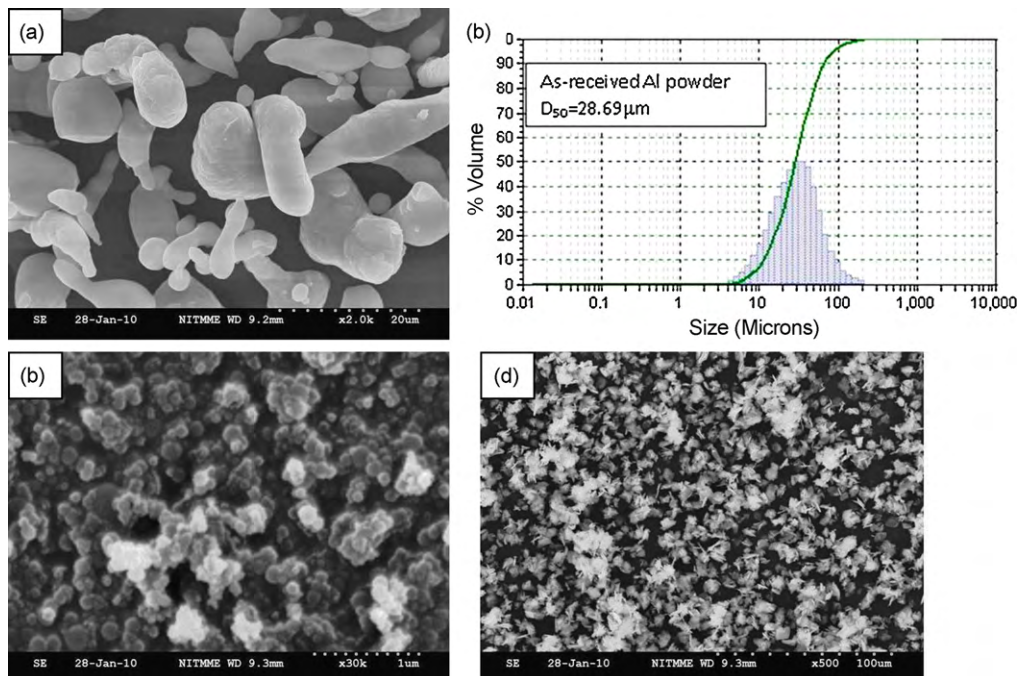
high-energy ball milling. Reports on enhanced hardness, strength and ductility of MMCs due to the presence of meta-stable, finer precipitation, resistance to high-temperature creep and grain refinement properties provide the importance to the selection of thermally stable nano-sized Al<sub>2</sub>O<sub>3</sub> and Y<sub>2</sub>O<sub>3</sub> particulates as reinforcements [27–30]. In the present study, the nano reinforcement of 1.3% volume fraction ( $v_f$ ) is kept constant for all the systems under investigation [24]. The role of hard nano-sized particle reinforcement and milling time on the powder surface morphology, particle shape, size and distribution, crystallite size and lattice strain for different composite powders were investigated. Differential thermal analysis and composition analysis were carried out on the synthesized aluminium composite powders.

## 2. Experimental procedures

### 2.1. Materials

The base material used in the present experimental investigation is Aluminium 6063 (Al 6063), whose nominal chemical composition (wt.%), purity and mesh size of the pure elemental powders supplied by Kemphasol, Mumbai, India is listed in Table 1.

Yttrium oxide (Y<sub>2</sub>O<sub>3</sub>) powder of 99.5% purity with a particulate size range of 25–50 nm, and aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) powder of 99.995% purity with a particulate size range of 40–50 nm supplied by Alfa Aesar, USA, were used as the particulate reinforcements. Volume fraction of 1.3% was used as reinforcement level in the present investigation for the synthesis of all systems of composite mixture.



**Fig. 1.** (a) Morphology of as-received aluminium powder (b) Particle size distribution of as-received aluminium powder. (c) Morphology of as-received nanoAl<sub>2</sub>O<sub>3</sub> powder (d) Morphology of as-received nanoY<sub>2</sub>O<sub>3</sub> powder.

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