

Chapter 5

Fabrication of Aluminium Metal Matrix Nanocomposites: An Overview



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1 Introduction

Improved fuel economy, reduced vehicle emission, increased safety, attractive styling options and better features of comfort and luxury to passengers at competitive cost are the challenges being faced by automotive and aerospace industries these days. This has prompted continuous research on developing light-weight materials with desired and tailored properties. Aluminium and its alloys have become obvious choice due to low cost availability, low density, better strength, corrosion resistance, near-infinite recyclability and casting ease. Aluminium composites in particular have attracted researchers due to properties like high strength and stiffness, increased resistance to wear and corrosion and improved high-temperature performance [6, 74]. Successful automotive applications of Al include transmission components, brake elements, structural parts like chassis, suspension, body parts including bumpers, doors and interiors [36, 79]. Improvement in properties of aluminium is affected by reinforcing with ceramic phase to produce so-called aluminium metal matrix composites (AMMCs). It is because aluminium and ceramics have vastly different properties making it possible to obtain desired property combinations. Large number of MMCs are now commercially being produced and used with major contribution of particulate-reinforced Al composites on mass basis [111]. Different micro/nano-scale particles such as TiB_2 , SiC , TaC , TiC , SiO_2 , TiO_2 , Si_3N_4 , TiN , Al_2O_3 and B_4C are used to produce metal matrix composites via different solid- and liquid-state fabrication routes.

Aluminium-based microcomposites have been successfully utilized in different sectors like ground transportation, aerospace, electronics, recreational goods industries for structural and wear resistance applications. It is also clearly demonstrated

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in several studies in recent past that nanoparticle reinforced composites outperform microcomposites in many respects. For instance, the compressive strength performance of A356 alloy composites reinforced with 20 μm and 50 nm size Al_2O_3 particles was compared [82] and reported 610 MPa strength for 3 wt% alumina nanocomposite as compared to 453 MPa measured for 10 wt% alumina microcomposite. But the critical issue in producing MMNCs is the uniform dispersion of nanoparticles in metal matrix because due to large surface area they tend to form clusters under the influence of Van der Waals force of attraction. During conventional liquid processing, limited wettability of ceramic particles and particle getting pushed by the solidification front leads to their agglomeration near grain boundaries and also formation of pores/voids which seriously affect properties diminishing the beneficial effects of nanoparticles.

Fabrication of nanocomposite is relatively complex on account of very fine size particles which are difficult to handle and introduce uniformly into the matrix. Researchers are continuously trying to promote suitable methods for low-cost defect-free production of nanocomposites with possible industrial scalability. But still most of these efforts are of laboratory scale. It is essential to promote commercial utilization of MMNCs which in turn may help in their developmental efforts. Most of the existing methods of fabricating MMNCs are similar to MMCs but being modified to tackle added challenges posed by nanoparticles. This paper takes an overview of the fabrication methods proposed and published in the literature regarding aluminium nanocomposites while highlighting their prominent features and challenges which may stand useful for students and researchers working in the area of nanocomposite fabrication.

2 Fabrication Methods

Different methods employed for fabricating aluminium metal matrix nanocomposites can be divided into three major groups, viz. solid-state processing, semisolid-state processing and liquid-state processing. Some attempts have been made to combine above principal methods for availing their benefits in the improvement of properties and so can be termed as hybrid methods. Some novel routes for nanocomposite fabrication have also been reported in recent times. Solid- and liquid-state processes are either *ex situ* or *in situ* type depending upon whether the reinforcement particles are generated during the fabrication process or are available for incorporation beforehand. *Ex situ* is when the hard/soft reinforcement particles in solid form are added externally to the solid or liquid metal matrix. *In situ* involves production of reinforcement particles inside the metal matrix during ongoing fabrication process as a result of reaction between the phase elements involved.

2.1 Solid-State Processing

Solid-state processing techniques of nanoparticle reinforced composites are typically based on the conventional powder metallurgy route and its modified versions in terms of powder size modification, compaction techniques, sintering methodologies and secondary process utilized for microstructure refinement. Material processing takes place below solidus temperature, and hence, it reduces undesirable reactions between the matrix and reinforcements which remain in solid state throughout the process.

2.1.1 Powder Metallurgy (PM)

In conventional powder metallurgy method, fine metallic powder and reinforcement particles in required relative amount along with additive lubricants are mixed in mechanical mixture. The mixed powder is then compacted using uniaxial/isostatic cold or hot compaction. Compaction pressure is typically maintained between 400 and 900 MPa considering reinforcement size and area of compact while holding period is varied from few to several minutes. For hot compaction, processing temperature in the range of 500–600 °C and pressure of 250–300 MPa is employed. Green compacts are then sintered usually in inert atmosphere for obtaining dense microstructure. Compact is heated to temperature near to the melting point of matrix material, for instance 550–650 °C for aluminium-based composites. Dense compacted specimens may be further subjected to secondary processes such as extrusion, rolling, heat treatment for microstructure refinement and uniform distribution of reinforcement particles. Powder metallurgy has benefits which include capability of producing near net shaped parts, possibility of incorporation of large volume reinforcement content and ability to manufacture parts in large batches, especially for the automotive sector. PM may also be applied to the matrix/reinforcement system which cannot be realized by liquid processing route. However, it suffers from the limitation on the size of part, industrial scalability, flexibility in tailoring properties, complexity as well as relative cost of manufacturing and high porosity which necessitates secondary processing such as extrusion, rolling and forging. Basic structural arrangement of the process is shown in Fig. 1.

Classical powder metallurgy route was employed [89] to produce Al-MWCNT nanocomposites. Authors used varying CNT contents (0.25–2 wt%) for mixing with pure Al powder of APS 22 µm after giving ultrasonic bath to reinforcements in ethanol for 15–90 min. Mixing of Al and CNT was done in Turbula for 60–600 min followed by uniaxial pressing up to 300 MPa. Green compacts were subjected to sintering at 500–640 °C for 30–90 min. Nanocomposites on examination displayed well dispersed and embedded CNTs for incorporation up to 0.75 wt% but with further addition of CNTs (1 and 2 wt%), large clusters developed. However, for 0.75 wt% CNT, measured values of tensile strength 196 MPa and hardness 50 Hv were 200 and 50% higher than those for unreinforced aluminium.

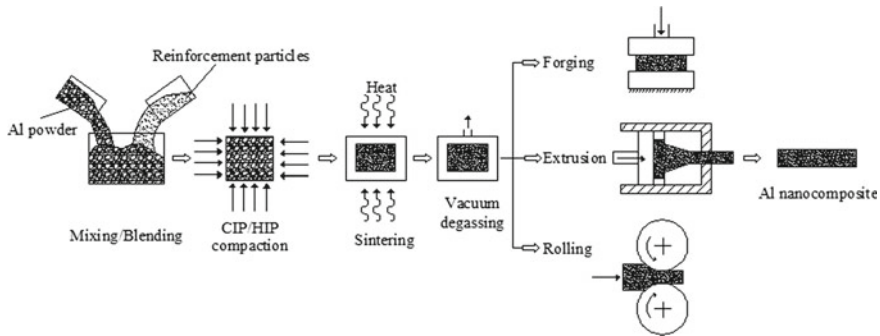


Fig. 1 Powder metallurgy (PM) process

Al-Si₃N₄ nanocomposite was synthesized [64] by powder metallurgy (PM) technique. They mixed 15 nm size Si₃N₄ particles with atomized aluminium powder of 20 μm size, hot pressed the mixture in vacuum at 600 °C and then extruded the compact at 420 °C with 20:1 extrusion ratio. They observed UTS, YS and % elongation values of 1 vol.% Si₃N₄ nanocomposite to be 180 MPa, 143.7 MPa and 17.4 in comparison to 176.1, 94.3 and 14.5 of Al-15vol.%SiC (3.5 μm) microcomposite, respectively, and the strength values to be much higher than respective values of pure aluminium (102.6, 67.8). They associated this significant rise in strength of nanocomposite to the reduced size of reinforcement and Orowan bowing mechanism of interaction between particles and dislocations.

On the similar line, Al/10 vol.% SiC (13 μm) microcomposite and Al-alumina (1–7 wt%, 50 nm) nanocomposites were fabricated [45]. After powder mixing, cold isostatic pressing (CIP) and extrusion produced 15 mm diameter bars. Finally, annealing heat treatment was done for all bars at 350 °C for 2 h. It was found that 31.8 HRF hardness of pure Al increased to 68.4 HRF for 7 vol.% alumina nanocomposite and was 48.6 HRF for 10 vol.% SiC microcomposite. Effect of grain refinement due to nanoparticles was witnessed as Al grain size of 4.6 μm was reduced with increasing content of nanoparticles and reached 1.2 μm for 4 vol.%. But with further addition of nanoparticles, the benefit of grain refinement diminished due to agglomeration of particles. Improvement in TS and UTS is also observed with respect to microcomposite and pure aluminium but at the cost of reduction in % elongation.

Aluminium nanocomposites containing SiC and Al₂O₃ particles of 200 nm and 60 nm size and up to 5 vol.% each were produced [59]. Particles were mechanically mixed with aluminium powder (99.7% pure, 10–100 μm) along with 0.5–1.5 wt% paraffin wax in blender, and the homogeneous mixture was then cold compacted by 500 MPa pressure followed by sintering the compact under argon atmosphere at 600 °C for 100 min. Composite billets were later extruded at 500 °C temperature with extrusion ratio of 2:1. Microhardness values of MMNCs were much higher than aluminium and increased with increasing reinforcement vol.% content and particle size. Al/SiC MMNC exhibited higher hardness and corrosion resistance than Al/Al₂O₃ MMNC and pure aluminium. Effective production of bulk MMNCs

by PM process however displayed particle agglomerations near grain boundaries with size varying between 1 and 5 μm for Al/ Al_2O_3 and 0.5 and 10 μm for Al/SiC MMNCs, respectively.

2.1.2 Mechanical Alloying (MA)

The issue of formation of particle clusters in traditional powder metallurgy can be effectively diminished by employing mechanical alloying technique for enhancing particle dispersion. MA involves high energy ball milling employed for repetitive cold welding, plastic deformation induced fracture and rewelding of powder particles (Fig. 2). Mechanical attrition in presence of nanoparticles accelerates fracture and welding process and on reaching equilibrium, produces equiaxed fine particles [43, 46] with random orientation which are then compacted and sintered to produce bulk parts. Mechanical alloying is unique in terms of forming nanometre size mixtures of immiscible elements, generation of uniformly dispersed intermetallic phases, solid solutions and alloys. Suryanarayana [93] described in detail the results of three mechanically alloyed systems, viz. Al- Al_2O_3 , TiAl-Ti₅Si₃ and MoSi₂-Si₃N₄, and demonstrated that MA is ideal for dispersing large volume fraction reinforcements of nanometre range in variety of metallic matrices. This promising technique of nanocomposite fabrication involving mechanochemical milling of various combinations of oxides, metals and especially carbides get influenced by processing parameters like miller type, ball-powder ratio, speed, time, process control agent (PCA) and milling environment.

Mechanical alloying method was used [75] to study the effect of different fraction (20, 30, 50 vol.%) alumina content as well as particle size variation 50 nm, 150 nm and 5 μm on the dispersion of particles in the aluminium (44 μm APS) powder

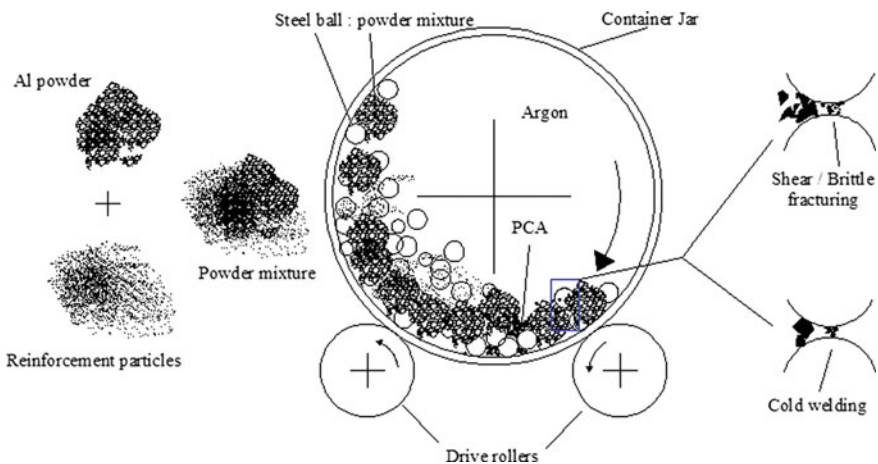


Fig. 2 Schematic diagram of mechanical alloying process

matrix. Al/alumina mixture was milled in SPEX 800 mixer with 0.5–1 wt% stearic acid as PCA under argon atmosphere and 10:1 steel ball–powder ratio. Powder was taken out at 5, 10, 15 and 20 h for characterization study and observed mechanical attrition induced continuous refinement of mixture with milling time. They successfully demonstrated that uniform distribution of particles with content fraction as high as 50 vol.% and size as low as 50 nm can be effectively achieved on milling for 20 h without phase transformation in metastable alumina.

In a research work [19], Al6061 powder with 10 and 20 vol.% ZrO₂ particles of APS 15 nm were mixed and subjected to high energy planetary ball milling at 200 rpm for 40 h in argon-filled container. Mixture was then uniaxially compacted at 600 MPa pressure and 380 °C temperature in 10⁻¹ bar vacuum condition. Microstructural characterization revealed the formation of nanocrystalline aluminium solid solution (50 nm grain size) with homogeneously dispersed ZrO₂ particulates. After hot vacuum compaction, grain size became 80 nm due to recrystallization with finer dispersions. Compression strength of 20 vol.% nanocomposite increased to 1000 MPa. Microhardness (applied load, 1 N) of 10 and 20 vol.% nanocomposites obtained at the end of 40 h milling were measured as 280 HV and 369 HV, respectively.

Similar investigations with high energy milling and consolidation methods like cold or hot isostatic compaction, uniaxial pressing or hydraulic pressure to obtain bulk composites with different material systems such as Al-SiC [20, 95, 100], Al-Al₂O₃ [2, 40, 41, 47, 71], Al-TiC [10, 39–41], Al-BN [29] are reported in the literature. Most of these involved sintering of green compacts in the temperature range of 400–600 °C for durations up to 3 h. Some other works have reported extrusion process been employed after compaction or sintering for obtaining finer and homogeneous microstructure of nanocomposite systems, viz. Al-AlN [99], Al-Al₂O₃ [3, 50], Al-SiC [21, 27, 51], Al-Al₃Mg₂ [76] and Al-TiB₂ [1]. They carried out extrusion at temperature around 500 °C with extrusion ratio up to 20:1. All these investigators have similar findings and common consensus as regards improvement in mechanical properties, hardness rise and refinement of structure with increase in reinforcement volume fraction and milling time.

2.1.3 Cryomilling

In conventional milling of powder mixture, frictional temperatures induce severe recovery and recrystallization of microstructures. Low-temperature processing suppresses such effect in cryomilling which is usually carried out in cryogenic medium like liquid nitrogen. So cryomilling is beneficial in obtaining refined nanocrystalline grain structure more rapidly [101] besides avoiding undesirable chemical reactions between reinforcements and matrix material.

Al-B₄C nanocomposite [107] was produced using cryomilling method. Al5083 alloy powder of APS 40 μm and boron carbide powder of 1–7 μm were mixed together with 0.2–1 wt% stearic acid. Milling of mixture was carried out for 8 h in attritor mill at 180 rpm while maintaining 32:1 ratio of ball to powder. Complete

immersion of powder mixture and milling balls was ensured inside the milling tank filled with liquid nitrogen. The tank was maintained at minus 180 °C temperature using K type thermocouple controlled nitrogen flow. On characterization, authors found uniformly dispersed B₄C particles in Al matrix with slight reduction in size and no particle agglomeration. Clean particle–matrix interface with no voids or cracks around is seen and more importantly no detrimental phases such as Al₃B₄₈C₂, AlB₂₄C₄ and Al₄C₃ were observed in the composite due to cryomilling which otherwise appear in high-temperature processing.

2.1.4 Microwave Sintering (MWS)

Microwave sintering process is advantageous for time and energy saving. It is considered as unique technique which involves instantaneous conversion of electromagnetic energy into thermal energy for efficient volumetric heating of powder compact. It avoids variation of microstructure along thickness of specimen as observed in any differential heating. Microwaves penetrate deep into powder compact, and the generated heat radiates outwards. MWS is found better for fabricating nanocomposites with enhanced properties due to merits like high heating rate, short processing time and homogeneous microstructure [78].

Very recently [63] Al–Al₃N₄ nanocomposite is fabricated by employing microwave sintering route. They took aluminium powder of APS 10 μm and blended with (0, 1, 2, 3 wt% fraction) Al₃N₄ particles of 15–30 nm size in planetary ball mill for 2 h at 200 rpm. The mixed powder was then compacted uniaxially at 50 MPa with 1 min holding time. Compacted specimens were later placed and subjected to heating at 10 °C/min for 30 min in microwave furnace at 550 °C. Authors reported uniformly dispersed silicon nitride particles and increase in UTS, YS, compressive strength and hardness of the microwave sintered specimens.

2.1.5 Spark Plasma Sintering (SPS)

Novel technique called spark plasma sintering is also known as field-assisted sintering (FAS) and plasma-assisted sintering (PAS). It is used for consolidating powders to relatively high density with fine crystalline structure by generating plasma spark between gaps of compacted powders via electric DC pulse discharge. The process is popular for low processing temperature, self-purification of particle surface and short sintering time compared to conventional PM. Moreover, it overcomes the drawbacks of traditional powder metallurgy method such as coarse microstructure, low adhesion, reduced strength and hardness at high temperatures [17]. Effective consolidation of nanostructured Al alloy composites by SPS is done by optimizing process parameters like temperature, applied pressure, heating/cooling rate and cyclic pulse duration.

In a recent research work, [15] Al–SiO₂ nanocomposites were densified containing 0, 3 and 6 wt% SiO₂ (50 nm) powder using spark plasma sintering technique. Under 50 MPa pressure, they raised temperature of the compact to 550 °C with heating rate

of 100 °C/min followed by holding period of 5 min and then cooling rate of 30 °C/min. Authors reported improvement in hardness and 25% rise in the tensile strength of Al-3%SiO₂ composite. But 6 wt% composite exhibited reduction in strength due to agglomeration of particles and intergranular mode of fracture. Several attempts to fabricate nanostructured aluminium composites [12, 52, 69, 80] have been reported in the last decade proving SPS to be effective method of powder compaction.

2.1.6 Friction Stir Processing (FSP)

Friction stir processing, a relatively new process under developmental stage and derived from friction stir welding, is employed, especially for fabricating surface nanocomposites. An exhaustive review paper on FSP has been published by Arora et al. [9]. At first, a groove of desired size is made in the composite and it is then filled by required amount of reinforcement particles. A blunt pin less non-consumable rotating tool is plunged into workpiece to close the groove encapsulating reinforcements inside. Later on, a rotating tool with pin is pushed inside the surface and moved along the groove to spread particles by strong stirring action. The pin can be moved several times along the groove for improving the particle distribution. Successful fabrication of surface composite by FSP depends on processing parameters like tool size, speed and number of passes. Uniform dispersion of particles and surface finish obtained are the main challenges of FSP process. Basic layout of FSP is shown in Fig. 3.

Sharifitabar et al. [87] for instance filled 50 nm size Al₂O₃ particles into groove (1 mm × 2 mm) made in 4 mm thick Al5052-H32 rolled plate and employed FSP to obtain surface nanocomposite. They applied different ratios of tool rotation to tool travel speeds from 8 to 100 rev/mm and tilt angle of pin from 2.5° to 5° to see their effect in producing defect-free stir zones. Hardened steel pin of diameter 5 mm, length 3.7 mm and shoulder size Ø13.6 mm was employed for stirring groove line area after initially closing the groove by pin-less tool. On finding optimum parametric conditions, they investigated the influence of number of passes on mechanical properties and specimen microstructure with or without Al₂O₃ powder. They suggested use of

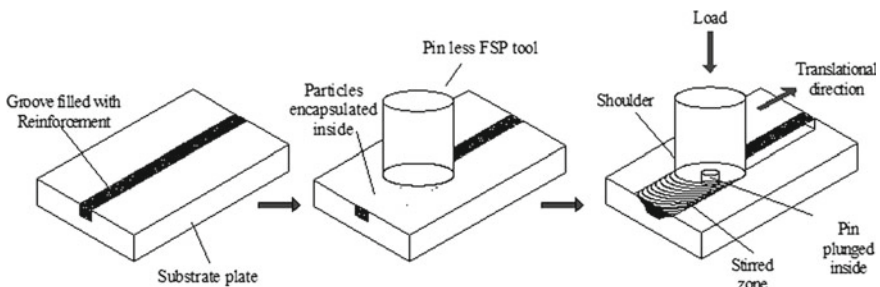


Fig. 3 Friction stir processing method

higher tilt angle of pin and high ratio of tool rotational to travel speed for obtaining defect-free surface composite. Also found that increasing number of passes resulted in improved mechanical properties like tensile and yield strength due to reduced grain size and better dispersed particles. Observations for aluminium nanocomposites like increased hardness, better particle dispersion with number of passes and improved mechanical properties due to refined grains caused by heavy plastic deformation and dynamic recrystallization employing optimized FSP process are reported by several researchers [38, 49, 58, 65, 85].

2.1.7 Accumulative Roll Bonding (ARB)

In this process, metallic sheets after wire brush cleaning to remove oxides and contaminants are stacked over each other with uniformly spread reinforcement particles in between. The stacked sheets are then rolled together to reduce the stacked thickness by up to 50% in single rolling cycle (Fig. 4). The roll bonded sheets are then cut into two pieces and stacked again for rolling in order to reduce the thickness by half. This process is continued several times and examined periodically to obtain uniformly dispersed particles embedded inside the bulk material.

Alizadeh and Paydar [7] fabricated SiC reinforced aluminium nanocomposites by accumulative roll bonding process. They took eight strips of 200 mm × 30 mm × 0.4 mm size made of Al1050 alloy and spread 5 μm size 1 vol.% SiC powder in between each two of them. Before it, all the strips were cleaned with acetone and subjected to wire brush cleaning using 90 mm circumferential diameter brush of 0.35 mm diameter wire at 14 m/s surface speed. Laboratory rolling mill having roll diameter 170 mm and 15 rpm rolling speed was used to reduce the initially

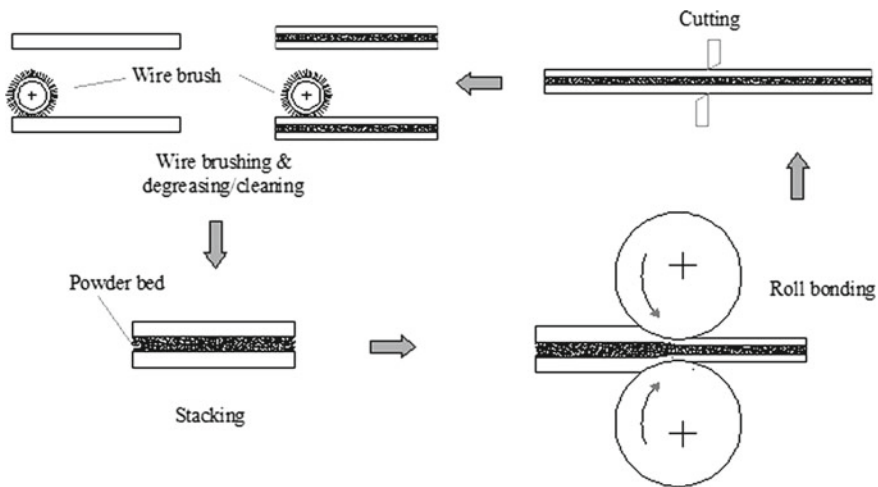


Fig. 4 Accumulative roll bonding (ARB) method

stacked thickness by 66%. Roll bonded strip was cut into two pieces and annealed for an hour at 350 °C. The heat-treated strips after cleaning and wire brushing were stacked again and rolled to reduce thickness by 50%. The process is repeated eight times with no additional reinforcements and heat treatment. The bulk nanostructured composite thus produced after eight accumulative ARB cycles was further examined for microstructure and mechanical properties. Microstructure exhibited uniformly dispersed SiC particles with ultrafine grains of 180 nm size. At the end of 8th ARB cycle, specimen tensile strength increased by 4.1 times to reach 243.3 MPa while elongation increased by 17%.

ARB process was originally developed and introduced in 1998 [81] and later on followed by different researchers for producing aluminium composite materials [90, 98, 106] with remarkably improved material properties on account of grain refinement and dislocation strengthening.

2.1.8 Laser Cladding

This process also termed as laser additive manufacturing is relatively recent method of producing nanocomposites with some reported publications in the last decade. In this effective method, nanocomposite is developed by additively depositing new layer onto substrate by laser melting blown reinforcement powders or already preplaced powder bed. Rapid heating and cooling experienced by deposited powder and substrate develop strong metallurgical bond-forming cladded nanocomposite without changing initial composition and properties of added materials. Different combinations of powder mixtures can be deposited on substrate to develop tailored surface with desired properties like improved strength, hardness, thermal stability, corrosion and wear resistance. Selection of optimized process parameters such as laser power, speed and beam diameter is required to avoid melting of nanoparticles [113]. This process is in the developmental stage, and issues like nanoparticle powder handling and delivery, agglomeration, particle dissolution, health hazards and fabricating 3D parts are being addressed. Better interface bonding between particles and matrix with improved properties of aluminium nanocomposites such Al-Al₂O₃ [32], Al-SiC [16], Al-TiB₂ [104] and Al-TiO₂/B₄C [62] have been demonstrated in research works.

Gu et al. [30] for instance produced TiC reinforced Al10SiMg alloy nanocomposite by laser cladding. YLR-200-SM ytterbium laser with power 200 W and 70 μm spot size was selected to deliver power of 80–140 W with scan rate of 100–150 mm/s. Aluminium substrate was first fixed to levelled platform in sealed chamber filled with argon gas. Mechanically alloyed mixture of TiC (50 nm) and AlSi10Mg (30 μm) powders was deposited onto the substrate by layering mechanism creating powder bed of 30 μm thickness. Specimen CAD data was used to scan powder bed selectively following raster scan pattern. The process was then repeated layer by layer until finally to get the bulk specimen of 20 mm × 10 mm × 6 mm size. Unreinforced specimens were also fabricated by the same process for comparing microstructure and tribo-mechanical properties. It was observed that near full density (98%) composites with hardness of 184.7 HV (20% higher than unreinforced alloy) and increased wear

resistance with friction coefficient as low as 0.28 were successfully developed by laser additive manufacturing process.

2.2 *Semisolid-State Processing*

Semisolid method consists of processing the metallic systems in between solidus and liquidus temperatures wherein the partial solid slurry contains nearly globular grains with 20–60% solid fractions. Due to semisolid condition, it is possible to produce castings having low shrinkage pores and less thermochemical degradation to reinforcements on account of low processing temperatures. Two types of semisolid processing methods, viz. compocasting and thixoforming, are seen for fabricating metal matrix nanocomposites.

2.2.1 **Compocasting**

Compocasting also termed as rheocasting involves producing semisolid slurry by lowering the molten metal temperature during casting process itself to reach between liquidus and solidus line. In this condition, the required amount of preheated reinforcements are added into the slurry and stirred vigorously for uniform incorporation. Compocasting is found suitable for producing aluminium nanocomposite with improved mechanical properties due to refinement of grains as demonstrated in the published literature [37, 42, 83].

In one of the reported works [23], A356 alloy was reinforced with (1, 2, 3 wt%) 50 nm Al_2O_3 particles using compocasting technique. They charged 3 kg of base aluminium alloy in graphite crucible at 630 °C. The melt was then degassed using hexachloroethane tablet and brought down to a temperature range of 590–605 °C to have semisolid melt slurry. Alumina nanoparticles wrapped in aluminium foils and preheated at 200 °C were introduced into melt slurry and stirred at 1000 rpm for 1 min. The slurry on pouring into preheated mould, developed nanocomposite cast specimen. Composite sample exhibited rise in strength, hardness and ductility by 34, 31 and 90%, respectively.

2.2.2 **Thixoforming**

In thixoforming process, a solid feedstock or slug is reheated to semisolid state and formed to shape in partially melted condition with liquid fraction between 30 and 50%. It can form complex near net-shaped components using thixotropic behaviour of materials in the semisolid condition thus saving manufacturing cost. Thixoforming process outperforms traditional casting in terms of reduced porosity because semisolid metal flow is not turbulent but laminar. Although the process is

found implemented for microcomposites, its application for fabricating metal matrix nanocomposites is not given consideration yet.

The only publication to date as per our knowledge is of the fabrication of A356/SiC and A356/TiB₂ nanocomposites [44]. SiC and TiB₂ of 20–30 nm size each were first mixed with A356 powder of APS 60 μm size and compacted to produce green tablets. The composite tablets were introduced into A356 melt to produce 0.8 wt% nanocomposites of SiC and TiB₂ particles each using ultrasonic stir casting process. The cast nanocomposites then were taken for thixoforming process. A356/SiC and A356/TiB₂ billets were placed in thixoforming press and induction heated to 575 °C (approximately 30% liquid fraction) in three steps of 500, 560 and 575 °C. Semisolid slurry was held for 9 min at each step to reduce temperature gradient of billet. The slurry was then forced into preheated (300 °C) tool steel die by applying ram with stroke velocity of 70 mm/s thus producing thixoformed A356/SiC and A356/TiB₂ nanocomposite samples.

2.3 *Liquid-State Processing*

In the liquid metallurgy route, matrix material of composite is heated well above its melting point and then solid reinforcement particles are added from outside into it or created inside the melt by affecting thermochemical reactions between composite constituents. Nanoparticles are dispersed inside the melt by employing techniques like mechanical stirring, electromagnetic stirring or ultrasonic vibrations. Liquid infiltration and disintegrated melt deposition are other liquid processing methods of composite fabrication.

Although liquid processing is widely studied due to relative simplicity and flexibility, it is identified to have some critical issues, especially in case of fabricating nanocomposites [11, 14, 54, 60] as briefed below. As nanoparticles provide large surface area, Van der Waals force of attraction induce them to agglomerate near the grain boundaries and fail to disperse uniformly in the matrix which effectively reduce their strengthening potential. It has been found that the aggregation rate of 20 nm size particles is four orders of magnitude higher than that of 1 μm size particles. Ceramics are found to have limited wettability with liquid aluminium which can create poor interfacial bonding between particles and matrix. Interface debonding occurs during plastic deformation affecting strength and stiffness of the composite. Nanoparticles on account of their large surface area increase the interfacial reactions manifold producing reaction products which at times may be undesirable and spoil composite properties. There is a critical size of nanoparticles below which if they added to the matrix will deteriorate material properties due to negative Hall–Petch effect. Also there is limit for addition of nanoparticles in the liquid melt above which if added form clusters reducing the effectiveness of dislocation hindrance and thereby lower the mechanical strength. However, it is to be noted that nanoparticles on adding homogeneously into the matrix in stable form can dramatically improve the mechanical properties as compared to microcomposites and unreinforced alloys.

Several studies reported so far have shown improvement in hardness, yield strength, compressive strength, wear and corrosion resistance of aluminium alloy because of uniform distribution of nanoparticles and refined microstructure. Nanoparticles are observed to restrict grain growth by acting as obstacles called Zener pinning effect.

2.3.1 Stir Casting

It is a widely used liquid-state processing method for fabricating composites for simplicity and low cost. A typical experimental layout of the method is shown in Fig. 5. In the process, metal alloy ingots are heated for complete liquification inside furnace crucible of the stir casting setup. The reinforcement particles are then dropped manually over the surface of melt which is then stirred for spreading the particles inside. Mechanical stirring rod carries impeller blades attached at the bottom and is dipped inside the melt to stir it before and after particle addition. Stirring action creates vortex inside the melt which helps to suck in particles due to differential pressure gradient. However, as aforementioned, it is usually difficult to distribute particles uniformly inside the melt because of their low wettability and tendency to cluster [34, 91] while the situation aggravates further for nanoparticles due to the increased surface area. Large density difference between particles and alloy can make the situation even more challenging as the particle can start moving to float or sink the moment stirring is stopped. To tackle with these issues, different ways and means have been tried like preheating the particles [92], coating or pretreating them to enhance wettability [13], modified stirring [31, 110], introducing particles with the help of master alloy powder [97] and addition of Mg [86] and K_2TiF_6 salt flux

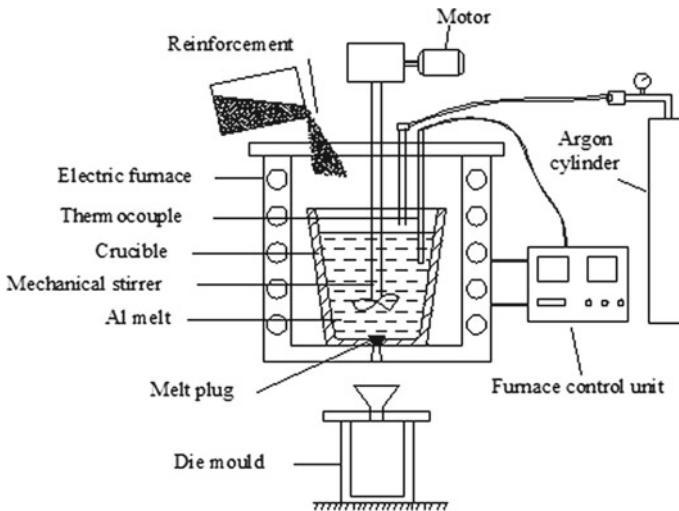


Fig. 5 Stir casting experimental setup

with some reported success. Air entrapment due to stirring can increase porosity in the cast composite. Hence, for better densification efforts like use of melt degassing tablet, particle addition using argon gas-assisted injection, continuous supply of inert gas inside the furnace, bottom pouring and vacuum-assisted casting are employed.

Mazahery and Ostadshabani [66] for instance followed stir casting route to develop A356/alumina nanocomposite reinforced with 0.75–5 vol.% alumina particles. They initially produced master alloy powder by mixing Al (16 μm) and alumina (50 nm) with Al/alumina ratio of 1.67 using ball milling process. A356 ingots weighing 450 g were charged into the furnace for melting at two temperatures, 800 °C and 900 °C. 1 wt% Mg was added for improving wettability. When vortex was created in the melt at 600 rpm stirring speed, the master alloy powder (1 g) packed in aluminium foil packets were dropped into melt one at every 20 s till the required volume fraction addition is achieved. Stirring was continued for 15 min while continuously passing argon gas in the crucible. Slurry was then poured into CI mould to cast composite specimen. Authors observed reasonable distribution of particles inside the matrix with few agglomeration sites. Tensile strength and compressive strength improved initially, and highest values were measured for 1.5 and 2.5 vol.% nanocomposites, respectively, but declined further. Porosity increased slightly with increase in nanoparticle addition. Moreover, % elongation decreased and reached to almost half for 5 vol.% composite.

2.3.2 Electromagnetic Stir Casting

Electromagnetic stirring a novel method also known as induction heating is useful for grain refinement and homogeneous addition of nanoparticles into the metallic melts by means of external magnetic field [26, 88]. In this process, electric current is passed through induction coils fixed around the crucible which create alternating magnetic field introducing Lorentz forces in the melt. These forces are found to generate vortex type flow pattern and strong stirring action to disperse nanoparticles by breaking their clusters. The process parameters like stirring time, electromagnetic frequency, temperature and current intensity are controlled for best results.

Yu [108] produced A356/SiC nanocomposite using electromagnetic stirring. He used induction heating unit which could convert 50–60 Hz, 3-phase power into single-phase high-frequency output. He charged 198 g A356 ingots into crucible and raised the temperature of melt to 750–800 °C. After adding 2 wt% SiC nanoparticles (50–60 nm) using aluminium foil capsules, the melt was treated to electromagnetic stirring with 60 Hz output frequency for 60 min. 2 wt% Mg was added for improving wettability. Uniform dispersion of particles with 22% rise in hardness of fabricated nanocomposite was observed. Successful preparation of Al/Al₂O₃ nanocomposite [53] has been reported recently.

2.3.3 Ultrasonic Stir Casting

Ultrasonic stir casting is observed as the most effective method for uniform distribution of very fine particles by breaking their clusters in melt, refined microstructure and improvement in wettability, especially of ceramics in aluminium [55, 61, 103]. It involves treating nanocomposite melt by ultrasonic waves with approximate frequency of 20 kHz using high power density ultrasound (100 W/cm^2). Typical schematic of ultrasonic stir casting setup is shown in Fig. 6. Sonotrode made of titanium or niobium alloy on account of high temperature stability is dipped in the melt to pass in high-frequency ultrasonic waves. It gives rise to two principal effects in the melt called as cavitation and acoustic streaming. During negative part of the ultrasonic wave, air bubbles are created in the melt under tensile stress. Under positive cycle, bubbles shrink under compressive stress of wave. Air entrapped in voids of clusters is believed to provide nucleation sites for bubble growth. On reaching critical size, bubbles burst creating local microsites of very high temperatures ($5000 \text{ }^\circ\text{C}$) and pressures (1000 atm) called hot spots. This effect of formation and collapse of transient hot spots is called as cavitation effect which can clean the particle surface for improved wettability. Due to high pressure gradient in several small places in the bulk melt, strong macroscopic flow circulation effect is created called acoustic streaming which help in dispersing loose particles. Moreover, as the melt is not agitated, air entrapment is diminished. This promising technique however suffers from the fact that very large volume of melt cannot be treated because of the need of corresponding ultrasonic high power source which limits its industrial scalability. Ultrasonic treatment of continuously flowing liquid can be seen as a possible solution for scaling up. But still the fact of particle movement and settling in the melt on account of density difference after stopping of ultrasonication prevails and can lead to marginal porosity.

Ultrasonic vibration-assisted stir casting process is employed for fabricating several aluminium-based nanocomposites reinforced with SiC, Al_2O_3 , B_4C , TiB_2 ,

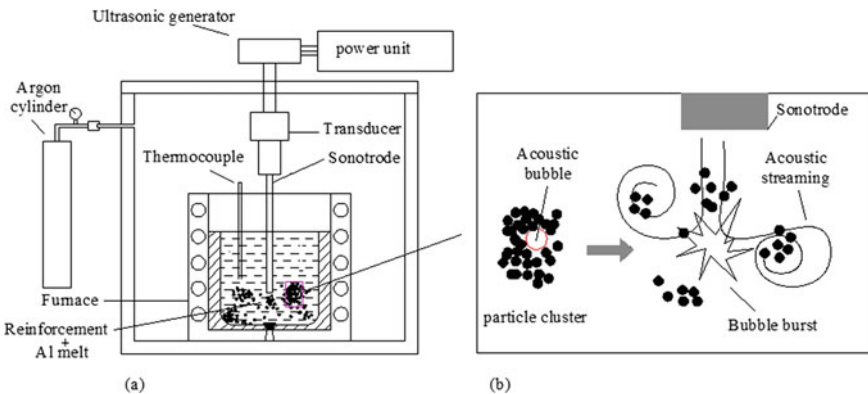


Fig. 6 a Schematic of ultrasonic stir casting setup; b mechanism of particle deagglomeration

SiO₂, GNPs and h-BN [5, 18, 24, 33, 48, 56, 68, 84]. Researchers have reported superior particle incorporation, wettability, improved densification, grain refinement and mechanical properties, for instance 92% rise in hardness for just 2% Al₂O₃ [68] and 134% enhancement in tensile strength for 5 vol.% GNP [5] incorporation. In a research work by Poovazhagan and Kalaichelvan [72], the effectiveness of ultrasonication treatment was exhibited on characterizing the fabricated AA6061-SiC nanocomposites. AA6061 alloy ingots weighing 800 g were melted in graphite coated SS crucible at 680 °C and chocked for 10 min. Mechanical stirring was performed before and after adding SiC particles in required volume fraction. Then, titanium sonotrode was dipped into the melt for performing ultrasonication for 45 min. Ultrasonic generator with 2 kW power producing 20 kHz frequency was used for melt treatment. After ultrasonic treatment, the melt temperature was again raised to 800 °C for improving its fluidity and poured in preheated steel die for casting to shape. Different nanocomposites containing 0.3–1.5 vol.% SiC were produced the same way. In microstructural investigation, EDS elemental mapping images revealed very good dispersion of particles. Tensile strength of just 1.25 vol.% composite increased by more than 100% while 1.5 vol.% composite displayed 74% rise in BHN value.

2.3.4 Liquid Infiltration

In liquid infiltration method, liquid metal is incorporated into the preformed porous solid of reinforcement under either the influence of applied pressure or allowed to seep in with time without external pressure. The slurry consisting of reinforcements and liquid binder carrier is formed to the required shape under pressure. Thereafter, preform is heat treated and dried for its dimensional stability. During liquid infiltration process, preform is placed into die and liquid metal is poured over it. Melt is then forced to infiltrate the preform by applying ram pressure. Liquid infiltration process is also referred as squeeze casting and may take the form wherein the composite slurry of metal and reinforcements is squeezed into the die under pressure. Contact angle between melt and nanoparticle is required to be less than 90° to ensure wetting. This process is preferred for being inexpensive and allows incorporation of as high as 50% nanoparticle fraction into the metal matrix. However, it takes long time for casting, especially in case of pressureless infiltration [116]. Aluminium nanocomposites reinforced with SiC, CNT and CuO [25, 96, 112] have been produced by squeeze casting method.

Geng et al. [28] produced hybrid nanocomposite of Al2024 alloy reinforced with SiC whiskers (20 vol.%) and SiC nanoparticles (2, 5, 7 vol.%, 35 nm) by squeeze casting method. They initially prepared homogeneous slurry of whiskers and fine particles of SiC mixed with alcohol by mechanical blending and dried to get solid hybrid preform. Alloy temperature was raised to 800 °C for melting while preform heated to 500 °C. Thereafter, molten alloy was forced into reinforcement preform at low infiltration pressure of 2 MPa and later solidified under pressure of 50 MPa. Tensile strength and elastic modulus of (20% SiC_w + 7 vol.% SiC_{np}) nanocomposite

were reported to be 620 MPa and 127 GPa, respectively, as compared to corresponding values of 206 MPa and 70 GPa for Al2024 alloy. But % elongation of alloy reduced from 17 to very low value of 0.77 due to reinforcement by this method.

2.3.5 Disintegrated Melt Deposition (DMD) and Spray Co-deposition

In disintegrated melt deposition process, superheated and stirred mixture of metal and reinforcement is allowed to fall under gravity from crucible through small nozzle into a chamber filled with inert gas. The falling slurry is disintegrated by two argon gas jets arranged normal to the metal flow which then solidifies on a metallic substrate (Fig. 7). Despite process being suitable for both Al and Mg alloys, it is specifically adopted for fabricating Mg-based composites. It is because Mg is highly oxidizable and DMD process reduces formation of oxides in the casting. The cast specimen obtained by this process is usually extruded in order to reduce porosity.

In a reported research work, Hemanth [35] (102) produced LM13-ZrO₂ (50–80 nm, 3–15 wt%) nanocomposites by DMD process. The preheated reinforcement particles (200 °C) were added to the superheated (720 °C) LM13 alloy melt and stirred

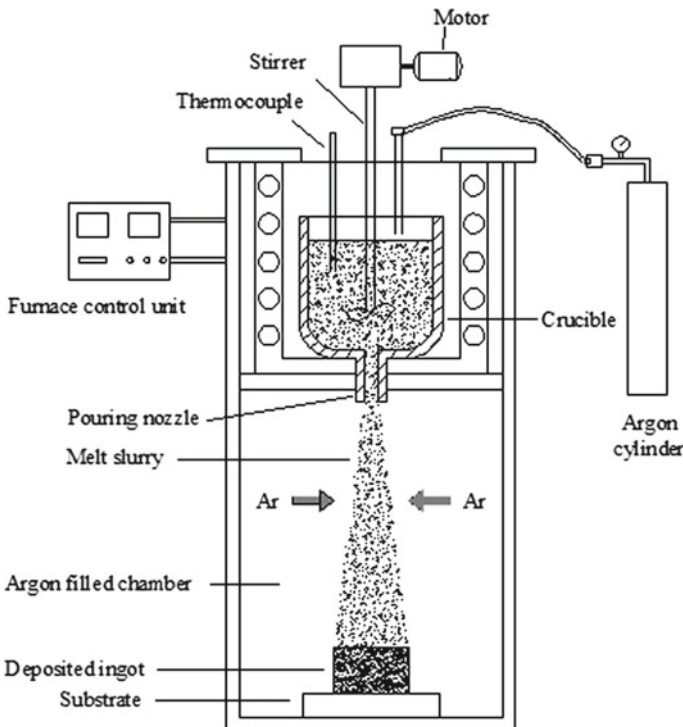


Fig. 7 Typical experimental setup of disintegrated melt deposition process

at 450 rpm speed for better dispersion. Fabricated nanocomposites were extruded by hydraulic press at 200 °C in order to refine microstructure. On characterization, they revealed much refined grain structure, uniformly distributed particles and minimal porosity. Besides, significant rise in hardness (128 Hv) and UTS (258 MPa) was seen for 12 wt% nanocomposite in comparison to 98 Hv and 170 MPa values measured for unreinforced LM13 alloy produced by the same method. Spray co-deposition [115] is a method close to DMD process. In this technique, molten metal stream is allowed to fall into inert chamber and is incorporated with reinforcement particles sprayed onto it through jets of argon gas while simultaneously disintegrating the falling mass. Layers of fallen metal droplets on substrate create composite material after solidification.

2.4 Hybrid/Combination Processing

Different processes available for nanocomposite fabrication are sometimes combined to draw in benefits of individual process. Such approach can improve product quality as no single process is perfect in all respects.

Some researches have combined mechanical alloying technique with liquid metallurgy route to fabricate AMMNCs [4, 67, 94, 114]. For instance, Tahamtan et al. [94] fabricated A206-5vol.% Al₂O₃ nanocomposite by adopting this hybrid route. They mixed powders of aluminium (74 µm), alumina and Mg (100 nm each) and ball milled the mixture up to 9 h to create master powder for reinforcement. Milling was performed in argon atmosphere with 1.7 wt% stearic acid as PCA. In addition, compacted powder tablets of 20 mm × 3 mm size were prepared for incorporation. Besides, they produced master metal matrix composites by melt process using as received alumina powder and also using milled powder. In this manner, four different forms of reinforcement addition, viz. as received alumina, master powder, master tablet and master composite, were created for producing different nanocomposite specimens. Later on, again two ways of fabrication such as semisolid stirring and liquid-state stirring were used. They heated base matrix to temperature of 745 °C for liquid processing and incorporated reinforcements slowly over period of 15–30 min with melt stirring at 400 rpm. Alumina powder and master alloy/composite were preheated at 400 and 200 °C, respectively, before addition to melt. Less melt turbulence was reported for addition of master alloy in place of nanoparticles leading to decreased porosity. Semisolid processing was performed at 640 °C temperature (20% solid fraction). Ball milling improved wettability and uniform distribution of particles, and decreased reaction products, and hence, tensile properties were enhanced.

Stir casting and cross accumulative roll bonding were combined [8] to produce Al-Al₂O₃ nanocomposites. Authors initially produced Al-2vol.% Al₂O₃ (100 nm) nanocomposite by stir casting method by employing argon gas-assisted particle injection into melt at 670 °C while stirring at 500 rpm. Cast specimen was then machined to 120 mm × 100 mm × 4 mm size specimens. All specimens after annealing at

540 °C for 2 h were cold rolled to obtain 100 mm × 100 mm × 1.5 mm strips. Two such strips were taken for roll bonding process after acetone bath, wire brush cleaning and the stack reduced to 50% thickness at the end of the first rolling pass. Bonded strip was cut into two pieces, cleaned them, stacked and subjected to second roll pass by changing the orientation to 90° with respect to previous pass. The process repeated eight times, and after every pass, the strip was characterized for microstructure and tensile properties. Cross orientation at every pass elongated the strip in both linear and transverse directions resulting in 71 nm crystal size. Tensile strength at the end of the eighth pass increased to 344 MPa (213% rise) and elongation % increased to 6.4 (205% rise).

Xiong et al. [102] combined pressureless infiltration and ball milling to produce Al-3%Mg/SiC nanocomposite having reinforced with 40 nm size SiC particles with varying volume (3, 6, 14) percentages. Al and Mg powder each of 70 μm size particles and SiC reinforcement were first ball milled in argon atmosphere for 10 h at 450 rpm and ball–powder ratio of 4:1. Powder was cold compacted at 50 GPa pressure to prepare preforms of size 40 mm × 40 mm × 80 mm. Base matrix material was later melted in furnace at 700 °C with addition of (NaCl + KCl) salt mix for reducing melt oxidation and held for 20 min. Preform was then added to melt, kept in suspension for 2 h for infiltration and allowed the slurry to solidify in the furnace itself. Authors detected MgO at the particle–matrix interface which they claimed to improve wettability. Tensile properties improved with retained ductility.

In another research work [22], rheocasting and squeeze casting were combined to fabricate A356/MWCNTs (0.5–2.5 wt%) nanocomposite. Initially, Al powder and MWCNTs were ball milled for 8 h at 200 rpm in presence of stearic acid. Blocks were then prepared by compacting mix at pressure of 70 MPa for 15 min duration in steel mould. A356 ingots were melted at 660 °C in argon atmosphere, and after degassing the melt using hexachloroethane tablet, the melt temperature was reduced to 601 °C (solid fraction 30%). On adding blocks, the semisolid slurry was stirred for 1 min at 750 rpm. It was next poured in preheated (250 °C) steel die at the same temperature and immediately squeezed to acquire mould shape. For the optimum 1.5 wt% MWCNT composite, tensile strength and elongation % enhanced by 50 and 280%, respectively, which was attributed to uniform particle dispersion and grain refinement.

2.5 *In Situ Methods*

Formation of inhomogeneous microstructure and detrimental reaction phases are the issues to be tackled for ex situ fabrication of nanocomposites. In situ fabrication involves thermochemical reactive processes between the constituents of composite material resulting in creation of very small, low range particulate reinforcements dispersed well in the entire microstructure. The advantages of the approach include formation of clean particle–matrix interface, thermodynamic stability, combination of variety of matrix materials and reinforcing particles, scalability and absence of

detrimental phases. Initially, the reactive constituents of composite material can be in various physical states such as solid–solid, solid–liquid, liquid–gas, solid–gas and liquid–liquid.

Recently, TiC nanoparticles are created in aluminium matrix by in situ process during mechanical alloying using nanodiamond as precursor [73]. They mixed powders of Al (30–100 μm), titanium (100 μm) and nanodiamond (4–6 nm) and milled the mixture in Retsch PM400 planetary mill at 300 rpm with ball–powder ratio of 10:1. Besides fine TiC particles, intermetallics such as TiAl_2 and TiAl were detected in the final mixture. Mechanical attrition activated combustion synthesis has been reported to produce $\text{Al}/\text{Al}_3\text{Ni} + \text{Al}_2\text{O}_3$ [77] and $\text{Al}/\text{TiC} + \text{Al}_2\text{O}_3$ [109] hybrid nanocomposites.

Direct melt reaction in situ technique was employed [105] to produce $\text{Al6061}/\text{ZrO}_2$ nanocomposites with varying (1–3) volume percentages. K_2ZrF_6 and KBF_4 inorganic salts were mixed in the molar ratio of 1:2:4 and ball milled to get homogeneous mix. Further, it was dehydrated for 2 h in oven at 250 $^\circ\text{C}$. The mixture in required proportion was added slowly to alloy melt at 851 $^\circ\text{C}$ and held the composition for 30 min in isothermal condition. Stirring was set for 3 min at 600 rpm and performed with a gap of 2 min in between. Melt was then poured at 750 $^\circ\text{C}$ to cast the specimen. Salts were observed to react with aluminium forming ZrB_2 particles in the matrix.

Significant reduction of TiB_2 particle size (800–23 nm) was achieved by ultrasonic stir treatment given to $\text{Al-4.4Cu}/\text{TiB}_2$ nanocomposite in situ melt produced [70] by direct melt reaction of aluminium with K_2TiF_6 and KBF_4 halide salts.

Al/AlN nanocomposite is reported [57] to be produced by evaporation of aluminium in the N_2/H_2 mixed atmosphere by plasma arc discharge and consolidating the collection of as-deposited particles by compaction followed by sintering.

3 Summary

This article is an attempt to overview the developments and issues of several different manufacturing techniques employed for obtaining bulk aluminium matrix nanocomposites. Powder metallurgy-based mechanical alloying and stir casting are the two widely used methods for producing AMMNCs. Aluminium nanocomposites are reported to exhibit exceedingly better properties than microcomposites and unreinforced monolithic alloys but their development is limited to lab-scale investigations only. However, considering the performance, nanocomposites have a great future and are likely to replace their micro-counterparts in various applications of automotive, aerospace, electronics, military, sports goods, etc. Agglomeration of nanoparticles is the most prominent issue and needs extreme care for dispersing them in the alloy matrix. Ultrasonic stir casting has so far been observed as the promising route in this regard but suffers from the industrial scalability aspect. Contamination free, careful handling of nanoparticles is essential for better results. Economical synthesis of nanoparticles and developing cost-effective ways of nanocomposite

fabrication should certainly motivate their practical usage. Different solid- and liquid-state processes available are useful and may be modified to produce near net-shaped parts so as to avoid difficulty in further processing imposed by hard reinforcements. Combining production routes have offered better results and can be explored further. Processing with nanoparticles is rather complex than micro-particles as increased surface-to-volume ratio creates challenges of particle clustering and increases the formation of reaction products, especially in liquid processing methods. In situ fabrication is a promising approach for AMMNCs due to formation of clean and strong interfaces as well as very fine dispersoids but not yet tried fully due to limited information.

Conflict of Interest None declared.

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