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Chapter

Synthesis of Magnesium Based Nano-composites

Srinivasan Murugan, Quy Bau Nguyen and Manoj Gupta

Abstract

Magnesium based nanocomposites are new lightweight and high-performance materials for potential applications in automotive, aerospace, space, electronics, sports and biomedical sectors primarily due to their lower density when compared to aluminum-based materials and steels. Synthesis of magnesium-based materials is relatively challenging and accordingly this chapter explicitly provides an insight into various techniques hitherto devised/adopted by various researcher for synthesizing magnesium based nano-composites (MMNCs). Overall processing of MMNCs often includes combination of primary and secondary processing. Primary processing fundamentally leads to the initial formulation and creation of MMNC ingots by solid, semi-solid or liquid state processing routes. This is followed by secondary processing that includes plastic deformation or severe plastic deformation to alleviate inhomogeneity, clustering of particles and fabrication defects to enhance the properties of the MMNCs. This chapter provides an insight into different fabrication methodologies, their benefits and limitations for MMNCs.

Keywords: magnesium, reinforcement, nano-composite, MMNC, synthesis

1. Introduction

Research on magnesium based-composites has seen sustainable growth in last four decades due to their light weight, higher strength to weight ratio, ductility, hardness, wear resistance and biodegradability [1, 2]. Magnesium based materials, in general, are currently targeted for applications in automotive, aerospace, electronics, sports and biomedical engineering. The driving force for intense research into magnesium-based nano-composites is to utilize them to mitigate global warming, energy consumption and land, air and water toxicity. The presence of reinforcement at nano-length scale leads to grain refinement leading to Hall-Petch strengthening and Orowan strengthening due to presence of nano-particles/fibers with diameter less than 100 nm [3].

The primary processing of MMNCs can be categorized in two groups i.e. ex situ and in situ routes [4]. In ex situ processing during fabrication of MMNCs the major issue is particle clustering. High surface energy navigates to poor wettability of particles/fibers with the matrix during liquid and semi-solid processing. These clustered particles lead to reinforcement distribution inhomogeneity in the matrix leading to inferior properties in as-cast state. Such non-uniform distribution can only be reduced through judicious secondary processing step.

The common liquid and semi-solid ex situ process are stir casting/melt stirring, ultrasound cavitation, disintegrated melt deposition (DMD) and Rheocasting. The solid-state ex situ syntheses are powder metallurgy (PM), severe plastic deformation (SPD) by accumulative roll bonding (ARB) and plastic deformation by friction stir processing. The in-situ processes eliminate the reinforcement clustering, since the reinforcements are distributed in the matrix by thermodynamic and chemical reaction during the process.

The MMNCs processed by ex situ route commonly exhibit microstructural defects such as dendrites, pores, and micro cracks. These require careful characterization followed by using a secondary processing technique that can target the property enhancement required by end application. The common secondary processes are thermal treatment, hot extrusion, hot rolling, equal channel angular pressing (ECAP) and cyclic extrusion and compression (CEC).

The prime objective of this chapter is to provide an overview of various processing methods used currently for synthesizing MMNCs, their benefits and limitations. Critical observations made by other researchers are also highlighted in this chapter.

2. Processing methods

The processing methodology for fabricating magnesium based nano-composites (MMNCs) normally includes coupling of primary and secondary processing. In primary processing (solid, liquid or two phase), the matrix (master alloy) and the reinforcements are blended together by the application of thermal or mechanical energy to form a composite. During primary processing some undesirable effects are introduced in the composites such as porosity and non-uniform distribution of reinforcement voids. To minimize these defects, secondary processing is utilized to attain a relatively homogeneous microstructure and enhanced mechanical properties.

2.1 Primary processing

Primary processing techniques are grouped into liquid state, semi-solid and solid state processing types. The liquid state processes are stir casting, ultrasonic cavitation (UST), disintegrated melt deposition (DMD), in-situ processing. Rheocasting is a semi-solid composite primary processing. The soil-state fabrication includes powder metallurgy (PM), accumulated roll bonding (ARB) and friction stir processing (FSP). These techniques are briefly introduced in following subsections.

2.1.1 Stir casting

Stir casting is one of the most common liquid-phase technique utilized for processing MMCs for almost last four decades. The schematic of stir casting setup is shown in the **Figure 1**.

For processing MMNCs, the Mg ingots are melted in a crucible made of graphite or steel at a temperature between 680 and 750 °C in an electric resistance or induction furnace. The liquid melt is mechanically stirred using a coated impeller. The coating is provided onto the impeller to avoid erosion by abrasion and chemical reaction. Predetermined amount of nano-reinforcement is introduced in molten metal along the side of the vortex. The reinforcements were distributed in the melt due to the difference in pressure from the inner to outer surface of the liquid
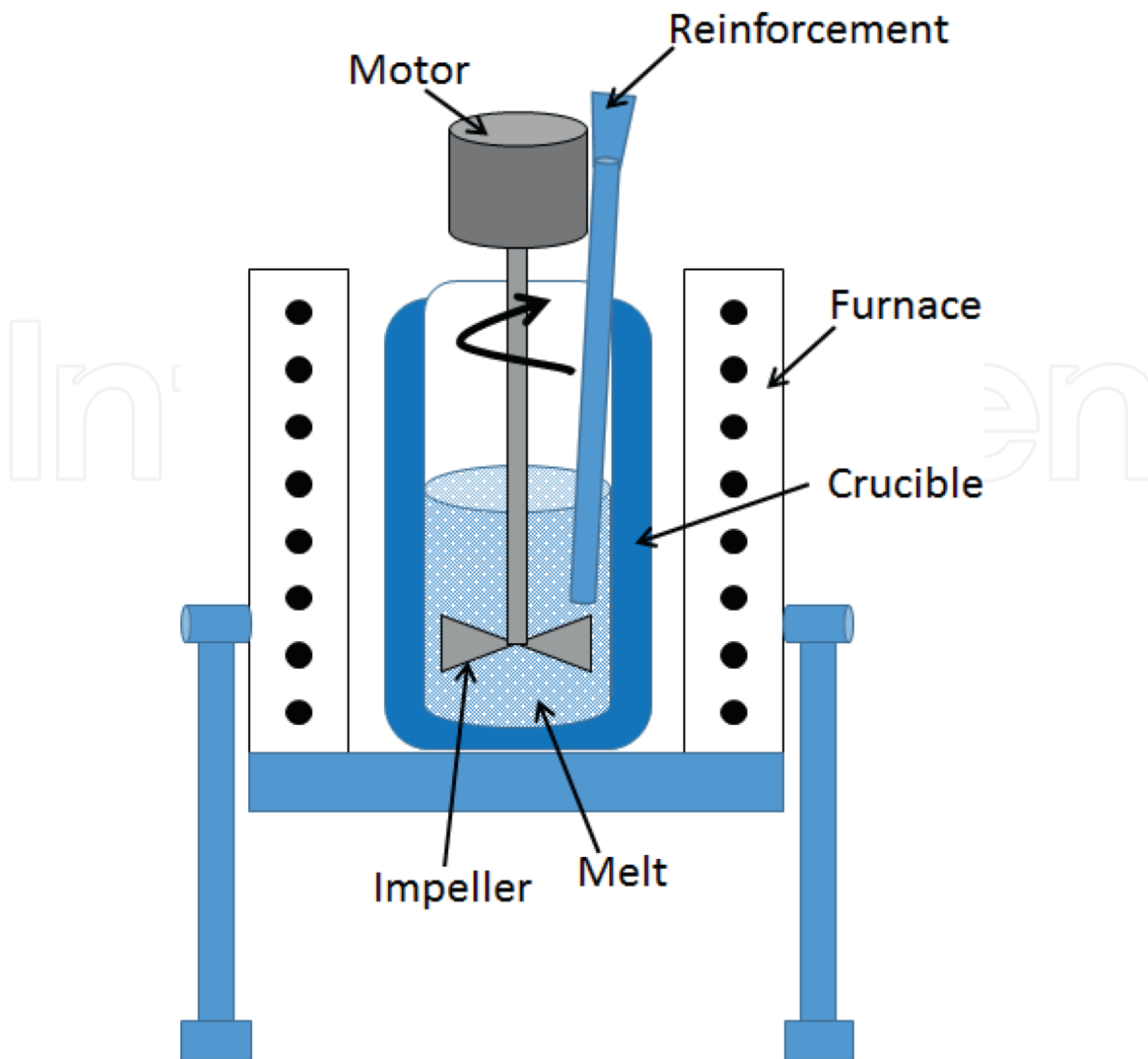
Figure 1.
Schematic of stir casting setup.

vortex. The vortex is shielded using inert atmosphere to avoid oxidation /ignition. Alternatively, to overcome agglomeration/clustering, the powders of magnesium and the nano reinforcement are ball milled together prior to addition. The liquid slurry is stirred for ~10 min to homogenize the mixture. After homogenization, the liquid slurry is poured into a permanent mold. MMNCs reinforced with Al_2O_3, SiC and CNTs are commonly synthesized using stir casting technique [5–16].

The benefits of stir casting include: (a) ease of processing, (b) economical, and (c) scalability to ensure large volume production. Disadvantages of stir casting includes: (a) unavoidable agglomeration of reinforcement and (b) porosity.

2.1.2 Ultrasonic cavitation method

One of the main drawback of the stir casting technique is its inability to avoid agglomeration of nano-reinforcement due to their larger surface energy. This causes inferior mechanical properties of the end composites. Ultrasonic (UST) cavitation method is a relatively more effective technique to disperse the reinforcement into the matrix material in MMCs [17]. By introducing the ultrasonic waves with power and frequency range of as low as 1.5–4.0 kW and 17.5–20 kHz the agglomerates can be fragmented, and a uniform distribution of reinforcement can be realized in the liquid melt. This method has been adopted so far to produce MMNCs reinforcing with CNTs, AlN, SiC, B_4C and Al_2O_3 [17–35]. The schematic of UST setup is shown in the **Figure 2**.
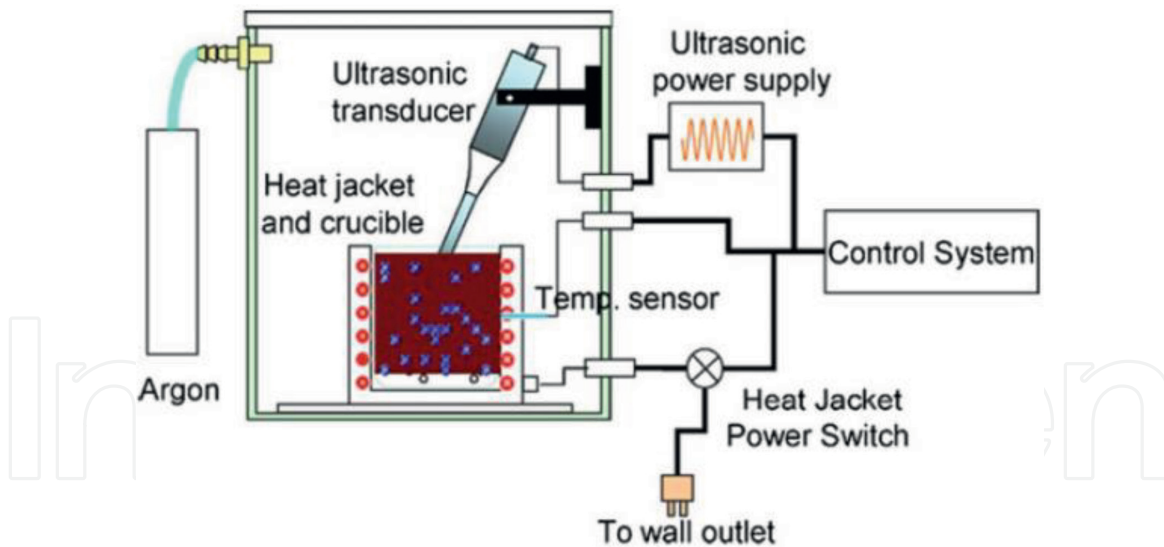
Figure 2.
Schematic representation of UST [4].

In the UST process, the Mg alloy is placed in a graphite crucible and heated to the desired temperature using a resistance/induction furnace. Predetermined amount of reinforcement is than added depending on the size of the particles. During processing, Mg melt is protected using argon gas (flow rate of 20 lpm) to avoid oxidation. High-intensity ultrasound shock waves [16] are supplied to disperse nano-particles thoroughly in the melt at semi-solid temperature. The ultrasonic processing temperature is chosen so as to ensure better flowability of the slurry in the mold.

The selection of material for sonotrode plays a vital role in MMNCs melting due to the erosion of sonotrode surface during melting of liquid metal. For better sonification of MMNCs, niobium (Nb) and titanium (Ti) are recommended by the researchers. Ti-based sonotrodes are widely used for UST treatment due to lesser costs when compared to Nb-based sonotrodes. To note that Nb-based sonotrode exhibit less variation in the Young's modulus as the function of temperature while Ti-based sonotrodes are very stable in MMNCs melt as Ti is insoluble in Mg. Earlier findings have indicated that the high-intensity UST vibration needs an intensity of 100 W/cm^2 [4]. For large scale volume production, the requirement of sonification is likely to be higher. The large-scale production of MMNCs require enormous power and frequencies. The key issue is reducing rate of sonification by decreasing the volume of the melt during UST. This can be achieved in two steps firstly, preparing the melt with reinforcement and secondly, passing the liquid melt into a sonotrode assisted UST chamber for fragmenting particle clusters. Further work is required in this area especially regarding scalability.

2.1.3 Disintegrated melt deposition method (DMD)

DMD technique is a hybrid technique that incorporates the concepts of casting, melt stirring and spray forming process [36–45]. In DMD, the processing steps involve:

a. Heating matrix material and reinforcement in a graphite crucible (ceramic bonded clay graphite) to a desired superheat temperature. Mg and its alloys (in the form of chips or turnings) and reinforcement are placed in alternative layers.

b. Stirring of reinforcement at ~ 450 rpm for 5–10 min using a Zirtex 25 coated stainless-steel impeller realize uniform distribution.
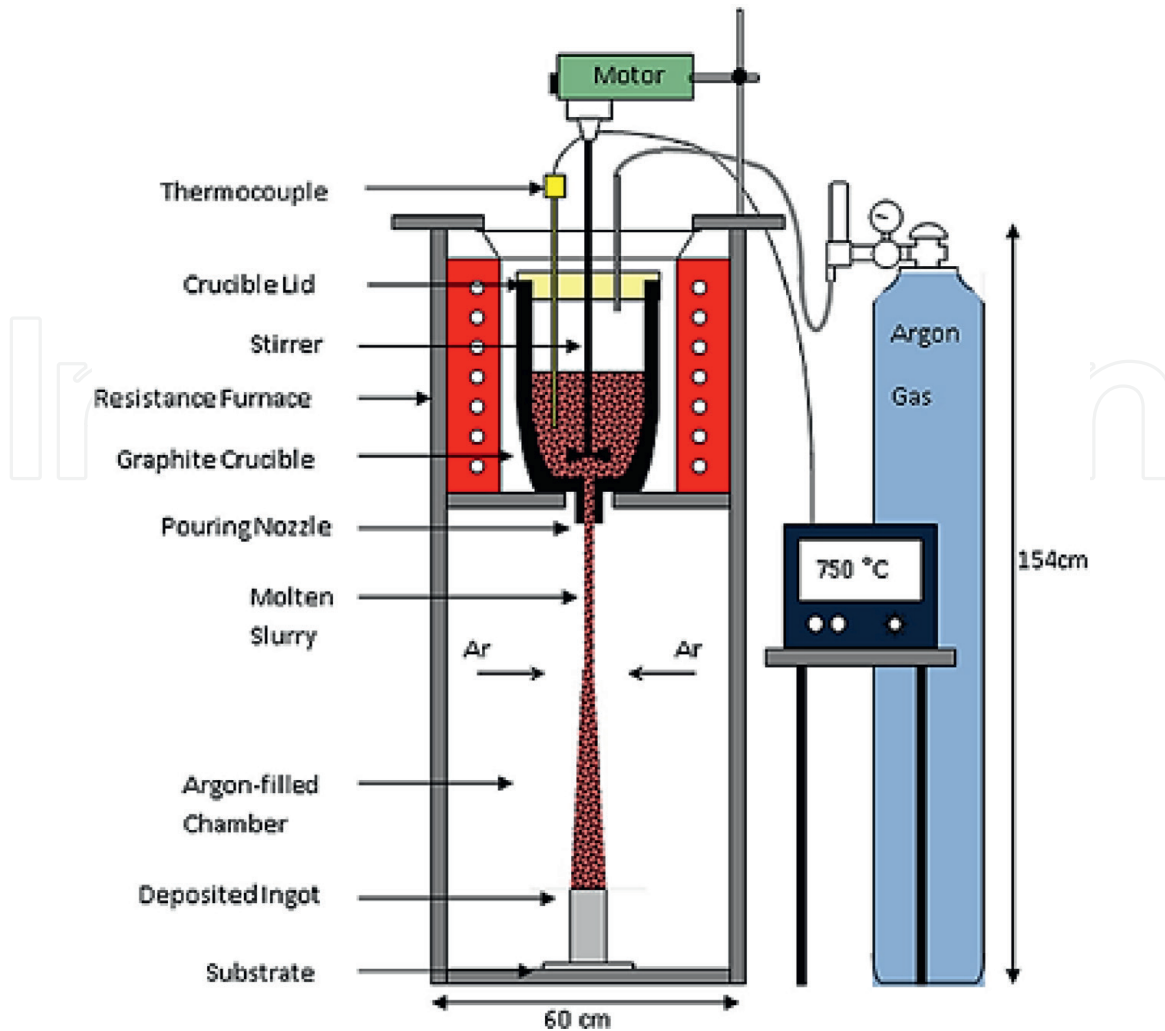
Figure 3.
Schematic drawing of DMD [35].

 c. Bottom pouring of the slurry and disintegration of slurry through 10-mm annular diameter graphite nozzle.

 d. Disintegration of slurry using two argon gas jets.

 e. Deposition of disintegrated slurry into a steel mold to get 40-mm ingots.

 DMD processing is carried out under argon inert atmosphere to minimize oxidation. Experimental setup of DMD process is shown in **Figure 3**. The advantages of the method include:

 a. Bottom pouring of the liquid slurry to ensure almost 100% yield.

 b. Disintegration of molten slurry using low pressure gas jets to ensure improved particle distribution and microstructural homogeneity.

 c. Use of argon gas rather than SF_6 (greenhouse gas) to prevent oxidation.

2.1.4 In-situ casting processing

 In-situ casting of MMNCs is a very versatile and an economical process to synthesize MMNCs. The reinforcements are formed and controlled by metallurgical

reactions between principal alloy and the additives [46–50]. The type and number of additives are chosen based on final formulation of the matrix and volume fraction of the reinforcement. Reaction temperature is a key parameter in process design of in-situ MMNCs to ensure the desired matrix and the reinforcement phase. An example of creating in-situ Mg-Zn/MgO composites includes the use of Mg and ZnO as starting materials and heating them to a predetermined temperature to ensure the feasibility of the following two reactions (Eqs. (1) and (2)):

$$Mg + ZnO => MgO + Zn \qquad (1)$$

$$Mg + Zn => Mg\!-\!Zn \qquad (2)$$

Chelliah et al. [46] synthesized magnesium-polymeric derived ceramic (PDC) silicon carbonitride (SiCNO) nano composite by liquid pyrolysis using stir casting technique. The liquid poly (urea-methyl-vinyl) silazane (PUVMS) was used to formulate magnesium nano-composite. In this method, magnesium was melted in a steel crucible using a resistance furnace at a temperature of 700 °C and shielded with Ar-5%SF$_6$ gas. The melt was stirred mechanically at 600 rpm to form vortex and the liquid PUVMS was injected to the melt. The melt was stirred for 15 min to ensure thorough pyrolysis. The liquid melt was bottom-poured into a rectangular metal mold preheated at 300 °C. Mg/nano- SiCNO composite was fabricated exhibiting uniform distribution of the reinforcement (**Figure 4**).

The benefits of in-situ MMNCs include: (a) uniform distribution of the reinforcement, (b) elimination of particle wettability issue, and (c) clean and strong matrix-particle interface. The disadvantages of in-situ techniques, in general, are scalability and the amount of reinforcements that can be created using the in-situ reactions.
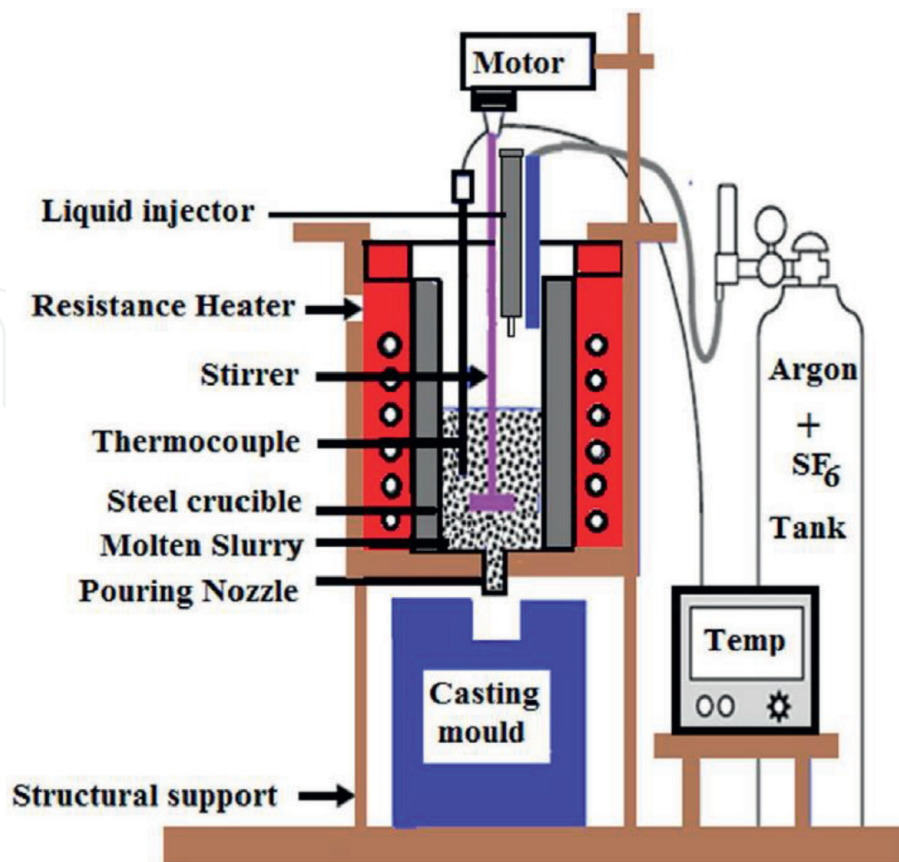

Figure 4.
Experimental setup of liquid pyrolysis stir casting [45].

2.1.5 Rheocasting technique

Rheocasting is a semi-solid casting method where the matrix is processed in liquidus-solidus (L-S) zone. In this so called semi-solid zone, the reinforcement particles are added, and the resultant slurry is thoroughly stirred to ensure uniform distribution of the reinforcement. Following stirring, the semi-sold composite melt is tapped into a permanent mold. Often cleaning and degassing of the slurry is carried out to avoid oxidation and formation of inclusions.

A MMNC of Mg (AZ91E) with Al_2O_3n (50 nm) was synthesized using a semi-solid Rheocasting process [51]. The Mg ingots were placed in boron nitride coated mild steel crucible. The melt was formed in the metal crucible at 750 °C using electric resistant furnace. The slurry was degassed using argon to avoid oxidation. The reinforcement (Al_2O_3n) was then added to the slurry at semi-solid (L-S) temperature (~590 °C). The melt slurry was stirred using a mechanical stirrer. The MMNCs slurry was subsequently poured into a permanent mold for further characterization.

The benefits of this technique include: (a) spheroidal/equiaxed grains and no dendrites, (b) less shrinkage and porosity and (c) lower operating temperature.

2.1.6 Powder metallurgy

Powder metallurgy (PM) [52] is one of the most common solid-state synthesis method for magnesium based nano-composites [53–60]. The steps followed in PM are shown in **Figure 5**. In the first step metal alloy and ceramic particle in powder form are blended/mixed together to get homogenous mixture. The mixing parameters are decided based on the density difference between metal/alloy and reinforcement powder. The blended powders are subsequently compacted using a cold press or hot press or hot isostatic press. Finally, the green compacts are sintered by heating to a predetermined temperature to regain mechanical properties. Near-net-shaped components with simple geometries can be fabricated by PM technique.
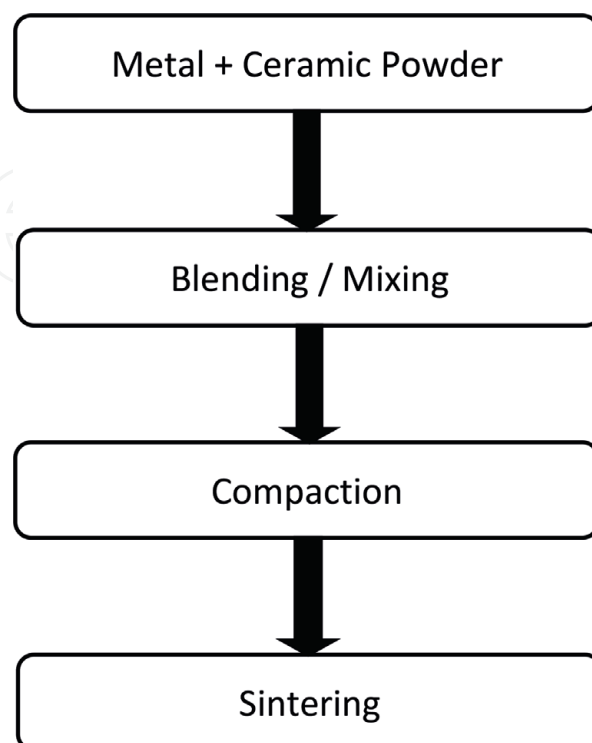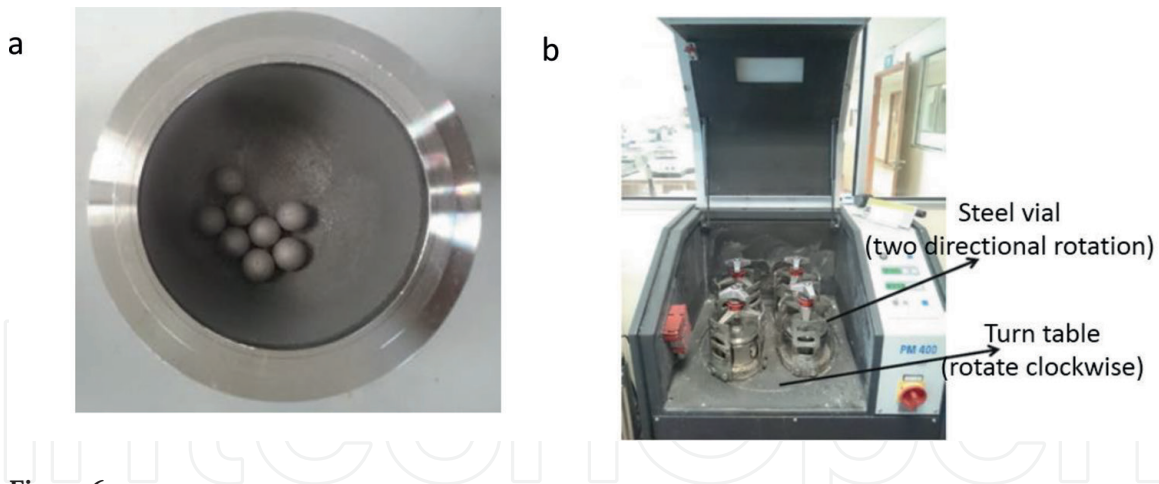

Figure 5.
Step in PM.

Figure 6.
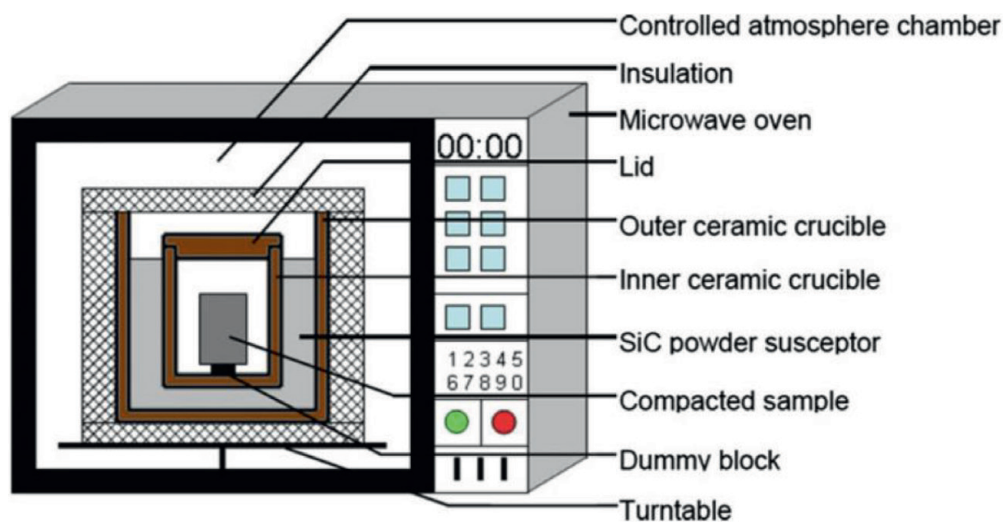*(a) Diagram shows the steel vial with steel balls and (b) planetary ball mill [2].*

Figure 7.
Hybrid microwave sintering setup [52].

Several magnesium-based alloys, conventional and nanocomposites (e.g. Mg/3wt%Al/0.1wt%GNP) have be synthesized using PM technique. Typical processing steps include blending or mechanical alloying the predetermined amounts of metal and ceramic powder with or without steel balls using a planetary ball mill at a speed of 200 rpm for 1 h. A typical planetary type ball mill setup is shown in **Figure 6**. The composite powders obtained from blending step are subsequently compacted using a 100 T hydraulic press to attain a billet of ~35 mm diameter and height of 40 mm. Compacted billets can be sintered using a conventional furnace or microwave based sintering. Heating time during microwave sintering is kept with 16 min. Conventional microwave 0.9 kW power and 2.45 GHz operating frequency can be used. The schematic diagram of microwave sintering set-up is shown in **Figure 7**.

2.1.7 Accumulative roll bonding

Accumulative roll bonding (ARB) is a solid-state severe plastic deformation technique used to produce layered composite stacked with either similar or dissimilar alloys sheet and reinforcements [61–64]. The final output of the ARB process depends on the process variables namely weight percent of the alloys and the reinforcement, number of rolling cycles and the temperature. The benefits of the process are grain refinement from fine to ultra-fine, fragmentation of reinforcement clusters and their subsequent uniform distribution. To realize best possible
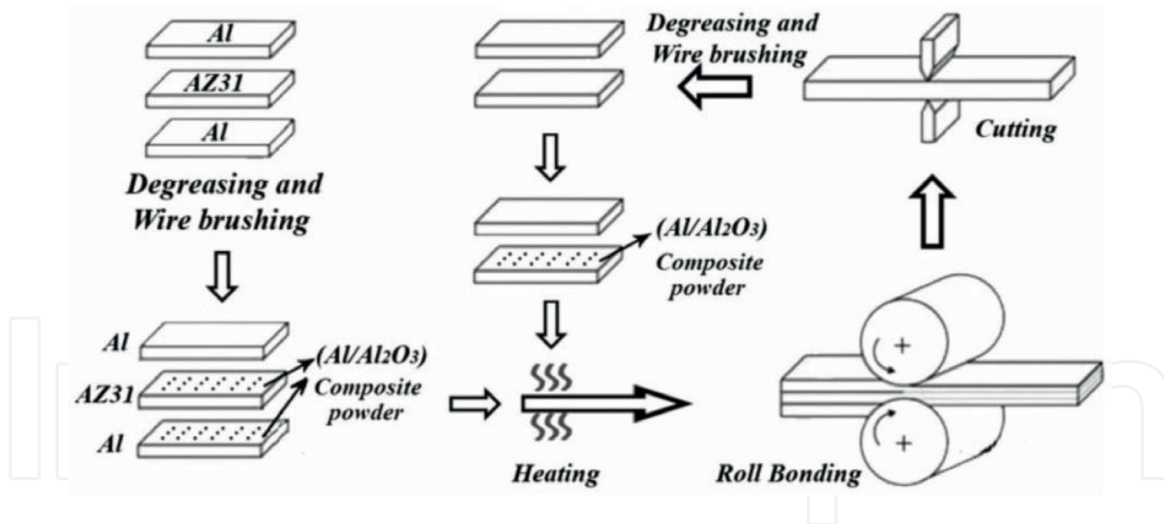
Figure 8.
Steps in synthesizing nano-composites using ARB [61].

results, the sheets need to be pretreated by grinding and polishing for descaling oxides and to make friction free surface. Degassing process is also often used. The schematic setup of the ARB is shown in **Figure 8**.

Monolithic aluminum (Al) and AZ31 magnesium [61] strips of 1 and 0.5 mm thickness were cut to 150 × 50 mm rectangular strips. The strips were annealed at 400 °C for 2 h and furnace cooled near ambient temperature to soften the strips prior to rolling. The sheets were ground, polished, degassed and cleaned prior to rolling. In a steel vial Al and nano-alumina (<50 nm) powder were purged along with 0.5 and 1 mm in diameter steel balls. Milling of Al-alumina powder was conducted at 300 rpm and ball-to-powder ratio of 20:1 for six cycles, each of 45 min duration with a dwell time of 15 min to eliminate undesirable rise in temperature. The milled Al-alumina particles were uniformly spread between the strips for better wettability. The stacking was done as Al/AZ31/Al with reinforcement powder in between. The stack was fastened by copper wires to avoid slipping. The assembly was preheated in the temperature range of 300–350 °C for 15 min in an air furnace. 50% reduction was maintained at each rolling stage. Rolling was carried out for four times.

Accumulative roll bonding has yielded good combination of properties due to enhanced microstructural aspects and is investigated further for scaling up.

2.1.8 Friction stir processing (FSP)

Friction stir processing is a solid-state plastic-deformation-based synthesis method (**Figure 9**). It can be used to build nano-composite layer/surface composite as well as bulk composites of limited thickness/dimensions. It often leads to uniform distribution of reinforcement and refined grain size [65–70]. The FSP process uses a shouldered rotating tool that pass over the matrix containing nano-ceramic particles. During the translational movement of tool, matrix get plastically deformed and reinforcement gets simultaneously incorporated in the matrix. The stirring action enables uniform distribution and refine grained structure of the fabricated composite.

The α-Al_2O_3 nano-particle reinforced AZ31 composite [65] was fabricated using friction stir processing technique. The Mg rectangular plates of 600 × 100 × 10 mm were cut for preparing the composite. The ceramic particles were spread into a groove of width and depth 1.2 × 5 mm cut in the plates as shown in the **Figure 8**. Two types of shouldered tools were used to form the composite, one pin less shoulder and the other shoulder with a pin of 5 mm height
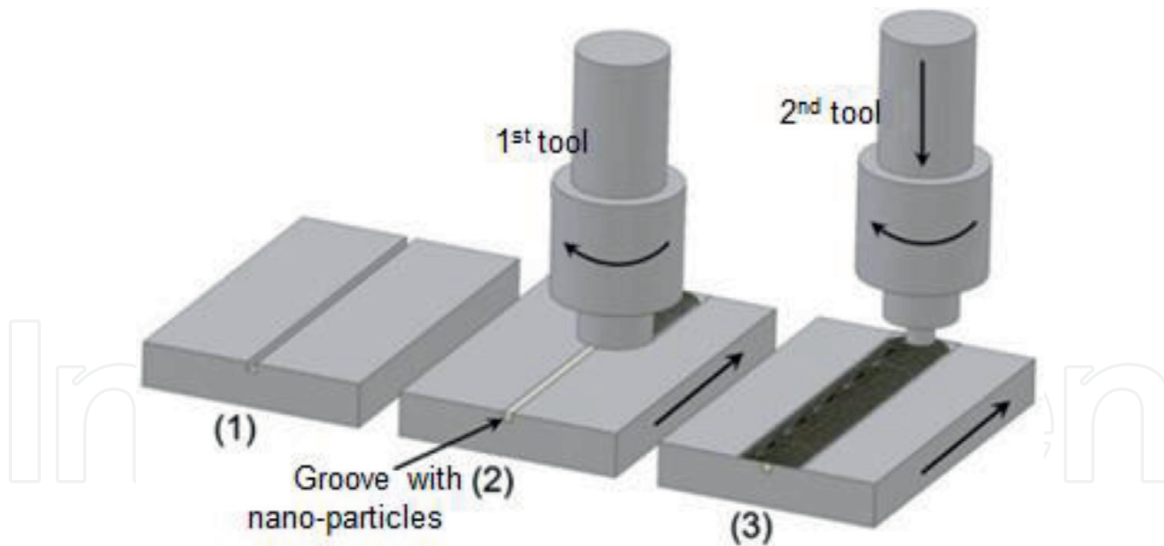
Figure 9.
FSP fabrication stages from 1 to 3 for Mg nano-composite [67].

and diameter of 6 mm. The pin less tool was fed first to prevent the nano-phase getting distorted from the groove. The second tool with pin was then passed to complete the process. The tool rotation (800–1400 rpm) and the traverse speed (45 mm/min) were varied to obtain the desired strength and structure of the composite. Higher hardness was observed due to grain refinement at higher tool rotational speed.

2.2 Secondary processing methods

Secondary processing is essentially performed on primary processed composites to eliminate/minimize defects and microstructural irregularities to enhance mechanical properties such as strength, hardness etc. Secondary processing methods include bulk deformation process such as extrusion/rolling/friction stir processing and severe plastic deformation processing methods such as equal channel angular pressing (ECAP) and cyclic extrusion and compression (CEC).

2.2.1 Extrusion

The MMNC billet is stressed under high pressure ram inside a die which quantitatively reduces the irregularities like pores, cavity, voids and cracks formed earlier in primary processing methods. Normally carried above recrystallization temperature, the hot extrusion processes can be categorized into;

1. Forward or direct extrusion

2. Backward or indirect extrusion

The difference in the two method is the flow of billet direction. The billet extruded on the Direction of the ram is direct extrusion while in the backward extrusion, the billet is extruded opposite to the direction of movement of ram. Typical forward extrusion process in shown in **Figure 10**. The key process parameters are extrusion ratio, temperature during extrusion and the speed of the ram. Extrusion ratio in the ratio between initial to the final cross section of the billet. The working temperature and environment should be optimally decided to eliminate oxidation during extrusion.
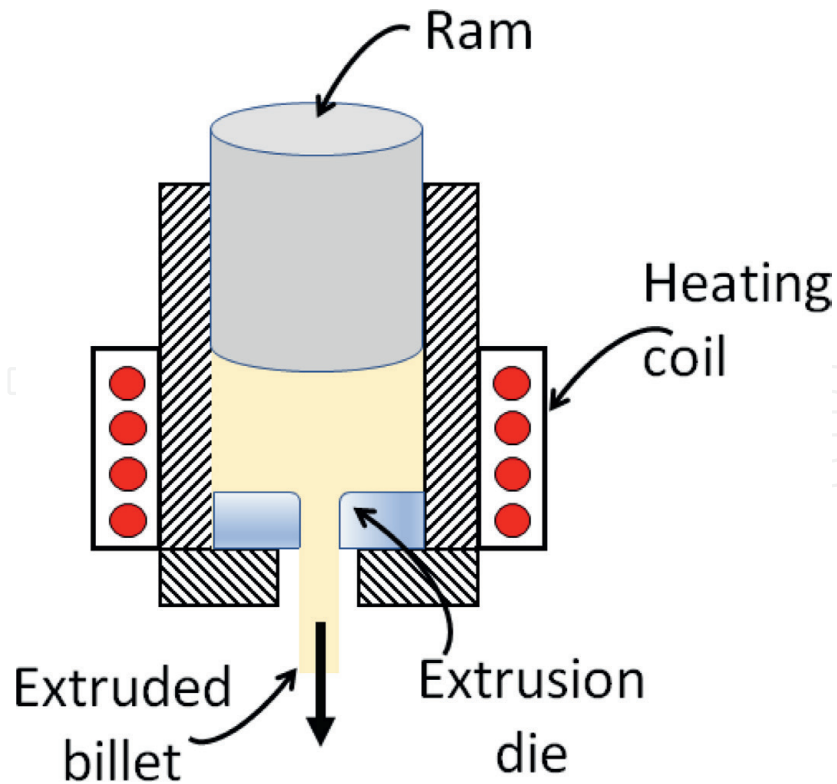
Figure 10.
Schematic representation of direct extrusion process.

2.2.2 Rolling

Rolling is a plastic deformation process in which the MMNCs are deformed by passing through set of high-pressure rolls. The MMNCs are deformed plastically by compressive stress and squeezing action between the rolls. The process enables the MMNCs to obtain fine grain microstructure and eliminates defects caused in the primary processing. The benefits include fine grain microstructure and enhanced mechanical properties of the material. The process parameters are percentage reduction, temperature and the number of passes to achieve the final thickness on the MMNCs. Rolling is also normally carried out a higher temperature to minimize the load required for plastic deformation.

2.2.3 Equal channel angular pressing (ECAP)

ECAP (**Figure 11**) is used to form ultrafine-grained (UFG) microstructure MMNCs. The MMNC billet is passed through two channeled die with identical cross section that intersect at an angle Φ (channel intersection angle). During the process the billet undergoes severe plastic shear deformation (SPD) without altering the geometrical cross-section. The process is normally repeated for several passes in order to attain UFG structure. Different routes followed namely A, BA, BC and C as discussed elsewhere [71]. Previous researcher [71] reported that when Φ is 90°, enhanced grain refinement is realized due to increase in the shear strain (γ). The shear strain for the individual passes can be obtained by:

$$\gamma = 2\,cot\left(\frac{\Phi}{2}\right) \qquad (3)$$

In an earlier research [16] conducted on Mg-9Al-1Si-1SiCn composite, ECAP was performed at a temperature of 360 °C. Route BC was chosen with
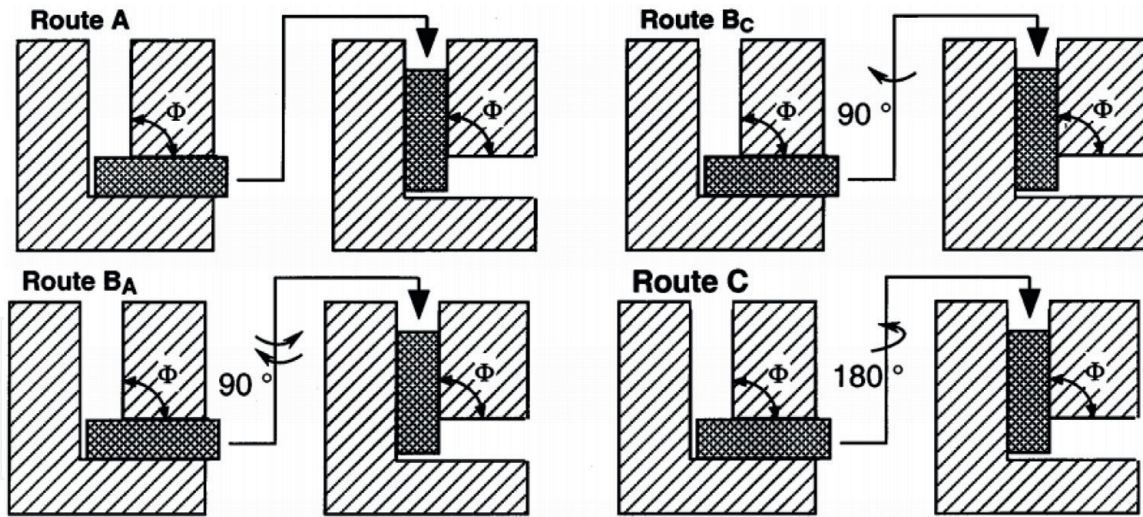
Figure 11.
Schematic diagram of four ECAP routes [71].

ram speed of 2 mm/min. Homogenization heat treatment was carried prior to ECAP. Homogenized ECAP composite billet exhibited superior ductility and tensile strength.

2.2.4 Cyclic extrusion and compression (CEC)

CEC is another type of SPD technique in which the MMNC ingot is passed through the annual die to attain a fine-grained microstructure shown in **Figure 12**. The fine-grained structure in the ingot is realized using optimal temperature and the number of reciprocating passes. A typical setup of the CEC is shown in the figure below.

AZ91D/SiCn nano-composite was fabricated with refined grain structure using CEC [72]. Fabrication using the CEC was performed by varying the operating temperature from 300 to 400 °C and up to eight passes were made. Superior hardness with refined grain structure were observed.
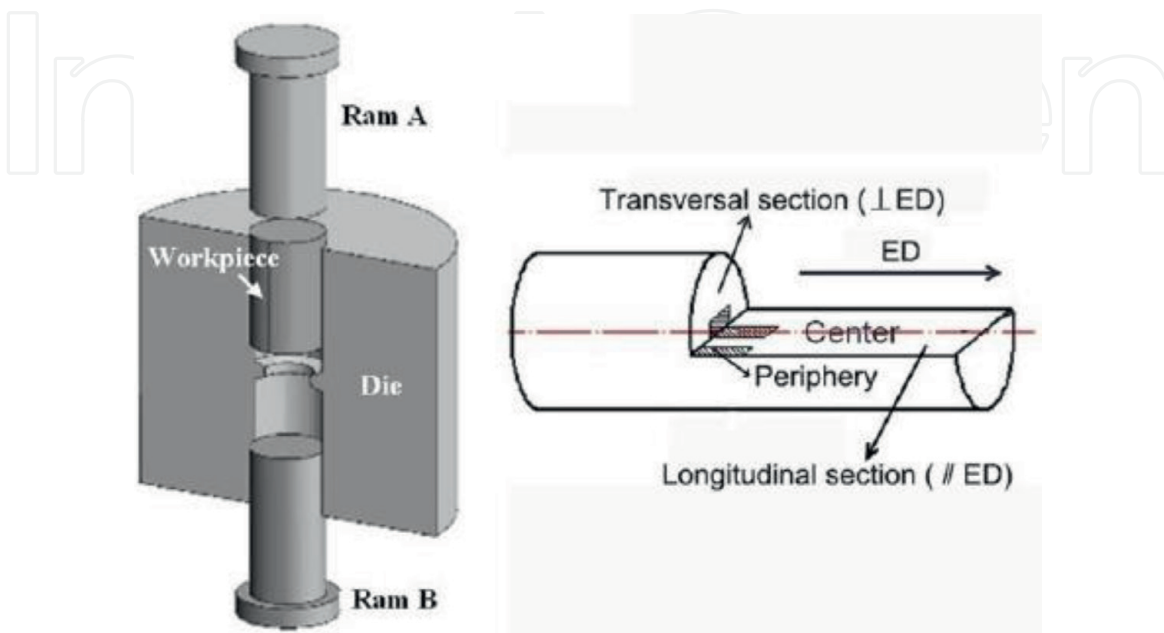

Figure 12.
Schematic diagram of CEC setup [35].

3. Conclusion

Present chapter provides an insight into the magnesium nano-composites that are emerging as potential candidates in many weight-critical engineering applications ranging from aerospace, automotive to sport industries. Not only they are significantly lighter than aluminum and titanium, they can also be processed using both conventional and advanced processing methods. Stir casting is the most traditional high-volume production technique capable of generating a uniform dispersion of nano-particles in the magnesium matrix. An improvement using the ultrasonic cavitation as a mean to disperse the nano-reinforcement is already attempted with promising results. Furthermore, disintegrated melt deposition technique has proved to be the most effective one because of its capability to well disperse the nano-additives and refine the microstructure which results in excellent mechanical properties. DMD is also a scalable technique. A different approach to create magnesium based nano-composites employing the chemical reaction to form nano-intermetallics during the casting period is called as in-situ casting. A semi-solid casting technique called Rheocasting applied slurry characteristics to mix the nano-particles into the matrix. The magnesium nano-composites could also be processed using powder metallurgical methodology where the raw matrix material and reinforcements are pre-mixed in powder forms using ball milling followed by compaction and sintering. Other solid-state processing techniques such as accumulative roll bonding and friction stir processing have shown tremendous promise as well. This chapter also introduces conventional and advanced secondary processing techniques such as extrusion, rolling, equiaxed channel angular pressing, and cyclic extrusion and compression. The utilization of secondary processing techniques along with primary processing techniques can lead to enhanced microstructural properties that are key to improved mechanical performance and reliability.

Author details

Srinivasan Murugan[1*], Quy Bau Nguyen[2] and Manoj Gupta[3]

1 Dhofar University, Salalah, Oman

2 Charles Darwin University, Australia

3 National University of Singapore, Singapore

*Address all correspondence to: smurugan@du.edu.om

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