

Chapter 2

Ex Situ Production Routes for Metal Matrix Nanocomposites

Abstract Among different production routes hitherto developed for the manufacturing of metal matrix nanocomposites, a distinction can be done depending upon the matrix state during the production process, which can be molten, solid or semi-solid. In this Chapter, an overview of ex situ production routes is given, highlighting their general potential and shortcomings. Relevant case studies on the most promising and widespread casting production routes will be discussed more in detail in Chap. 3.

2.1 Liquid State Processes

Compared to other methods, liquid state MMNCs processing routes are attractive due to the fact that they are relatively simple, cost effective and potentially scalable to industrial level for the production of near-net shape components [1–3]. Liquid routes include stir casting, ultrasonic assisted casting, infiltration techniques and disintegrated melt deposition.

Stir casting. Stir casting is one of the most widespread liquid-based technique employed for MMNCs production, due to its simplicity and cost effectiveness; moreover, it is suited to be applied to large volumes of metal [4–6]. The reinforcing phase is usually added to the matrix and distributed in the molten state by applying mechanical stirring through an impeller. MgO, Al₂O₃, ZrO₂ and SiC nanoparticles as well as CNTs have been added to aluminium and magnesium matrices by stir casting method [1, 2, 7–10]. A basic layout of the process is shown in Fig. 2.1.

The molten alloy together with the dispersed particles may be used for sand casting or permanent mold casting. Due to the low wettability of nanoparticles within the molten matrix, their tendency to agglomerate, and the differences in density between particles and matrix, it is usually difficult to obtain a homogeneous distribution of the nano-reinforcement. This results in clustering of nanoparticles [1, 2, 4, 11–13]. Moreover, due to the air entrapment induced by the rotating stirrer, a high porosity content can characterize the composite and, furthermore, undesired chemical reactions at the matrix/reinforcement interface may occur [2, 14].

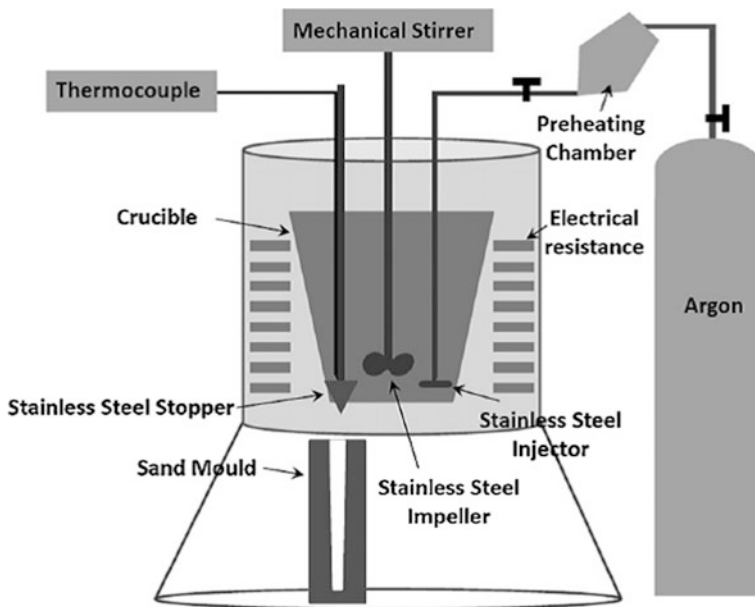


Fig. 2.1 Typical stir casting experimental set-up [2]

Particles may be added to the matrix directly into the molten stream while filling the mold or through an inert carrier gas; the dispersion may also be obtained through the so-called vortex method [4], consisting in vigorously stirring the melt, generating a vortex at the melt surface. The particles are introduced at the side of the vortex and subsequently transferred to the liquid matrix by the vortex, due to the pressure difference between the inner and the outer surface of the melt. As to eliminate the initial clustering and diminish wetting issues, nanoparticles may be pre-dispersed through ball milling on the surface of metal powders (Al or Mg based). The composite powders are then added to the molten metal and stirring is employed: during melting of the matrix powders, nanoparticles are released in the molten matrix. Nanocomposites reinforced with Al_2O_3 , SiC and CNTs have been produced in this way [1, 3, 13, 15–19].

Ultrasonic assisted casting. Ultrasonic assisted casting is reported to be effective in eliminating particle clusters which are generated due to agglomeration tendency and low wettability of nanoparticles [12, 20]. It consists of treating the melt with ultrasonic waves (usually in the frequency range 18–20 kHz) during or after adding the reinforcing phase; the treatment has been used for the production of Al and Mg based nanocomposites reinforced with SiC, Al_2O_3 , B_4C , CNTs and AlN [11, 12, 21–26]. A typical set up for ultrasonic treatment, in which a ultrasonic irradiating sonotrode is directly dipped into the melt, is shown in Fig. 2.2. Non-contact ultrasonic treatment was also applied for the production of Al/ Al_2O_3 nanocomposite by means of an ultrasonic chamber [22].

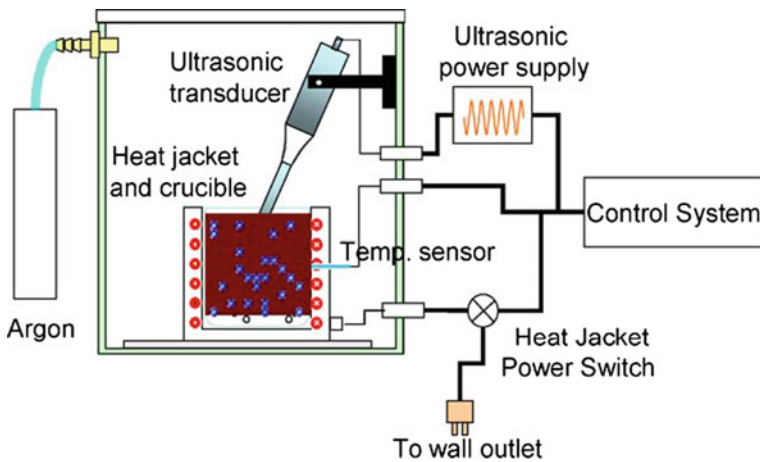


Fig. 2.2 Ultrasonic assisted casting set-up [23]

The sonotrode is usually made of titanium alloy (Ti6Al4V), stainless steel or niobium based alloy (e.g. C-103), due to their efficiency in transmitting ultrasounds and dimensional stability at elevated temperatures [27, 28]. Although costly, Nb sonotrodes possess higher chemical inertness when in contact with the molten metal matrix [26, 29], withstanding high processing temperatures with minimum ultrasonic cavitation induced erosion [30, 31]. To this end, Ti-based sonotrodes behave better than steel-sonotrodes and are widely used due to lower costs as compared to Nb ones.

Ultrasonic cavitation can produce transient (in the order of nanoseconds) micro “hot spots” with temperatures of about 5000 °C, pressures above 1000 atm, and heating and cooling rates above 10^{10} K/s [32]. Since nanoparticle clusters are loosely packed, the entrapped air inside the voids in the clusters, will serve as nuclei for cavitation [26]. The strong heating and cooling rates induced during the process, as well as the pressure gradient, are believed to be able to break nanoparticle clusters and to remove impurities from the particles surface. Despite its efficiency in dispersing nanoparticles and destroying clusters, ultrasonic technique seems quite difficult to be scaled up for industrial applications, as the volume of castings are limited to the power of the ultrasonic source. It has been reported, in fact, that high-intensity ultrasonic vibration requires at least an ultrasonic intensity of 100 W/cm^2 , while a fully developed cavitation occurs in the molten aluminium alloys when the threshold value of 80 W/cm^2 is achieved [33]. However, a possible solution for scaling up the process could be represented by melt flow processing, which involves forcing the melt to flow in a ultrasonic treatment chamber so that a small sonotrode can treat a large metal volume [34].

Infiltration process. Infiltration process involves the pressure-assisted injection of liquid metal into a porous preform (Fig. 2.3). This technique has been widely adopted for the production of composites with micro sized reinforcement, e.g. SiC

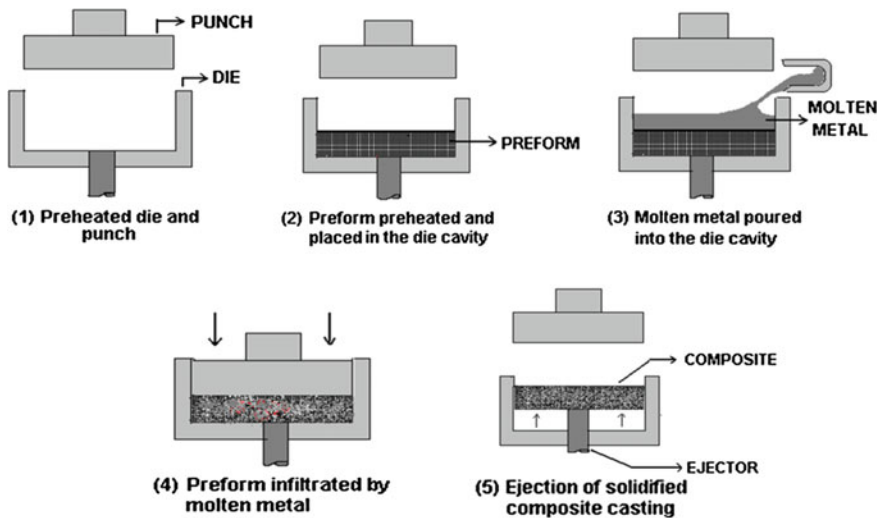


Fig. 2.3 Schematic of the infiltration method [134]

particles and foams, TiC, glass fiber, Al_2O_3 , AlN, and Al_4C_3 [35–40]. The preform is generally obtained by preparing a slurry consisting of a binder, a liquid carrier and the reinforcement phase, and then subjecting the slurry to filtration. Subsequently, the preform is dried and heat-treated to remain dimensionally stable during the pressure-assisted molten metal infiltration [41]. Infiltration process was adopted by Babu et al. [42] to produce Mg-based composites by infiltrating graphite nanofibers and alumina short fibers hybrid preforms. Recently, infiltration technique has been applied in a pressureless configuration, involving the spontaneous infiltration of the ceramic preform without the aid of externally applied pressure, nor vacuum [43]. The technique results to be more cost-effective than the traditional infiltration process, as it does not require expensive equipment, although long infiltration time are required. Al alloy based nanocomposites reinforced with SiC particles were produced by combining ball-milling and cold pressing to produce the preform, prior to pressureless infiltration [44, 45]. A similar technique was adopted by Zhou as to produce Al-based composites reinforced by CNTs, by melting and infiltrating LY12 alloy (Al–Cu–Mg) into the CNT–Al–Mg preforms, by using an infiltration time of 5 h [43]. With these techniques, processed metal volumes are limited by long infiltration times and preform costs.

Disintegrated melt deposition. Disintegrated melt deposition (DMD) technique was adopted to disperse nano-reinforcement in molten magnesium alloys. The process, developed for the synthesis of near-net shape discontinuously reinforced MMCs [46] is a combination of casting and spray processes. It involves incorporating the ceramic particles by stirring the molten alloy with an impeller as the particles are added. The resulting composite slurry is then disintegrated by jets of inert gas at a typical superheat temperature of 750 °C and subsequently deposited

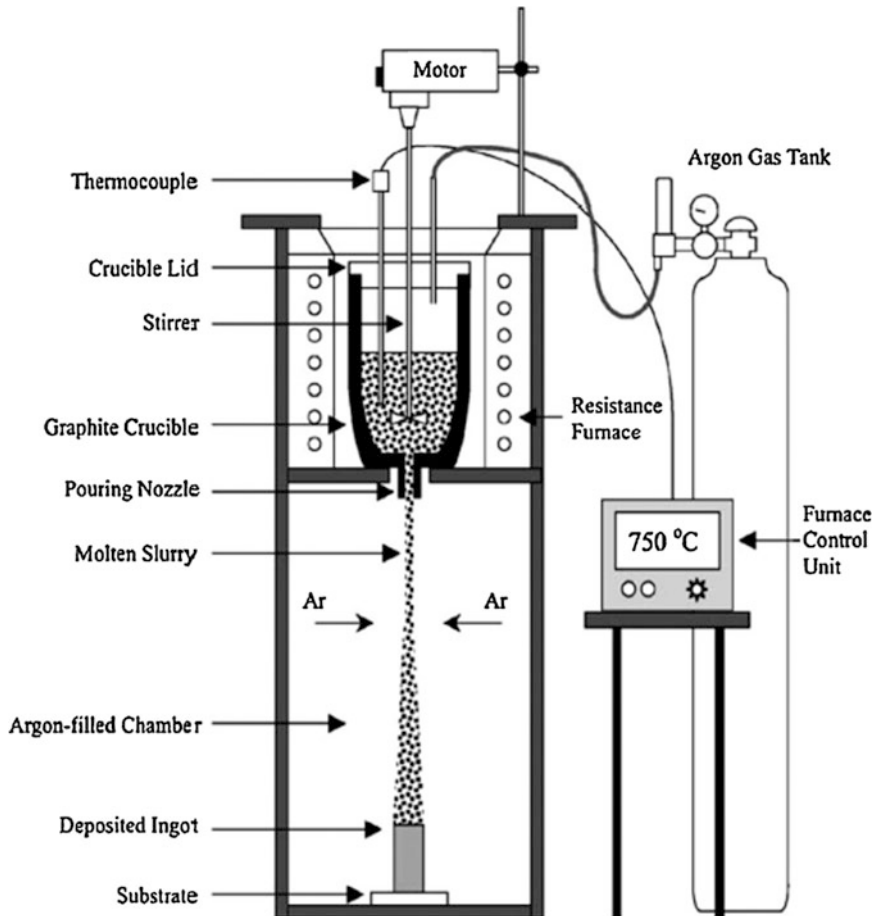


Fig. 2.4 Schematic of the DMD process [49]

on a metallic substrate. The ingot can be then extruded (Fig. 2.4). Although the method is suitable for making both Al- and Mg-nanocomposites, most of the studies are pertained to Mg-nanocomposites. This is possibly due to the reason that it can overcome the major drawbacks observed during conventional processing of Mg-materials, viz., (i) oxides in the final product due to the highly oxidisable nature of Mg and (ii) retention of reinforcement particles in the crucible, due to the density differences between the Mg-matrix and reinforcement. These together can give rise to impurities, insufficient volume fraction of reinforcement and non-uniform dispersion, which can seriously impair the properties. As DMD is a bottom-pouring technique it can ensure elimination of oxide entry into the deposited product and complete utilization of the reinforcement. Various nanoparticles such as Al_2O_3 , SiC , Y_2O_3 , B_4C , BN , ZrO_2 , ZnO etc. as well as multi-walled carbon nanotubes

(MWNT) have been added to Mg/Mg-alloys to produce Mg nanocomposites [47–51]. In most cases, the microstructural characterization of the extruded composites showed uniform distribution of the ceramic reinforcements, good interfacial integrity between the ceramic reinforcement and the metallic matrix and limited amounts of porosity. Due to these reasons, the mechanical properties of the Mg-nanocomposites have often shown significant improvement in terms of both strength and ductility [52].

High pressure die casting. Among the traditional liquid processes, aside from stir casting and semi-solid casting techniques, high pressure die casting (HPDC) has also been applied for the production of MMNCs. In general, the process enables obtaining more detailed components as compared to gravity and low pressure die casting methods. The molten metal is forced into the die cavity under pressure, and both filling speeds and solidification rates are particularly high. For this reason, HPDC is characterized by fast cycle times, which may range from seconds to several minutes, depending on size and wall thickness of the casting. On the other hand, the process inevitably induces gas entrapment due to the highly turbulent flow of metal in the cavity [53, 54]. Very few works have been reported on the application of HPDC for the production of MMC, especially in the case of nanocomposites. HPDC was applied by Li et al. [9] to manufacture CNT-reinforced Al-based composites. The method allowed obtaining an increase of both tensile stress and elongation to failure as compared to the unreinforced alloy.

2.2 Solid State Routes

Solid-state processes for the production of bulk nanocomposites are based on powder metallurgy. Matrix/reinforcement wetting issues related to nanoparticles are clearly and noticeably diminished with respect to liquid and semi-solid routes [55]. To some extent, similar to primary liquid and semi-solid processes, PM processes enable to produce near-net shape components. Competitive advantages of this processing route include the capability of incorporating a higher volume fraction of reinforcement, as well as the possibility to produce matrix/reinforcement systems not obtainable by traditional liquid casting routes. PM is also of great interest for manufacturing of large batches, typically for the automotive industry, although usually of small sized components [56]. Major drawbacks include the high cost of the powders and the high amount of porosity which could characterize the final product, requiring further working steps (secondary processes) such as extrusion, rolling, or forging [57].

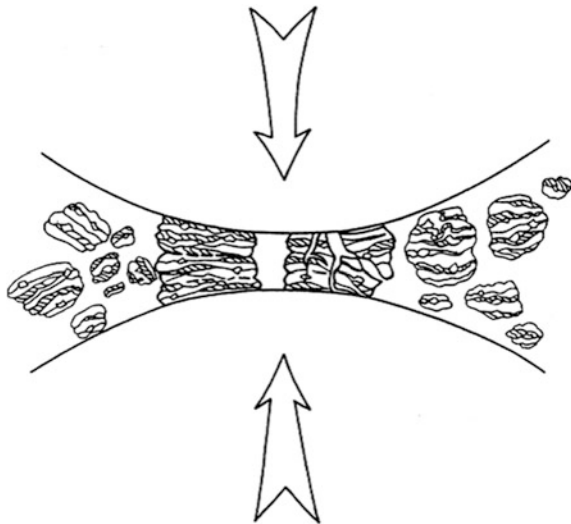
Powder metallurgy. Powder Metallurgy (PM) processes usually involve the following phases: blending of matrix alloy and reinforcing phase powders; compacting the blend, usually by cold pressing; degassing the compacted structure to remove volatile contaminants (lubricants, mixing and blending additives), water vapor and gases; green compacts can be then consolidated by different routes such as direct sintering, hot isostatic pressing (HIP), vacuum hot isostatic pressing,

hot extrusion or cold sintering [58]. Severe plastic deformation processes, such as equal channel angular pressing (ECAP), have been also applied to consolidate composite powders of e.g. Al7075 alloy and nano-TiO₂ [59].

Mechanical alloying. In the traditional PM process, the aim of the blending is simply to mix the powders without inducing material transfer between the mixed components. It is possible, however, to perform a high energy mixing through milling media, as to eliminate the voids between the matrix and the reinforcement powders, by incorporating hard ceramic particles into the matrix powder through a solid-state bonding [57]. For example, in mechanical alloying (MA), matrix and reinforcement are fused together by inducing cold welding, fracturing, and re-welding of the powder particles [55, 60, 61]. The strengthening of metallic alloys is achieved through grain size refinement and dispersion of nanometric particles. During the process, a small quantity of the base powders are loaded into a sealed container, together with the grinding media, then blended through agitation at high speed for a predetermined amount of time (Fig. 2.5). As the kinetic energy of the grinding balls depends on their mass and velocity, dense materials such as stainless steel or tungsten carbide are preferred. Main process parameters, influencing the quality of the composite, comprise ball-to-powder ratio (BPR), time and rotational speed of milling. After being milled, powders are compacted, degassed and consolidated.

A process control agent (PCA, usually referred to as lubricant or surfactant) is usually added while milling the powders, aiming to minimize the effect of cold welding and consequent formation of large powder clusters. Methanol, stearic acid, and paraffin compounds may be used for this purpose [62]. During the continuous severe plastic deformation, a refinement of the internal structure of the powders to the nanometer scale may occur, resulting in the production of nanostructured

Fig. 2.5 Mechanical alloying processing technique: milling action on the powders [61]

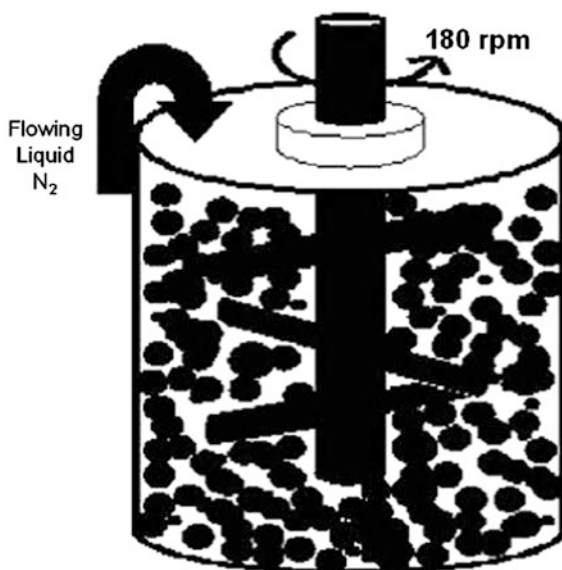


powders [60, 63]. The mechanisms of grain refining has been evaluated by Fecht [63]. The deformation is first localized in shear bands with high dislocation density. This initial phase is followed by annihilation and recombination of dislocations; as a result, nanometric sized grains may be formed during the milling process. As a third stage, the sub-grain boundary structure is transformed to randomly oriented high-angle grain boundaries. In the process, contamination of the powders must be carefully controlled. Possible sources of contamination are the milling tools, milling atmosphere, as well as the process control agent. During consolidation, impurities may influence microstructural evolution and grain growth, leading to a possible decrease of mechanical properties of the resulting composite [57, 61, 64, 65]. The milled powders obtained from mechanical alloying may also be employed as reinforcing particles for casting processes [1, 3, 13, 15, 16].

Mechanical alloying can also be accompanied by a solid state reaction, aimed to produce fine dispersion of oxides, nitrides and carbides in the light alloy matrix [56, 66–68]. In this case, the process is usually defined as *reaction milling* (RM). In order to allow the reaction to occur, the process control agent can be absent or a suitable milling atmosphere can be used to introduce reagents, i.e. oxygen, argon, nitrogen, or simply air [60]. Sometimes the PCA could be itself part of the reaction process [69].

In *cryomilling*, the milling phase is carried out at cryogenic temperatures (Fig. 2.6) or, in some cases, within a cryogenic medium, as liquid nitrogen [62]. A PCA (e.g. stearic acid) can be used to avoid severe sticking. During traditional milling process, the temperature increases due to the frictional heating. As a result, severe recovery and recrystallization of fine microstructures may occur [61]. On the contrary, when cryomilling is applied, recovery and recrystallization are suppressed

Fig. 2.6 Schematic of cryomilling process [62]



by the extremely low milling temperature, enhancing the beneficial effects of mechanical milling and leading to finer grain structures and more rapid grain refinement. As a result, nanocrystalline grain structures may be obtained [62]. Moreover, detrimental chemical reactions between matrix and reinforcement are also suppressed at such low temperatures [57, 60].

Microwave sintering. In microwave sintering, the principle of heating and sintering is fundamentally different. Microwave heating is a volumetric heating process involving conversion of electromagnetic energy into thermal energy, which is instantaneous, rapid and highly efficient [52]. It is unlike the conventional sintering processes wherein thermal energy is transferred from the outer surface of the materials to the inner surface. In microwave sintering, heat is generated from within the materials and radiates outward due to the penetrative power of the microwaves [52]. Metal in the form of powder will absorb microwaves at room temperature and will be heated very effectively and rapidly. The microwave energy is absorbed by the materials and is not dependent on the heat transfer from the outer surfaces. It is observed that in this method, higher temperatures exist at the core whereas the surfaces experiences lower temperatures [52].

Any method that gives rise to differential heating (core-to-surface or surface-to-core) in the sample causes variation in microstructure along the thickness of the sample, and would usually result in poor properties. To eliminate such a disadvantage, hybrid microwave heating has been developed, which is described below.

Bi-directional hybrid microwave sintering. In this process, microwave susceptors such as SiC particles/rods are used to assist in the reduction of thermal gradient during sintering [70]. Such a type of microwave heating set-up is shown in Fig. 2.7 [71], (which uses a simple household microwave oven) wherein the compacted metal/composite powder billets are placed in the inner crucible and SiC powder is placed between the inner and outer crucibles. SiC powder absorbs

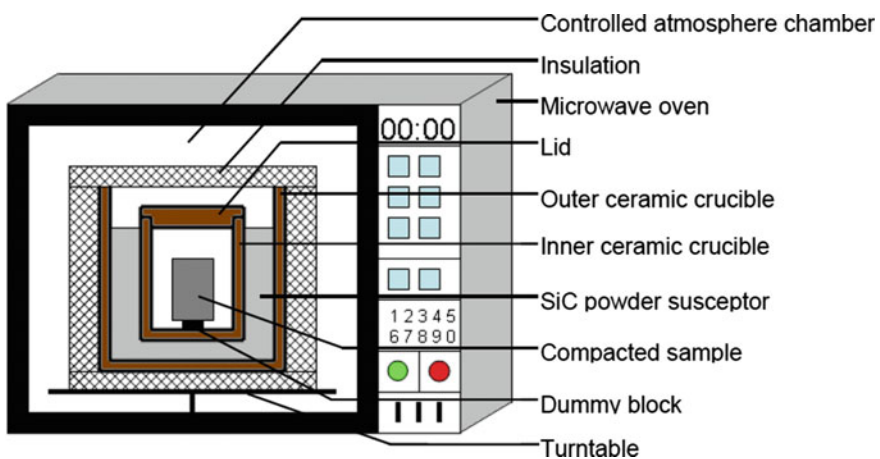


Fig. 2.7 Schematic of the bi-directional hybrid microwave-assisted sintering [71]

microwave readily and heats up quickly, providing radiant heat to externally heat the compacted billets (preforms). On the other hand, the compacted billets themselves absorb microwave and are heated from within. Thereby, uniform heat is experienced along the thickness of the specimen and the core-to-centre thermal variation is minimized [70].

The method has been used successfully for sintering light-metal (both Al and Mg) alloys and composites. Especially in the case of nanocomposites, as the bi-directional hybrid microwave sintering reduces the thermal variation, high sintering temperatures ($\sim 620\text{--}650\text{ }^{\circ}\text{C}$) can be utilized for a short period of time, which is almost close to the melting point of these metals [52, 70]. This ensures good bonding of the nanoparticles within the preforms and eliminates porosity at the particle/matrix interface, which is a major concern in conventionally sintered materials [70, 72]. The resulting microwave sintered nanocomposite product is dense with fine microstructural characteristics. The advantages of hybrid microwave energy for materials processing include: (i) time and energy savings, (ii) rapid heating rates, (iii) fine microstructures and hence improved mechanical properties and better product performance, (iv) does not require inert atmosphere, even for readily oxidisable materials such as Mg, and (v) lower environmental impact [52, 70].

Using the bi-directional hybrid microwave sintering technique nano-reinforcements such as Al_2O_3 , SiC, CNT, Y_2O_3 , BN etc., have been incorporated into pure Mg matrix. The sintered products are usually characterised for their microstructure and mechanical properties after hot-extrusion. In most studies, the density of the composites is much closer to the theoretical density indicating minimal porosity, a significant advantage that has given rise to better microstructural and mechanical characteristics, including enhanced ductility [70].

2.3 Semi-solid State Processes

Semi-solid casting processes involve the shaping of a partially solid mixture (slurry) with relatively small near-globular grains at solid fractions between 20 and 60 %. As a common advantage, semi-solid processes are in general characterized by low shrinkage and porosity, non-turbulent filling and lower processing temperature. They can be divided into two main groups of processes, namely thixo-processes and rheo-processes (compocasting).

Thixoprocessing. In thixo-processes a proper solid feedstock is reheated and partially melted. The base material is generated by allowing a liquid melt to partially solidify under controlled conditions (low superheat and rapid cooling, usually combined with significant convection in the liquid), as to induce the formation of crystals in the slurry. The feedstock may be produced in a variety of ways such as with mechanical stirring during solidification as in rheocasting [73–75], continuous casting combined with magneto-hydrodynamic stirring for grain refining [73, 76], and ultrasonic treatment [73, 77, 78] again for grain refinement. Other methods to

prepare a fine grained non-dendritic material is by spray casting [73, 79] and low-superheat casting [73, 80] processes. Many of the processes employ intense chemical inoculation to maximize the efficiency of the above mentioned processes, particularly magneto-hydrodynamic stirring and low-superheat casting [73, 81]. Usually, the semi-solid material is then injected into hardened steels dies as final stage process. Being the slurry obtainable in several ways, the process results to be tailorable and optimized for the production of MMNC [73, 82]. Although thixo-processes would offer a great possibility to premix nanoparticles into the feedstock through different production methods, to the authors' best knowledge, very few efforts strictly referring to thixocasting technology for MMNCs, aiming at the production of a globular microstructure, have been hitherto reported in the literature [31]. Nevertheless, it should be observed that several production routes involved the preparation of premixed ceramic/metallic or CNT/metallic feedstocks to be then diluted in the molten matrix [1, 3, 15, 16, 83, 84]. In the present review, such studies are reported in the stir casting or compocasting sections respectively in case the matrix is in its liquid or semisolid state.

Rheoprocessing. On the other hand, in rheo-processes, a special feedstock is not required, and the semi-solid slurry is generated starting from the liquid state by cooling the molten metal during the casting process itself. In common for all rheo-processes is that they are easier to implement in a foundry as they involve standard equipment for melting, transport, treatment, degassing and handling. The key difference between the various approaches is in the slurry making process, where great efforts are being made to create a robust on-demand slurry-making capability. Among rheo-processes techniques, it is worthwhile mentioning the ***New Rheo Casting process (NRC)***, which relies on a cooling slope to generate the initial slurry [85]. The molten metal is poured at low superheat (about 10 K) onto the side of a holing cup and a large amount of very small crystals are formed. The slurry is then held for a pre-set time in the cup, allowing the crystals to grow and spheroidise without additional shearing or stirring. Just before pouring, the temperature of the slurry is homogenised [86]. As a variation to the precedent, the ***Hong-Nano Casting method (H-NCM)***, [87], uses an electro-magnetic field in the pouring and cooling stages. This modification helps in homogenizing the temperature and increase the overall heat transfer, resulting in fast cooling and copious nucleation—approximately 1000 times higher than in the NRC process. Further, the ***Rheo Die Casting process (RDC)***, also known as Twin Screw Rheo Moulding (TSRM), involves the use of twin screws for mixing, providing a high amount of shearing. The molten metal is cooled at a controlled rate. The high level of shear is thought to break oxides into small, round particles which are well dispersed in the entire cast component [88, 89]. The slurry may be generated by letting the melt passing through a conversion reactor (a cooled copper or iron block with a twisting channel inside, causing the melt to cool and partially solidify under shear) in the so called ***Continuous Rheo-conversion Process (CRP)*** [89]. Other processes developed so far for semisolid metal processing are the ***Sub-Liquidus (SLC)*** [90, 91] and ***Semi Solid Rheo (SSR)*** casting processes [92], ***GISS*** process, ***Rapid Slurry Forming (RSF)*** [93, 94], ***Semi-Solid Metal (SSM)*** and ***ATS*** processes [95].

Although a multitude of different rheo-processes have been developed for semi-solid processing of metals, it appears that very little research has been conducted on adapting and using these methods for the production of MMNCs. In particular, as a result on literature survey, the most widespread technique employed is the so called compocasting route, which is a rheocasting process involving the injection of nanoparticles into semisolid state alloys.

Depending on the state of the matrix during casting operations, it is possible to distinguish between semisolid–semisolid (SS) and semisolid–liquid (SL) routes. In the first one, during casting operations, the matrix is partially liquid, while in the second one, the slurry containing nanoparticles is heated up to the fully liquid state before being poured into the mold [96]. Similarly to stir casting route, a vortex may be generated so as to introduce the particles within the semi-solid slurry. As concerning the metallic matrix to be processed through compocasting technique, it is a favourable feature to present a wide temperature interval for solidification (i.e. difference between solidus and liquidus temperatures). For example, as concerning aluminium alloys, one of the most used casting alloys is A356 alloy, which in fact solidifies in a temperature interval of 43 °C [96]. Compocasting is generally thought to be a processing route allowing to obtain quite uniform distribution of reinforcing particles, as well as to enhance particle wettability [97, 98]. It has been reported, in fact, that the primary solid particles which are formed in the semi-solid slurry are able to mechanically entrap the reinforcing phase and to prevent their gravity segregation, as well as to reduce their agglomeration tendency [99]. Moreover, the lower porosity which is usually observed in experimental studies, is attributed to the better wettability between the matrix and the reinforcement particles as well as the lower volume shrinkage of the matrix alloy [98]. Despite these advantages, some agglomerates, inevitably induced by the high surface-to-volume ratio, as well as to Van der Waals interactions are still reported [96].

Compocasting has been applied to produce MMNCs employing reinforcing phases such as SiC, CNTs and Al_2O_3 in aluminium matrices [100–102], while SiC has been used as to strengthen Mg alloys [103–110]. Some authors, such as Chen et al. [103], reported having successfully introduced nanoparticles within the matrix at the semi-solid state, while dispersing them through ultrasonication at the fully liquid state. In general, as a result of comparative studies, compocasting process is reported to induce less porosity in the composites and to obtain, at equal reinforcement fraction, higher mechanical properties than the traditional stir casting process [111–113].

2.4 Hybrid Methods and Other Routes

Nanocomposites can also be prepared through a combination of the above-mentioned processes. Often in these methods, powder-metallurgy techniques are utilized prior to producing the nanocomposite (e.g. by liquid-state or compocasting routes). Selective alloying element(s) of interest (micron/nano-sized

metal powders of suitable weight/volume fraction) are prepared in form of master powders by: (i) either mixing/blending with the reinforcement nanoparticles, or (ii) mechanical alloying using a ball-mill, which are then incorporated into the melt [114, 115]. In non-reactive powder mixtures, each of these intermediary steps is performed to ensure uniform distribution of the constituent elements in the liquid metal and further to avoid agglomeration of particles, which usually occurs when nanoparticles are added directly to the melt [114]. In reactive powder mixtures, the mechanical alloying process induces chemical reaction between the powders (such as intermetallic phase formation), which are controlled via processing parameters employed during mechanically alloying, and depends on the formation enthalpy [115].

It should be noted here that in the traditional method of adding the reinforcements/alloying elements to the melt (i.e. direct addition), the reaction of the constituent elements is controlled by their reaction with the molten metal, and hence usually forms intermetallic phases with matrix as the main constituent. This usually makes the matrix brittle [114, 115]. In contrast, the intermediary step of pre-processing of the constituent elements (alloying element/reinforcement) brings about a major advantage of the formation of matrix-free intermetallic phase and upon its addition to the melt, enhances the mechanical properties of the developed material by acting as a strengthening phase [114, 115]. Multiple-reinforcements (metal/ceramic reinforcements with micron/submicron/nano-sizes) can also be considered, as the intermediary process can be more effective. In the case of multiple reinforcements, wherein nano-sized particles are present along with micron-sized particles having irregular shape, size and sharp corners it has been observed that in the pre-processing step, the large-sized irregular particles (micron-sized) breakdown to small-sized (almost uniform) with rounded edges. By the reason that the shape/size of the large-sized irregular particles change during pre-processing step, the stress concentration reduces, thereby providing enhanced resistance to fracture [114, 115].

2.4.1 Other Processes

Friction Stir Processing (FSP). FSP is based on friction stir welding and was initially used to produce surface-reinforced composites [116]. In recent years, research efforts are being attempted to use the process as an alternative route to incorporate nanoparticles into the metal matrix to form bulk nanocomposites. During FSP, a rotating tool with a shoulder and a pin is plunged into the surface of the work piece (the desired base matrix) with grooves filled with the desired volume fraction of nanoparticles. As the tool rotates, it feeds forward to cover the region of interest [116]. A schematic showing the FSP is given in Fig. 2.8 [117]. Most studies have shown significant hardness improvement of the produced nanocomposites. However, the process is still in the developmental stage, as the uniform dispersion of nano-sized reinforcements remains a challenge and the thickness of the

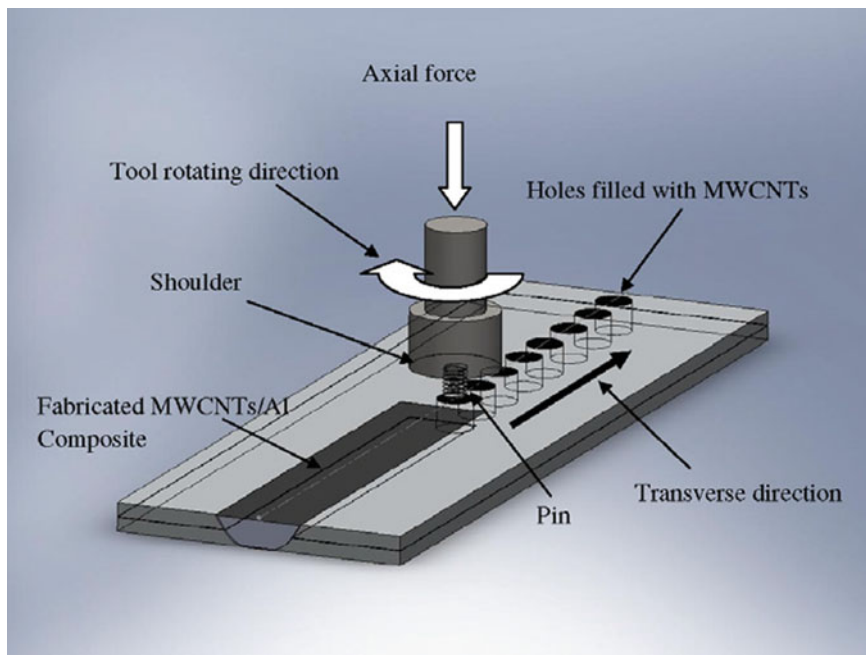


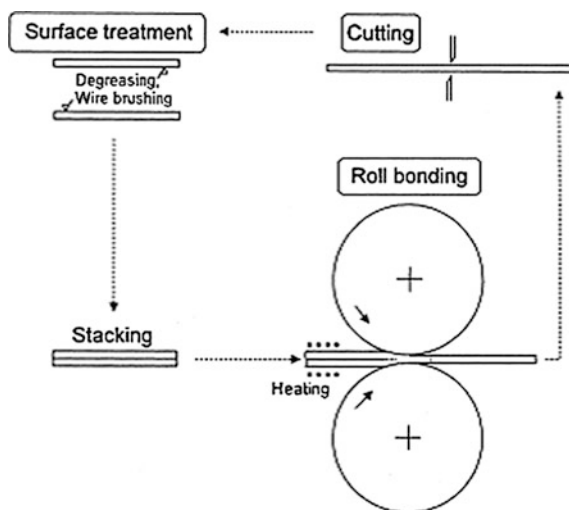
Fig. 2.8 Schematic of friction stir process (FSP) for Al/CNTs nanocomposites [117]

component is limited by the action of the stirring tool; either Al- [117–119] and Mg- [120, 121] based nanocomposite have been produced by FSP by incorporating ceramic or carbon based nano-reinforcement.

Accumulative roll bonding. Accumulative roll bonding (ARB) is a solid state method which enables to produce MMCs in the form of sheets, and was firstly developed in 2008 by Alizadeh et al., who produced nano-structured Al-SiC composites [122]. The method belongs to severe plastic deformation processes and, in addition to producing MMCs, allows to obtain nanostructured and ultrafine grained materials [123–125]. ARB process consists in roll bonding stacked sheets (about 50 % of thickness reduction), then cutting the roll-bonded material and rolling it again after stacking the pieces over each other [123] (Fig. 2.9).

Particles are usually distributed on the sheets before processing; by increasing the number of cycles, an enhancement in particles uniformity is usually reported, as well as a decrease in porosity content and an increase in the bonding strength between matrix and reinforcement [123]. As a result, mechanical properties of the ARB processed unreinforced metals and composites have been reported to increase (YS, compressive strength, wear resistance) [126–129]. Micrometric as well nano-sized reinforcing particles (e.g. Al_2O_3 , SiC, SiO_2 and B_4C particles) have been added to metallic matrices through ARB process [126, 130–132].

Fig. 2.9 Schematic of accumulative roll bonding (ARB) process [124]



Accumulative roll bonding (ARB) was also used to improve bonding of Multi-walled Carbon NanoTubes MWCNTs with the matrix and to reduce their agglomeration tendency by producing Al–MWCNT composite stripes to be then diluted in a molten Al-matrix of appropriate composition followed by squeeze casting [133].

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Jayalakshmi, S.; Morri, A.; Rotundo, F.; Toschi, S.; Singh,
R.A.

2017, XII, 164 p. 89 illus., 31 illus. in color., Hardcover

ISBN: 978-981-10-2680-5