Indian Journal of
Advances in Chemical Science

Article

Synthesis of Light Metal Nanocomposites: Challenges and Opportunities

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ABSTRACT

Light metal matrix nanocomposites (LMMNCs) are advanced materials, in which nanosized ceramic particles are reinforced in aluminum/magnesium matrices. In conventional metal matrix composites (MMCs), the incorporation of micron-sized reinforcements usually contributes to the high hardness and ultimate strength when compared to the unreinforced base material. However, most of these composites do not show plastic deformation (little or no yield) and exhibit a drastic reduction in ductility. This poses a major limitation for MMCs to be used in real-time applications. To overcome this drawback, composites with nanoscale reinforcements are being developed. From research studies, it has been established that LMMNCs are better materials as they show improved strength as well as high ductility resulting in enhanced toughness. However, for improvement in properties to occur, the nano-reinforcements should be distributed uniformly in the matrix, without any clustering or agglomeration. Hence, the greatest challenge in obtaining high-performance nanocomposites and realizing their application potential invariably lies in obtaining uniform distribution of nanoparticles. In this paper, the state-of-the-art processing methods employed in the advancement LMMNCs and the challenges encountered are presented.

Key words: Light metal matrix composites, Ductility, Nano-reinforcements, Processing techniques.

1. INTRODUCTION

The focus of aerospace and automotive industries has turned toward lightweight materials due to the depletion of oil reserves, increasing demand for fuel efficiency, and regulations on emission [1]. While aluminum (Al) and magnesium (Mg) metals are suitable in terms of being lightweight, their alloys do not fully meet the requirements in terms of properties. For this reason, composites are often preferred, as the incorporation of strong and stiff ceramic constituents (e.g., SiC, Al_2O_3, and B_4C) provides significant improvement in properties. However, despite these advantages, the major drawback that restricts the wider use of these materials is their poor ductility, i.e., low toughness. The poor ductility (little or no yield/plastic deformation) arises due to: (i) The presence of hard but brittle ceramic reinforcement phase, (ii) the micron size of the reinforcements that cause particle clustering during processing, and (iii) formation of undesirable chemical reactions at the reinforcement/matrix interface.

In this context, incorporation of nano-sized reinforcements to create light metal matrix nanocomposites (LMMNCs) is a promising alternative [2]. Nanoparticles can give rise to significant enhancement in strength properties due to the "dispersion strengthening like" effect, along with ductility retention/enhancement, giving rise to composites with enhanced toughness. However, the ability to achieve uniform distribution of the reinforcement (without agglomeration or clustering) plays a major role in defining the properties, which, in turn, is dependent on the processing route employed [3]. To address this concern, concerted research and development efforts are focused toward (i) making significant changes in the existing processes, (ii) introducing new processes, and (iii) adopting methods that are currently being used for altogether different manufacturing purposes. In this paper, the research trends in the processing of LMMNCs, the difficulties encountered, and the opportunities for the future are highlighted.

2. EXPERIMENTAL

Conventional metal matrix composites (MMCs) are produced by liquid-, solid-, and semi-solid-state processes. These production routes are also suitable for nanocomposites production. The choice of the processing route depends on several factors such as the reinforcement type, its distribution, matrix-particle bonding, control of matrix microstructure, process simplicity, and cost-effectiveness. The synthesizing methods discussed in the paper include stir casting, squeeze casting, ultrasonic-assisted casting, disintegrated melt deposition (DMD**)**, bidirectional microwave sintering, spark plasma sintering (SPS), and friction stir processing, as referred from Haque *et al*. [4], Ghomashchi and Vikhrov [5], Uozumi *et al*. [6], Mula *et al*. [7], Gupta *et al*. [8], Abramov *et al*. [9], Suslick *et al*. [10], Gupta and Sharon [11], Tun and Gupta [12], Saheb *et al*. [13], and Weglowski and Pietras [14].

3. RESULTS AND DISCUSSION

3.1. Liquid-state Processes

Liquid-state processing techniques are attractive as they are relatively simple, inexpensive and can be scalable to industrial level. The important liquid-state processing routes include stir casting, ultrasonic-assisted casting, infiltration techniques, and DMD method.

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ISSN NO: 2320-0898 (p); 2320-0928 (e)
DOI: 10.22607/IJACS.2019.703004

Received: 25th May 2019;
Revised: 18th July 2019;
Accepted: 25th July 2019

3.1.1. Stir casting

Stir casting process is usually used to produce conventional MMCs. In this process, the reinforcements, usually in the form of particles, are incorporated into molten metal followed by casting. Nanocomposites with uniform distribution of particles can be achieved by (i) the vortex created by mechanical stirring using a rotor that rotates the stirrer immersed in the liquid metal and (ii) by the stirring effect caused by the injection of an inert gas into the molten metal incorporated with the reinforcement (Figure 1) [4].

3.1.1.1. Challenges

The major issues related to this process are as follows: (i) Entrapment of gas, (ii) presence of slag in melts (that lead to high porosity and microdefects), (iii) non-desirable chemical reactions at the interface, and (iv) poor wettability of the nano-reinforcement with molten matrix causing particle agglomeration (cluster formation and non-uniform distribution of reinforcement). Such issues would cause severe deterioration of properties. Hence, the process parameters should be effectively standardized.

3.1.2. Squeeze casting/infiltration process

In this process, the composites are produced by the infiltration of a molten alloy into a ceramic fiber/particle perform followed by solidification [5]. The introduction of molten metal into the preform can be achieved either through the application of pressure or without it. In pressureless infiltration, the molten metal is poured on to the ceramic fiber bundles that are placed in a die and allowed to solidify. The solidified composites are then hot pressed to achieve 100% density. In contrast, the pressure infiltration process can be carried out by either gas infiltration or by squeeze infiltration. In gas infiltration, vacuum or inert gas atmosphere is utilized to bring forth infiltration. Its advantages are improved wettability due to the increased surface activity of reinforcement in vacuum environment, removal of entrapped gas, and near net shape of components. Drawbacks include phase segregation and chemical reaction between matrix/fiber. Squeeze infiltration process involves infiltration of molten metal into a preform using hydraulic pressure. By this method, the drawbacks of phase segregation and interface reaction can be eliminated due to the application of hydraulic pressure. Furthermore, the pressure application increases solidification rate and produces fine-grained material.

3.1.2.1. Challenges

The preparation of preforms is a major challenge. For nanoscale reinforcements, usually, only carbon nanotubes (CNTs) are used as preforms for they have dimensional anisotropy (i.e., aspect ratio) [6]. Improperly made preforms can cause local inhomogeneous distribution of CNTs causing large variation in volume fraction within the composite. Further, if the preform preparation is not sound (e.g., less compaction pressure during perform making), it may distort/break during squeeze infiltration.

3.1.3. Ultrasonic-assisted casting

Particle agglomeration/cluster formation in nanocomposites can be eliminated effectively using this method [7]. Conventional mechanical stirring methods usually cause agglomeration. In comparison, in the ultrasonic-assisted method, ultrasonic wave (frequency range: 18–20 kHz) is employed during or after adding a reinforcing phase into the molten metal, which creates a uniform stirring effect (Figure 2) [8].

The principle involves the use of high-intensity ultrasonic waves that can generate transient cavitation [9] which causes a pressure gradient within the bulk of a molten metal, giving rise to stirring effect. The high-intensity ultrasonic waves induce the formation, growth, pulsating, and collapsing of tiny bubbles in the liquid phase. In every cycle, bubbles collapse in $<10^{-6}$ s, producing micro "hot

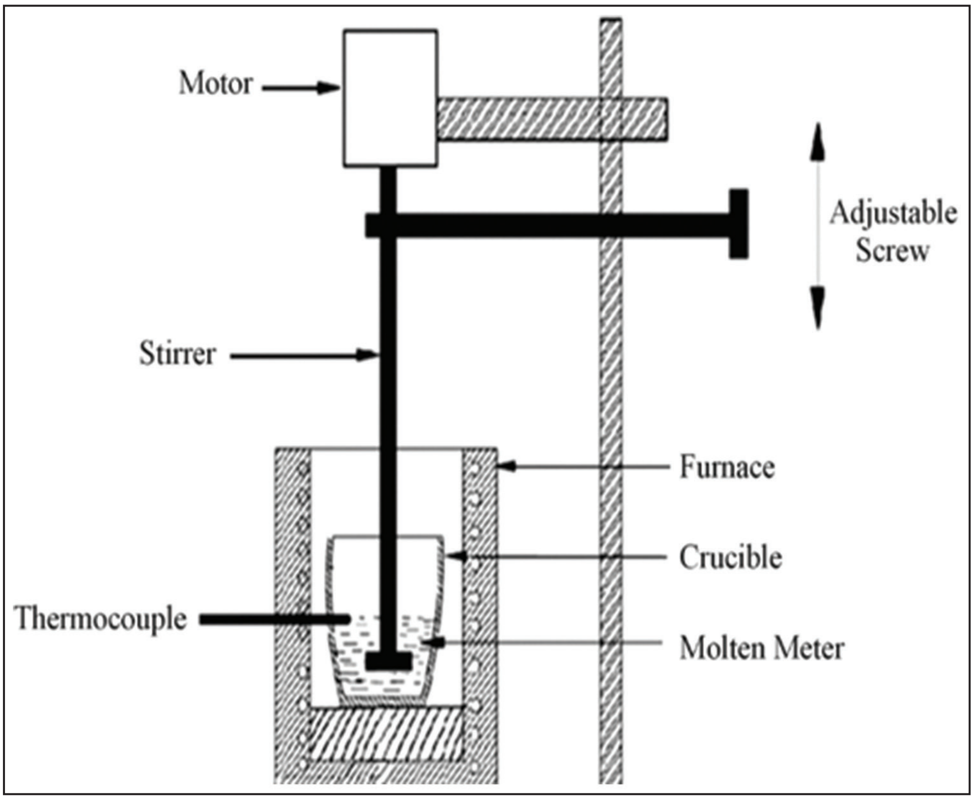

Figure 1: Schematic of stir casting method [4] (©2014 Scientific Research. Open Access).

spots" that can reach temperatures of ~5000°C, pressures of ~1000 atm, and heating/cooling rates >1010 K/S during microseconds transient [10].

When used for making nanocomposite, the entrapped air in voids of particle clusters would act as nuclei for cavitation, which would eventually break the clusters while achieving uniform dispersion. Gases/impurities are also removed due to the high pressure and temperature created, thereby enhancing the wettability. This method is extremely successful in producing composites with uniform dispersion of nano-reinforcements.

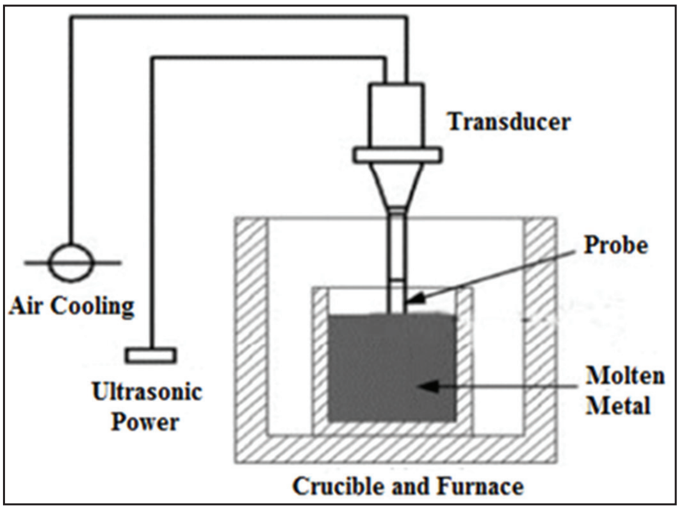

Figure 2: Schematic diagram showing the ultrasonic-assisted casting process [8] (©2013, Interscience. Open Access).

3.1.3.1. Challenges/drawbacks

The method is most suitable for laboratory scale due to small volume of melt. For large-scale production, probe size with higher source power is required.

3.1.4. DMD technique

DMD technique is a unique method which incorporates the combined advantages of gravity die casting and spray forming [11]. DMD process employs higher superheat temperatures and lower impinging gas jet velocity, which makes it different from the spray process. In this process, the molten metal/alloy with nanoparticles is stirred at a predetermined velocity and time using an impeller. The resulting composite slurry is then made to exit from the bottom of a crucible, followed by disintegration of melt by jets of inert gas at a superheat temperature of 750°C. It is finally deposited onto a metallic substrate (Figure 3). Due to melt disintegration by gas, higher solidification rate and fine-grained structure are achieved.

It is well-known that during melting of Mg by conventional methods, the following issues are encountered: (i) As Mg is highly oxidizable in nature, the presence of oxides cannot be avoided and (ii) as most of the reinforcements used are denser than Mg, they tend to settle at the bottom of crucible. These give rise to the presence of impurities, insufficient reinforcement volume fraction, and non-uniform reinforcement dispersion [11]. The major advantage of DMD is that, being a bottom-pouring technique, it can effectively eliminate oxide entry into deposited products and does not retain reinforcement in crucible, thereby enabling

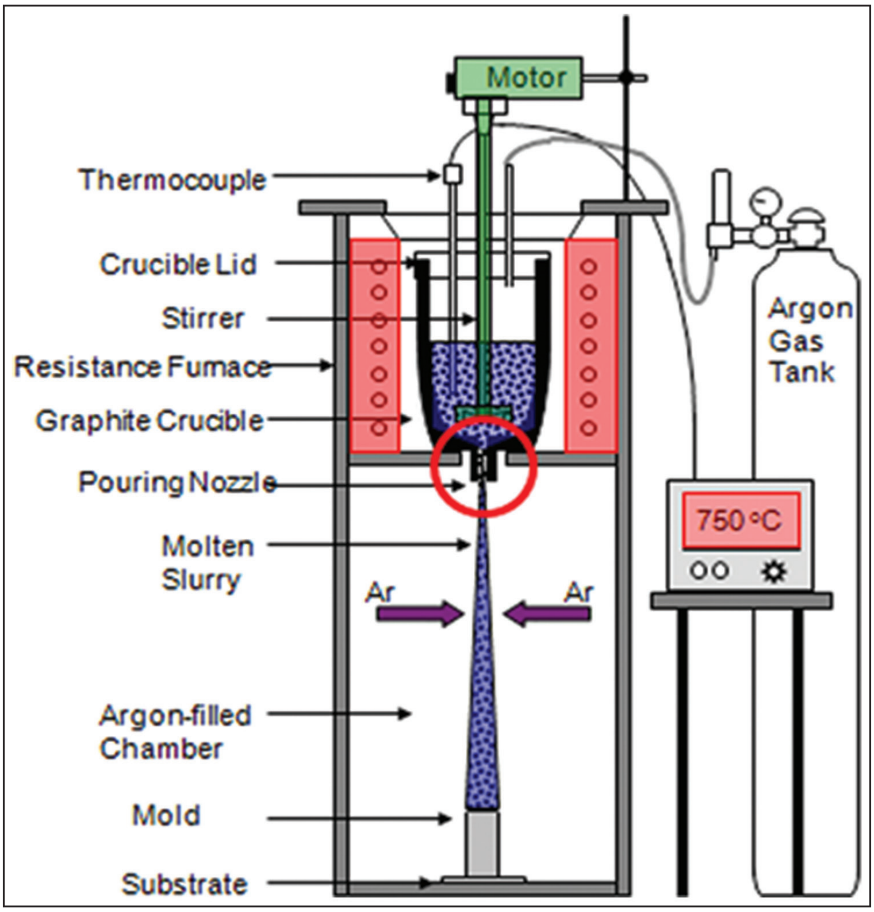

Figure 3: Schematic of the disintegrated melt deposition technique.

their actual volume fraction in the composite. Further, the disintegration of molten metal by inert gas results in higher solidification rate.

Salient features of the process are as follows:

- Combined advantages of gravity die casting and spray forming processes.
- Does not require separate melting and pouring units.
- Absence of oxides/slag and least metal wastage.
- Facilitate incorporation of nanoparticles.
- Does not retain nano reinforcements in crucible.
- Efficient process producing materials having fine-grained structure and minimal porosity.
- However, it should be noted that DMD is a primary process, after which a secondary process such as extrusion is usually employed. By this method, Mg nanocomposites have been successfully produced at laboratory scale.

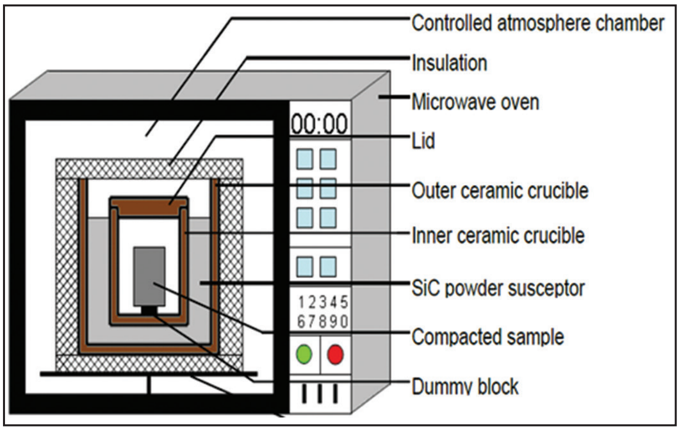

Figure 4: Schematic showing the bidirectional, microwave-assisted rapid sintering set-up [12] (©2007, Elsevier. Used with permission).

3.1.4.1. Challenges/drawbacks

(a) As DMD is a primary process, it is more suitable to produce MMNC ingots that can be used as precursors for making wrought products and (b) due to the process set-up/equipment design, it is difficult to be automated.

3.2. Solid-state Processes

3.2.1. Microwave sintering

Microwave heating can be defined as a volumetric heating process that involves the conversion of electromagnetic energy into thermal energy [11]. Unlike the "furnace sintering" process, in regular microwave sintering, heat is generated from within the materials and is then radiated outward due to the penetrative power of microwaves. Due to this phenomenon, microwave sintered materials exhibit higher temperatures at the core than at the surface causing a thermal gradient. This causes variation of microstructure and properties. Such microstructural inhomogeneity can be overcome using "bidirectional hybrid microwave-assisted rapid sintering" method (Figure 4) [12].

In this process, the reduction of thermal gradient during sintering is achieved using SiC particles/rods (microwave susceptors). In this method, two crucibles are used. SiC powder is packed in between the inner and outer crucibles, and the compacted metal/composite powder billets are placed in the inner crucible. SiC powder absorbs microwave readily, due to which it heats up rapidly providing radiant heat. This heat externally heats the compacted billets; while the compacted billets get heated internally by themselves as they absorb microwave. This "bidirectional heating effect" prevents the core-to-surface thermal gradient [11]. This gives rise to high sintering temperatures (620–650°C) that can be generated within a short period of time (12–14 min). Note that these are almost close to the melting points of Al and Mg. Such high temperature results in enhanced wettability and reduced porosity in the nanocomposite. Some of the merits of the process are

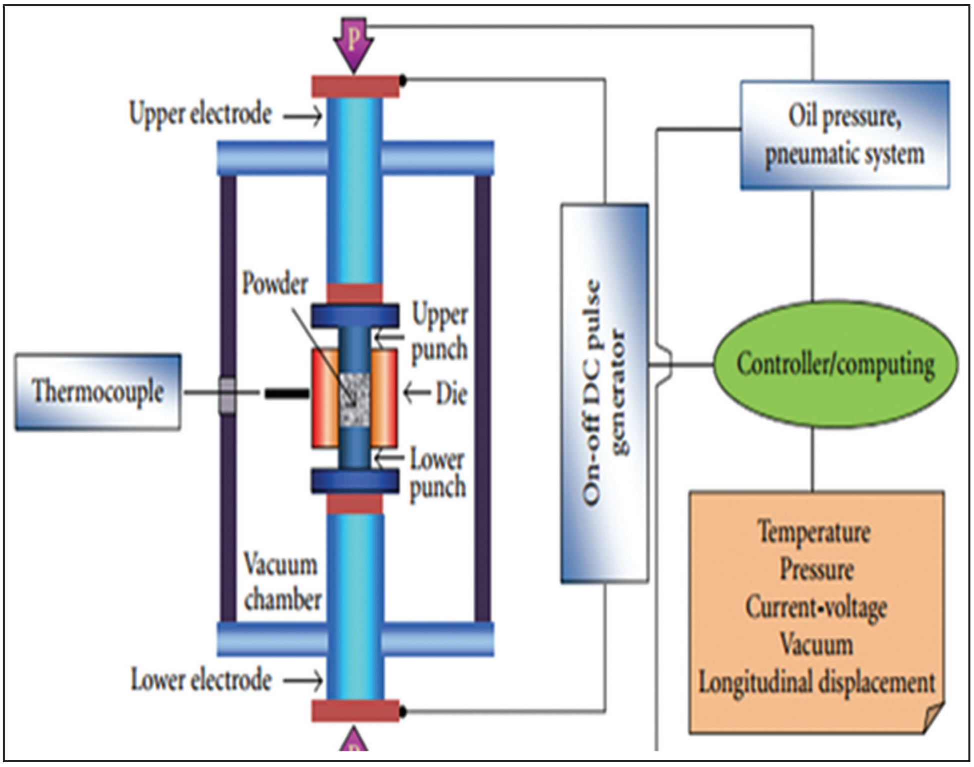

Figure 5: Process set-up of spark plasma sintering [13] (©2008, Hindawi Publishing. Open Access).

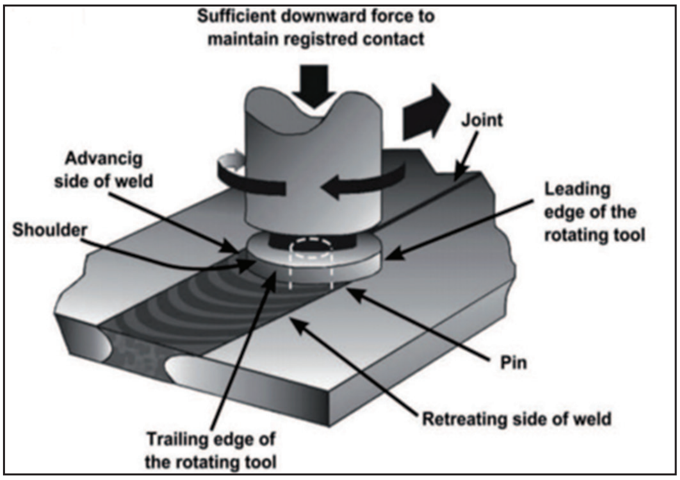

Figure 6: Method of surface composite production using friction stir process [14] (©2011, De Gruyter Open. Open Access).

as follows: (i) High and rapid heating rates, (ii) low sintering time, due to which even for oxidizing materials like Mg, does not require inert atmosphere during sintering, (iii) high sintering temperature that results in relatively low porosity, and (iv) fine microstructure and better mechanical properties [11].

3.2.1.1. Challenges

At present, the process is widely used only at research scale, usually followed by extrusion. The process (i) is limited in specimen dimensions, (ii) requires calibration of sintering time and temperature for varying specimen thicknesses, and (iii) needs to be investigated for the effectiveness of "bidirectional heating" for complex-shaped sintered parts, without any requirement of secondary process.

3.2.2. Spark plasma sintering

The major drawbacks in conventional sintering are (i) porosity and (b) matrix grain growth during hot working that weakens mechanical properties. The SPS is an effective non-conventional sintering method used to obtain fully dense materials with fine grain size [13]. In SPS, the densification is facilitated using a current. The process uses the Joule heating effect, wherein a pulsed DC current is directly passed through a graphite die and composite powder compact (Figure 5). The heat generated is used to densify the powder compacts near to its theoretical density. Like microwave heating, in SPS too, the heat generation is internal and facilitates high heating rates (up to ~1000 K/min), making the sintering process very fast (within a few minutes). Such rapid sintering facilitates complete densification of powder without coarsening [13].

3.2.2.1. Challenges/drawbacks

(i) Very expensive, (ii) temperature calibration is not accurate, and (iii) more suitable for symmetrically shaped specimens.

3.2.3. Friction stir processing

FSP is based on friction stir welding and is used to produce surface composites [14,14]. First, groves are made in the matrix (workpiece), in which required volume fraction of nanoparticles is filled. During FSP, a rotating tool is plunged into the surface of the workpiece (Figure 6) [14]. As the tool rotates, over the groves, it generates heat by friction over the region of interest and mixes/incorporates the reinforcement into the surface of the matrix. In recent years, efforts are being made to use this process to form bulk nanocomposites. However, the major challenge is to obtain uniform dispersion of nanosized reinforcements.

4. FUTURE OPPORTUNITIES

From the available literature, it could be seen that the nanocomposites show significant improvement in hardness, strength, and ductility when they were produced after careful standardization and judicious selection of process parameters (by any of these processing methods) [11]. To utilize these enhanced properties in real-time applications, first, upscale production and component level production should be initiated. Furthermore, most of the studies on property evaluation are related to basic microstructural and mechanical properties (hardness, tensile, compressive tests, and to some extent wear tests). It is worthwhile to investigate other industry critical properties such as fatigue, creep, corrosion/oxidation, and impact resistance/crash-worthiness, which would contribute to technological advancement of the field of nanocomposites.

5. CONCLUSION

1. Nanocomposites are promising materials to replace conventional metal/alloys and their composites with micron-sized reinforcements.
2. Most of the liquid-state processes used for micron-sized reinforced composites can also be adopted for making nanocomposites.
3. Uniform dispersion of nano-reinforcements devoid of agglomeration/clustering is the major requirement to achieve high-quality nanocomposites.
4. Judicious selection of processing method and standardization of parameters is essential for producing high-quality nanocomposites.
5. Nanocomposites produced usually have high ductility and high toughness.

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**Bibliographical Sketch*

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