Available online at
www.ijacskros.com

Indian
Journal of Advances in
Chemical Science

Indian Journal of Advances in Chemical Science S1 (2016) 283-288

Synthesis of Light Metal Nanocomposites: Challenges and Opportunities

S. Jayalakshmi[1]*, R. Arvind Singh[2], M. Gupta[3]

[1]Department of Aeronautical Engineering, Bannari Amman Institute of Technology, Sathyamangalam - 638 401, Tamil Nadu, India. [2]Department of Mechanical Engineering, Bannari Amman Institute of Technology, Sathyamangalam - 638 401, Tamil Nadu, India. [3]Department of Mechanical Engineering, 9 Engineering Drive 1, National University of Singapore (NUS), 117576, Singapore.

Received 22nd April 2016; Revised 14th May 2016; Accepted 17th May 2016

ABSTRACT

Light metal matrix nanocomposites (LMMNCs) are advanced materials in which nanosized ceramic particles are reinforced in Al/Mg 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 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 a 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 light-weight 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 light-weight, 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, B_4C) provide a significant improvement in properties. However, in spite of 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 nanosized reinforcements to create light metal matrix nanocomposites (LMMNCs) is a promising alternative [2]. Nanoparticles can give rise to a 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 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

*Corresponding Author:
E-mail: jayalakshmi.subramanian@gmail.com
Phone: +91-9566784068

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), bi-directional microwave sintering, spark plasma sintering (SPS), and friction stir processing (FSP), as referred from [4-14].

3. RESULTS AND DISCUSSION

3.1. Liquid-state Processes

Liquid state processing routes are attractive as they are relatively simple, cost-effective, and are potentially scalable to an industrial level. The main processing routes include stir casting, ultrasonic assisted casting, infiltration techniques, and DMD method.

3.1.1. Stir casting

Stir casting also known as "vortex method" is widely used to produce nanocomposites. It involves incorporation of reinforcements (in particle form) into molten metal, followed by casting. Homogeneous distribution of reinforcement is achieved by (i) A rotor rotating in a liquid metal that creates a vortex or (ii) by injection of gas carrying the reinforcement into the liquid metal (Figure 1) [4]. Finely distributed slurries produced are then shaped by conventional casting techniques.

- Challenges

Some of the issues associated with this process are: (i) Gas entrapment, (ii) slag in melts (that lead to high porosity and micro-defects), (iii) Undesired chemical reactions at the interface, (iv) Low wettability of the nanoreinforcement with molten matrix that increases the tendency of particles to agglomerate (formation of nanoparticle clusters, non-uniform distribution of reinforcement). Such issues would cause severe deterioration of properties. Hence, to successfully implement this process, careful standardization of process parameters should be properly selected.

3.1.2. Squeeze casting/infiltration process

The squeeze casting/infiltration process involves the infiltration of a molten alloy into a ceramic fiber/particle preform followed by solidification [5]. The introduction of molten metal into a preform could be achieved either through pressureless infiltration or by infiltration under pressure. In pressureless infiltration, ceramic fiber bundles are first placed in the die. The molten metal is poured onto it and allowed to solidify. The solidified composites are then hot pressed to achieve 100% density. In contrast, the pressure infiltration process is carried out in two ways, namely via gas infiltration and squeeze infiltration. In gas infiltration, vacuum, or inert gas atmosphere is utilized to bring forth infiltration. Advantages include an increase in the wettability due to the increased surface activity of reinforcement in a vacuum environment, elimination of gas entrapped, and achieving near-net shaped components. Disadvantages are segregation of phases and reaction between matrix/fiber due to the slow nature of the process. 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 encountered in gas infiltration can be eliminated due to the application of hydraulic pressure (as it increases solidification rate).

- Challenges

The preparation of preforms is a major challenge. For nanoscale reinforcements, carbon nanotubes (CNTs) are used as preforms for they have dimensional anisotropy (i.e. aspect ratio) [6]. Improperly made preforms can cause the local inhomogeneous distribution of CNTs causing large variation in volume fraction within a solidified composite. Furthermore, in case that the preform is not well prepared (e.g., insufficient binder) it has the tendency to break during application of squeeze pressure.

3.1.3. Ultrasonic-assisted casting

This method is effective in mitigating particle cluster formation in nanocomposites that occur due to low wettability and high tendency of agglomeration of nanoparticles [7] Agglomeration is usually encountered in conventional stirring methods such as mechanical stirring. In contrast, ultrasonic-assisted method employs subjecting melts with ultrasonic waves (frequency range: 18-20 kHz) during or after adding a reinforcing phase. This is followed by casting (Figure 2) [8].

The principle involves the use of high-intensity ultrasonic waves that can generate transient cavitation and acoustic streaming in liquids [9]. Acoustic streaming causes pressure gradient within the bulk of molten metal that produces the stirring effect. During cavitation, cyclic high-intensity ultrasonic waves induce the formation, growth, pulsating, and collapsing of tiny bubbles in the liquid phase. At every cavitation cycle, bubbles implosively collapse in $<10^{-6}$ s, producing micro "hot spots" that can reach temperatures of ~5000°C, pressures of ~1000 atm, and heating/cooling rates >1010 K/S during microseconds transient [10].

During cavitation of nanocomposite melt, air entrapped in voids of particle clusters serves as nuclei for cavitation that can break the clusters thereby providing a uniform dispersion. The high pressure and temperature developed also removes gasses/impurities and enhances the wettability of nanoreinforcement. This method is extremely successful in producing composites with uniform dispersion of nanoreinforcements.

- Challenges

For large scale production, it requires to up-scale the probe size with higher source power to ensure its effect over a large volume of melts.

3.1.4. DMD technique

DMD technique is a liquid-state processing method, which has the combined advantages of gravity die casting and spray forming [11]. Unlike in the spray process, DMD process employs higher superheat temperatures and lower impinging gas jet velocity. This process involves stirring of nanoparticles with predetermined stirring velocity and time using an impeller when the metal/alloy is in a molten state. 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 and is finally deposited onto a metallic substrate (Figure 3). The disintegration of composite melt ensures higher solidification rate and fine-grained structure.

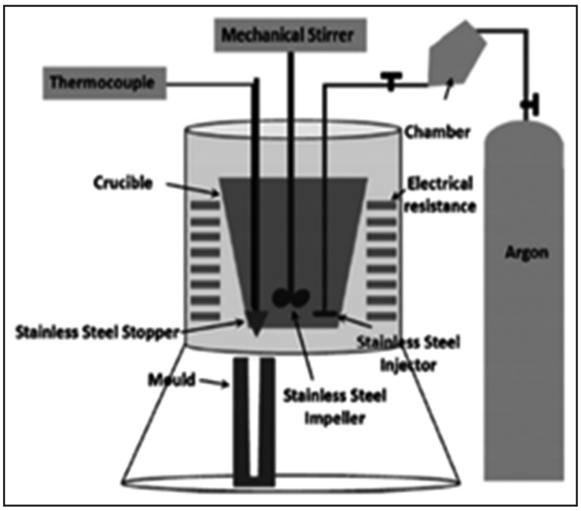

Figure 1: Schematic of Stir casting (vortex) method [4] used for the production of nanocomposites.

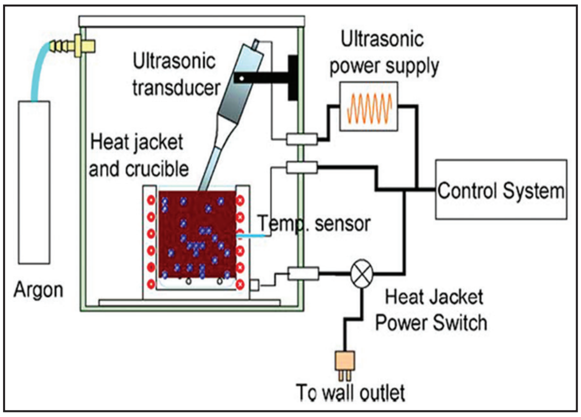

Figure 2: Schematic diagram showing the ultrasonic-assisted casting process [8].

Particularly during Mg materials production via conventional methods, persisting issues commonly encountered are (i) Presence of oxides in the final product and (ii) retention of reinforcement particles in crucibles (i.e., most of the reinforcement are denser than Mg, and hence has the tendency to settle at the bottom of crucible). These issues give rise to impurities, insufficient reinforcement volume fraction and non-uniform reinforcement dispersion [11]. Given that DMD is a bottom-pouring technique, it ensures (i) Effective elimination of oxide entry into deposited products, (ii) complete utilization of the reinforcement, and (iii) higher solidification rates owing to disintegration of molten metal by an inert gas.

Salient features of the process are:

- Combined advantages of casting and spray forming processes.
- Eliminates the requirement for separate melting and pouring units.
- Removes oxides/slag and least metal wastage.
- Flexibility of incorporation of nanoparticles.
- Eliminates the retention/settling of nano-reinforcements in crucible.
- High process yields and gives rise to fine-grained materials with minimal porosity.

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.

- Challenges

(a) Not suitable to produce cast components as the method is suitable to produce MMNC ingots to be used as precursors for making wrought products, (b) difficult to be automated and to be used for continuous casting operations unless modifications are made in equipment design.

3.2. Solid-state Processes

3.2.1. Microwave sintering

Microwave heating is a volumetric heating process that involves the conversion of electromagnetic energy into thermal energy [11]. Unlike in conventional 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, which results in variation of microstructure and properties. To avoid such occurrence, a "bi-directional hybrid microwave-assisted rapid sintering" has been developed (Figure 4) [11].

In this process, microwave susceptors such as SiC particles/rods are used to assist in the reduction of thermal gradient during sintering. The compacted metal/composite powder billets are placed in the inner crucible, and SiC powder is placed in between the inner and outer crucibles. As SiC powder absorbs microwave readily, it heats up providing radiant heat that can externally heat the compacted billets; the compacted billets themselves absorbs microwave and get heated from within/internally thereby preventing core-to-surface thermal gradient [11]. Due to this reason, high sintering temperatures (620-650°C) can be generated within a short period (12-14 min) that are almost close to the melting points of Al and Mg, by the virtue of which enhanced wettability and reduced porosity can be achieved. Advantages of this process include: (i) Rapid heating rates, (ii) low sintering time (even with Mg, the process does not require an inert atmosphere, (iii) lesser porosity, and (iv) fine microstructure and better mechanical properties [11].

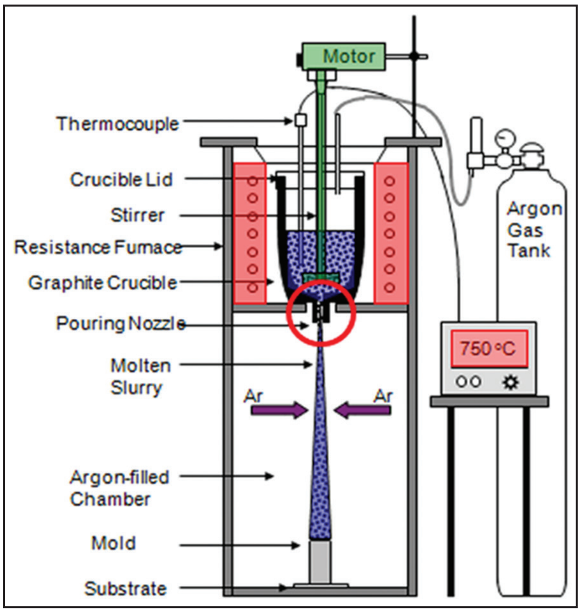

Figure 3: Schematic of the disintegrated melt deposition technique.

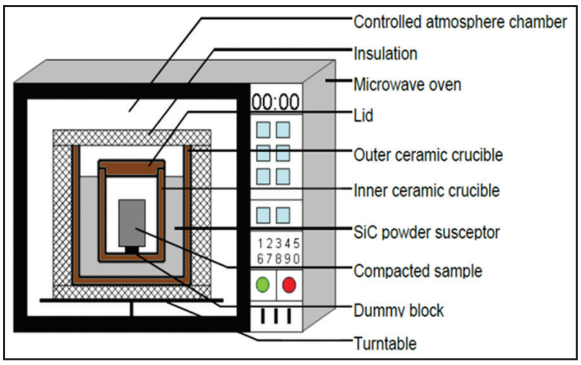

Figure 4: Schematic showing the bi-directional, hybrid microwave-assisted rapid sintering set-up.

- Challenges

(i) Currently, it is carried out only at research scale (ii) limited in specimen dimensions (iii) calibration of sintering time and temperature is required for varying specimen thicknesses.

3.2.2. Spark plasma sintering

The major drawbacks in conventional sintering are (i) porosity (b) matrix grain growth during hot working that weakens mechanical properties. The SPS is an effective non-conventional sintering method for obtaining fully dense materials with refined grain size [12]. In SPS, the densification is facilitated by the use of a current. A pulsed DC current is directly passed through a graphite die and composite powder compact (Figure 5). Joule's heating effect plays the role in densifying powder compacts achieving near theoretical density. In SPS, the heat generation is internal, and it facilitates high heating rates (up to ~1000 K/min), making the sintering process very fast (within a few minutes). The speed of the process ensures densification of the powders without coarsening [12].

- Challenges

(i) Expensive (ii) temperature calibration is not accurate (iii) more suitable for symmetrically shaped specimens.

3.2.3. FSP

FSP is based on friction stir welding and is used to produce surface composites [13]. During FSP, a rotating tool is plunged into the surface of a workpiece (matrix) with grooves filled with nanoparticles of required volume fraction (Figure 6) [14]. As the tool rotates, it covers the region of interest. In recent years, efforts are being made to use this process to form bulk nanocomposites. However, obtaining uniform dispersion of nanosized reinforcements still remains a challenge.

4. FUTURE OPPORTUNITIES

The available literature on the properties of nanocomposites shows that with any of these processing methods (carried out after overcoming the challenges and drawbacks), significant improvement in hardness, strength, and ductility are obtained [11]. To utilize these properties in real-time applications, studies pertaining to up-scale production, and component level production should be initiated. Currently, most of the studies on property evaluation

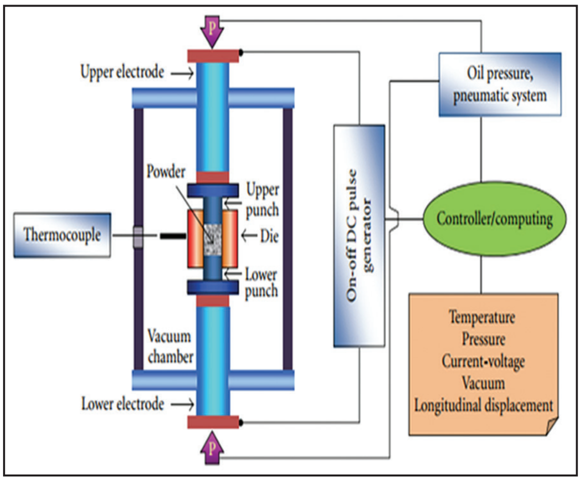

Figure 5: Process set-up of spark plasma sintering (SPS) [12].

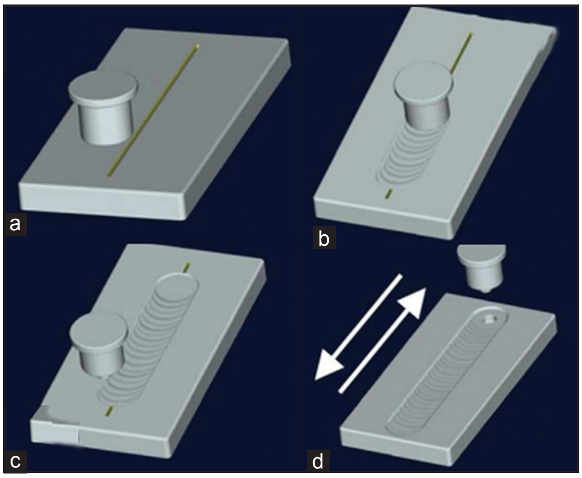

Figure 6: (a-d) Method of surface composite production using Friction stir process [14].

are related to basic microstructural and mechanical property evaluation (hardness, tensile, and compressive tests). It is worthwhile to investigate other industry-critical properties such as fatigue, creep, corrosion, oxidation, impact resistance, crash-worthiness, a high temperature which would contribute to the scientific and technological advancement of the field of nanocomposites. With the emphasis on processing-structure-property relationship, hybrid materials development can be carried by which properties unseen before can be realized.

5. CONCLUSION

1. Nanocomposites are promising materials to replace conventional metal/alloys and their composites with micron-size reinforcements.
2. Most of the liquid-state processes used for micron-size 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 absolutely essential in producing high-quality nanocomposites.
5. Nanocomposites produced usually have high ductility and high toughness.

6. REFERENCES

1. K. Kainer, (2006) ***Metal Matrix Composites: Custom-made Materials for Automotive and Aerospace Engineering,*** New York: John Wiley & Sons.
2. S. M. Choi, H. Awaji, (2005) Nanocomposites – A new material design concept, ***Science and Technology of Advanced Materials*, 6:** 2-10.
3. C. Suryanarayana, N. A. Aqeeli, (2013) Mechanically alloyed nanocomposites, ***Progress in Materials Science*, 58:** 383-502.
4. T. Noguchi, K. Asano, S. Hiratsuka, H. Miyahara, (2008) Trends of composite casting technology and joining technology for castings in Japan, ***International Journal of Cast Metals Research*, 21:** 219-225.
5. M. Ghomashchi, A. Vikhrov, (2000) Squeeze casting: An overview, ***Journal of Materials Processing Technology,* 101:** 1-9.
6. H. Uozumi, K. Kobayashi, K. Nakanishi, T. Matsunaga, K. Shinozaki, H. Sakamoto, M. Yoshida, (2008) Fabrication process of carbon nanotube/light metal matrix composites by squeeze casting, ***Materials Science and Engineering: A,* 495:** 282-287.
7. S. Mula, P. Padhi, S. Panigrahi, S. K. Pabi, S. Ghosh, (2009) On structure and mechanical properties of ultrasonically cast Al–2% Al2O3 nanocomposite, ***Materials Research Bulletin*, 44:** 1154-1160.
8. Y. Yang, X. Li, (2007) Ultrasonic cavitation based nanomanufacturing of bulk aluminum matrix nanocomposites, ***Journal of Manufacturing Science and Engineering*, 129:** 497.
9. O. Abramov, (1994) ***Ultrasound in Liquid and Solid Metals***, Boca Raton, FL: CRC Press.
10. K. S. Suslick, Y. Didenko, M. M. Fang, T. Hyeon, K. J. Kolbeck, W. B. McNamara, (1999) Acoustic cavitation and its chemical consequences, ***Philosophical Transactions A,* 357:** 335-353.
11. M. Gupta, N. M. L. Sharon, (2011) ***Magnesium, Magnesium Alloys, and Magnesium Composites***, Hoboken, NJ: John Wiley & Sons.
12. N. Saheb, Z. Iqbal, A. Khalil, A. Hakeem, N. Al Aqeeli, T. Laoui, R. Kirchner, (2012) Spark plasma sintering of metals and metal matrix nanocomposites: A review, ***Journal of Nanomaterials,* 2012:** 1-13.

13. K. Sun, Q. Y. Shi, Y. J. Sun, G. Q. Chen, (2012) Microstructure and mechanical property of nano-SiCp reinforced high strength Mg bulk composites produced by friction stir processing, ***Materials Science and Engineering: A***, **547:** 32-37.
14. C. Lee, J. Huang, P. Hsieh, (2006) Mg based nanocomposites fabricated by friction stir processing, ***Scripta Materialia,*** **54:** 1415-20.

***Bibliographical Sketch**

Dr. S. Jayalakshmi is a Professor at the Department of Mechanical Engineering, Bannari Amman Institute of Technology (BIT), Sathyamangalam, India, from 2014 till date. She received her PhD from the Indian Institute of Science (IISc), Bangalore, India in 2002. Prior to joining BIT, she was a Research Fellow at the Department of Mechanical Engineering, National University of Singapore (NUS). At NUS, she was actively involved in the design and development of new Mg-based alloys/nanocomposites. Earlier, from 2004 to 2009 she was a Visiting Scientist at the Korea Institute of Science & Technology (KIST), Seoul, South Korea. She has extensive experience in investigating the processing, characterization and structure-property relation of metallic materials including light metal alloys/composites, bulk metallic glasses and metallic amorphous alloys for hydrogen-related energy applications. Her current research interests include high entropy alloys, creep and tribological investigation of nanocomposites. She is also the recipient of Indian National Academy of Engineering (INAE) Best Thesis Award for her M.Tech project. She has more than 80 technical publications in international journals/conferences from her research works.