# **Recent development in the fabrication of metal matrix-particulate composites using powder metallurgy techniques**

Y. B. LIU, S. C. LIM, L. LU, M. O. LAI

*Department of Mechanical and Production Engineering, National University of Singapore, 10 Kent Ridge Crescent, Singapore 0511* 

It is advantageous to fabricate metal matrix-particulate composites (MMPCs) using powder metallurgy (PM) because the fabricated composites possess a higher dislocation density, a small sub-grain size and limited segregation of particles, which, when combined, result in superior mechanical properties. The various PM-related processes currently in use in the fabrication of MMPCs, are reviewed, outlining the common problems encountered in each of these fabrication processes. The more recently developed PM techniques to fabricate M M PCs are also discussed.

# **1. Introduction**

Metal matrix-particulate composites (MMPCs) as a group of advanced materials have been developed over the last twenty years [1, 2]. These materials exhibit a unique set of microstructures and properties not found in either monolithic ceramics or metals. Many methods have been developed to produce different metal-based composites [3-8]. Successful industrial applications have been reported and a series of MMPCs fabricated using the various methods are now commercially available [1, 9, 10]. Properties of MMPCs depend mainly upon the microstructure and properties of the matrix materials, the nature of particles, the distribution, size and shape of particles, and the interfacial behaviour between particles and matrix. Unfortunately, most particles employed to yield MMPCs possess poor wettability with the matrix metals, resulting in a poor distribution of reinforced particles and debonding at the particle-matrix interface. In order to enhance wettability, some "luxurious" techniques, such as surface coating by nickel, copper or titanium, have been introduced to preprocess the particles before fabrication. This preprocessing of particles raised the cost of MMPCs further, limiting their wider commercial applications.

Some recently introduced MMPCs, using modern high-performance materials such as tungsten, molybdenum, niobium and tantalum as the metal matrix, are difficult to fabricate by the conventional liquid metallurgy process owing to the high temperatures involved [10, 11]. In these cases, powder metallurgy (PM) with the near-net-shape capability is more attractive and it has become the most important fabrication technique for this group of MMPCs. The PM route also reduces the forming and machining cost correspondingly [10]. Furthermore, MMPCs with a high dislocation density, a small sub-grain size and limited recrystallization can be fabricated, resulting in superior mechanical properties [5, 9-12]. Another advantage of the PM processing is the easy attainment of a uniform distribution of particles in the metal matrix compared to other fabrication techniques, because segregation of particles is a common problem found in cast MMPCs [3, 4].

This paper surveys the various PM techniques available to fabricate MMPCs, focusing attention on the recent developments in some of these techniques. It is believed that the continual development and refinement of these techniques will result in the fabrication of cheaper, but higher quality, MMPCs, stimulating further industrial application of these materials.

# **2. Fabrication of green compacts of MMPCs**

The conventional method of manufacturing in powder metallurgy involves blending or mixing, compaction and sintering. This is known as primary manufacturing. The secondary manufacturing is to deform further the PM products by extrusion, rolling or other metal-working methods. In this section, we will discuss the first two steps in the primary manufacturing of MMPCs: blending or mixing, and compaction.

# 2.1. Blending or mixing

Before blending or mixing, it is important to ensure that a proper selection of the materials is carried out. For example, Hunt *et al.* [13, 14] have reported that for aluminium-based composites, a ratio of SiC particle size to aluminium powder size influences the mechanical properties of composites produced. The maximum toughness in SiC-particle-reinforced MB78 (7000 series aluminium alloy) is clearly dependent on SiC to aluminium powder size ratio. Another important factor in materials selection is the size of the



*Figure 1* Configurations at (a) 0, (b) 60, (c) 120 and (d) 290 cycles from Monte Carlo simulations of the shaking of a ternary mixture of particles: the shaking amplitude is equal to the diameter of smallest particles: after [16].

reinforcement particles. Bhanuprasad *et al.* [15] reported that a decrease in SiC particle size from 14.5  $\mu$ m to 1.5  $\mu$ m resulted in an increase in the tensile strength (UTS) of a PM A1-20 vol % SiCp composite from 148 MPa to 190 MPa after annealing. Other factors to consider during the materials selection process include mechanical behaviour, chemical stability, thermal mismatch and cost [5].

The next step, blending or mixing, is just as important, because it controls the final distribution of reinforcement particle and porosity in green compacts after compaction, which strongly affects the mechanical properties of PM materials produced. Segregation and clustering are the common problems associated with the present state-of-the-art blending or mixing methods. The phenomenon of segregation is inherent to any loose powder configuration which is subjected to mechanical blending [16, 17]. The reason for segregation and clustering includes different flow characteristics between metal powders and reinforcement particles, and the tendency of the agglomeration of particles to minimize their surface energy [7].

As a general rule, a larger particle size will lead to a better degree of distribution. The shape of particles also influences the blending result: it is easier to mix metal powders with spherical particles than with irregular-or flake-like particles. The segregation behaviour of different sized particles can be seen in Fig. 1 which shows the Monte Carlo simulations of shaking a ternary system with equal area fractions of each species  $[16, 17]$ . The larger particles rise to the top as a result of the shaking because the larger particles are moved upwards as smaller particles fill voids created beneath the larger particles. Segregation is complete after 290 cycles when the largest and the intermediate sized particles are fully separated after all the smallest particles shifted to the bottom. Furthermore, the effect of different densities between particles and metal powders of similar size is significant during blending: the lighter particles tend to stay at the top, while the heavier particles primarily segregate to the bottom  $[5, 7]$ .

The segregation and clustering during blending can be overcome by a technique developed during the 1960s called mechanical alloying (MA) [18]. Mechanical alloying is a dry, high-energy ball-milling process for producing composite metal powders with a fine



*Figure* 2 A schematic representation of the repeated fragmentation and coalescence processes characteristic of mechanical alloying. The wordings suggest the various events that can be imagined to occur as they depend on the impact angle; after [23].

controlled microstructure [19-22]. A schematic representation of mechanical alloying is shown in Fig. 2 [23,24]. This process consists of the repeated fracturing and rewelding of a mixture of particles and metal powders by high-energy compressive-impact forces to yield a uniform distribution of particles and metal powders with a satisfactory microstructure after compaction. In addition to the strengthening caused by uniformly dispersed fine particles, the fine grain size and the high dislocation density of the metal matrix, as a result of work hardening of metal powders  $[18-24]$ , also contribute to the strengthening of these MMPCs. Some processing details and properties of mechanically alloyed composites have been reported [20-22, 25-28].

## 2.2. Compaction

Following the blending or mixing operation, the mixture is then pressed at room temperature in a die at pressures which make the powders adhere to each other to form a green compact with an appropriate density. This process is called cold compaction. In this process, the densities of the green compacts should be

TABLE I Techniques to preprocess the particles before the fabrication of MMPCs

Techniques	Particles	Matrix alloys
Metallic coatings on particles	Ni, Cu- or Ti-coated graphite, SiC, $Al_2O_3$ , fly ash, mica	Al and Al alloys Cu and Cu alloys
Ceramic coatings on particles	BN, TiN-coated $Al_2O_3$ or Sic	Mo, Cu and Cu alloys
Addition of reactive or binding elements or chemicals	Mg, P for graphite Cr, Ti for graphite Ca for graphite	Al and Al alloys Cu and Cu alloys Iron and iron alloys

carefully controlled to ensure that the pores are well connected if the outgassing process is to be employed later [5].

The outgassing process is an operation to remove the adsorbed or chemically bonded water and other volatile species through the combined action of heat, vacuum and inert-gas flushing  $[5]$ . It has been reported [29] that outgassing strongly affects the adhesion of the powders which ultimately determines the ductility and ultimate tensile strength of the MMPCs produced. Mohn [30] has reported the effect of operating temperatures on the outgassing process in SiC particle-reinforced aluminium 6061 alloy. The outgassing results for iron-, nickel-, copper- and silverbased composites under vacuum conditions were reported by Bowen and Hickam [31].

In the conventional method of compaction, a pressure is usually applied in one direction, resulting in an uneven distribution of consolidation, and sometimes, insufficient densities. This affects the subsequent processing of the green compacts, such as sintering and secondary manufacturing. To control the quality of green compacts better, isostatic pressing techniques were developed such as cold, warm and hot isostatic pressing (C, W and HIPing) [32, 33]. Cold isostatic pressing (CIPing) is mainly used to press powders under a high pressure. The advantages of CIPing over the other compaction methods include the uniformity of density of the compacts achievable regardless of the size and shape of powders; controllable shrinkage; and limited residual stresses resulting from the wall friction in one-dimensional pressing. Fabrication of green compacts using cold, warm or hot isostatic pressing techniques have been reported [5,6,8,32-35]. The other promising development in the isostatic-processing techniques is a high-productivity system called dry-bag pressing. This particular process, which utilizes a cassette system to load the powders, is so-called because the press, unlike other isostatic presses, contains the pressurizing liquid inside a flexible membrane, thus keeping the mould dry. Asari *et al.* [32] and Lewis [34] have demonstrated separately that combining CIPing and HIPing, or combining HIPing with sintering may produce higher quality PM products at a suitably high productivity.

## 3. Sintering and other **consolidation methods**

## 3.1. Sintering of MMPCs

Usually, properly prepared green compacts are next

sintered. The controllable parameters in this stage are the sintering temperature and sintering atmosphere. The commonly occurring problems are the presence of oxide films, the imperfect distribution of particles and sweating during liquid-phase sintering, and poor strength in solid-phase sintering  $[1, 7]$ . The tendency for liquid metals to sweat out during sintering is due to the poor wettability of particles by liquid metal. An inadequate bonding between particles and metals at the sintering temperature results in poor strength. To increase the wettability between particles and matrix has become one of the major concerns in the fabrication of MMPCs, especially for those composites that contain soft particles such as graphite  $[1, 7, 36]$ .

Some techniques have been developed to overcome the poor wettability and interaction between the reinforcement particles and the metallic matrix, as shown in Table I. For example, the contacting angle of nickel-coated graphite particles is reduced to  $60^{\circ}$  compared to  $160^{\circ}$  for uncoated ones [3] thereby greatly enhancing their wettability. Another advantage of coated particles is their ability to avoid reaction with some metals during the fabricating process. A recently introduced technique of surface modification is to mill ceramic particles with 0.1% copper powders before a conventional blending process [15]. The tensile strength of an aluminium based composite containing 20 vol % of such pre-processed SiC particles is about 20% higher than that of the composite containing the same volume fraction of unprocessed SiC particles [15]. Some ceramic coatings can successfully protect particles from reacting with metals. These include a coating of boron nitride for SiC particles. This coating also eliminates the reaction between SiC and copper powders during sintering [37]. Another method to improve the wettability between graphite and ironbased metals is to add a small amount of calcium in the form of calcium-silicon alloy to the powder mixture of iron-graphite composites. This method has been reported to increase the volume fraction of graphite in the MMPCs up to 90% without sweating during sintering [1].

# 3.2. Hot pressing methods

Sometimes, consolidation methods other than direct sintering are used. Hot isostatic pressing (HIPing) and vacuum hot pressing (VHPing) are the usual techniques employed here. Sargent *et al.* [38] have reported that for Al  $6061-30$  wt %  $\text{SiC}_p$  composites,



*Figure* 3 A schematic representation of the vacuum hot pressing; after [5]. (a) Load powder into VHP die and assemble; (b) heat mixture/die in vaccum, outgas; (c) compact into billet; (d) strip billet from die.

HIPing followed by extrusion resulted in a pronounced reduction in porosity and an increased strength (about 25% higher) as compared to the conventional route of extrusion.

A schematic representation of the VHPing process is given in Fig. 3 [5]. Under a proper sintering temperature, gradual consolidation is accomplished by the maintainance of a constant pressure. The selection of the consolidation temperature is a compromise between the need to minimize the necessary pressure to produce optimum density and degradation of the powder matrix.

Zhou *et al.* [39] have tested Al 2024-15 vol %  $\rm{SiC}_n$ composites made by VHPing followed by hot extrusion and they found significant improvements in the mechanical properties of these composites.

#### 3.3. High-energy high-rate processing

A recent progress in the consolidation of rapidly quenched powders containing fine distribution of ceramic or graphite particles is known as high-energy high-rate processing (HEHR) [40-44]. A schematic representation of the HEHR processing is shown in Fig. 4. Persad and co-workers [40-43] have reported that HEHR processes using a homopolar generator (HPG) is an attractive processing approach with the following desirable characteristics: (1) very fast processing to minimize time for internal oxidation; (2) rapid heating and cooling rates associated with the pulsed joule heating; (3) possible micro-encapsulation

*Figure* 4 A schematic representation of the die assembly used in HEHR processing; after [40].

Fixed platen

Alumina

Moving platen

**I**  High**power** I generator

**1** 

**l**  $\begin{array}{ccc} \downarrow & \downarrow & \downarrow \ \downarrow & \downarrow & \downarrow \end{array}$ 

ode~-~'~~ Alub:na

Steel backup $\left| \begin{array}{c} | \\ | \end{array} \right|$   $\left| \begin{array}{c} | \\ | \end{array} \right|$  Powder  $r_{\text{ing}}$   $\leftarrow$   $\leftarrow$ 

**i ..... A t** 

Hydraulic **press** 

Electrode<br>(plunger)

by preferential heating and local melting; and (4) a dense product with improved mechanical properties which can be achieved by simultaneous forging during discharge. Persad *et al.* [41] have concluded that in A1-SiC composites, the short-time-at-temperature approach offers the opportunity to control phase transformation and the degree of microstructural coarsening which is not possible using other powder processing methods. Many MMPCs have been produced using the HEHR processing; these include copper-graphite  $\lceil 40, 42 \rceil$ , aluminium-SiC  $\lceil 41 \rceil$ , nickel-molybdenum-boride [43] and tungsten-based composites [42, 44].

#### **3.4.** Resistance sintering

Another sintering technique similar to the HEHR process is resistance sintering. This technique has been used to sinter fibre-reinforced  $[45]$  or  $\text{SiC}_p$ -reinforced [46] aluminium alloys. In this process, a low-voltage high-amperage current is applied through the powder compact in a stainless steel die in which the compact is being compressed simultaneously. Instead of a homopolar generator, a welding machine with a capacity of 45-65 kVA is employed as the power source [45, 46]. Almost full densification can be achieved under a proper pressure during resistance sintering. Because the sintering process is so fast (less than 1 s), no controlled atmosphere or vacuum is needed. The mechanical properties of such composites are excellent. A compressive yield stress of 500-700 MPa and compressive ultimate strength of 600-800 MPa can be reached for Al-30 vol %  $\mathrm{SiC}_p$  composites [46].

#### **3.5.** Dynamic consolidation

Dynamic consolidation (sometimes known as shock wave consolidation) is a more recently developed technique to produce materials using rapidly solidified or amorphous-structured powders. This method consists of using explosives or a high-velocity punch to impact the powders, causing instantaneous compaction [34]: The advantages of this method.are relatively low bulk temperatures, nearly random crystallographic texture in the final products, and shock hardening [47]. A shock wave can be introduced into the



*Figure 5* A schematic representation of the processing sequence for metal-ceramic composites. (a) Compact to about 80% of theoretical density; (b) treatment in hydrogen atmosphere, removing oxide layers from metal particles and metallization of surface layers of ceramic particles; (c) cold sintering to full density. A, Matrix; B, pores; C, ceramic particles; after [48].

materials in three ways: (1) by a propellent or compressed gas in a gas gun; (2) by the direct application of explosives; or (3) by the impact of a projectile accelerated by explosives. Much higher pressures and interparticle melting during shocking result in almost full densification. It has been reported that the tensile strength of an as-shocked steel (304 stainless steel) is higher than that produced by a conventional casting method [47].

## 3,6. Cold sintering

Cold sintering is an alternative ambient-temperature consolidation method suitable for the consolidation of rapidly solidified powders or mechanically alloyed powders [48, 49]. In this method, a much higher pressure (about 2-5 GPa) is used to consolidate the powders to almost full density ( $> 99\%$ ) and this process is schematically illustrated in Fig. 5 [48]. In the illustration, a mixture of metal powders and ceramic particles is pressed to yield a green compact containing interconnected pores (Fig. 5a). The compact is then treated in hydrogen to remove the oxide layers from the surfaces of metal powders and to metallize the surfaces of ceramic particles (Fig. 5b). The third step of this process is to cold sinter the compact under a high pressure to produce a final product with almost full density (Fig. 5c).

## **4. Unconventional PM processing techniques**

#### 4.1. Spray deposition

A conventional plasma spray can be used to consolidate metal powders in an inert-gas low-pressure chamber (5-500 torr, 1 torr = 133.322 Pa) to control the deposition quality. The temperature of metal powders, the degree of melting and the reaction with the plasma gases will determine the properties of the deposited products [50-52].

In the case of MMPCs fabrication, liquid metal and dispersoid powders are co-sprayed through an atomizer on a substrate to form billet, disc, strip or laminated structures. Particles of  $1-500 \mu m$ in size have been used with a metal flow rate of  $0.25-2.5$  kg s<sup>-1</sup> to produce composites with 1-45 vol % particles  $[7, 50-54]$ . Aluminium-, iron-, steel-, nickel-, titanium-, copper-, tantalum-, cobalt- **and** 



*Figure* 6 A schematic representation of the stages during impact of droplets and SiC particles at the deposition surface; after [57].

niobium-based metals or alloys have been utilized to fabricate MMPCs using this method [7, 50-54]. By using the spray-deposition method, Hasegawa and Takeshita [53] have produced stainless steel-based composites (containing  $0.4\% - 2.12\%$  Al<sub>2</sub>O<sub>3</sub> or ZrO<sub>2</sub> particles) which have superior mechanical properties compared to that of the base steels. One of the advantages of the spray process is that it combines the blending and consolidation operations, promising major savings in the cost of production. Another development in spray-deposition techniques is the socalled variable co-deposition of multiphase materials (VCMM) [55-58]. The VCMM process was developed in view of the attractive combinations of structures and properties that could be achieved in the composites. Although this process is still under development, some details have been reported in recent publications [55-58]. Gupta *et al.* [57] have given a process model to explain this complex process of deposition, as shown in Fig. 6.

#### 4.2. Rapid solidification/powder metallurgy methods

In order to enhance the mechanical properties of PM composites, some new approaches have been reported to combine different processes with powder metallurgy. One of the techniques is known as rapid solidification/powder metallurgy (RS/PM) [29, 59-62]. The advantages of the RS/PM technique include superior strength and resistance to corrosion, elevated temperature stability and the development of low-density alloys such as aluminium-lithium-based alloys [29].

TABLE II Rapid solidification techniques, morphologies and cooling rates [59, 63, 64]

Techniques	Product type	Typical dimensions (dia.) $(\mu m)$	Typical cooling rate $(Ks^{-1})$
Gas atomization	Smooth, spherical powder	$50 - 100$	$10^2 - 10^3$
Water atomization	Rough, irregular powder	$75 - 200$	$10^{2}-10^{4}$
<b>USGA</b>	Smooth, spherical powder	$10 - 50$	$\leq 10^6$
<b>PMRS</b>	Spherical powder	$< 1 - 50$	$> 10^6$

USGA, ultrasonic gas atomization; PMRS, plasma melt and rapid solidification.

Some common PM techniques of rapid solidification are shown in Table II [59,63,64]. Krishnamurthy *et al.* [60] have reviewed recent progress in RS/PM in the fabrication of light-metal MMPCs such as aluminium-, titanium- and magnesium-based composites where significant improvement in the properties of aluminium- and magnesium-based particle composites have been achieved [60-62].

## 4.3. Powder **forging**

Powder forging (combining forging with PM) is another method developed to improve the mechanical properties of PM composites. This method allows the direct forming of a mixture of metal powders and particles into a near-net-shape product, resulting in good material yield and a simplified process. The fabrication of 2024 A1 alloy composites containing 20 vol % fine SiC particles (about  $5 \text{ µm}$ ) using this method has been reported [65]. After a two-stage powder forging, the composites maintain a high strength under an elevated temperature (623 K). Almost full densification can be achieved using this technique.

Rack and Piper [66] have reported a combined method of liquid sintering and forging to produce aluminium-based SiC whisker composites. In their method, a mixture of aluminium alloy powder and SiC whiskers is cold compacted and followed by vacuum hot pressing in a mushy liquid-solid zone to eliminate the large clusters which tend to limit the mechanical behaviour of the composites.

# **5. PM fabrication of MMPCs using extrusion**

## 5.1. Extrusion methods

Extrusion has received much attention in the PM fabrication of MMPCs. This process is originally a part of the secondary manufacturing techniques [5, 10]. However, recent developments have combined this process with cold compaction or sintering [67-]. The advantages of such combinations include: (1) an improvement in mechanical properties and performance by structural refinement and minimisation of segregation; (2) a more homogeneous distribution of particles in the metal matrix; (3) the ability to form wrought structures directly from powders and hence the possibility to remove the sintering process; and (4) higher productivity and lower cost. Fig.  $7 \times 67$ ] shows three examples of hot extrusion methods. Fig. 7a shows a conventional method of extrusion, whereas a

loose powder mixture can be directly extruded as shown in Fig. 7b. If powders are extruded with a metal can (Fig. 7c), the canned billet can be evacuated leaving the powders in a vacuum environment. This is followed by heating the sealed billet at elevated temperatures without atmospheric control.

## 5.2. Development of new MMPCs by **extrusion**

A new composite material called "polymet alloy" has been developed using the extrusion method [68]. In fabricating this material, aluminium and polymer powders are blended and vacuum hot pressed at 343 °C after outgassing at 232 °C. An extrusion ratio of 32:1 is employed with the intention to form a highly aligned and uniformly distributed polymeric structure in the aluminium matrix. The yield strength of these polymets is higher than the standard alloy, although they suffer from low ductility.

A similar approach has been used to fabricate a new group of PM MMPCs, called *in situ* composites [69]. In this method, elongated reinforcement phases are created by deformation processes such as extrusion, drawing and rolling. Fig. 8 [69] shows an example of such a MMPC fabrication process. In order to form elongated, fibrous or lamellar reinforcements in the MMPCs, the particles used have to remain ductile during extrusion [69, 70].

# **6. Conclusion and prospects**

Fabrication processes of metal matrix-particulate composites using powder metallurgy can be divided into three major operations: blending including materials selection, compaction including the process of outgassing, and sintering. The current knowledge and common problems, in each of the above operations have been reviewed separately. New developments in fabrication techniques made in recent years have been enumerated and discussed in detail. These new techniques include mechanical alloying, cold, warm and hot isostatic pressing, preprocessing on particles, vacuum hot pressing, resistance sintering, dynamic consolidation, cold sintering, high-energy high-rate processing, spray deposition, variable codeposition of multiphase materials, rapid solidification/powder metallurgy, and powder forging. A comparison of mechanical properties: of  $AI-SiC_p$ composites fabricated using some of the methods discussed in the earlier sections is shown in Table III; the mechanical properties of MMPCs fabricated by



*Figure 7* Hot extrusion methods for metals and composites; after [67].





*Note:* Some data were taken from average values. E, Young's modulus; UTS, ultimate tensile strength; YS, yield strength; El. elongation; VHP, vacuum hot pressing; EXT, extrusion; SD, spray deposition; HIP, hot isostatic pressing; PM, powder metallurgy; CIP, cold isostatic pressing; PF, powder forging; MA, mechanical alloying.

 $\rm{``VHP} + \rm{EXT}$  [10].  $\rm{``SD} + \rm{Hot}$  EXT [55].  $\rm{``EXT}$  [38].  $\rm{``PM} + \rm{EXT}$ .  $\rm{``EXT}$  [60].  $\rm{``PM}$  [55].  $\rm{``DWA}$  composites (PM) [7t].  $\rm{``HIP} + \rm{EXT}$ .  ${}^{\prime}$ ALCOA composites (CIP + VHP + EXT) [71].  ${}^{\prime}$ NOVAMET composites (MA + PM) [21].  ${}^{\prime}$ VHP + Hot EXT [39].  ${}^{\prime}$ PM + EXT [60]. "ALCOA composites (PM) [8]. "Powder forging [65].



*Figure 8* A schematic representation of the formation of tungsten filaments in a nickel matrix during extrusion; after [69].

these techniques are clearly higher (especially the tensile strength and stiffness) than those produced using conventional PM methods. These newly developed techniques have greatly expanded the potential applications of PM in the fabrication of MMPCs.

The current limitations to wider industrial applications of MMPCs are still the inadequate toughness and high cost of these materials. The low ductility and fracture toughness of MMPCs are mostly caused by the premature failure of the interfacial bonds, as a result of poor wetting and interracial reactions between metal powders and reinforcement particles. These problems are expected to be solved by improvements in the low-temperature and high-pressure processes. Some new techniques developed to fabricate high-performance MMPCs have so far increased the cost of these final products because of the inclusion of expensive equipment and operations, such as complex blending processes, powder handling and consolidation. One of the most important directions for future research into the fabrication of PM MMPCs is, therefore, to develop ways to lower the processing cost without compromising the mechanical properties. This barrier is expected to be overcome in the not-toodistant future.

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