Progress of Flake Powder Metallurgy Research

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Abstract: This paper reviewed several recent progresses of the new powder metallurgy technology known as flake powder metallurgy (FPM) including different processing routes, conventional FPM (C-FPM), slurry blending (SB), shift-speed ball milling (SSBM), and high-shear pre-dispersion and SSBM (HSPD/SSBM). The name of FPM was derived from the use of flake metal powders obtained by low-speed ball milling (LSBM) from spherical powder. In this case, the uniformity of reinforcement distribution leads to increased strength and ductility. Powder is the basic unit in PM, especially advanced PM, and its control is key to various new PM technologies. The FPM is a typical method for finely controlling the powder shape through low-energy ball milling (LEBM) to realize the preparation of advanced material structures. The present paper represents a review of the main results of research on FPM and indicates the potential for future studies devoted to the optimization of this processing route.

Keywords: flake powder metallurgy; processing; rolling; strength; ductility

1. Introduction

With the rapid development of metal matrix composites (MMCs) and especially aluminum matrix composites (AMCs), traditional casting, infiltration, plastic forming, and other methods showed a limited ability to meet the requirements of preparation and processing of new aluminum matrix composites. New powder metallurgy technology, large plastic deformation, and material addition manufacturing have a high controllability and unique role in the preparation and processing of AMCs, which has been widely noted by advanced researchers.

In recent decades, many efforts such as powder metallurgy (PM) [1–3], severe plastic deformation (SPD) [4–8], nanoscale dispersion (NSD) [9,10], in situ chemical vapor deposition (CVD) [11,12], and ball milling followed by post-sintering processes such as forging, extrusion, or rolling [13–19] have been undertaken to fabricate MMCs, especially AMCs. Among all the proposed techniques, the PM route has been identified as very promising in order to obtain optimal second phase distributions [20–22]. The basics of this route comprise the deformation, cold-welding and balanced stages [23]. At the deformation stage, ductile Al powders flatten as a consequence of intense collisions of steel balls with powder particles, consequently providing a larger specific surface compared to spherical particles. Reinforcement clusters are fractured and then dispersed onto the surface of the flattened powder [24–26]. During the cold-welding stage, flattened powders are cold-welded into coarser reinforcement-metal powder particles decorated by reinforcement at the welded seams. It should be noted that once reinforcements are inserted, their further dispersion becomes more and more difficult. Therefore, the minimum necessary time for cold-welding should be precisely set [27,28].

Powder metallurgy (PM) technology can control the interface of the composite at temperatures well below the melting point of the aluminum matrix, thereby allowing for avoidance of the natural agglomeration of micro-reinforcements into the melt and the
floating or settling of reinforcements due to differences between the density values of the melt and the second phase. At the same time, the AMCs have a smaller matrix structure than the liquid phase method. As the basic unit of PM technology, control of powder is key to various new PM technologies.

Some technologies use the reaction activity resulting from the high specific surface area of powder and the external input energy. The dispersion of nano-reinforcements is accomplished through an in situ reaction, such as the reaction of spray deposition of atomized droplets and reactant [29] or the mechano-chemical reaction of powder and reactant through high-energy ball milling [30]. Some PM technologies can disperse the reinforcement, control the shape of the powder and refine the powder grains through high deformation during ball milling, such as mechanical alloying of the reinforcement by long-term high-energy ball milling (HEBM) and low-temperature ball milling such as cryo-milling [31–33]. The HEBM route has demonstrated its effectiveness in the destruction of the oxides layers, providing optimal interface bonding between the second reinforcing phase and the matrixes. However, the severe deformation due to ball milling can lead to remarkable damage in very brittle reinforcements such as CNTs.

In general, most of the new PM technologies have been developed to realize the new component design or structural design of AMCs. For example, flake PM (FPM) is a typical method for fine control of powder shape LEBM of mostly ≤ 200 rpm [24,27,34–36], and then for the preparation of the composite structure. The method not only is characterized by a very high specific surface area and flat surface, but also provides sufficient space for the surface in situ reaction [37,38] and surface dispersion [26,39,40] to the reinforcement, and can induce flake nano-grain (NG) or ultrafine grain (UFG) in the powder. This fine microstructure can then be retained in the bulk material. Depending on whether or not the flake shape is destroyed during the powder consolidation process, a nano-reinforced body dispersion structure [25,40–43] or a layered configuration [44–47] can be formed, respectively.

The obtaining of sound mechanical properties in nano-reinforced AMCs can be achieved only if second-phase optimal dispersion is realized during processing [3,24,48,49]. The second requirement effectively maintains the structural integrity of the nano-dispersions, especially for CNT and graphene [27,50,51]. The third requirement is interfacial bonding between the nano-dispersal surfaces and the matrix material [19,25,52]. It is interesting to note that the alignment of the nano-dispersoids has a remarkable influence on the mechanical properties of metal matrix composites [53]. The new PM technologies, namely FPM, as a powder-smart severe plastic deformation (SPD) method will enable us to overcome the obstinate roadblocks in UFG alloys and MMCs caused by poor dislocation storage and weak strain hardening ability. FPM, as a bottom-up approach, could fulfill a flaky powder geometry known as the building blocks of bulk advanced materials, which was hitherto believed impossible.

The PM route for MMCs production comprises the following main steps: powder processing and consequent powder consolidation. Metal powders are blended with reinforcements by means of techniques such as conventional FPM (C-FPM) [35,47,54], slurry blending (SB), vapor-based synthesis, shift-speed ball milling (SSBM), and high-shear pre-dispersion and SSBM (HSPD/SSBM), which are then followed by various consolidation routes of the mixture such as hot extrusion [27,55], spark plasma sintering [47,56,57], friction stir processing [58,59], hot rolling [19,46,60], and others [5,36]. Significant advances can be underlined in the field of solid-phase powder metallurgy (PM) techniques [47]. The PM based methods include HEBM [19,46,55], FPM [24,34], liquid phase ball milling [24,61], nanoscale dispersion methods [9,10], molecular level mixing [62,63] and in-situ CNTs production combined with low-energy ball milling [11,37,64], and others [65]. These methods sometimes require large time consumption when used on an industrial scale. Therefore, it is fundamental to review recent advances in powder-based processing techniques such as FPM. This review specifically focuses on the different FPM approaches, namely C-FPM,
SB, SSBM, and HSPD-SSBM. After reviewing the recent advances in FPM approaches, the potential for further optimization and development is indicated.

The main developed powder metallurgy routes are based on the employment of starting spherical powders in order to allow dense compaction of the formed blocks. By contrast, FPM is based on the employment of flake-shaped reinforcements that can be aligned by deformation through hot compaction and rolling or extrusion. During flake powder metallurgy, the main processing steps are the precise preparation of flake reinforcements, their proper mixing, consolidation, and alignment.

From the crystal plasticity perspective, the BM process is known as a micro-plastic deformation process, affected by rotational speed and rotational time \[2,27,66,67\]. Generally, the collision forces between the milling balls and reinforcement and matrix powders with all their complexity, including compressing and shearing forces, are the origin of the BM process evolution \[27,68\]. Specifically, compression leads to the powder’s deformation, flattening and fragmentation, as well as final cold welding. In contrast, shearing effectively promotes the breaking of contamination skins such as alumina skin in Al powders and reinforcement clusters, resulting in uniform dispersion of reinforcements.

2. Conventional Flake Powder Metallurgy (C-FPM)

The FPM via typical dry milling is introduced as powder BM in a dry jar. Specifically, the ingredients blended in the jar are reinforcement, matrix powder, steel balls, and usually a processing agent such as stearic acid, ethanol or similar. The process of flaking for spherical metal powders under the C-FPM route is performed through micro-rolling (ball milling) the starting powders (Figure 1a).

![Figure 1. (a) Schematic of micro-rolling process, (b) SEM images of CNT/Al flakes under C-FPM route.](image)

In current practice, for the first time, the LSBM process is considered a kind of rolling process, but on a micro scale—namely, micro-rolling—because: (1) the LSBM process causes plastic deformation of initial powders, thus leading to significant effects on the geometry of the powders; specifically, particles flatten via a process which is similar to the one which is performed in the conventional rolling process, (2) milling balls can be considered similar...
to conventional rollers but with a much smaller size, and (3) despite the difference in the source of the generation of imposed stresses, deformation occurred originating from shear stresses upon the powders.

The Al powders are flattened into flakes with a large aspect ratio and tabular morphology as a consequence of the micro-rolling process, and are then assembled into astonishingly well-aligned macroscopic assemblies, even under common processes, such as spark plasma sintering and hot extrusion, as shown in Figure 1b.

In previous studies, C-FPM was adopted by using ball-milled Al flakes to achieve an appropriate level of reinforcement dispersion into the matrix. Choi et al. [2] reported that LSBM led to low energy Al powders flattening and to less damage to CNTs. Due to the long duration of metal flattening, reinforcement can be uniformly distributed without excessive damage. Morsi et al. [35], using C-FPM, investigated the effect of milling time on flackening effect and on the CNTs distribution and reported that the final shape of the flakes varies with the milling time and the CNTs percentage. Jiang et al. [69] concluded, from studying Al$_2$O$_3$/Al biomimetic nanolaminated composite, that the flake-shaped building blocks achieved by the micro-rolling process are the key factor for the excellent properties of bulk MMCs.

Furthermore, Zhang et al. [70] found that mechanical properties strongly depend on the flakes’ thickness. With a thickness of 500 nm, they found an optimal balance between strength and ductility in the formed composites, despite numerous reports that C-FPM could not effect a significant improvement in the balance of mechanical properties due to a reduced uniform level of reinforcement distribution in the matrix as the most important factor in the final properties of MMCs.

3. Flake Powder Metallurgy via Slurry Blending (FPM-SB)

The strategy of using a slurry-based dispersion process of the reinforcement has emerged as a mighty new route in overcoming the prerequisites needed to produce advanced AMMs [39,71]. In this technique, the particles’ morphology is modified from three-dimensional spheres to two-dimensional flakes. Subsequently, the surface is modified through organic solutions such as polyvinyl alcohol (PVA) or ethanol. In this way, the flakes can easily attract the second phases onto their surfaces. The produced composite is then compacted and sintered, as illustrated in Figure 2a.

In this technique, to break the reinforcement clusters, ultrasonic energy is used. This is obtained through the well-known sonication process. Ultrasonication is an effective method to break clusters of nanoparticles or nanocarbon such as CNTs or graphene. Recently, functionalization of the flake surface has been employed for the modification of the surface of flake metal powders. This was developed in order to improve the compatibility between the reinforcement and the matrix. In this regard, Jiang et al. [24,26,39] adopted a novel strategy for a slurry-based process to solve the potential incompatibilities between the matrix and the CNTs. Normally, polyvinyl alcohol (PVA) is employed for the surface modification of the Al powders surfaces in order to favor the bonding with CNTs. However, in this case, larger surfaces were demonstrated to be more effective in bonding with the one-dimensional nanotubes. All of these solutions allow for the optimization of the potential reinforcement percentage to be introduced into the composite. The nanotubes bonding to the metallic flakes can be improved by surface modification through PVA. This reinforces the bonding, leading to improved final mechanical properties [39]. Figure 2b shows CNT/Al flake powders with large flat surfaces after the FPM-SB route and Figure 2c exhibits uniform CNT distribution onto the flake powders thanks to the beneficial geometrical compatibility of the Al powders with the 1-D nature of the CNTs. Chen et al. [61], by using a combination of BM and a solution approach, namely, solution ball milling (SBM), obtained an improved homogenization of CNTs accompanied by very low reinforcement damage. Recently, Fan et al. [25] showed that shorter milling times of pre-dispersed CNT/Al nanoflake powders prepared from a slurry-based dispersion process
lead to homogeneous distribution and very high-strength interfacial bonding. This was attributed to physical bonding and partial reaction bonding.

Figure 2. (a) Schematic of slurry-based dispersion process, (b,c) SEM images of CNT/Al flakes under FPM-SB route.

As a matter of fact, the FPM-SB CNT/Al nanocomposite showed tensile stress of 375 MPa with strain to fracture around 12%, very high when compared to corresponding material produced via conventional PM (330 MPa strength and 6% ductility, Figure 3a) [24,26]. The high-level ordering nanolaminates of flake-shaped building blocks was reported as the key factor in significantly increased tensile strength and ductility when compared with nanocomposites with the same matrix and with non-uniform distribution of CNTs. Moreover, Figure 3b depicts that the advantage of FPM over other methods such as nanoscale dispersion (NSD) [9], low-energy blending (LEB), and others can be obtained through the comparison of the strengthening effects of CNTs®. In fact, an incredible strengthening of the efficiency of CNTs is obtained through FPM thanks to the homogeneous and individual distribution of CNTs in the Al matrix, originating from the high compatibility of Al powders with CNTs [24,72].

Despite the numerous advantages of FPM via slurry blending, through the application of the abovementioned method to fabricate AMCs with high nano-reinforcement percentages of CNT [3], SiO2 [73], etc., the ductility of the composite also decreased dramatically. Thanks to high nano-reinforcement percentages, various layers of nano-reinforcement can be bonded and overlapped through slurry-based dispersion, even if this leads to difficult bonding between neighboring Al nanoflakes. Hence, the main goal of the FPM processing route for the production of MMCs is the combination of reinforcement dispersion with the structural properties and matrix-second phases bonding.
Figure 3. Tensile properties of CNT/Al nanocomposites (a) loading with 1 vol.% CNTs and fabricated by conventional PM and flake PM (inset shows the relevant strengthening efficiencies of CNTs), (b) The strengthening efficiency and tensile strength for CNT/Al composites fabricated by various methods, the data were drawn based on recent reviews [22,24,48,74,75].

4. Flake Powder Metallurgy via Shift Speed-Ball Milling (FPM-SSBM)

The correlation between the CNTs distribution, consequent bonding and final mechanical strength of the composites has been the subject of in-depth study by many scientists [35,65,67,76–80]. Here, the different BM conditions (low- and high-energy) affecting the final properties of AMCs are illustrated in order to optimize the different milling strategies. To this end, recently, Xu et al. [27] proposed a new flake powder metallurgy route via a SSBM strategy. In this strategy, LSBM was applied for very long times in order to obtain aluminum flat flakes with very uniform distribution of reinforcements without damage. The results are compared with those of HSBM in order to produce cold-welded lamellar particles with acceptable CNT/Al bonding and inter-flake bonding, as shown in Figure 4a. Specifically, LSBM allowed for mild ball-to-powder contact and very slow flattening of spherical metal powders into flake-shaped powders. This provided enough time for nano-reinforcements to be optimally dispersed on the surface of metal flakes with low damage to structure integrity of nano-reinforcements, especially carbon-based ones such as CNT or graphene; HSBM showed much more pronounced collisions and cold welding of Al flakes into cake-like particles with a clear laminated structure, as seen in Figure 4b,c, leading to clustered nano-reinforcements within the welded metal particles. In this case, very pronounced damage to reinforcements resulted.

Further developments demonstrate that SSBM, where different powder deformation mechanisms are obtained as a consequence of various milling speeds, produces a high strength of up to 1.5% of CNTs. The strength and ductility levels are much higher compared to sole LSBM or HSBM [27]. As a matter of fact, the optimal combination of reinforcement distribution, structural integrity and matrix-second-phase bonding can be obtained through a new SSBM process known as FPM-SSBM.
Most composites exhibited a strength–ductility dilemma and these data are all placed under a typical “banana shaped” curve (Figure 5). However, the strength–ductility results of the SSBM route for CNT/Al composite overcome the blue belt in the graph, demonstrating improved mechanical properties. Therefore, FPM-SSBM is a very effective route for this materials’ processing. Comparing previous studies with FPM-SSBM revealed that, with conventional milling processes, it was difficult to fabricate CNT/Al composite with uniform CNT dispersion and good bonding without reducing CNT integrity [72]. In contrast, SSBM enhanced the mechanical property of CNT/Al composites by combining the advantages of LSBM, which included mild ball-to-powder collision and slow flattening of spherical Al powders into flakes, with those of HSBM, which included a much stronger collision and cold welding of Al flakes, and avoiding the adverse factors introduced by LSBM and HSBM, which were unhealed insufficient bonding between Al flakes, and many local brittle areas with CNT clusters or forming brittle Al$_4$C$_3$, respectively. Hence, FPM is known as a smart technology of SPD materials based on a bottom-up approach.
Jiang et al. [81] fabricated graphene nanosheet (GNS)/Al composites via FPM-SSBM. They showed high strength and optimal ductility (295 MPa and 13%, respectively). They also showed that by precisely tailoring LSBM plus HSBM, very low damage in GNSs is achieved.

Very recently, Fu et al. [43] studied the trimodal grain structure fabricated by the FPM-SSBM technique in CNT/Al-Cu-Mg composites and reported that FPM-SSBM is a practical and feasible strategy for designing a heterogeneous grain structure to improve the mechanical properties of the AMCs. However, owing to the strong van der Waals force of attraction among reinforcements, the starting reinforcement is typically in clusters of tens of microns in diameter. This can be more deleterious when the size scale of the reinforcement is in nanometers. Therefore, control of nano-reinforcement dispersion during LSBM in SSBM process is the key to preparing high-performance nano-reinforced MMCs, especially AMCs.

5. Flake Powder Metallurgy High-Shear Pre-Dispersion and SSBM (FPM-HSPD/SSBM)

De-agglomeration of CNTs before ball milling results in an optimal solution to gain good reinforcements dispersion. However, this good dispersion is difficult to obtain via the traditional SB route because of the easier re-aggregation of second phases during drying. Presently, many AMCs’ processing routes show some limitations in the reinforcement dispersion, especially in the case of high reinforcement percentages. In addition, as the reinforcement percentage increases, the control of interfacial reactions becomes more difficult [65]. The PM-based methods include high-energy ball milling [19,46,54], flake powder metallurgy [24,34], liquid-phase ball milling [24,61], nanoscale dispersion method [9,10], molecular level mixing [62,63] and the in-situ formation of CNTs in combination with low-energy ball milling [11,37,64], etc. [65]. Nevertheless, these solutions require
energy and time consumption which is incompatible with industrial production. Therefore, pre-dispersion optimized methods are fundamental in this direction [34,52,69,82].

It seems that FPM results in the best route, with optimal control of the process and high mechanical properties, even if there is still space for further improvement. A combined route of high-shear pre-dispersion and shift speed ball milling is proposed to fabricate AMCs, as shown in Figure 6a. In this technique, the spherical metal powder and reinforcement are first homogenized for a short time (about 30 min at a speed of 300 rpm [42]), and then experience severe and tailorable shear stress through the high-shear pre-dispersion process at a preferred speed of approximately 2000–2500 rpm for 15 min [49]. A spherical CNT/Al composite powder is obtained. Then, the as-prepared powders are processed via the SSBM route to obtain cold-welded lamellar particles, as shown in Figure 6b,c.

Indeed, the subsequent HSBM process causes the flakes obtained during LSBM to be cold-welded quickly into laminated particles. In fact, not only can the inter-flake bonding

\begin{figure}
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\caption{(a) The schematic of fabrication CNT/Al composite via FPM-HSPD/SSBM technique: High-shear pre-dispersion process coats CNTs uniformly onto spherical Al powders, shift-speed ball milling process redistributes CNTs into Al flakes [42], (b,c) CNT/Al laminated particle, and a side cross-section view of particle after FPM-HSPD/SSBM route.}
\end{figure}
be improved, the CNTs that initially attached onto the Al surface via the HSPD process can also be effectively embedded into the Al matrix and strengthen the interface bonding between CNTs and the Al matrix. Subsequently, the as-prepared composite particles are pressed and sintered to fabricate the bulk AMCs.

Recently, many routes for the dry coating of particles were realized in order to produce advanced materials by coating the surface of larger core (host) particles with fine shell (guest) particles through mechanical forces [83,84]. Among the various routes, mechanofusion (MF) provides an exceptional solution [85] by attaining a considerable capacity to produce composite powders with the desired tailor-made properties for advanced MMCs [42,85,86]. This processing route is expected to achieve the dry and uniform combination of nano-sized CNTs and micron-sized metal powders. Recently, Chen et al. [42] showed that dry particle coating (also termed high-shear pre-dispersion) combined with the FPM route not only improves the dispersion efficiency of CNTs in the ball milling process, but also well preserves the structural integrity of CNTs compared to other PM routes [42,87].

Very recently, Sadeghi et al. [49] investigated the factors influencing the dispersion uniformity and structural integrity of CNTs during processing by using smart mechanical powder processing based on the mechanofusion system. According to their results, the high-shear pre-dispersion process as smart mechanical powder processing is a relatively simple and environmentally friendly approach to produce AMCs powder with the desired tailor-made balance of dispersion and structural integrity of nano-reinforcements in a metallic matrix.

6. Promising BM Techniques

In addition to the production methods of flake powder mentioned above, two other techniques based on controlling ball movement—namely, electrical discharge-assisted mechanical milling (EDAMM) and magnetic mechanical milling—may be introduced here. The key point applied in these two advanced techniques is related to control of the pattern of ball movement: the former incorporating high-voltage, low-current electrical discharges and the latter using the external magnetic field. In both techniques, the applied conditions cause rapid fracture rates, enhanced mechano-chemical reactions and novel reaction paths [88–90]. However, achieving flake powder using EDAMM and magnetic mechanical milling strongly depends on processing parameters such as discharge type (glow and spark [91]) and operation mode (shearing and impact [89]).

The EDAMM technique works on the principle of interface transformation rather than diffusional transformation, the main underlaying mechanism in conventional BM, as shown in Figure 7a. Indeed, narrow and micro-sized gaps between individual balls, as well as between balls and the chambering wall accompanied by a high-voltage electrical impulse, are basically governed by the type of electrical discharge that is formed. The mechanisms associated with these types of milling with electrical discharge—either glow (cold) or spark (hot)—are highly complex, and more literature on processing parameters under different electrical conditions is required. Moreover, according to the current knowledge, there is a general consensus on the fact that the electrical discharge condition induces large thermal and mechanical stresses within powder particles which, in turn, cause combined effects such as local melting, partial evaporation and Joule heating [88,90,91]. The morphology of the achieved powder has a dominant shape, mostly flake, followed by circular and angular (irregular).
On the other hand, in the magnetic mechanical milling technique, an external magnet controls the ball motion and restricts particle–ball interaction to adjust the operation mode from low-energy shearing to high-energy impact (Figure 7b). In fact, the spatial and temporal profiles, as two main parameters of the magnetic field, control the balls' movement paths, and thus the impact and shearing energy imposed upon the powders. On one side, in shearing mode, by decreasing the intensity of magnetic field and the frequency of the rotating chamber, the balls probably both rotate and oscillate around an equilibrium position at the bottom of the milling chamber. On the other side, in impact mode, the external magnetic field restricts the ball movement path to the vertical plane by the cell walls [92].

7. Conclusions and Vista

Numerous attempts, both technically and scientifically, have been carried out to provide a reasonable extent of dispersity, as this is the first and foremost factor in enhancing properties of MMCs. Surface coating techniques, both wet-coating methods and dry-coating techniques, are known to be effective and feasible techniques to de-agglomerate and fabricate nanocomposite powders. Notably, the direct particle coating technique, or smart powder processing technique, has been developed in recent years, and utilizes high shear or impact forces to disperse and coat the nano-reinforcements to the surface of the micron-sized powders, thereby obtaining composite powders with various core–shell (host–guest) structures. Such an approach can provide a high level of reinforcement dispersity, which has been used in various industrial sectors to alter the properties of powder particles, such as flowability, dispersibility, particle shaper/sphericity, sinterability, solid-phase reactivity, etc. The recently developed FPM method achieved a uniform dispersion of high content of nanoparticles on the surface of metallic nanoflake powders via the SB, the SSBM, and HSPD/SSBM methods. However, to de-agglomerate nanoparticles, chemical agents or long-duration ball milling are always needed during these processes, making the fabrication complex or energy-intensive.

Further studies in the technical sector should focus on combining direct particle coating technology with the FPM route, that is, using flake powder as the host particles for dry particle coating techniques. This route is expected to solve the problems of de-agglomeration of the nanoparticles and the uniform dispersion of high-volume fraction nano particles in the meantime.
Author Contributions: Conceptualization, Writing, Data curation (B.S.); Conceptualization, Writing, Supervision (P.C.). All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data presented in this study are available on request from the corresponding author.

Conflicts of Interest: The authors declare no conflict of interest.

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