

LECTURE NOTES ON MANUFACTURING AND PROCESSING OF COMPOSITE MATERIALS

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1. Introduction

There are more than 50,000 materials available to engineers for the design and manufacturing of products for various applications. These materials range from ordinary materials (e.g., copper, cast iron, brass), which have been available for several hundred years, to the more recently developed, advanced materials (e.g., composites, ceramics, and high performance steels). Due to the wide choice of materials, today's engineers are posed with a big challenge for the right selection of a material and the right selection of a manufacturing process for an application. It is difficult to study all of these materials individually; therefore, a broad classification is necessary for simplification and characterization.

These materials, depending on their major characteristics (e.g., stiffness, strength, density, and melting temperature), can be broadly divided into four main categories

- Metals,
- Plastics,
- Ceramics, and
- Composites.

We tend to think of the latter half of the twentieth century as the “composite age”. In some ways this is realistic and gives us a feeling of continuity from former “material based ages” such as the stone, bronze and iron ages. Certainly the last 50 years have been associated with some remarkable developments in composite materials.

Composite materials have been utilized to solve technological problems for a long time but only in the 1960s did these materials start capturing the attention of industries with the introduction of polymeric-based composites. Since then, composite materials have become common engineering materials and are designed

and manufactured for various applications including automotive components, sporting goods, aerospace parts, consumer goods, and in the marine and oil industries. The growth in composite usage also came about because of increased awareness regarding product performance and increased competition in the global market for lightweight components.

Among all materials, composite materials have the potential to replace widely used steel and aluminum, and many times with better performance. Replacing steel components with composite components can save 60 to 80% in component weight, and 20 to 50% weight by replacing aluminum parts.

2. Definition of Composite Materials

The word 'composite' in composite materials means that two or more materials are combined together in a certain order on a macroscopic level to form a new material with different and attractive properties. The reason alloys are not considered in this category is because they are homogeneous on the macroscopic level.

A composite material consists of two phases:

- Primary
 - Forms the matrix within which the secondary phase is imbedded.
 - Any of three basic material types: polymers, metals, or ceramics.
- Secondary
 - Referred to as the imbedded phase or called the reinforcing agent.
 - Serves to strengthen the composite. (fibers, particles, etc.).
 - Can be one of the three basic materials or an element such as carbon or boron.

Composite materials consist of a bulk material called the matrix, and a filler of some types such as fibers, whiskers or particles. Composite materials are conventionally classified into three categories viz. polymer matrix, metal matrix and ceramic matrix depending on the matrix employed. Furthermore, composites can be divided into classes in various manners. One simple classification scheme is to separate them according to reinforcement forms particulate-reinforced, fiber-reinforcement, or laminar composites. Fiber reinforced composites can be further divided into those containing discontinuous or continuous fibers.

Although the continuous-fiber-reinforced metal matrix composites (MMCs) have the best combination of mechanical properties, particulate reinforced MMCs are superior from the viewpoint of cost-performance tradeoff. Compared to fiber reinforced MMCs, the particulate-reinforced MMCs possess improved ductility, reduced anisotropy of mechanical properties, as well as ease of secondary working with conventional metal working techniques. The availability of inexpensive particulates as reinforcements serves as an added advantage.

In particulate strengthened metal matrix composites, the strengthening is due to both the matrix as well as the dispersed phase. While the matrix provides strengthening by restricting the crack growth, thus avoiding the brittle failure, the dispersed phase acts as an impediment for dislocation movements. This type of strengthening is similar to precipitation hardening but for the fact that the strengthening effect is not as pronounced as with precipitation hardening. But because the particles that are dispersed into the matrix are inert, the strengthening is retained at elevated temperatures. Also, precipitation hardening is limited to the small amount of the dispersing phase. On the other hand, particulate reinforced composite can have significant percentage of the particulate and provide outstanding wear resistant properties.

The properties of a composite depend on the following:

- Properties of constituent phases.
- Relative amounts of constituents.
- Geometry of the dispersed phase.
 - Shape of particles.
 - Particle size.
 - Particle distribution.
- Interfacial reactions between constituents.

Several properties of the composites may be assessed by the rule of mixture (ROM), which states that the property of the composite is the sum of products of the property of an individual constituent and its volume fraction in the composite. For example:

$$\text{Density: } D_c = D_r V_r + D_m V_m$$

where, D stands for density and subscripts c , r and m stand for composite, reinforcement and matrix respectively. This theoretical calculation can be used to check whether the composite produced has the optimum properties. Modulus, Strength, Electrical properties may sometimes be estimated with ROM as the first approximation. Only particulate reinforced metal matrix composites will be dealt with here. Discussion on other types of composites is beyond the scope of this chapter.

3. Classification of composite material

- Metal Matrix Composites (MMCs)
 - Include mixtures of ceramics and metals, such as cemented carbides and other cermets, as well as aluminum or magnesium reinforced by strong, high stiffness fibers.
- Ceramic Matrix Composites (CMCs)
 - Least common composite matrix. Aluminum oxide and silicon carbide are materials that can be imbedded with fibers for improved properties, especially in high temperature applications.
- Polymer Matrix Composites (PMCs)
 - Thermosetting resins are the most widely used polymers in PMCs. Epoxy and polyester are commonly mixed with fiber reinforcement.
- Matrix material serves several functions in the composite
 - provides the bulk form of the part or product
 - holds the imbedded phase in place
 - shares the load with the secondary phase
- The Reinforcing Phase

Is an important material form for metals and ceramics range in size from microscopic (less than 1 micron) to macroscopic (greater than 1 micron).

- In the microscopic size range and proportion of imbedded material of 15% or less, the particles result in strengthening the matrix.
- In the macroscopic size range and proportion of imbedded material of 25% or more, the particles serve to share the load with the matrix material.

- This form of composite strengthening occurs in cemented carbides, in which tungsten carbide (80%) is held in a cobalt binder.

4. Nano-composites materials

Nano-composites are materials that incorporate nanosized particles into a matrix of standard material. The result of the mixing nanoparticles with matrix gives a drastic improvement in properties such as mechanical strength, toughness, electrical and thermal conductivity. The effectiveness of the nanoparticles can appear if the material added is only between 0.5 and 5% by weight.

Nanoparticles have an extremely high surface to volume ratio, which dramatically changes their properties when compared with their bulk-sized equivalents. It also change the way in which the nanoparticles bond with the bulk material. The result is that the composite can be many times improved with respect to the component parts.

Nano-composites can dramatically improve properties like:

- Mechanical properties including strength, modulus and dimensional stability.
- Electrical conductivity.
- Thermal stability.
- Chemical resistance.
- Surface appearance.
- Optical clarity.

Today, the industrial applicability of nano-composites is widely spread due to the improvements of the mechanical characteristics of such materials.

Some applications fields of these materials are:

- Automotive engine parts as mirror housings, door handles, engine covers and fuel tanks.
- Impellers and blades for vacuum cleaners, power tool housings, mower hoods and covers for portable electronic equipment such as mobile phones.
- Thin-film capacitors for computer chips.
- Oxygen and gas barriers.
- Food packaging.

5. Metal Matrix Composites (MMCs)

Metal matrix composites (MMCs) have been around for a long time, but have only been recognized as legitimate engineering composite materials in the second half of the twentieth century. From their humble research beginnings, MMCs have gone from "niche" materials to several high performance applications in aerospace, electronic packaging, automotive, and recreational products. There has been a myriad of new and exciting advances and applications in the field. Examples are carbides for machining of materials in product engineering, noble metal composite systems for contacts in electronics and electro-technology, copper-graphite sliding contacts for generators and electric motors and multi-compound systems for brake linings in high speed brakes. After the massive effort in recent years to develop metal matrix composites (MMCs) with light metal matrixes the successful application of these materials has taken place in traffic engineering, especially in automotive and transport technology.

Metal matrix composites (MMCs), like all composites; consist of at least two chemically and physically distinct phases, suitably distributed to provide properties not obtainable with either of the individual phases. Generally, there are two phases, e.g., a fibrous or particulate phase, distributed in a metallic matrix. Examples include continuous Al_2O_3 fiber reinforced Al matrix composites used in power transmission lines; Nb-Ti filaments in a copper matrix for super conducting magnets; tungsten carbide (WC)/cobalt (Co) particulate composites used as cutting tool and oil drilling inserts; and Sic particle reinforced Al matrix composites used in aerospace, automotive, and thermal management applications.

A legitimate question that one might ask is: Why metal matrix composites? The answer to this question can be subdivided into two parts: (a) advantages with respect to unreinforced metals and (b) advantages with respect to other composites such as polymer matrix composites (PMCs). With respect to metals, MMCs offer the following advantages:

- Major weight savings due to higher strength-to-weight ratio.
- Exceptional dimensional stability (compare, for example, SiC, /Al to Al).

- Higher elevated temperature stability, i.e., creep resistance.
- Significantly improved cyclic fatigue characteristics.

With respect to PMCs, MMCs offer these distinct advantages:

- Higher strength and stiffness.
- Higher service temperatures.
- Higher electrical conductivity (grounding, space charging).
- Higher thermal conductivity.
- Better transverse properties.
- Improved joining characteristics.
- Radiation survivability (laser, UV, nuclear, etc.).
- Little or no contamination (no out-gassing or moisture absorption problems).

5.1 Types of Metal Matrix Composites (MMCs)

All metal matrix composites have a metal or a metallic alloy as the matrix. The reinforcement can be metallic or ceramic. In some unusual cases, the composite may consist of a metallic alloy "reinforced" by a fiber reinforced polymer matrix composite (e.g., a sheet of glass fiber reinforced epoxy or aramid fiber reinforced epoxy).

In general, there are three kinds of metal matrix composites (MMCs):

- (i) Particle reinforced MMCs
- (ii) Short fiber or whisker reinforced MMCs
- (iii) Continuous fiber or sheet reinforced MMCs

Fig.1 shows, schematically, the three major types of metal matrix composites:

Continuous fiber reinforced, short fiber or whisker reinforced, particle reinforced, and laminated or layered composites. The reader can easily visualize that the continuous fiber reinforced composites will be the most anisotropic of all. Table1 provides examples of some important reinforcements used in metal matrix composites as well as their aspect ratios (length/diameter) and diameters.

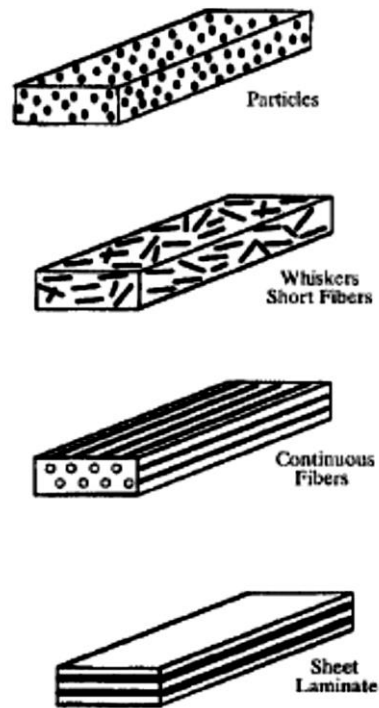


Fig. 1 Different types of metal matrix composites

Table 1 Typical Reinforcements Used in Metal Matrix Composites

Type	Aspect Ratio	Diameter, μm	Examples
Particle	1– 4	1– 25	SiC, Al ₂ O ₃ , BN, B ₄ C, WC
Short fiber or whisker	10 – 10000	1– 5	C, SiC, Al ₂ O ₃ , Al ₂ O ₃ +SiO ₂
Continuous fiber	>1000	3 – 150	SiC, Al ₂ O ₃ , C, B, W, Nb-Ti, Nb ₃ Sn

Particle or discontinuously reinforced MMCs (the term discontinuously reinforced MMCs is commonly used to indicate metal matrix composites having reinforcements in the form of short fibers, whiskers, or particles) have assumed special importance for the following reasons:

- Short fiber or whisker Continuous fiber Particle reinforced composites are inexpensive vis-a-vis continuous fiber reinforced composites. Cost is an important and essential parameter, particularly in applications where large volumes are required (e.g., automotive applications).

- Conventional metallurgical processing techniques such as casting or powder metallurgy, followed by conventional secondary processing by rolling, forging, and extrusion can be used.
- Higher use temperatures than the unreinforced metal.
- Enhanced modulus and strength.
- Increased thermal stability.
- Better wear resistance.
- Relatively isotropic properties compared to fiber reinforced composites.

Within the broad category of discontinuously reinforced composites, metal matrix composites made by liquid metal casting are somewhat cheaper to produce than powder metallurgy composites. There are two types of cast metal matrix composites:

- Cast composites having local reinforcement.
- Cast composites in the form of a billet having uniform reinforcement with a wrought alloy matrix. Such composite billets are forged and /or extruded, followed by rolling or other forming operations.

5.2 Characteristics of Metal Matrix Composites (MMCs)

One of the driving forces for metal matrix composites is, of course, enhanced stiffness and strength. There are other characteristics, which may be equally valuable. As examples, we can cite the ability to control thermal expansion in applications involving electronic packaging. By adding ceramic reinforcements, one can generally reduce the coefficient of linear thermal expansion of the composite. Electrical and thermal conductivity characteristics may be important in some applications. Clearly, superconductors require superconducting characteristics. The metallic matrix provides a high thermal conductivity medium in case of an accidental quench, in addition to holding the tiny superconducting filaments together. Other important characteristics that may be of immense value include wear resistance (e.g., in WC/Co composites used in cutting tools or oil drilling inserts and SiC/Al rotor in brakes). Thus, although one commonly uses the term reinforcement by particle or fibers in the context of metal matrix composites, it worth pointing out that strength enhancement may not be the most important characteristic in many applications. In the chapters

that follow we explore these and other unique and important attributes of metal matrix composites.

6. Fabrication of metal matrix composites (MMCS)

Metal matrix composites can be made by liquid, solid, or gaseous state processes. In this chapter we describe some important processing techniques for fabricating MMCs.

6.1 Liquid state processing

Metal matrix composites can be processed by incorporating or combining a liquid metal matrix with the reinforcement. There are several advantages to using a liquid phase route in processing. These include near net-shape (when compared to solid state processes like extrusion or diffusion bonding), faster rate of processing, and the relatively low temperatures associated with melting most light metals, such as Al and Mg. The most common liquid phase processing techniques can be subdivided into four major categories:

- *Casting or liquid infiltration*: This involves infiltration of a fibrous or particulate perform by a liquid metal. In the case of direct introduction of short fibers or particles into a liquid mixture, consisting of liquid metal and ceramic particles or short fibers, is often stirred to obtain a homogeneous distribution of particles. In centrifugal casting, a gradient in reinforcement particle loading is obtained. This can be quite advantageous from a machining or performance perspective.
- *Squeeze casting or pressure infiltration*: This method encompasses pressure-assisted liquid infiltration of a fibrous or particulate perform. This process is particularly suited for complex shaped components, selective or localized reinforcement, and where production speed is critical.
- *Spray co-deposition*: In this process the liquid metal is atomized or sprayed while a particle injector introduces ceramic particles in the spray stream to produce a granulated mixture of composite particles. The composite particles are then consolidated using another suitable technique, such as hot-pressing, extrusion, forging, etc.

• *In situ processes*: In this case, the reinforcement phase is formed in situ either by reaction during synthesis or by controlled solidification of a eutectic alloy. We now discuss each of these processing classes in detail.

6.1.1 Casting

Conventional Casting

Casting of MMCs can typically be accomplished with conventional equipment used to cast metallic alloys. It is typically used with particulate reinforcement because of the difficulty in casting fibrous performs without pressure. The particles and matrix mixture are cast into ingots and a secondary mechanical process, such as extrusion or rolling, is applied to the composite, see Fig. 2.

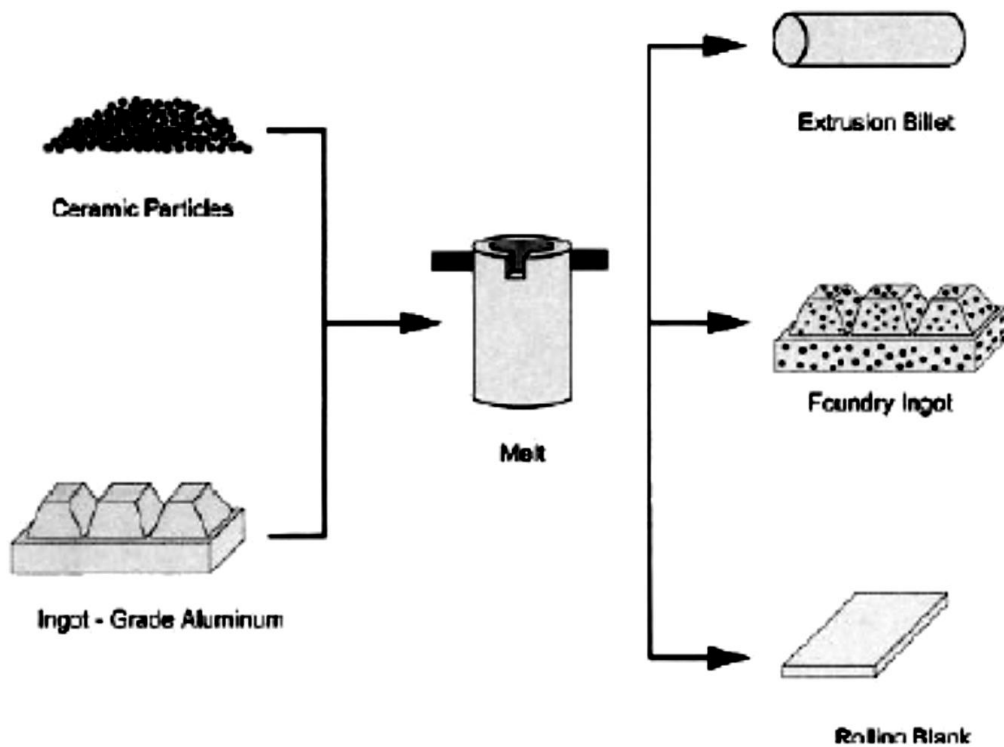


Fig. 2 Conventional casting route for processing particle reinforced MMCs

Stirring of the composite melt is often required, Fig.3. The density of Sic (3.2 g/cm³), for example, is higher than that of Al (2.2 g/cm³), so the particles will sink unless the melt is agitated. Alternating currents in a magnetic field and mechanical vibration have also been used to improve wetting and permeability of the reinforcement in the liquid matrix.

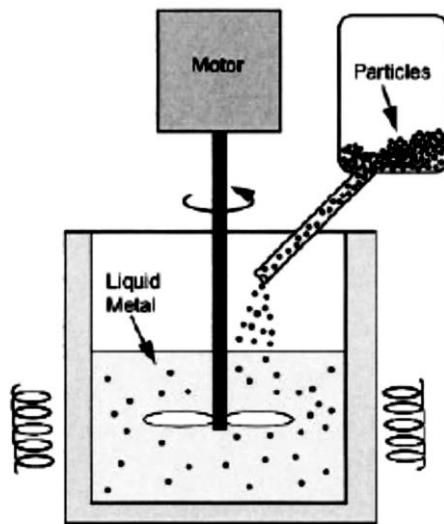


Fig. 3 stirring of composite melts with ceramic particles to minimize settling of the particles during processing

Centrifugal Casting

One of the disadvantages of MMCs with ceramic reinforcement is that they are typically more difficult to machine than the unreinforced alloy. In centrifugal casting, optimal placement of the reinforcement can be achieved by inducing a centrifugal force immediately during casting, Fig. 4, to intentionally obtain a gradient in reinforcement volume fraction. In brake rotors, for example, wear resistance is needed on the rotor face, but not in the hub area. Thus, in areas where reinforcement is not as crucial, such as in the hub area, machining would be easier without the reinforcement.

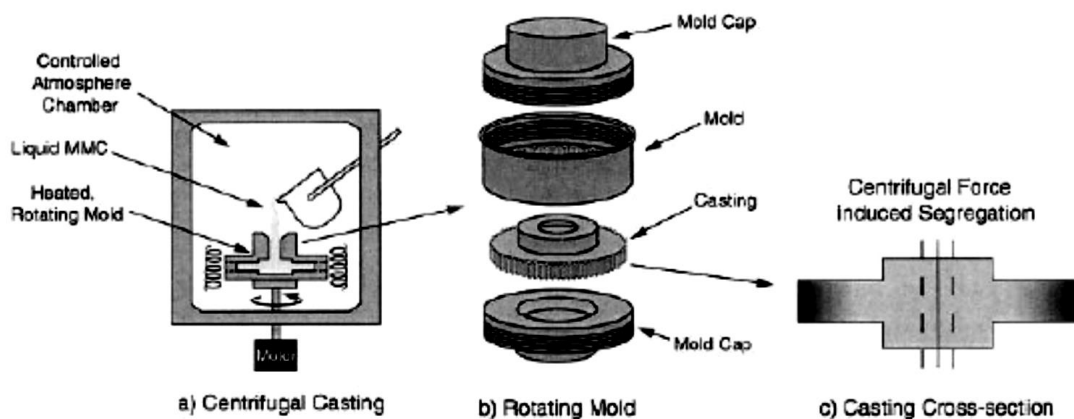


Fig. 4 (a) Schematic of centrifugal casting process, (b) rotating mold, and (c) cross section of finished casting with intentionally-segregated reinforcement

6.1.2 Liquid Infiltration

Liquid Infiltration

Another pressure less infiltration process consists of reactive or non-reactive infiltration of a reinforcement preform. In this process particulate or fibrous filler is infiltrated with pure Al or Al-Mg alloy, Fig.5. Alloy matrices may also be formed in this process. For example, when pure Al is intermingled with Mg particles (in addition to the ceramic reinforcement particles) an Al-Mg alloy matrix is formed. The process is conducted in N₂ atmosphere to minimize interfacial reactions, particularly since the pressure less process involves long infiltration times at high temperatures. When pure Al is infiltrated, the infiltration temperatures are between 700-800°C, while infiltration of Al-Mg alloys is conducted between 700-1000°C (Lloyd, 1997).

Typical infiltration rates are less than 25 cm/h.

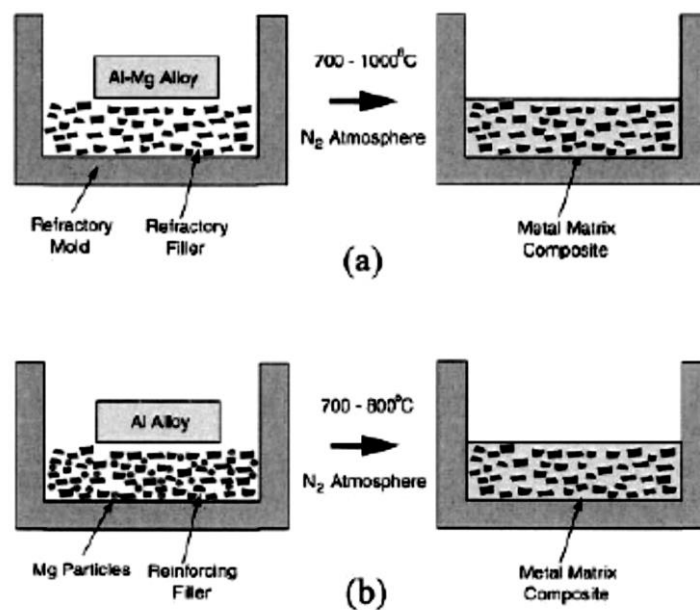


Fig. 5 Pressure less infiltration of MMCs: (a) Alloy matrix infiltration of particulate preform and (b) pure matrix infiltration of metallic alloy particle and ceramic particulate preform

Squeeze Casting

Squeeze casting or pressure infiltration involves forcing the liquid metal matrix into a short fiber or particulate preform. The main advantages of this method over conventional casting are the shorter processing times (which is of particular interest

for production of materials in high volumes), ability to fabricate relatively complex shapes, minimal residual porosity or shrinkage cavities due to the applied pressure, and minimization of interfacial reaction products between reinforcement and matrix (due to the shorter processing times). Before infiltration takes place, the reinforcement preform must be prepared. Fig. 6 shows two processes for making the preform: (a) press forming and (b) suction forming. In the press forming process, aqueous slurry of fibers is agitated and poured into a mold, pressure is applied to squeeze out the water, and the preform is dried. In the other process, suction is applied to the mixture that consists of the reinforcement, fiber, and water. The mixture is then demolded and dried.

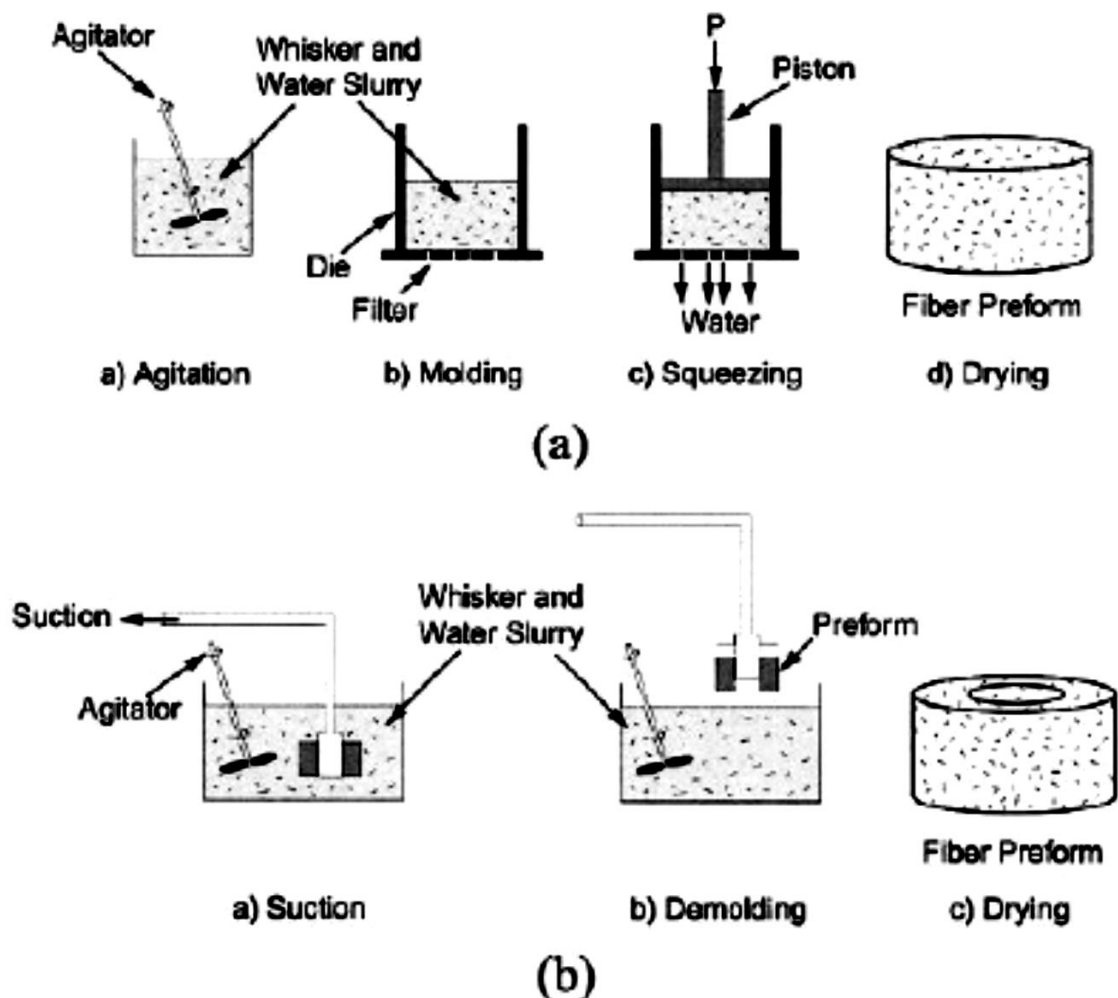


Fig. 6 Processes for making particulate preforms: (a) press forming and (b) suction forming

In order to obtain infiltration of the preform, the molten metal must have a relatively low viscosity and good wettability of the reinforcement. A schematic of the liquid infiltration process is shown in Fig.7. The reinforcement preform is placed in a mold, and the liquid is poured into a preheated die located on the bed of a hydraulic press. Infiltration takes place by mechanical force or by using a pressurized inert gas. Applied pressures on the order of 70-100 MPa are typically used. Having the preform temperature lower than that of the matrix liquid us temperature is highly desirable in order to minimize interfacial reaction and to obtain a fine matrix grain size.

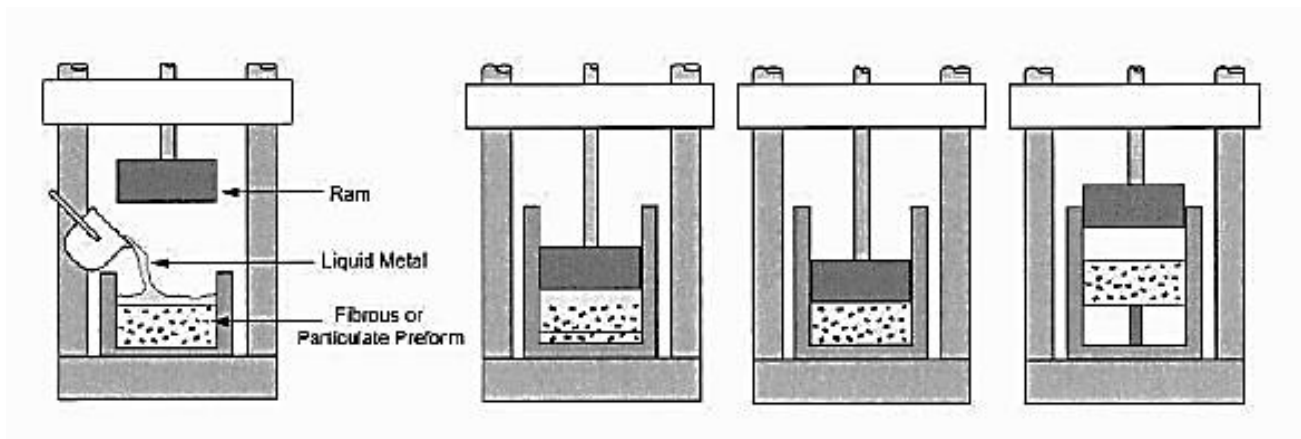


Fig. 7 Schematic of squeezes casting process.

6.1.3 Spray Co-deposition

Spray deposition has been used for some time to fabricate metallic alloys in powder form. The metal or alloy is melted and the liquid stream is atomized with water or an inert gas. Rapid solidification of the liquid takes place, resulting in a fine solid powder. This technique has been modified, by injecting reinforcement particles or co-depositing the particles with the matrix alloy, Fig.8. The advantage of this technique is the high rate of production, which can approach 6-10kg/min, and the very fast solidification rate, which minimizes any reaction between particle and matrix. The as-processed billet is not fully dense, so secondary processing is required to fully density and homogenizes the composite.

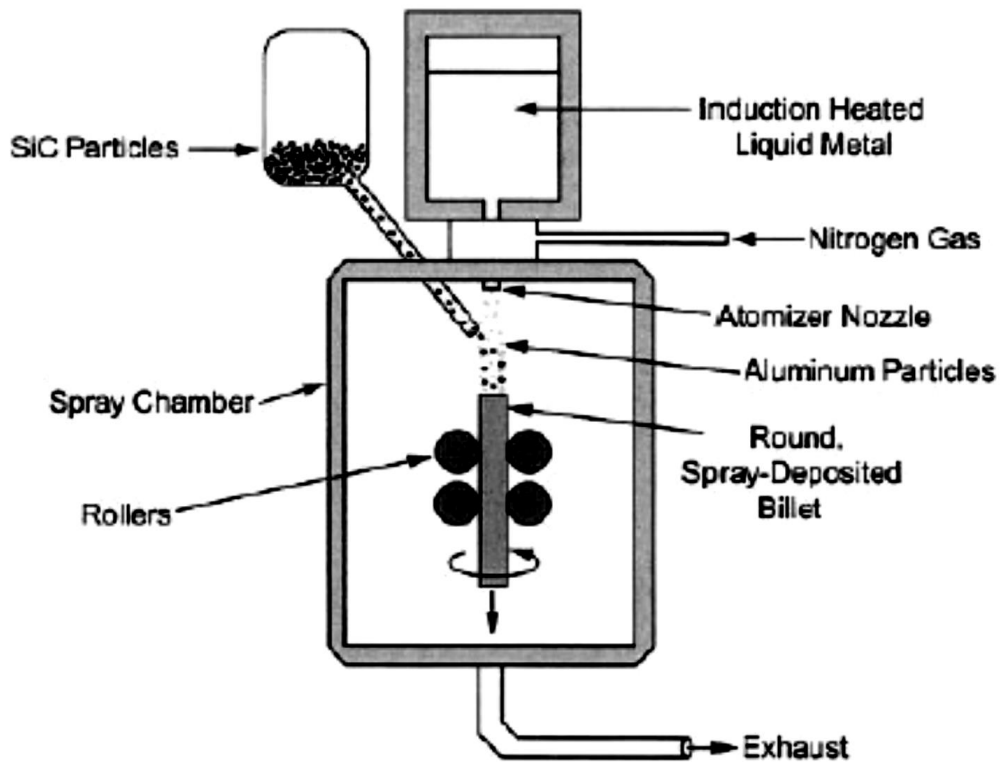


Fig. 8 Spray co-deposition of Sic particles and A1 liquid droplets, to form composite particles

6.1.4 In situ Composite Materials

In situ metal matrix composites (MMCs) is multiphase material whose reinforcing phases are synthesis by chemical reaction during fabrication, while a conventional ex situ MMCs is fabricated by directly adding reinforcements into its matrix. The in situ chemical process has several advantages over ex situ chemical process, such as more homogenous reinforcement, excellent surface bonding, pure interphase, and appears to be a suitable method for preparing metal–ceramic nano-composite.

In-situ and Ex-situ Techniques

In-situ is multiphase material whose reinforcing phases are formed by chemical reaction during fabrication.

Ex-situ is fabricated by directly adding reinforcements into its matrix.

The in-situ chemical process has several advantages over ex-situ process

- More homogenous reinforcement.
- Excellent Surface bonding, good strength and ductility.

- Good electrical properties.
- A suitable method for preparing metal–ceramic nano-composite.

In situ composite material is a material class, which uses the reactions of different components during the production to produce new components. Here source materials are the different metal or alloy powders as well as other powder shaped components. The reaction between components can thus happen both during mixing the powders and in the subsequent processing. However, a minimum of energy must first be introduced to get the reaction going. This can take place, for example, with the thermal energy of hot isostatic pressing, with a subsequent thermal treatment after extrusion, or by high kinetic energy in the case of the mechanical alloying. In the latter case the process is known by the abbreviation, MSR (mechanically induced self-propagating reaction). Takacs gives an overview regarding the procedures applied and the possible reaction partners etc. Table 2 shows a composition of possible reaction systems. These reactions are usually exothermic reactions, which run independently after activation. The components react completely with each other in relation to the percentage by volume and in a matrix, specified before; a new phase develops with a high percentage by volume, which then takes over the reinforcing function.

Table 2 Examples of reaction systems of in situ reactions during powder atomization in the manufacture of composite powders

<i>Gas–liquid reaction</i>	<i>Liquid–solid reaction</i>
$\text{Cu[Al]} + \text{N}_{2/\text{O}_2} \rightarrow \text{Cu[Al]} + \text{Al}_2\text{O}_3$ $\text{Fe[Al]} + \text{N} \rightarrow \text{Fe[Al]} + \text{AlN}$ $\text{Fe[Al]} + \text{N}_{2/\text{O}_2} \rightarrow \text{Fe[Al]} + \text{Al}_2\text{O}_3$ $\text{Fe [Ti]} + \text{Fe [C]} \rightarrow \text{Fe} + \text{TiC}$ $\text{Fe [Ti]} + \text{Fe[B]} \rightarrow \text{Fe} + \text{TiB}_2$	$\text{Ti} + \text{SiC} \rightarrow \text{Ti[Si]} + \text{Ti[C]}$ $g \text{ Fe[Ti]} + \text{Xr}_{\text{xN}} \rightarrow g \text{ Fe} + \text{TiN}$ $\text{Cu[Al]} + \text{CuO} \rightarrow \text{Cu} + \text{Al}_2\text{O}_3$
<p>Liquid–solid reaction</p> $\text{Cu[Ti]} + \text{Cu[B]} \rightarrow \text{Cu} + \text{TiB}_2$	

6.2 SOLID STATE PROCESSING

The main drawback associated with liquid phase techniques is the difficulty in controlling reinforcement distribution and obtaining a uniform matrix microstructure. Furthermore, adverse interfacial reactions between the matrix and the reinforcement are likely to occur at the high temperatures involved in liquid processing. These reactions can have an adverse effect on the mechanical properties of the composite. The most common solid phase processes are based on powder metallurgy techniques. These typically involve discontinuous reinforcements, due to the ease of mixing and blending, and the effectiveness of densification. The ceramic and metal powders are mixed, is statically cold compacted, and hot-pressed to full density. The fully dense compact then typically undergoes a secondary operation such as extrusion or forging. Novel low-cost approaches, such as sinter forging, have aimed at eliminating the hot pressing step, with promising results.

6.2.1 Powder Metallurgy Processing

Powder processing involves cold pressing and sintering, or hot pressing to fabricate primarily particle- or whisker-reinforced MMCs. The matrix and the reinforcement powders are blended to produce a homogeneous distribution. The most common method in manufacture of MMC is by first mixing the existing powders, matrix metal and reinforcement with each other. This can happen in the dry condition, with the help of dispersion agents and/or controlled atmospheres or by mechanical alloying (MA). Fig. 9 show schematically the operational sequence of the powder metallurgy production.

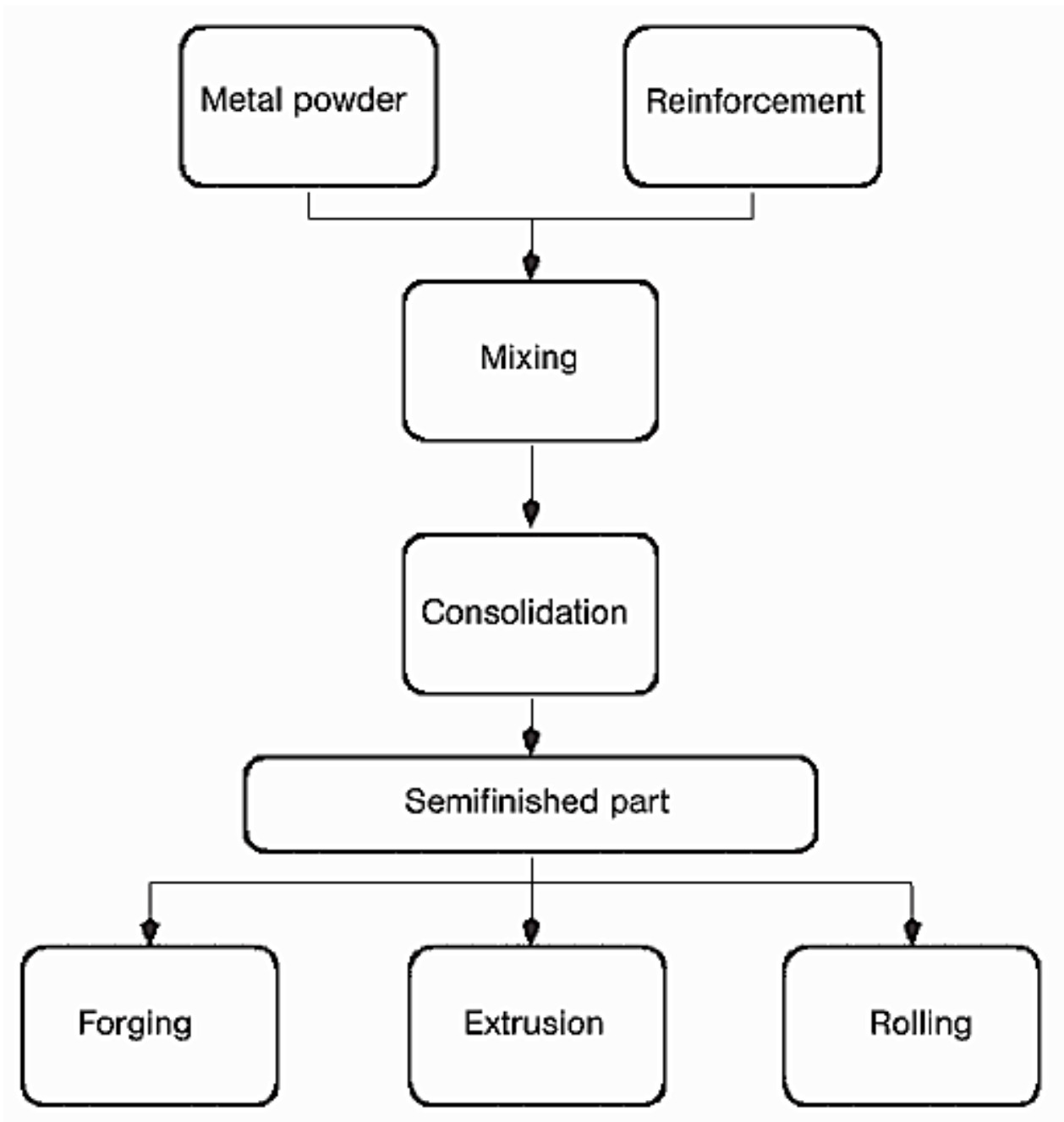


Fig. 9 Schematic presentation of the manufacturing process for MMCs

6.2.2 Mechanical alloying and milling

Two different terms are commonly used in the literature to denote the processing of powder particles in high-energy ball mills. Mechanical Alloying (MA) describes the process when mixtures of powders (of different metals or alloys/compounds) are milled together. Material transfer is involved in this process to obtain a homogeneous alloy. On the other hand, mechanical milling of uniform (often stoichiometric) composition powders, such as pure metals, intermetallics, or pre-alloyed powders, where material transfer is not required for homogenization, is termed Mechanical

Milling (MM). The advantage of MM over MA is that since the powders are already alloyed and only a reduction in particle size and/or other transformations need to be induced mechanically, the time required for processing is short.

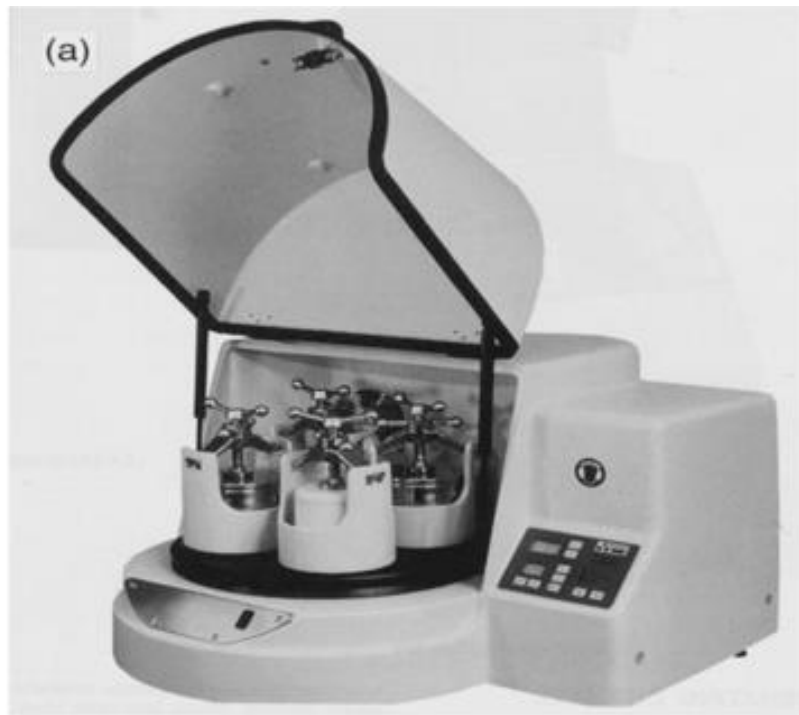
Reaction Milling (RM) is the mechanical alloying process accompanied by a solid-state reaction. In this process, the powder is milled without the aid of any process control agent to produce fine dispersions of oxides and carbides in aluminum. The dispersion of carbides is achieved by adding lamp-black or graphite during milling of aluminum. Adjusting the oxygen content via close control of the milling atmosphere (oxygen, argon, nitrogen, air, etc.) produces the oxides.

6.2.2.1 The process of mechanical alloying

The actual process of MA starts with mixing of the powders in the right proportion and loading the powder mix into the mill along with the grinding medium (generally steel balls). This mix is then milled for the desired length of time until a steady state is reached when the composition of every powder particle is the same as the proportion of the elements in the starting powder mix. The milled powder is then consolidated into a bulk shape and heat treated to obtain the desired microstructure and properties. Thus the important components of the MA process are the raw materials, the mill, and the process variables. We will now discuss the different parameters involved in the election of raw materials, types of mills, and process variables.

6.2.2.2 Types of mills

Different types of high-energy milling equipment are used to produce mechanically alloyed powders. They differ in their capacity, efficiency of milling and additional arrangements for cooling, heating, etc. A detailed description of the different mills available for MA can be found in Planetary ball mills in Fig. 10 and Attritor mills in Fig. 11.



(b)

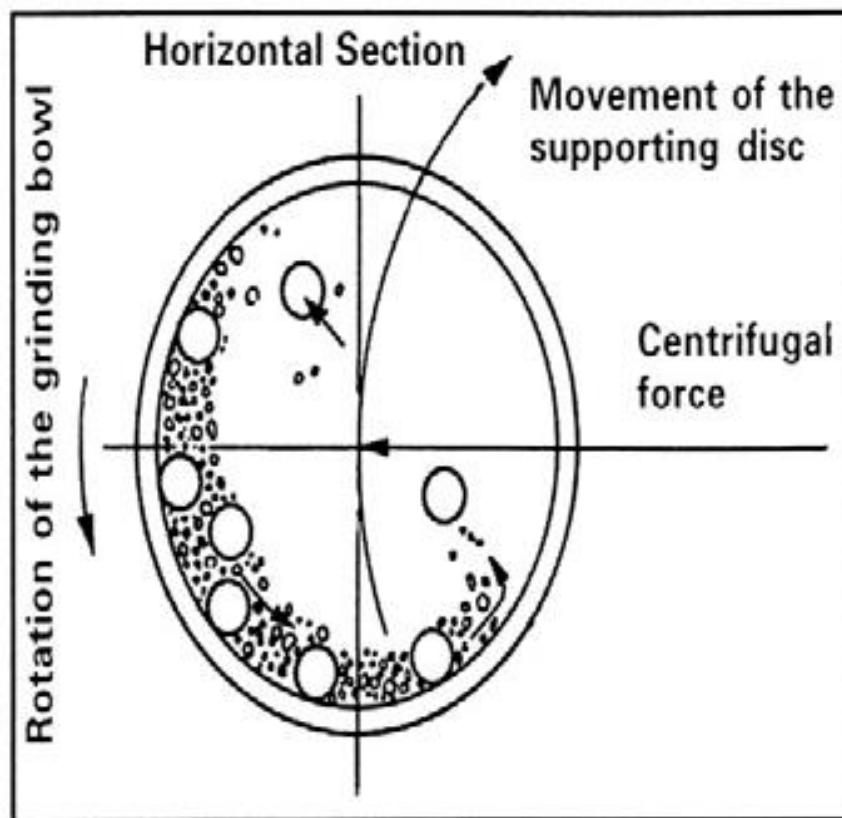


Fig. 10 (a) Fritsch Pulverisette P-5 four station ball mill. (b) Schematic depicting the ball motion inside the ball mill. Courtesy of Gilson Company, Inc., Worthington, OH

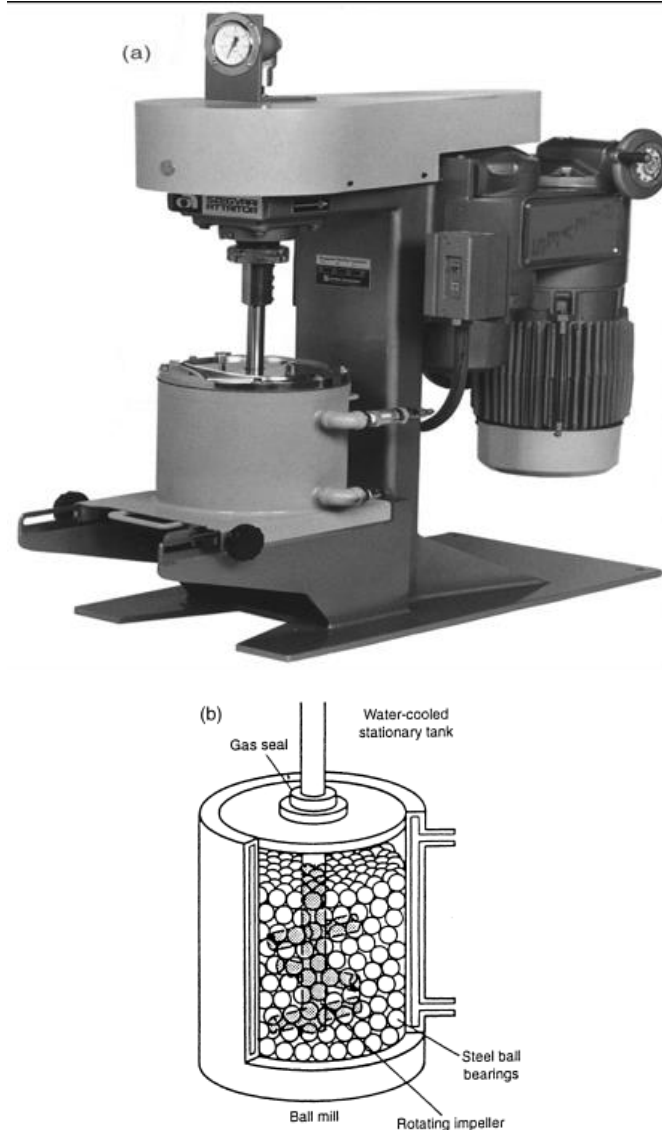


Fig. 11 (a) Model 1-S attritor. (b) Arrangement of rotating arms on a shaft in the attrition ball mill. Courtesy of Union Process, Akron, OH

6.2.2.3 Process variables

Mechanical alloying is a complex process and hence involves optimization of a number of variables to achieve the desired product phase and/or microstructure. Some of the important parameters that have an effect on the final constitution of the powder are:

- type of mill,
- milling container,
- milling speed,
- milling time,
- type, size, and size distribution of the grinding medium,

- ball-to-powder weight ratio,
- milling atmosphere,
- process control agent, and
- Temperature of milling.

All these process variables are not completely independent. For example, the optimum milling time depends on the type of mill, size of the grinding medium, temperature of milling, ball-to-powder ratio, etc..

6.2.2.4 Mechanism of alloying

During high-energy milling, the powder particles are repeatedly flattened, cold-welded, fractured and re-welded. Whenever two steel balls collide, some amount of powder is trapped in between them. Typically, around 1000 particles with an aggregate weight of about 0.2 mg are trapped during each collision Fig. 12. The force of the impact plastically deforms the powder particles leading to work hardening and fracture. The new surfaces created enable the particles to weld together and this leads to an increase in particle size. Since in the early stages of milling, the particles are soft (if we are using either ductile-ductile or ductile-brittle material combination), their tendency to weld together and form large particles is high.

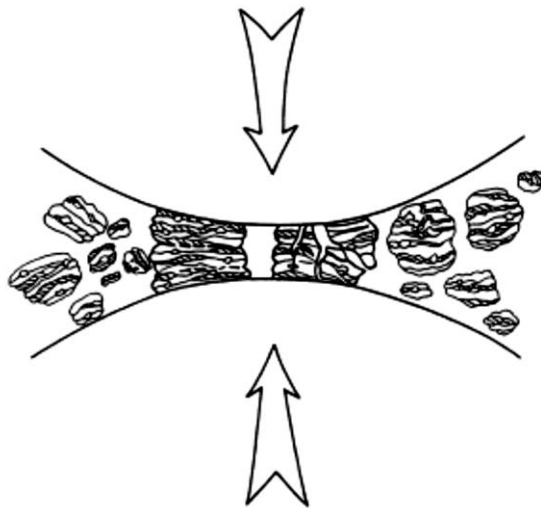


Fig. 12 Ball-powder-ball collision of powder mixture during mechanical alloying

After milling for a certain length of time, steady-state equilibrium is attained when a balance is achieved between the rate of welding, which tends to increase the average particle size, and the rate of fracturing, which tend to decrease the average composite

particle size. Smaller particles are able to withstand deformation without fracturing and tend to be welded into larger pieces, with an overall tendency to drive both very fine and very large particles towards an intermediate size. The particle size distribution at this stage is narrow, because particles larger than average are reduced in size at the same rate that fragments smaller than average grow through agglomeration of smaller particles Fig. 13.

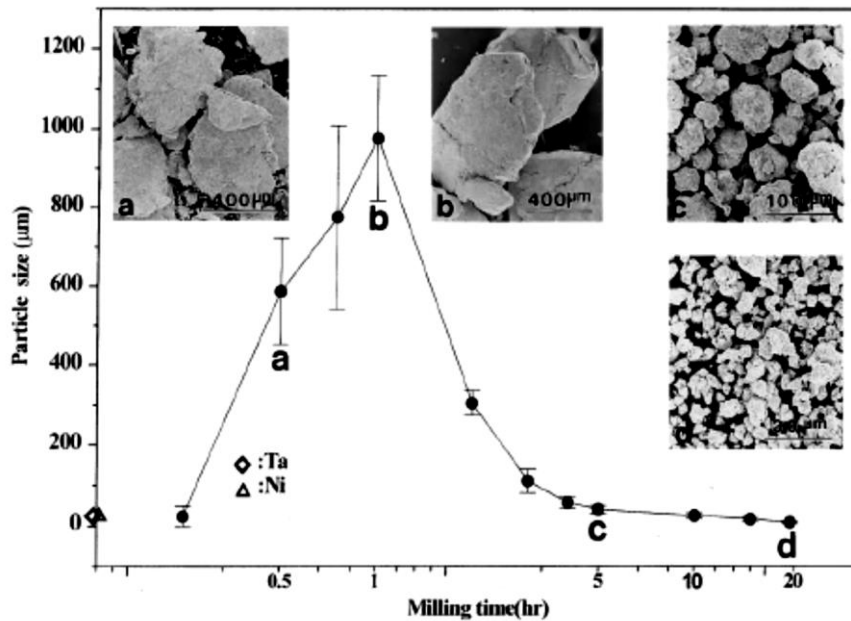


Fig. 13 Narrow particle size distribution caused by tendency of small particles to weld together and large particles to fracture under steady-state conditions

The specific times required to develop a given structure in any system would be a function of the initial particle size and characteristics of the ingredients as well as the specific equipment used for conducting the MA operation and the operating parameters of the equipment. In a few minutes to an hour, the lamellar spacing usually becomes small and the crystallite (or grain) size is refined to nanometer dimensions Fig. 14. The ease with which nanostructured materials can be synthesized is one reason why MA is extensively employed to produce nano-crystalline materials. As mentioned above, it is possible to conduct MA of three different combinations of metals and alloys: (i) ductile-ductile, (ii) ductile-brittle, and (iii) brittle-brittle systems. Therefore, it is convenient to discuss the mechanism of MA also under these categories.

Table 3 gives an overview of MMCs which are produced by mechanical alloying as well as their processing and applications.

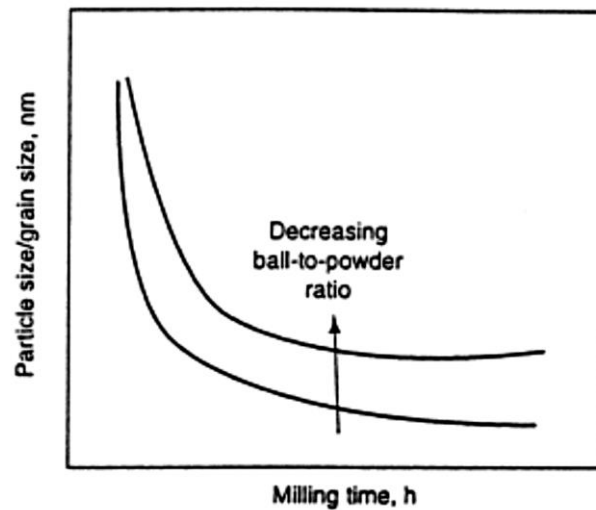


Fig. 14 Refinement of particle and grain sizes with milling time, rate of refinement increases with higher milling energy, ball-to-powder weight ratio, lower temperature, etc.

Table 3 Overview of MMC powders produced by mechanical alloying (MA), their processing and application.

<i>System</i>	<i>Manufacture</i>	<i>Processing</i>	<i>Application</i>
ODS Ni alloys	MA	HIP, extrusion, hot and cold forming, heat treatment	high temperature area, engine industry, glass processing, air and aerospace industry, chemical plant engineering
dispersion hardened Al (Al/Al ₂ O ₃ , Al/Al ₄ C ₃)	MA	CIP, heat treatment (vacuum), extrusion, hot and cold forming	engine industry, high absorbing materials, optical devices, high temperature batteries, compressors, fans
dispersion hardened Cu alloys	MA inner oxidation	axial pressing, CIP, sintering extrusion, hot and cold forming, heat treatment	contact materials, electro-mechanical components, springs
Cu-W	MA	axial pressing, CIP, sintering, extrusion, MIM	spot welding electrodes, contact materials, carrier for electronic components
dispersion hardened Ti alloys	MA	CIP, HIP, extrusion, hot- and cold forming	air and aerospace industry, medical technology

6.3 Compaction and Sintering Characteristics

In the classical powder metallurgical production technique, consolidation follows the manufacture of the powder and powder mixtures. There a variety of techniques have been developed, which are applied alone or in combination. The following techniques are most important:

- Sintering.
- Extrusion.
- Hot-pressing.
- Hot isostatic pressing (HIP).
- Cold isostatic pressing (CIP).
- Forging.
- Rolling.
- Metal injection molding.
- Production of coatings.

The basic steps of powder processing are powder production, compacting of powder, and sintering. The last phase involves heating the “preform” part to a temperature below its melting point, when the powder particles lose their individual characteristics through an inter-diffusion process and give the part its own overall physical and mechanical properties. Sintering lowers the surface energy of the particles by reducing their (surface) areas through inter-particle bonding.

6.3.1 Compacting

Bulk powder can be (automatically) transformed into (“green”) performs of desired geometry and density through compacting prior to their sintering. The first step in this process is effective mixing of the multi-material powder. At this stage, lubricant, in the form of fine powder, is also added to the mixture (for reduced friction) if the powder is going to be formed in a closed die. Most compacting operations, with the exception of processes such as slip casting and spray forming, are carried out under pressure: die compacting, isostatic compacting, powder rolling, extrusion of powder, and powder injection molding (PIM). Pressure-assisted compacting can be further

categorized into cold (at ambient temperature) and hot (material-dependent enhanced-temperature) compactions.

Bulk powders are compressible materials as the pressure is increased, the fraction of voids in the powder rapidly diminishes and the particles deform under (first elastic and then) plastic mechanisms Fig. 15. The denser the preform is, the better are its mechanical properties and the less dimensional variation during sintering.

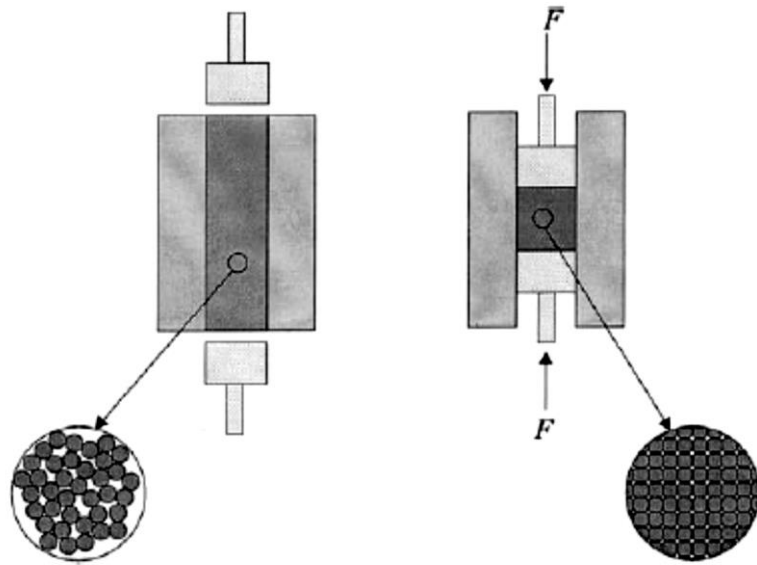


Fig. 15 Compacting of powder.

6.3.1 Sintering

Sintering, the last stage in powder processing, is the thermal bonding of particles into a coherent, primarily solid structure. The mechanical properties of the original green compacted part are significantly improved through the elimination of the pores and the increase in density. However, it should be noted that the former phenomenon occurs at the expense of shrinkage and undesired dimensional changes. Thus maximum densities should be obtained at the pre-sintering compaction phase.

Sintering forms solid bonds between the particles, reducing the surface energy through grain growth and elimination of pores. Individual grain boundaries normally disappear by the end of the sintering process, and what remains behind is a solid cross section with distributed pores Fig. 16. As is further shown in Fig. 17, individual grain boundaries are assumed to disappear through a neck growth process, in which two particles coalesce into a single larger particle.

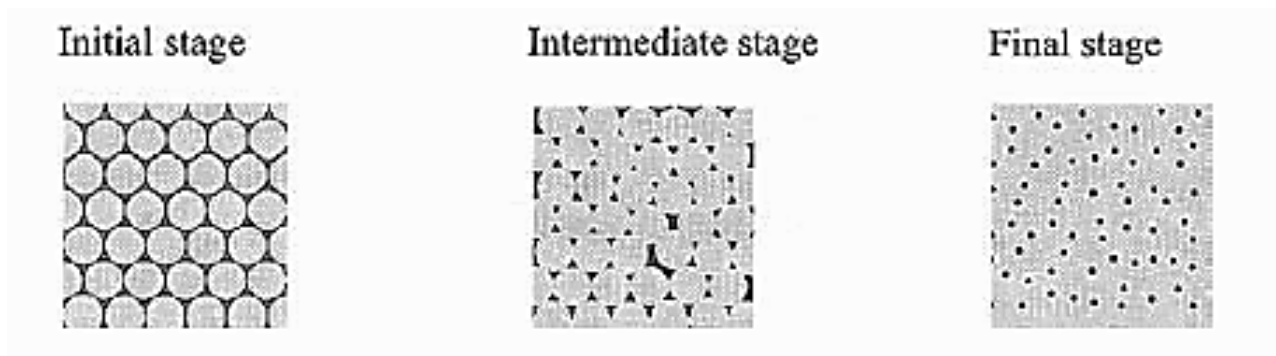


Fig. 16 Sintering as a function of time

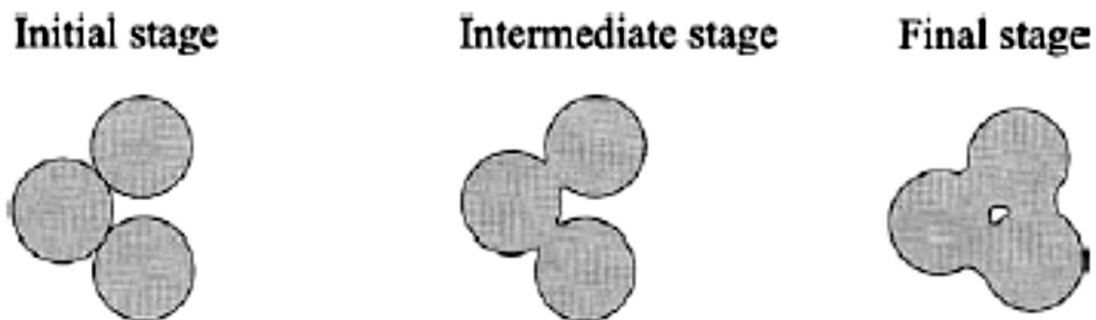


Fig. 17 Neck growth in sintering.

Two mass-transport mechanisms contribute to grain bonding: surface transport and bulk transport. The former yields neck growth at lower temperatures without a change in particle spacing. Although bulk transport also contributes to neck growth, mass densification is the primary characteristic of this mechanism, which is achieved through volume diffusion, plastic flow, and viscous flow at high temperatures.

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