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Review of recent studies in magnesium matrix composites

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In this paper, recent progress in magnesium matrix composite technologies is reviewed. The conventional and new processes for the fabrication of magnesium matrix composites are summarized. The composite microstructure is subsequently discussed with respect to grain refinement, reinforcement distribution, and interfacial characteristics. The mechanical properties of the magnesium matrix composites are also reported. © *2004 Kluwer Academic Publishers*

1. Introduction

Magnesium alloys have been increasingly used in the automotive industry in recent years due to their lightweight. The density of magnesium is approximately two thirds of that of aluminum, one quarter of zinc, and one fifth of steel. As a result, magnesium alloys offer a very high specific strength among conventional engineering alloys. In addition, magnesium alloys possess good damping capacity, excellent castability, and superior machinability. Accordingly, magnesium casting production has experienced an annual growth of between 10 and 20% over the past decades and is expected to continue at this rate [1–3]. However, compared to other structural metals, magnesium alloys have a relatively low absolute strength, especially at elevated temperatures. Currently, the most widely used magnesium alloys are based on the Mg-Al system. Their applications are usually limited to temperatures of up to 120°C. Further improvement in the high-temperature mechanical properties of magnesium alloys will greatly expand their industrial applications. During the past decades, efforts to develop high temperature magnesium materials have led to the development of several new alloy systems such as Mg-Al-Ca [4], Mg-Re-Zn-Zr [5], Mg-Sc-Mn [6] and Mg-Y-Re-Zr [7] alloys. However, this progress has not engendered extensive applications of these magnesium alloys in the automotive industry, either because of insufficient high temperature strength or high cost.

The need for high-performance and lightweight materials for some demanding applications has led to extensive R&D efforts in the development of magnesium matrix composites and cost-effective fabrication technologies. For instance, the magnesium matrix composite unidirectionally reinforced with continuous carbon fiber can readily show a bending strength of 1000 MPa with a density as low as 1.8 g/cm^3 [8–10]. The superior mechanical property can be retained at elevated temperatures of up to 350–400°C [11–13]. Moreover, composite materials are flexible in constituent selection so that the properties of the materials can be tailored. The major disadvantage of metal matrix composites usually lies in the relatively high cost of fabrication and of the reinforcement materials. The cost-effective processing of composite materials is, therefore, an essential element for expanding their applications. The availability of a wide variety of reinforcing materials and the development of new processing techniques are attracting interest in composite materials. This is especially true for the high performance magnesium materials, not only due to the characteristics of composites, but also because the formation of a composite may be the only effective approach to strengthening some magnesium alloys. Mg-Li binary alloys at around the eutectic composition, for example, are composed of HCP (α) and BCC (β) solid solution phases. The dissolution of Li into Mg causes a minor solution strengthening effect without the formation of any Mg-Li precipitates during the cooling process [14]. Thus, heat treatment based on phase transformation cannot be applied to improve their properties. Efforts to strengthen this binary system by producing LiX ($X=$ Al, Zn, Cd etc.) type precipitates have not been successful because these precipitates tend to overage easily, even at room temperature [15–17]. In contrast, the incorporation of thermally stable reinforcements into composite materials makes them preferable for high temperature applications. The potential applications of magnesium matrix composites in the automotive industry include their use in: disk rotors, piston ring grooves, gears, gearbox bearings, connecting rods, and shift forks [2]. The increasing demand for lightweight and high performance materials is likely to increase the need for magnesium matrix composites. This paper reviews recent studies on the processing, microstructure, and mechanical properties of magnesium-matrix composites.

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2. The processing of magnesium matrix composites

A key challenge in the processing of composites is to homogeneously distribute the reinforcement phases to achieve a defect-free microstructure. Based on the shape, the reinforcing phases in the composite can be either particles or fibers. The relatively low material cost and suitability for automatic processing has made the particulate-reinforced composite preferable to the fiber-reinforced composite for automotive applications.

2.1. Conventional processing

Due to the similar melting temperatures of magnesium and aluminum alloys, the processing of a magnesium matrix composite is very similar to that of an aluminum matrix composite. For example, the reinforcing phases (powders/fibers/whiskers) in magnesium matrix composites are incorporated into a magnesium alloy mostly by conventional methods such as stir casting, squeeze casting, and powder metallurgy.

2.1.1. Stir casting

In a stir casting process, the reinforcing phases (usually in powder form) are distributed into molten magnesium by mechanical stirring. Stir casting of metal matrix composites was initiated in 1968, when S. Ray introduced alumina particles into an aluminum melt by stirring molten aluminum alloys containing the ceramic powders [18]. A typical stir casting process of magnesium matrix composite is illustrated in Fig. 1 [19]. Mechanical stirring in the furnace is a key element of this process. The resultant molten alloy, with ceramic particles, can then be used for die casting, permanent mold casting, or sand casting. Stir casting is suitable for manufacturing composites with up to 30% volume fractions [19, 20] of reinforcement. The cast composites are sometimes further extruded to reduce porosity, refine the microstructure, and homogenize the distribution of the reinforcement. Magnesium composites with various matrix compositions, such as AZ31, Z6 [21], CP-Mg (chemically pure magnesium) [22], ZC63 [23], ZC71 [24], and AZ91 [25], have been produced using this method.

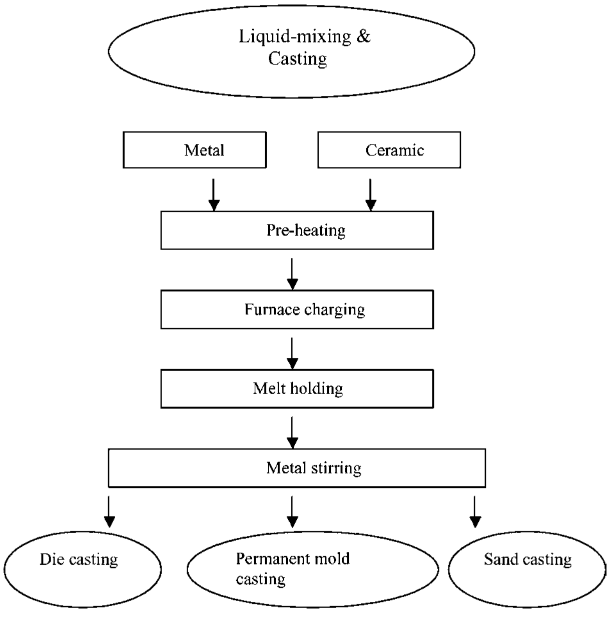

Figure 1 The process of stir casting [19].

A homogeneous distribution of secondary particles in the composite matrix is critical for achieving a high strengthening effect because an uneven distribution can lead to premature failures in both reinforcement-free and reinforcement-rich areas. The reinforcement-free areas tend to be weaker than the other areas. Under an applied stress, slip of dislocations and initiation of microcracks can occur in these areas relatively easily, eventually resulting in failure of the material. In the areas of significant segregation or agglomeration of normally highly brittle hard particles, weak bonds are formed in the material which can lead to the reduced mechanical properties.

A major concern associated with the stir casting process is the segregation of reinforcing particles which is caused by the surfacing or settling of the reinforcement particles during the melting and casting processes. The final distribution of the particles in the solid depends on material properties and process parameters such as the wetting condition of the particles with the melt, strength of mixing, relative density, and rate of solidification. The distribution of the particles in the molten matrix depends on the geometry of the mechanical stirrer, stirring parameters, placement of the mechanical stirrer in the melt, melting temperature, and the characteristics of the particles added [25, 26].

An interesting recent development in stir casting is a two-step mixing process [27]. In this process, the matrix material is heated to above its liquidus temperature so that the metal is totally melted. The melt is then cooled down to a temperature between the liquidus and solidus points and kept in a semi-solid state. At this stage, the preheated particles are added and mixed. The slurry is again heated to a fully liquid state and mixed thoroughly. This two-step mixing process has been used in the fabrication of aluminum A356 and 6061 matrix composites reinforced with SiC particles. The resulting microstructure has been found to be more uniform than that processed with conventional stirring.

The effectiveness of this two-step processing method is mainly attributed to its ability to break the gas layer around the particle surface. Particles usually have a thin layer of gas absorbed on their surface, which impedes wetting between the particles and molten metals. Compared with conventional stirring, the mixing of the particles in the semi-solid state can more effectively break the gas layer because the high melt viscosity produces a more abrasive action on the particle surface. Hence, the breaking of the gas layer improves the effectiveness of the subsequent mixing in a fully liquid state.

Another concern with the stir casting process is the entrapment of gases and unwanted inclusions. Magnesium alloy is sensitive to oxidation. Once gases and inclusions are entrapped, the increased viscosity of the vigorously stirred melt prevents easy removal of these detriments. Thus, the stirring process needs to be more judiciously controlled for a magnesium alloy than for an aluminum alloy in order to prevent the entrapment of gases and inclusions.

In principle, stir casting allows for the use of conventional metal processing methods with the addition of an appropriate stirring system such as mechanical stirring; ultrasonic or electromagnetic stirring; or centrifugal force stirring [28]. The major merit of stir casting is its applicability to large quantity production. Among all the well-established metal matrix composite fabrication methods, stir casting is the most economical (Compared to other methods, stir casting costs as little as one third to one tenth for mass production [29, 30]) For that reason, stir casting is currently the most popular commercial method of producing aluminum-based composites. However, no commercial use of stir casting has been reported on magnesium matrix composites.

2.1.2. Squeeze casting

Although the concept of squeeze casting dates back to the 1800s [31, 32], the first actual squeeze casting experiment was not conducted until 1931 [33]. Fig. 2 illustrates the process of the squeeze casting of a magnesium matrix composite [2]. During squeeze casting, the reinforcement (either powders or fibers/whiskers) is usually made into a preform and placed into a casting mold. The molten magnesium alloy is then poured into the mold and solidified under high pressure. Compared with stir casting, squeeze casting has the advantages of allowing for the incorporation of higher volume fractions (up to 40–50%) of reinforcement into the magnesium alloys [2], and the selective reinforcement of a portion of a mechanical component. Numerous magnesium matrix composites such as SiCw/Mg [34], SiCw/AZ91 [35], Mg_2Si/Mg [36], have been produced using this technology.

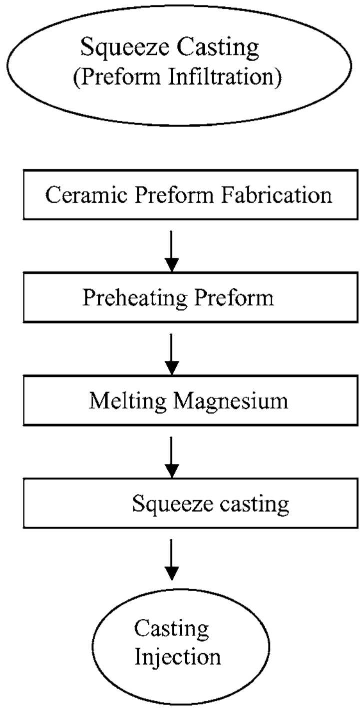

Figure 2 The process of squeeze casting [2].

The applied pressure is the primary variable which affects the microstructure and mechanical properties of the casting. Under high pressure, several unique phenomena take place in the solidifying melt. The first is the shift of the freezing temperature. According to the Clausius-Clapeyron equation $\mathrm{d}T/\mathrm{d}P = T_\mathrm{f}(V_\mathrm{s} - V_\mathrm{l})/L_\mathrm{f}$ (where T_f is the equilibrium freezing temperature of the material, V_s the specific volume of solid, V_l the specific volume of liquid, and L_f the latent heat of solidification), the solidifying temperature of an alloy depends on: the amount of pressure applied, the difference in its liquid and solid specific volumes, and solidification latent heat. It has been found that the eutectic temperature and composition of Al-Si alloy was changed from 660°C and 12.6%Si to 613°C and 17.4%Si respectively at 1300 MPa [33]. The $\mathrm{d}T/\mathrm{d}P$ of pure magnesium has been calculated to be 0.0647°C/MPa [37]. The second effect of the high pressure is the increased cooling rate due to the enhanced heat transfer that results from the closer contact between the mold walls and the solidifying melt. A study on the effects of pressure on the solidification of some eutectic alloys showed that the cooling rate was increased from 11°C/s for permanent mold casting to 282°C/s for squeeze casting [38]. The use of high pressure also introduces effective compensation for the solidification contraction. Under high pressure, the shrinkage in a solidifying ingot can be filled. The resulting material has finer grains and a higher density which lead to a greater strength and especially to an improved ductility of the castings. The ultimate tensile strength and hardness of a squeeze cast Mg-4.2% Zn-RE alloy were improved by 15 to 40% over those produced by permanent mold casting, and the tensile and hardness properties of the Mg-4.2% Zn-RE alloy reinforced with alumina fibers were increased by a factor of two when compared to the permanent mold cast alloy [39]. The high pressure also eliminates risers and feeders needed in normal gravity casting and thus increases casting yield. At the same time, the inherent castability of the alloy becomes less important under high pressure. In addition, squeeze casting is a near-shape process with little or no need for subsequent machining.

In the magnesium matrix composites, however, the pressure for squeeze casting has to be properly controlled because an excessively high pressure may produce a turbulent flow of molten magnesium, causing gas entrapment and magnesium oxidation [40]. The excessively high pressure can also damage the reinforcement in a composite material and reduce the mechanical properties of the composites [41]. Thus, a two-step squeeze casting, consisting of infiltration at low pressure and solidification at high pressure of the matrix alloy has been successfully performed to fabricate a SiC_w/ZK51A magnesium matrix composite [40]. The shortcomings of the squeeze casting process lie mainly in the constraints on the processing imposed by the casting shape, its dimensions and its low suitability for large quantity automatic production.

TABLE I Comparison of stir casting, squeeze casting, and powder metallurgy [29]

Method	Working range	Metal yield	Reinforcement fraction (vol%)	Damage to reinforcement	Fabrication cost
Stir casting	Wide range of shape, larger size (up to 500 kg)	Very high, >90%	∼30	No damage	Least expensive
Squeeze casting	Limited by preform shape, (up to 2 cm height)	Low	∼45	Severe damage	Moderately expensive
Powder metallurgy	Wide range, restricted size	High	–	Fracture	Expensive

2.1.3. Powder metallurgy

A variety of magnesium matrix composites have been fabricated through powder metallurgy such as SiC/AZ91 [42–44], TiO_2/AZ91 [45], ZrO_2/AZ91 [45], SiC/QE22 [46], and B_4C/AZ80 [47]. In the powder metallurgical process, magnesium and reinforcement powders are mixed, pressed, degased and sintered at a certain temperature under a controlled atmosphere or in a vacuum. The advantages of this processing method include the capability of incorporating a relatively high volume fraction of reinforcement and fabrication of composites with matrix alloy and reinforcement systems that are otherwise immiscible by liquid casting. However, this method requires alloy powders that are generally more expensive than bulk material, and involves complicated processes during the material fabrication. Thus, powder metallurgy may not be an ideal processing technique for mass production.

The fabrication methods described above are well established and embody the mainstream of the manufacturing routes for magnesium matrix composites. A comparative evaluation of these three traditional metal matrix composite processing techniques is provided in Table I [29].

2.2. Other processing techniques

In addition to the three well-established synthesis methods described above, a number of other techniques have been explored for the fabrication of magnesium matrix composites, including *in-situ* synthesis, mechanical alloying, pressureless infiltration, gas injection, and spray forming.

2.2.1. In-situ synthesis

Unlike other fabrication methods of the composite material, *in-situ* synthesis is a process wherein the reinforcements are formed in the matrix by controlled metallurgical reactions. During fabrication, one of the reacting elements is usually a constituent of the molten matrix alloy. The other reacting elements may be either externally-added fine powders or gaseous phases. One of the final reaction products is the reinforcement homogeneously dispersed in matrix alloy. This kind of internally-produced reinforcement has many desirable attributes. For example, it is more coherent with the matrix and has both a finer particle size and a more homogenous distribution. However, the process requires that the reaction system be carefully screened. Favourable thermodynamics of the anticipated reaction is the pre-requisite for the process to be applicable. Reasonably fast reaction kinetics are also required to make the fabrication process practical.

TABLE II The physical properties of Mg_2Si [56]

Material	Density (g/cm^3)	CTE (RT-100°C) (10^{-6} K^{-1})	E-modulus (GPa)	Melting point (°C)
Mg_2Si	1.88	7.5	120	1085

In recent years, the *in-situ* synthesis method has been extensively studied for aluminum matrix composites [48–55]. However, for magnesium matrix composites, this technique is still relatively new.

The Mg-Mg_2Si system is probably the first magnesium matrix composite fabricated by *in-situ* synthesis. The physical properties of Mg_2Si, which made it a desirable candidate for reinforcement, are listed in Table II [56]. The high melting point, high elastic modulus, and low density make Mg_2Si a desirable candidate for reinforcement. An analysis of the thermophysical and mechanical properties of Mg-Si system indicated its potential for high temperature application as a lightweight material [57]. In addition, the low solubility of Si in magnesium alloys indicates an easy formation of Mg_2Si in the magnesium matrix. The maximum solubility of Si in magnesium is only 0.003 at.% with the formation of an intermetallic Mg_2Si phase [58]. Thus, the Si added to the magnesium alloy can either readily react with the magnesium during the melting process or can precipitate from the matrix during the cooling process in the form of an intermetallic Mg_2Si phase. The abundance and low cost of Si explains why the Mg-Si alloy is the earliest system studied for *in-situ* synthesis of a magnesium matrix composite.

The drawback for Mg_2Si is its tendency to form coarse needle-shaped Mg_2Si phase at high concentration of Si during conventional casting which can reduce the mechanical properties of the final material. Various efforts have been made to modify the microstructure and improve the mechanical properties of the Mg-Mg_2Si *in-situ* composite.

Squeeze casting is one of the early attempts in this field. However, the high hardness of Mg_2Si imposes serious difficulties in casting Mg-Mg_2Si with a high content of Mg_2Si [57, 59].

The second fabrication method is the ingot casting of the Mg-Si alloy, followed by hot extrusion [60–62]. The hot extrusion greatly refines the matrix grains and the Mg_2Si phase and improves the homogeneity of the Mg_2Si distribution. The major finding in this series of experiments is that the cast materials, Mg-high Si ($\geq$10 wt%) alloys, showed lower tensile strength values

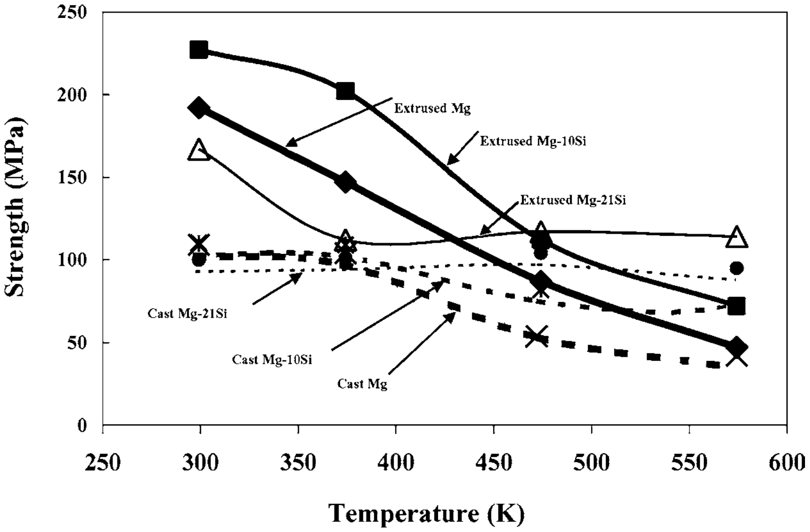

Figure 3 The tensile strength of extruded and cast Mg-Si alloys [60].

at temperatures of up to 100°C, as compared to the pure Mg and Mg-low Si (<10 wt%) alloys, whereas the strength at 300°C increased with an increasing Si content. The mechanical properties, too, of the cast material are improved by hot extrusion. The resulting tensile strengths of these various Mg-Si alloys are shown in Fig. 3 [60]. Alloying an Mg-Si system with aluminum and zinc has also been found to effectively increase its strength.

Rapid solidification is another process used to improve the mechanical properties of the Mg_2Si/Mg composite [63, 64]. As a result of the high cooling rate, both magnesium matrix grains and the precipitated Mg_2Si phase have been found to be dramatically refined. Table III lists the mechanical properties of an *in-situ* Mg_2Si/Mg composite [64] produced by the rapid solidification of a magnesium alloy Mg-10.6 wt%Si-4.0 wt%Al, during which process about 20 vol% of fine Mg_2Si particles were precipitated. A composite such as this has outstanding mechanical properties, with a specific strength conspicuously greater than that of magnesium alloys AZ91 and ZK60, aluminum alloy 7075, and even greater than that of titanium alloy Ti-6Al-4V. Obviously, the very fine microstructure of the rapidly solidified Mg_2Si-Mg composite matrix also plays an important role in producing the superior performance.

A more recent study, published in 1998, has shown that a mixture of MgO and Mg_2Si particulates can be *in-situ* produced in Mg-Li matrix at temperatures of 750–800°C by reactions of $4Mg + SiO_2 = 2MgO + Mg_2Si$ [65]. The particles are 2–5 μm in diameter and have a uniform distribution in the matrix. Another publication, which appeared in 2000, has successfully *in-situ* fabricated an Mg_2Si reinforced magnesium matrix composite by using gas pressure infiltration of a hybrid preform with AZ31, AZ91, and AE42 magnesium alloys [66]. The preform is composed of 7 vol% C-fibers and 4 vol% of Si particles bonded with SiO_2. The reactions occurring during the infiltration process are $2Mg + Si = Mg_2Si$, and $4Mg + SiO_2 = 2MgO + Mg_2Si$. The resulting materials have shown excellent creep strength. This research is ongoing. The most recent work has successfully produced magnesium matrix composites reinforced with Mg_2Si by mechanically milling elemental Mg, (Al), and Si powders. The results of this work are discussed in the next section.

Apart from the Mg_2Si-Mg system, a number of other *in-situ* systems have also been explored. These include an Mg-MgO composite formed by the reaction between Mg and B_2O_3 [67], an Mg-TiC composite formed by the reaction of Mg with Ti and C [68], and an Mg-TiB_2-TiB composite formed by the reaction of Mg with KBF_4 and K_2TiF_6 [69]. However, compared with the research on the Mg_2Si-Mg system, studies on these new *in-situ* magnesium composites are still in their inception stage. *In-situ* synthesis is a relatively new approach to magnesium matrix composite fabrication. The unique features of the process are expected to stimulate more research activities in this field.

2.2.2. Mechanical alloying

Mechanical alloying, which was developed in the late 1960s [70], is a process in which raw powders are

TABLE III The mechanical properties of *in-situ* Mg_2Si/Mg composite [64]

Materials	UTS (MPa)	0.2% proof stress (MPa)	Elongation to failure (%)	Specific strength (MPa)	Specific proof stress (MPa)
Mg-11Si-4Al	506	455	2	281	253
AZ91C (T4)	240	70	7	133	39
ZK60A (T5)	365	305	11	203	169
A7075 (T6)	573	505	11	205	180
Ti-6Al-4V (T6)	1166	1030	7	261	233

mixed with high energy milling balls, with or without additives, in an inert atmosphere. (Strictly speaking, the mechanical alloying is also a powder metallurgy process.) During the mixing process, the powders go through repeated cold welding and fracturing until the final composition of the very fine powders corresponds to that of the initial charge [71]. Along with the refining of powders, some solid state chemical reactions may also occur, driven by the high mixing energy. Thus, materials of unique microstructures and properties can be produced during the mechanical alloying process. This technique has been extensively applied to generate oxide dispersion strengthened alloys (nanocomposites) [72–74].

Mechanical alloying can take different forms. The first is the mechanical milling of a mixture of metal and ceramic powders, such as Al and SiC [75], whereby the grinding process reduces the dimensions of matrix grains and particles. The second method forms a nanocomposite using mechanical mixing to reduce an oxide powder with a metal powder, such as in the mixture of Al and CuO powders [76]. The third method involves directly milling elemental precursors to form secondary particles, such as in the Nb-Si-Ti-C system [77]. Due to the reactions occurring during mixing, the latter two methods can also be considered to be *in-situ* syntheses of the composite. Mechanical alloying has been extensively studied for the fabrication of magnesium matrix composites. Major reinforcements used in the formation of magnesium matrix composite by mechanical alloying include: silicide (Mg_2Si) [78–81], carbide (TiC) [82], boride (Ti_3B_4) [83], and oxide (MgO) [84]. Functional magnesium matrix composites such as hydrogen storage materials have also been fabricated by mechanical alloying [85, 86].

During the mechanical alloying process, the raw powders such as Mg, Si, Ti, C, and/or TiC are first mixed according to the target material and method used. During milling, the particles are refined with or without reactions taking place, depending on the powder system selected. After the high energy mixing, the powders are usually consolidated by vacuum hot pressing or by hot extrusion. If there are any solid-state reactions, they can also take place during this stage.

Solid-state reactions can also occur during the milling process. For example, TiC can be formed directly by mixing and milling Ti and C powders with high energy balls, and Mg_2Si can be formed from Mg and Si powders. In the Mg-Ti-C system, the solubility of Ti and C in magnesium is negligible and neither powder reacts with magnesium to form compounds. In addition, Ti and C can readily form TiC by mechanochemical reaction during milling [87, 88]. The as-milled Mg-Ti-C nanocomposite exhibited a matrix grain size ranging between 25 and 60 nm with a dispersion of ultra-fine nanometer-sized TiC particles (3–7 nm). Moreover, the Mg-Ti-C nanocomposite showed remarkably high ductility [82]. Another study on the same mechanically alloyed Mg-Ti-C system revealed that the reaction in the early stage of mechanical alloying is solely a refinement of graphite. No graphite/titanium reaction was observed. The next stage is the formation of TiC from refined graphite and Ti, which can be accelerated by heat treatment [68]. In the Mg-Si system, the formation of Mg_2Si from elemental Mg and Si powders can be achieved within 10 h of mechanical alloying. The amount of Mg_2Si formed increases with the increasing milling duration and annealing temperature. Mg_2Si with a grain size of 22 nm is stable up to 390°C. The nanostructured Mg-Al alloy reinforced with Mg_2Si can also be formed using mechanical alloying. Intermediate phase $Al_{12}Mg_{17}$ has been synthesized during mechanical alloying and decomposed during post annealing at 300°C. A stable Mg-Al solid solution reinforced with Mg_2Si has been obtained after annealing [78]. The resulting nanocomposite has a tensile strength and a yield strength of 350 MPa and 300 MP, respectively [79].

Mechanical alloying may also be a reduction process. In order to form a nanosized MgO reinforcing particle, powders of pure Mg and Mg-Al (AZ91) alloy can be mechanically alloyed with the addition of metal oxide (MnO_2 and Fe_2O_3). During milling, the MnO_2 and Fe_2O_3 powders decompose and then form MgO and intermetallic aluminide compounds. After extrusion, the matrix grains of the magnesium matrix composite can be as fine as 100–200 nm while the dispersed compounds are of less than 20 nm in size. In such an Mg-Al-MnO_2 system, the 0.2% proof stress and specific strength of the as-extruded material were found to be around 600 and 300 MPa, respectively, in a compression test [84].

2.2.3. Pressureless infiltration

Fabricating magnesium matrix composites through spontaneous or pressureless infiltration is relatively new as compared with pressure infiltration (squeeze casting). During the infiltration process, molten alloys flow through the channels of the reinforcement bed or preform under the capillary action. Certain criteria have to be met for the spontaneous infiltration to occur.

A SiC/Mg composite has been attained using this method [89]. The experimental set-up of the spontaneous infiltration is shown schematically in Fig. 4.

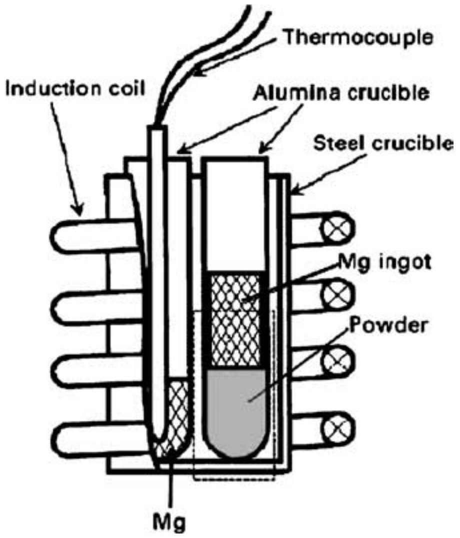

Figure 4 The setup of Mg infiltration in mixed SiC and SiO_2 powders [89].

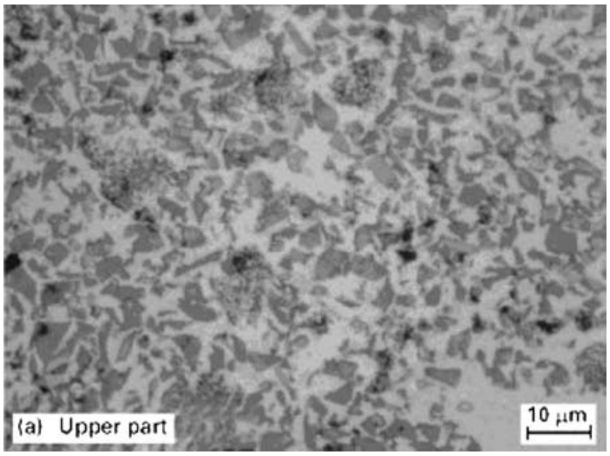

Figure 5 The microstructure of the infiltrated SiC/Mg composite [89].

SiC particles and infiltration agent SiO_2 powders were mixed and placed in an alumina crucible, then placed in a steel crucible. Another alumina crucible, which contained pure magnesium, was set beside the infiltration alumina crucible to monitor the temperature during the infiltration process. When the system was heated, the pure magnesium ingot, which was set on top of the powder mixture, melted and spontaneously infiltrated the powder mixture. The microstructure of the infiltrated SiC/Mg composite within which the SiC particles of a high volume fraction were distributed very evenly, is shown in Fig. 5.

The mechanism for spontaneous infiltration is considered to be the high temperature generated at the infiltration front resulting from the reactions between magnesium and SiO_2 [89]. The infiltration behaviour depends mainly on the SiO_2 content and the powder size. Without the SiO_2 there was no infiltration, and the minimum SiO_2 content needed to start the infiltration process increased with a decease in the SiC powder size. The proposed pressureless mechanism implies the existence of other infiltration agents if they can react with Mg to produce a high temperature. This was confirmed by the fact that TiO_2 can also be used as an infiltration agent [90].

2.2.4. Gas injection

Particulate reinforced metal matrix composites were incepted in the 1960s, when Ni-coated graphite powders were injected into an Al alloy melt with N_2 gas to form a reasonably uniform distribution in the final castings which solidified at moderately rapid rates [91]. More recently, this method has been employed to synthesize a magnesium matrix composite [92] where SiC and Al_2O_3 particles of various sizes were transported pneumatically through a tube or lance below the bath surface of a molten AZ91 alloy at 720–730°C with a carrier gas of either Ar or N_2. The ceramic powders were transported by the injection gas under a flow rate of 3.0–3.5 l/min from a screw powder feeder at a rate of 30–40 g/min. The reinforcing particles were found to have a reasonably uniform distribution in the AZ91 matrix after being injected with N_2. The maximum volume fraction of the injected SiC particles was about 17%, and the SiC particles were, for the most part, evenly distributed in the cast alloys, in spite of a number of clusters and agglomerates of the particulates.

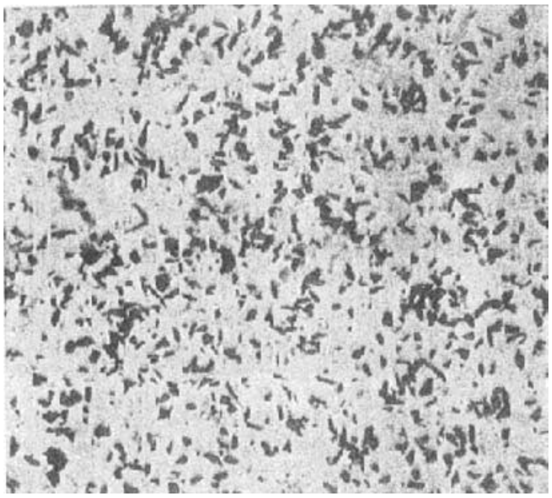

Figure 6 The microstructure of gas injected 17 vol%SiC/Mg composite [92].

The microstructure of a 17 vol% SiC/Mg composite produced by gas injection is shown in Fig. 6. Although some clusters formed, in general, the SiC particles were distributed uniformly. The mechanical properties of an injection-manufactured composite are illustrated in Fig. 7. With some exceptions, the addition of SiC particles increased the strength and elastic modulus of the material. The addition of 5 vol% SiC reduced the strength while 8 vol% maximized the strength. This variation in mechanical properties may be related to the formation of SiC clusters, which can cause cracking under a relatively low applied load. Optimized processing may minimize or eliminate the SiC clusters. Gas injection can be an economical method of fabricating magnesium matrix composites, but further research is needed to further explore this particular technology.

2.2.5. Spray forming

Spray forming or spray deposition is a process during which an atomized stream of molten material droplets is directed onto a substrate to build up bulk metallic materials. For a metal matrix composite, reinforcing particles are injected into the stream of the atomized matrix materials. The droplet velocities typically average about 20–40 $m \cdot s^{-1}$, and inhomogeneous distributions of ceramic particles are often present in the spray formed metal matrix composite [93].

A number of studies [94–97] on the fabrication of magnesium matrix composites using the spray forming method have examined the relationships between the spray processing parameters, the microstructure, and the mechanical properties of the composites. The process parameters were found to exert a considerable influence on the microstructure and properties of a SiC particle (8–12 μm) reinforced QE22 alloy [94]. But, due to the high cooling rate, the sprayed composite usually shows microstructural features typical of rapid solidification processes such as fine grains, porosity, and absence of brittle phases at the SiC/matrix interface

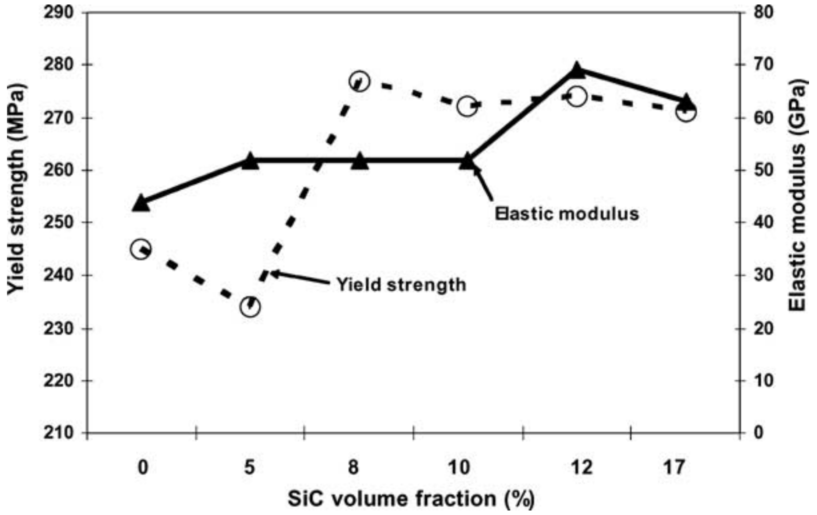

Figure 7 Yield strength and elastic modulus of injected SiC/AZ91 composite [92].

[97]. Furthermore, in some SiC particulate dispersed Mg-10% Ce and Mg-5%Ca alloy composites formed by spray deposition, the materials have a relative density greater than 95% which can be further improved to greater than 99% by hot extrusion. The minimum size of the sprayed SiC particles is even smaller than 1 μm. The elastic modulus and hardness were also appreciably increased by the dispersion of SiC particles [96].

2.3. Wettability

A number of factors can affect the processing of a magnesium matrix composite. Wettability between the reinforcement and the matrix alloy is one of the critical factors in the liquid processing. When the wettability is low, the mechanical agitation force in stirring casting, the pressure in squeeze casting, and the catalyst in infiltration are indispensable to overcome the surface energy barrier so that the reinforcement phases can be distributed into the molten alloy and the liquid metal can penetrate the reinforcement bed to form a strong bond.

The wettability between the matrix in the molten state and the reinforcement material depends on several elements. These include the intrinsic properties of the material such as the surface energy of the matrix and the reinforcement [98], and the surface condition of the particles such as the amount of oxidation and contamination. Wettability can also be improved by increasing the surface energy of the solid, decreasing the surface tension of the molten metal, and decreasing the particle-matrix interface energy [99, 100] through coating of the particles, adding additives to the melt, heating and cleaning of the particles, and subjecting the melt to ultrasonic irradiation.

A moderate modification in the matrix alloy composition can sometimes considerably alter its wettability with the same reinforcement [101, 102]. This is usually a result of the formation of a transient layer between the particles and the liquid matrix. The transient layer has a smaller wetting angle and surrounds the particles with a structure similar to those of both the matrix alloy and particles. Experimental investigation has demonstrated that in pure Al and Mg molten metals, a spontaneous rejection of Si_3N_4 particles took place at 7–8 vol% of the particles, while in Al and Mg alloys with 10% silicon, the rejection of Si_3N_4 particles did not occur until the volume fraction of Si_3N_4 particles reached 17–18% [101]. The distribution of SiC particles in the composite can be greatly improved by either the addition of Mg metal just prior to introducing the ceramic particles and/or by prior mixing of the ceramic with a mixture of zirconia and magnesia of a proprietary composition [102].

Pre-treatment of the reinforcement phase is another approach to enhance wettability. For example, coating graphite powders/fibers with Ni [100] or SiO_2 has been employed to improve wettability between the particles and aluminum alloys [103]. A metal coating on ceramic particles increases the overall surface energy of the solid and improves the wettability by changing the contact angle from metal-ceramic contact to metal-metal contact. More recently, coating SiC particles with Cu and Ni has also been used to prepare a pure Mg matrix composite [104]. However, due to the intrinsic higher wettability between SiC particles and magnesium alloys as compared with that between SiC and aluminum, the coating of SiC is used less frequently in the magnesium matrix composite. Cleaning of the reinforcement surface also helps to enhance wettability because the presence of a thermodynamically stable oxide layer (like Al_2O_3) on the reinforcement preforms inhibits the wetting and infiltration [105]. Thus, the degree of cleanliness of the reinforcement must be checked before its addition to the molten metals. Heat treatment of the particle prior to its addition is another common technique used to degas its surface for a better wetting. In short, the pre-treatment of the reinforcing materials depends on the characteristics of particles and matrix alloys, and on the type of processing methods employed.

Interface reactions between the matrix and the reinforcement also directly influence their wettability. It has been revealed that, in some aluminum systems, an improved wettability at the interface can be achieved by a chemical reaction that forms spinels or oxides isostructural with spinels ($MgAl_2O_4$) [106]. The thickness and uniformity of the interfacial reaction layer can be controlled by adjusting the melt temperature and the degree of melt agitation as well as the reinforcing phase residence time in the molten metal. The interfacial reaction is also affected by some other factors, which are discussed in later sections.

Much of the research to date on improving the wettability between a ceramic reinforcement and a matrix of the metal matrix composite are based on an aluminum matrix. Although less information is available on magnesium matrix composites, the approaches used in aluminum matrix composites may be used for magnesium matrix composites.

3. The microstructure of magnesium matrix composites

The key features in the microstructure of a composite material resulting from the interaction between the matrix and the reinforcement usually include the type, size, and distribution of secondary reinforcing phases, matrix grain size, matrix and secondary phase interfacial characteristics, and microstructural defects. The mechanical properties of the composite materials are strongly influenced by these factors.

3.1. Types of reinforcement

Two types of reinforcing materials have been investigated for magnesium matrix composites. The first and most widely used is ceramic. The other is metallic/intermetallic.

Ceramic particles are the most widely studied reinforcement for magnesium matrix composites. Some common properties of ceramic materials make them desirable for reinforcements. These properties include low density and high levels of hardness, strength, elastic modulus, and thermal stability. However, they also have some common limitations such as low wettability, low ductility, and low compatibility with a magnesium matrix. Among the various ceramic reinforcements, SiC is the most popular because of its relatively high wettability and its stability in a magnesium melt, as compared to other ceramics.

The shape of reinforcement is another factor affecting the reinforcing effect. In a magnesium matrix composite, the most commonly used reinforcements assume a shape of short fiber/whisker, or particle, or a mixture of these two configurations. Short fiber/whisker reinforced magnesium alloys usually show better mechanical properties than the particle reinforced magnesium alloy with some degree of anisotropic behaviors. The strengthening effect depends on the characteristics of the strengthening mechanism. To overcome the barriers of relatively high cost and the anisotropic properties associated with fiber reinforcement, some recent efforts have been made to reduce the fiber cost by developing a new fibrous material and using hybrid reinforcements that incorporate particles into fibers. For instance, because the cost of aluminum borate whiskers is about only 10% of that of SiC whiskers [107], this material has been used recently and shows promise for commercial applications of the magnesium matrix composite. The size of the reinforcement used has ranged from nanometers to micrometers.

Because metallic solids will generally have a much better wettability with liquid metals than ceramic powders [108], the reinforcing of a magnesium matrix with metallic/intermetallic particulates has recently been examined. Elemental metal powders, such as Cu, Ni, and Ti particulates with a diameter of a few micrometers, have been used as reinforcement agents in magnesium matrix composites because of their high melting points and very low solubility in magnesium [109–112]. The advantages of the metallic reinforcements lie in their high ductility, high wettability and high compatibility with the matrix as compared with ceramics, and their great strength and elastic modulus as compared to the magnesium matrix. A major concern in the use of the elemental metallic powders is that their relatively high density could compromise the lightweight of the magnesium-based composites. The higher specific strengths that the metallic powder reinforced composites have shown as compared to the ceramic reinforced ones indicated that the density increase was compensated for by the increased reinforcing effect of the metallic powders [109-112]. Since the high density of the metal powders can also cause mixing difficulties, the process has to be carefully designed and controlled to minimize the segregation of the reinforcements. Recently, the *in-situ* formation of aluminum based intermetallic particles such as Al_3Ni, Al_3Fe, and other transitional metal aluminides [113] as a reinforcement for lightweight metal composites, has been proposed. These intermetallics have similar benefits as the metallic reinforcement with a much lower density. The methods of *in-situ* formation of these particles are yet to be developed.

It is worthwhile to mention an interesting recent discovery associated with the formation of hard amorphous or quasi-crystalline phases at grain boundaries of some magnesium alloys [114–117]. Depending on the alloy composition, the amorphous or quasi-crystalline phases are reported to form under both rapid cooling and standard casting conditions. Furthermore, the amorphous or quasi-crystalline phases cannot be removed by annealing. The high thermal stability of the amorphous or quasi-crystalline phases has led to increased strength of magnesium alloys at elevated temperatures. However, the formation mechanism and the thermal stability of the amorphous and quasi-crystalline phases have to be further confirmed and better understood before any real application should be attempted.

3.2. Matrix

Various magnesium alloy systems have been used as the matrix for composites. In addition to the normal crystalline alloys predominantly used as the matrix, the recent developments in magnesium alloys, with high glass formation ability, has also triggered the investigation of magnesium composites to fully explore the unique mechanical properties of amorphous metals.

3.2.1. Amorphous matrix

The strength of magnesium metallic glasses is two to four times greater than that of commercial magnesium alloys [118]. Normally, amorphous metals are formed at a very high cooling rate, which has prevented the industrial production of amorphous metals. Some magnesium alloys developed recently exhibited a high glass formation ability and high thermal ability. These glass alloys can be cast with conventional techniques while maintaining their amorphous state at relatively high temperature [119]. Studies have also revealed that the presence of nanoscale precipitates or homogeneously distributed fibers or particles can further improve the mechanical properties of the glass metals [120–122]. Based on these findings, a magnesium composite with an amorphous matrix has been produced by mechanical alloying of powder mixture of $Mg_{55}Cu_{30}Y_{15}$ with MgO, CeO_2, Cr_2O_3, or Y_2O_3 oxide particles [123]. The addition of these oxide particles does not significantly affect the formation tendency of an amorphous matrix. However, they do affect the thermal stability of the amorphous matrix.

The mechanical properties of this amorphous magnesium matrix composite are excellent. The composite containing 5 vol% Y_2O_3 possessed a room temperature strength of 709 MPa, 1.5–2.5 times greater than that of the conventional crystalline magnesium alloys. The addition of oxide particles increased the fracture strength by 100–150 MPa and Young's modulus by 28 GPa. The improvement in mechanical properties of the amorphous matrix magnesium composite was more pronounced at elevated temperatures where the amorphous matrix was stable. The yield strength of the composite containing 5 vol% oxide at 150°C was almost the same as at room temperature while the yield strength corresponding to 20 vol% oxide addition exceeded that at room temperature.

While the strengthening mechanism in the amorphous matrix composite is not well understood, in general it has been attributed to the change in the deformation mechanism of the matrix caused by the presence of oxide particles. At room temperature, the deformation of an amorphous material is governed by a localized shear band in a depth of several tens of nanometers. At high temperatures, the deformation becomes more homogenous because each volume element contributes to the deformation. Thus, the addition of oxide particles changes the homogenized deformation by the interaction with the matrix [123]. Consequently, the homogeneity of the particle distribution is believed to be more important in an amorphous alloy matrix composite. Further studies are needed to gain a better understanding of the exact microscopic interaction mechanisms between the particles and the matrix.

3.2.2. Crystalline matrix

Mg-Al alloys such as AM60 and AZ91 are presently the most prevalent magnesium alloys utilized in the automotive industry. They are also the most widely studied matrix for magnesium-based composites. Other magnesium materials, such as pure magnesium, Mg-Li alloy, and Mg-Ag-Re (QE22) alloys, have also been employed as a matrix material, although less frequently.

Grain refinement is a key principle in the strengthening of engineering alloys. The yield strength of a material normally varies proportionally with the reciprocal square root of its grain size, as depicted by the well-known Hall-Petch equation: $\sigma = \sigma_0 + Kd^{-1/2}$, where σ is the yield stress, σ_0 the yield stress of a single crystal, K a constant, and d the grain size. The value of K generally depends on the number of slip systems and is greater for HCP metals than for FCC and BCC metals [124]. Consequently, HCP metals exhibit a higher strength sensitivity to the grain size. A study on the effects of grain size on the strength of magnesium alloy AZ91 and aluminum alloy 5083 indicates that the yield stress of the Mg alloy is lower than that of the Al alloy with a grain size larger than 2.2 μm. However, the yield stress of the Mg alloy becomes greater when the grain size is smaller than 2.2 μm, as shown in Fig. 8 [125].

In a metal matrix composite, the secondary reinforcing phase can significantly influence the grain size of the matrix. Some studies [126–128] reported significant grain refinement in the matrix in the SiC particle reinforced AZ91 magnesium alloys, as shown in Fig. 9

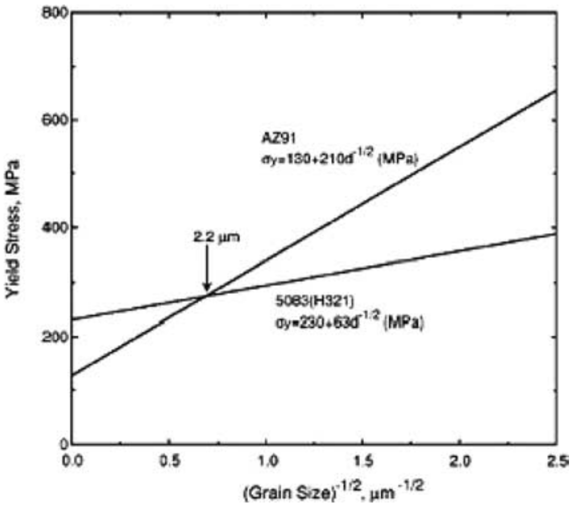

Figure 8 The yield strength and grain size of AZ91 and 5083 alloys [125].

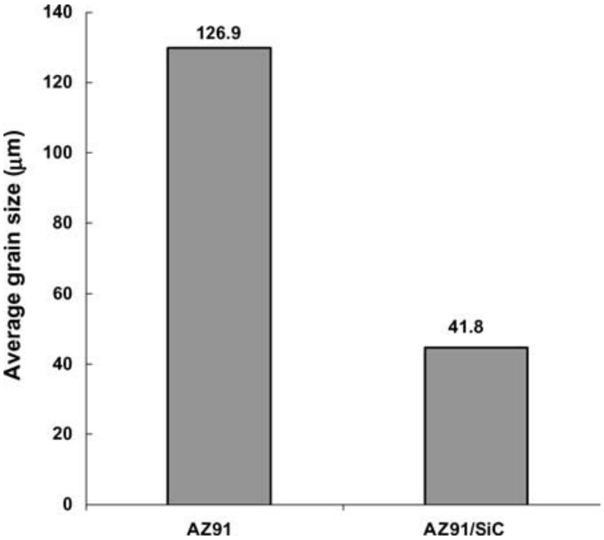

Figure 9 The grain size of AZ91 and SiC/AZ91 composite [126].

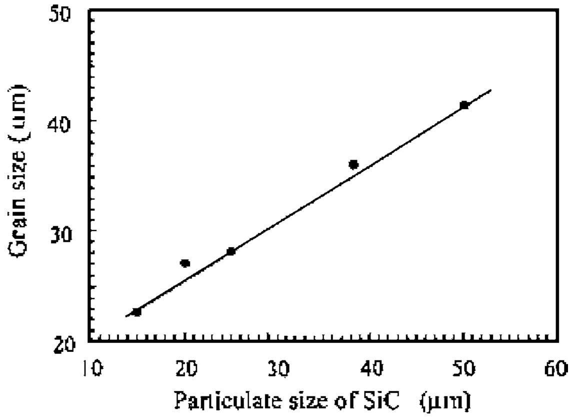

Figure 10 SiC particle size and magnesium composite matrix grain size [128].

[126]. The grain refinement effect has been attributed to heterogeneous nucleation of the primary magnesium phase on SiC particles and the restricted growth of magnesium crystals caused by the presence of rigid SiC particles. The heterogeneous nucleation mechanism is supported by the fact that the smaller the SiC particles, the finer the grains of the composite matrix, as shown in Fig. 10 [128]. This is because more nucleation sites are provided.

On the other hand, it has also been reported that the primary magnesium phase could not heterogeneously nucleate on the surface of SiC particles, and thus the SiC particles would not refine the matrix grains [129]. Another study on the microstructures of a metal matrix composite further suggests that the matrix grains may coarsen if no heterogeneous nucleation exists [130]. That particular study reports that, whereas the liquid flow is an important condition to form fine grain microstructures in castings, the reinforcement hinders the convection of the liquid metal.

The conditions and mechanisms for heterogeneous nucleation of magnesium on the SiC particles were investigated in a recent research paper [131]. The solidification microstructure of a 15 vol% SiC/Mg-Al-Zn composite that was investigated in the study showed that a majority of the SiC particles were pushed by the primary magnesium phases and segregated at the grain boundaries. At the same time, about 3% SiC particles were entrapped in the magnesium grain. The formation of the microstructure was discussed from the perspective of cooling, geometrical similarity, and surface defects of the SiC particles and the magnesium matrix.

Firstly, SiC particles have a lower thermal conductivity and heat diffusivity than the magnesium melt. During the cooling process the temperature of SiC particles is somewhat higher than that of the surrounding magnesium melt. The SiC particles with the higher temperature would heat up the surrounding magnesium melt, and thus retard its solidification. In such a situation, primary magnesium could not nucleate at the SiC particle surfaces, and the latter would be pushed by the solidifying primary magnesium [131].

Secondly, both magnesium and SiC have a hexagonal lattice and exhibit a close match in lattice parameters in certain orientations. The lattice parameters are $a = 0.32094$ nm, $c = 0.52107$ nm for magnesium, and $a = 0.307$ nm, $c = 0.1508$ nm for SiC, respectively [132]. The disregistry between the magnesium phase and the SiC particle is 2.3% in the $(10\underline{1}0)_{\mathrm{Mg}}//(0001)_{\mathrm{SiC}}$ crystallographic orientation. Because heterogeneous nucleation would normally occur when the disregistry is less than 5%, the primary magnesium phase can nucleate on the SiC particle surfaces in this orientation. However, since SiC particles are generally polycrystalline, the exposed atomic planes may not always be able to serve as substrates for the heterogeneous nucleation of the primary magnesium phase. This also helps to explain why most of SiC particles were pushed by the primary phase into the last freezing zone in the magnesium composite [131].

In the magnesium matrix composite, eutectic phases also form during the solidification process. These eutectic phases are able to wet the SiC particles and heterogeneously nucleate on the SiC substrate. The defects in the SiC particles—such as stacking faults, dislocations, and pits or grooves—would also act as favourable sites for heterogeneous nucleation [131].

In comparison to the ceramic particle reinforced magnesium matrix composite, the elemental metallic powder reinforced magnesium matrix composites are characterized by a relatively uniform distribution of the metal particles. This enhanced homogeneity of the metal powder distribution results not only from the good wettability occurring between the reinforcing particles and the matrix, but also from the various engineering controls of the process such as a thin layered (sandwich) arrangement of raw materials when loaded in the crucible before melting, and the judicious selection of the stirring parameters [109–112].

3.3. Interfacial characteristics

The interface between the matrix and the secondary reinforcing phase plays a crucial role in the performance of composite materials. The key features of the interface are the chemical reactions and the strength of bonding.

3.3.1. Interfacial chemistry

Interfacial reactions in the magnesium matrix composite are predominantly determined by the composition of the matrix and the reinforcement materials. A comparison study of the interfacial reactions in pure magnesium and AZ91 alloy based composites reinforced with SiC particles has evinced the effect of a matrix alloy composition on the particle/matrix interfacial phenomena [133]. In the pure Mg based composite, SiC particles were stable and no reaction products were found at the interface. On the other hand, in AZ91 based composites the particle/matrix interfacial reactions were confirmed by the presence of a Mg_2Si phase. The involved reactions were $4Al + 3SiC = Al_4C_3 + 3Si$ and then $Si + 2Mg = Mg_2Si$. Increasing the Al content in the alloy promoted the first reaction while increasing the Si content reduced the reaction [133]. Porosity also influences the interfacial reactions between the matrix and the reinforcing phases. For example, the reaction

$3Mg + Al_2O_3 = 3MgO + 2Al$ in an AZ 91 alloy reinforced with 20 vol% Al_2O_3 short fibers is not only influenced by the volume fraction of the fibers; but also by the fiber microstructure and porosity [134]. Porosity might have increased the surface area and thus promoted the reaction.

The surface cleanliness of the raw materials is another factor affecting the interface chemical reactions. The reactions occurring in a squeeze cast AZ91 matrix composite with SiC fibers are $2Mg_{(l)} + O_{2(g)} = 2MgO_{(s)}$ and/or $Mg_{(l)} + O = MgO_{(s)}$, where O_2 is produced from the absorption on the SiC fiber surface [35]. The parameters of the casting process such as melting temperature and holding time have also been found to change the interface reactions in the magnesium matrix composite [135]. Higher temperature normally accelerates interfacial reactions, as governed by the Arrhenius law. The degree of interfacial reactions can also change the microstructure [135]. To obtain composite materials with the desired microstructure and properties, the interfacial reaction should be controlled through selecting an appropriate matrix alloy, conducting an appropriate surface treatment of the reinforcement, and correctly controlling the process parameters.

The interface reactions in the SiC/QE22 composite are of particular interest. QE22 alloy has a good strength and creep resistance at elevated temperatures due to the formation of high melting point precipitates. However, unlike other magnesium matrix composites, the addition of SiC particles has been found to reduce the mechanical properties of the material [136–138]. This phenomenon has been attributed to the SiC and QE22 matrix interface features. The QE22 alloy has several types of precipitates such as Mg_3 (Ag,Nd), round α-Nd phase, rod-like Zr-Ni phase, MgO, and Mg-Nd GP zone. The very fine Mg-Nd GP zones play a key role in the precipitation hardening of the alloy. In the SiC/QE22 composite, all of the above-mentioned precipitates, except for the coherent GP zone, were observed. Furthermore, a pronounced precipitation of Nd-rich phase occurs at the SiC/matrix interfaces. Thus, the lack of GP zones in the composite may be attributed to the matrix depletion of Nd due to the precipitation of Nd-rich phases at the interfaces. This enhanced precipitation of Nd-rich phases at the SiC/matrix interfaces can adversely affect the creep behavior. Matrix depletion caused by the interfacial precipitation can produce inhomogeneous distribution of precipitates and a deficiency in the matrix precipitate microstructure, leading to composite weakening. Additionally, interfacial sliding may be another creep mechanism acting in the composite. As a result of interfacial sliding, many cavities can occur at the interfaces, giving rise to the macroscopic cracks and debonding of the matrix/SiC interfaces [137, 139].

Interface reactions also take place in the elemental metallic powder reinforced magnesium matrix composite. In the composites with Cu and Ni reinforcement, reactions occur at the Mg/Cu or Mg/Ni interfaces forming Mg_2Cu and Mg_2Ni intermetallics, respectively, leading to a reduction in the particle size of Cu and Ni powders. However, because of a higher wettability and compatibility, the matrix/reinforcement interface is free from such defects as debonding or microvoid [109–112].

3.4. Porosity and inclusions

Porosity and inclusions are detrimental to the mechanical properties of magnesium matrix composites. The existence of porosity in magnesium matrix composite can remarkably reduce the creep resistance of the materials [140]. At low porosity levels, the degree of damage to the mechanical properties caused by the porosity is the sum of that from each pore, in which case the tensile strength is found to be a linear function of the porosity density. This occurs because the distribution of the stress fields around each pore does not overlap [141]. When the porosity volume fraction reaches a certain level, the stress fields of the pores overlap with each other, and the tensile strength of the material is no longer linearly affected by the porosity [142].

The porosity in a composite may arise from a number of sources. These include: the entrapment of gases during mixing, hydrogen evolution, and the shrinkage of the alloy during its solidification. The entrapment of gases depends mainly on the processing method, such as mixing and pouring. Holding time and stirring speed as well as the size and position of the impeller can also significantly affect the porosity formation [143]. The hydrogen production is mainly the result of the reactions between the absorbed H_2O and Mg melt. Usually some water vapour is absorbed on the surface of the added fibers or particles. Once entering the melt, the water vapour can react strongly with Mg, forming MgO and releasing H_2. Although gas porosity in casting is much more sensitive to the volume fraction of the inclusions than to the amount of dissolved H_2 [144], the recommended practice is to thoroughly dry the raw materials before adding them to the magnesium melt for the purposes of both safety and quality control. In the magnesium matrix composite, the presence of relatively large amounts of fibers/particles may impose a serious porosity problem if the reinforcement is not properly degased prior to its addition to the melt. This is especially true for finer particles due to the larger number of specific surface areas involved.

Inclusion is another major microstructural defect that is deleterious to material properties. The inclusions normally encountered in magnesium alloys include magnesium oxide and nitride, Na, Ca, Mg, K-based chlorides, magnesium-based sulfide, fluoride, and sulfate [145]. The processing of some metal matrix composites requires melt stirring. Some of the conventional methods for removing inclusions, such as flux refining and gas sparging and settling, may no longer be suitable for processing the metal matrix composites. Due to the high oxidation potential of the magnesium and the limitations of the oxidation protection, the inclusion content in cast magnesium alloys is usually 10–20 times higher than that in aluminum alloys [145]. In addition to the inclusion density, the inclusion size is also important in determining the mechanical properties of the composite materials. It was observed [36] that larger inclusions are normally more harmful to the material's properties.

Thus, care must be taken to prevent the formation of inclusions in the magnesium matrix composites.

4. The mechanical properties of magnesium matrix composites

Improving such mechanical properties as tensile strength, Young's modulus, creep resistance, and fatigue resistance, is usually the major attraction of composite materials. Nevertheless, the improvement in strength and rigidity resulting from the addition of the reinforcing phases to the matrix is normally at the cost of some other properties. Thus, the total benefit of the improvements in certain mechanical properties of the composite materials has to be weighed against the reduction in other properties and the additional cost.

4.1. Tensile strength and elastic modulus

With the addition of a reinforcement phase, both tensile strength and Young's modulus of the magnesium alloys are, in general, increased. Within a certain range, both the yield strength and the elastic modulus of the magnesium matrix composite increase linearly with the increase in the volume fraction of the composite reinforcement [129]. Hybrid reinforcements, which involve more than one kind of particles or whiskers, have an even greater strengthening effect than a single reinforcement [146].

Particle strengthening, work hardening, load transfer, and grain refinement of the matrix alloy by the reinforcement phases are the key strengthening mechanisms in magnesium composites. The dispersion of fine and hard particles in the matrix drastically blocks the motion of dislocations and thus strengthens the material. Work hardening takes place when the composite is strained. The strain mismatch between the matrix and the reinforcement usually generates a higher density of dislocation in the matrix around the reinforcement, thus strengthening the material. Load transfer is a very important strengthening mechanism, especially for the fiber-reinforced composites. If the bonding between the matrix and the reinforcement is strong enough, the applied stress can be transferred from the soft matrix to the hard fiber/particle phases. Due to the much higher strength of the secondary hard phases, the relatively soft matrix is protected. As discussed earlier, magnesium strength is highly sensitive to its grain size. Thus, grain refinement contributes to the great strength at room temperature for both Mg alloys and the Mg matrix composite.

Under an applied load, the stress built up in the magnesium composite can be relaxed by the cracking of the reinforcement [147]. In a fractured SiC/Mg composite, the SiC particulate fracture was observed to be the predominant form of localized damage under tensile loading [148]. The fracture of the composite was dominated by the cracking of the reinforcing particulates that were present in the magnesium alloy metal matrix. Final fracture occurred as a result of crack propagation through the alloy matrix between particulate clusters. The size of the ceramic reinforcing particles is important here. In an investigation of the influence of SiC size and volume fraction on the AZ91D mechanical properties, the addition of 15 μm SiC particles was found to increase fatigue resistance while the addition of 52 μm SiC particles reduced fatigue resistance due to the high brittleness of SiC [149]. This difference depends on the extent to which the matrix and reinforcement can deform cooperatively. A finer secondary phase can produce a more cooperated deformation within the matrix. In a 10 vol%-SiC/MB2 magnesium matrix composite, it has been found that after a high strain deformation, the matrix around small SiC particles (2 μm) had a fine grain microstructure and a strong bonding with these particles. In contrast, cavities were produced around the big SiC particles (5 μm). This is because the stress built up around the small particles can be more easily relaxed by cooperative deformation during the tensile test [150]. Another advantage of the finer secondary phase is that it offers more heterogeneous nucleation sites for the solidification of the matrix magnesium alloy, which leads to a finer matrix grain size. Research results have shown that the strength of a magnesium matrix composite does not monotonically increase with decreasing particle size, as the particle strengthening relies greatly on the stress built up around the particles by lattice distortion.

The stress built up in the metal matrix composite may also be relaxed by debonding along the reinforcement/matrix interfaces [151]. In particular, when the matrix and reinforcement interface is a relatively weak region of the material, the composite may fail prematurely at the interfaces. In this case, the addition of any secondary hard phase actually can reduce the material's strength. Research focusing on an AZ91 magnesium alloy reinforced with 15 vol% SiC, TiB_2, TiC, TiN, AlN and Al_2O_3 has detected such an occurrence with AlN reinforcement, as shown in Fig. 11, where a comparison is made between the ultimate tensile strengths and hardness of these composites [152]. This research also shows that the decrease in the tensile strength of AZ91, which has been attributed to excessive chemical reactions, different powder size distribution and wetting conditions, was caused by the addition of AlN.

The relative strengths of the matrix and interfaces can also be temperature dependent. The tensile behaviors

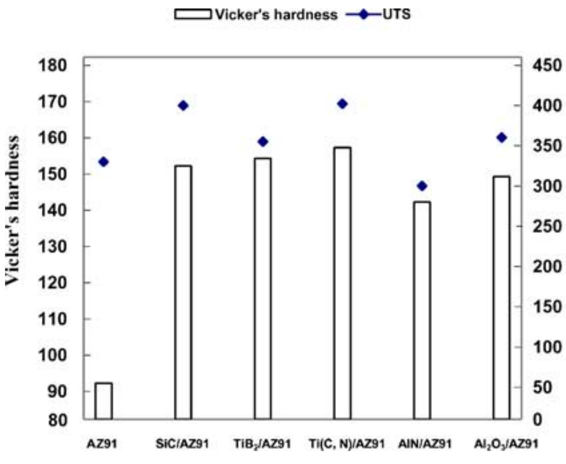

Figure 11 Strength and hardness of AZ91 reinforced with different particles [152].

of an AZ91 matrix composite reinforced with randomly oriented short carbon fibers showed that the failure mode of the composite changed from fiber/MgO interface dominated failure to failure at the MgO/matrix interface when the testing temperature is increased from room temperature to 200°C or higher [153].

In contrast to the composites reinforced with brittle ceramic hard phases, premature fractures of the reinforcement are not encountered in the elemental metallic powder- reinforced magnesium matrix composites. The high compatibility between the thermal and the mechanical properties and the good wettability between the matrix alloys and the secondary phase apparently leads to a highly cooperated deformation and a strong interface between the composite matrix and its reinforcement. As a result, the crack initiation in such composites, which originates from the matrix rather than from the secondary phase and interface [109–112], is noticeably delayed. The high toughness of the material is particularly advantageous when the applied load is impacted and large. Therefore, the introduction of strong and stiff high melting point metallic reinforcements in both pure magnesium and commercial grade magnesium alloys will significantly improve the overall mechanical properties, especially the strength and specific strength of the material, as shown in Table IV [109–112].

Crack initiation and propagation in the reinforcement-free areas in the matrix are often observed in composite materials [90]. In this case, the matrix plays a more important role in determining the deformation and fracture behavior of the composites. A comparison between pure magnesium and AZ91 reinforced with 10–20 vol% of Al_2O_3 fibers has evinced that the AZ91 matrix composite always exhibited a higher tensile yield strength, but the degree of strength improvement was greater in a pure magnesium composite. This phenomenon has been ascribed to the higher mechanical properties of AZ91 in comparison to pure magnesium [154]. Accordingly, processes that modify the matrix microstructure can affect the mechanical properties of the bulk composite. Indications are that the thermal cycling of a SiCw/ZK60 magnesium matrix composite may first recover and age the matrix and then degrade the tensile properties of the material [155].

TABLE IV Mechanical properties of various Mg based materials [109–112]

Materials	0.2%YS (MPa)	UTS (MPa)	Ductility (%)	Specific YS	Specific UTS
Mg	100	258	7.7	58	148
Mg/2%Cu	281	335	2.5	148	177
Mg/4%Cu	355	386	1.5	170	184
Mg/7%Cu	–	433	1.0	–	195
Mg/2%Ni	337	370	4.8	177	194
Mg/3%Ni	420	463	1.4	203	224
Mg/6%Ni	–	313	0.7	–	131
Mg/2%Ti	163	248	11.1	90	137
Mg/4%Ti	154	239	9.5	81	126
AZ91	263	358	7.2	145	197
AZ91/4%Cu	299	382	6.2	142	181
Mg/30%SiCp	229	258	2	105	118
AZ91D/10%SiC$_p$	135	152	0.8	69	77
AZ91D/15%SiC$_p$	257	289	0.7	126	142

4.2. Creep behavior

A magnesium alloy has a relatively low creep resistance, especially at high temperatures. The high creep rate of magnesium alloys usually results from grain boundary slide and dislocation slip at both basal and non-basal planes of the magnesium [156]. Therefore, obstructing the grain boundary slide and dislocation slip by precipitating hard phases at the grain boundary, or within the grain, is a key approach to developing high temperature magnesium alloys.

The addition of a hard secondary phase into a magnesium matrix to form composites is an effective way to improve the creep resistance of magnesium alloys [134, 138, 157–159]. The creep life of AZ91 and QE22 matrix composites reinforced with Al_2O_3 short fibres is an order of magnitude longer than the unreinforced alloy at low stresses [138, 159]. Like unreinforced magnesium alloys, the creep deformation of a magnesium matrix composite can also be depicted by the equation of $\varepsilon = K\sigma^{\mathrm{Napp}}\exp(-Q_{\mathrm{app}}/RT)$, where ε is the minimum strain rate, σ the true stress, K a creep constant, N_{app} the apparent stress exponent, Q_{app} the apparent activation energy, T the absolute temperature, and R the gas constant [160]. Both the apparent stress exponent and the apparent activation energy are significantly affected by the level of stress applied to the composite. At low stresses (<30 MPa), the apparent stress exponent and apparent activation energy of pure Mg reinforced with 30 vol%Y_2O_3 particles are 2 and 48 kJ mol^{-1} respectively, while at high stresses (>34 MPa), these parameters become much higher ($n = 9$–15, $Q = 230$–325 kJ mol^{-1}) [160]. Compared with that of unreinforced magnesium alloys, the minimum creep rate of a magnesium matrix composite has a different stress dependency. The values of N_{app} for monolithic AZ91 and QE22 alloys decrease slightly with the decreasing applied stress while N_{app} increases with the decreasing applied stress for the AZ91 and QE22 alloys reinforced with 20 vol% Al_2O_3 fibres [159].

The creep fracture of a composite material is a complicated process. In short-fiber reinforced composites, the fracture modes include fiber failure, debonding between the fiber and the matrix, fiber sliding and pulling out, as well as cavitation and matrix cracking [161]. The damage to the fibers caused during material sample preparation has been found to be significantly deleterious to the creep resistance of AZ91 magnesium matrix composites reinforced with alumina short fibers [162].

Creep also changes the microstructure of a magnesium matrix composite. After creep in an $Al_2O_{3(f)}$/AZ91 composite, there is a fine continuous precipitation and a coarsening of the $Mg_{17}Al_{12}$ intermetallic phase due to the Al enrichment in the matrix near the alumina fibres. Similarly, in a SiC/QE22 composite, the SiC particles act as nucleation centers in the precipitation process, promoting the precipitation of the Al_2Nd, $Mg(Ag)_{12}Nd$, and Mg_3Ag phases [138].

Load transfer is a key mechanism for the enhanced creep resistance of a magnesium matrix composite. It is generally accepted that the creep deformation within a metal matrix composite is controlled by the flow in the matrix material [158]. Creep occurs in a matrix alloy through the generation and motion of dislocations, grain boundary slide, and microstructural damage at high stress levels through debonding at the reinforcement—matrix interface and fracturing of the individual reinforcements. Load transfer from the matrix to the reinforcement, usually referred to as indirect strengthening, improves the creep resistance by redistributing the stress in the material and reducing the effective stress acting on the matrix. The direct strengthening in a magnesium matrix composite is based on the interaction between the reinforcement and the dislocations in the matrix, including the Orowan stress associated with a bowing between the particles [163], the back stress associated with the local climb of dislocation over the obstacles [164, 165], the detachment stress needed to separate a dislocation from an attractive particle [166], and the stress associated with the dissociation of the lattice dislocation into interfacial dislocation [167]. Some experimental results indicate that the load transfer contributes more to the improved creep resistance of magnesium matrix composite than to the dislocation-particle interactions. The $Al_2O_{3(f)}$/AZ91 and $Al_2O_{3(f)}$/QE22 composites showed a creep performance which was superior to their unreinforced counterparts at low stress levels. Nevertheless, the level of this improvement decreased as the amount of applied stress increased, and at high stress levels there was little or no effect on the reinforcement to the lifetime. At stresses higher than 100 MPa, the creep of the composites seemed to be similar to that of unreinforced matrix alloy, regardless of the experimental temperature [159]. This suggests the dominance of the load transfer, interface, and control of the void/crack formation in the creep strengthening of the magnesium matrix composite, as this is the most probable cause of the sudden weakening of composites. An *in-situ* observation of microcracks initiating at the fiber-matrix interfaces in an AZ91 matrix composite at high stress intensity factor levels [41] is consistent with the sudden weakening of the $Al_2O_{3(f)}$/AZ91 and $Al_2O_{3(f)}$/QE22 composite materials at stresses higher than 100 MPa. In addition, a microstructural study of the AZ91 and QE22 alloys reinforced with 20 vol% Al_2O_3 fibers reveals no substantial changes in the matrix microstructure due to the presence of the reinforcement [159]. This implies the importance of load transfer in creep strengthening magnesium matrix composites where the indirect strengthening results from the interaction of the dislocations and the reinforcement which inevitably produces different microstructural characteristics.

The most critical determinant of a composite material creep life is the chemistry of the reinforcing and matrix materials because a poor combination of reinforcement and matrix can lead to a weakening of the matrix. For example, the addition of SiC particles into QE22 alloy has been found to result in a significant reduction rather than an improvement in its creep resistance [138]. As discussed earlier, this abnormality has been examined and ascribed to the alteration of the matrix microstructure caused by interface reactions. Therefore, the right combination of composite matrix and the corresponding reinforcement is of paramount importance. The shape of the reinforcement is also important. The materials reinforced with fibers and particles respectively display different creep performances even using the same matrix. The addition of SiC particles into AZ91 alloy also improves its creep resistance. However, compared with the improvement in the creep resistance caused by the addition of Al_2O_3 short fibers, the SiC particles merely produce a moderate improvement [138]. This difference in the reinforcing effects of the fibers and the particles is due to the fact that the load transfer is more effective in a fiber-reinforced material. However, the particle-reinforced magnesium matrix composite has a lower material and fabrication cost as well as isotropic microstructures and properties. The thermal stability and distribution of the reinforcing materials are also important factors that affect the creep behavior of magnesium matrix composite. An AZ61-Si-P alloy reinforced with thermally stable Mg_2Si particles has shown a much higher creep resistance than that of an AZ91 alloy that is strengthened by thermally unstable $Mg_{17}Al_{12}$ particles, even though the volume fraction of $Mg_{17}Al_{12}$ particles in the alloy is higher than that of Mg_2Si in the composite [168]. Other features of the reinforcement, such as size, volume fraction [169], shape of the reinforcement [170], and the interfacial bond of the reinforcement with the matrix [171] have also been shown to be critical to the creep performance of a composite material. However, the studies of the creep behavior of magnesium matrix composites are not as extensive as with aluminum matrix composites.

4.3. Ductility

The hard secondary phases in magnesium matrix composites have a two-fold effect. First, when these phases are present in magnesium matrix composites they can reduce their ductility by preventing plastic deformation. On the other hand, the particles can produce a grain refining effect that improves ductility. The net effect of the hard particles is, in general, to reduce ductility. This happens in both particle-reinforced [128] and fiber-reinforced composites [159]. In contrast with the ceramic reinforced magnesium matrix composites, the elemental metallic powder-reinforced magnesium matrix composites show a much better ductility because of the reduced possibility of the breaking of the particles and interface, as shown in Table IV [109–112].

The reduced ductility in composites with a hard secondary phase is also evident in the interactions between the reinforcement and the dislocations. It is obvious that the resistance to the dislocation motion of the hard particles reduces the ductility of the composite materials. A research study examining the superplastic behavior of a fine-grained ($\sim$2 μm) WE43 magnesium alloy containing spherical precipitates ($\sim$200 nm) within grains revealed a superplasticity with an elongation-to-failure of over 1000 percent at 400°C. Dislocations were observed to interact with the particles within the grains. Data analysis based on the constitutive equation for superplastic flow demonstrated that the normalized strain rate for a particle-strengthened WE43 alloy was about 50 times lower than that of the same WE43 magnesium alloy without the hard precipitates. It would seem that the existence of intragranular particles diminishes the superplastic flow [172]. However, when compared to the breaking of large brittle particles and weak interface, the influence of very fine precipitates on the ductility may be less of a factor in a magnesium matrix composite.

However, the high brittleness of secondary particles does not necessarily mean that the composite with hard phases will always have a low ductility. In fact, grain refinement by the secondary phase can result in superplasticity in a magnesium matrix composite, even with highly brittle secondary phases. For example, a ZK60A magnesium alloy reinforced with 17 vol% of SiC particles showed a total creep elongation of 200–350% at the temperature range of 350–500°C [173]. Another study has revealed a low temperature superplasticity with an elongation to failure of 300% for the same material at 175–202°C due to the refined grains (about 1.7 μm) [174]. This low-temperature superplasticity is of significance for practical industrial applications of the magnesium matrix composite.

The secondary particles not only refine grains during the solidification process of the composite materials by providing heterogeneous nucleation sites, but also they retard the grain growth during mechanical processing and deformation processes at high temperatures. The composite studied in reference 173 was initially extruded to obtain a fine microstructure in the matrix with an average grain size of around 0.5 μm. At elevated temperatures, the grains in alloys tend to coarsen. However, with the presence of fine hard particles, the growth of matrix alloy grains is obstructed, and the fine structure is retained at high temperatures. There is a large amount of grain glide in such a fine microstructure, which contributes to its superplasticity. The role of the hard particles (also refined by the extrusion) in the composite is simply to impede grain growth and help to maintain the superplasticity. A uniform distribution of the hard particles in the matrix is a precondition for such a function. Although grain refinement can improve the strength and ductility at room temperature, it may accelerate creep deformation at elevated temperatures. Fine hard particles may improve the strength of magnesium alloys and also stabilize the grains at high temperatures, allowing for grain boundary slide. Compared to aluminum matrix composites, it may be more difficult to attain high creep resistance for magnesium matrix composites because of the higher grain boundary diffusion rate in magnesium. However, it is still possible that a combination of excellent strength, ductility, and creep resistance can be realized, along with the addition of secondary hard phases.

5. Summary

A significant amount of time and effort has been devoted to the research and development of magnesium matrix composites in recent years. Various techniques have been developed and applied to the processing of magnesium matrix composites, such as stir casting, pressure and pressureless infiltration, powder metallurgy, gas injection and *in-situ* formation of reinforcement in the matrix. Key factors affecting the performance of the magnesium composites are the matrix composition; the chemistry; the shape, size, and distribution of the reinforcements; and the bonding strength at the reinforcement/matrix interface. High strength in the composites is normally achieved at the cost of compromised ductility. Nevertheless, grain refinement is an effective way of improving ductility and strength at ambient temperatures. However, caution has to be taken in using fine-grained materials at elevated temperatures because creep resistance can be adversely affected by the fine grain size. The acceptance of the magnesium matrix composites as engineering materials depends not only on the performance advantages of the materials, but also on the development of cost-effective processing technologies for these materials.

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