



Recent progress in Mg–Li matrix composites

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Abstract: Mg–Li matrix composites are one of the ideal structural materials in the fields of aerospace and military due to their high specific strength and stiffness, good damping and wear resistance, and small thermal expansion coefficient. The preparation technologies of Mg–Li matrix composites including powder metallurgy, pressure infiltration, stir-casting, foil metallurgy, and in-situ synthesis were introduced, and their advantages and disadvantages were compared. The common matrix alloys and reinforcements for Mg–Li matrix composites as well as the structure and performance of typical composites were mainly summarized. Then the interface chemistry between matrix and reinforcement was briefly reviewed. Finally, some problems existing at present and the possible solutions were discussed.

Key words: Mg–Li alloy; composites; preparation; reinforcement; interface chemistry

1 Introduction

Mg–Li alloy, known as the lightest alloy with density between 1.25 and 1.65 g/cm³, is one of the most ideal and promising metal structural materials in the fields of aerospace, military, 3C industry, and medical equipment because of its tempting advantages, such as high specific strength and stiffness, good damping and formability, and excellent electromagnetic shielding performance [1–3]. With the worldwide shortage of energy, the demands for lightweight materials and devices in many industries are urgent. Since the density of Mg–Li alloys is only about 1/2 and 3/4 that of Al and Mg alloys, and much less than that of new aeronautic Al–Li alloys [4], the application of Mg–Li alloys will be a realistic approach for reducing the mass of devices, lowering the manufacturing cost, and saving energy, especially in the aerospace field. However, low strength and worse creep resistance make Mg–Li alloys lack competitive advantage compared with Al alloys and other Mg alloys [5–7]. Fortunately, some explorations have discovered that composite approach could diminish these weaknesses to meet the demands of rapidly developed science and technology for lightweight materials. This technology could not

only enhance the strength and stiffness by stress transfer between matrix and reinforcement, but also improve the damping capability and abrasive resistance [8].

Nevertheless, Mg–Li matrix composites are still at exploratory stage and without mature manufacture technologies, due to many hurdles such as severe interface reaction and chemical incompatibility, high manufacturing cost, and undesirable performance. Therefore, Mg–Li matrix composites still need tremendous researches. In this work, the research progress of Mg–Li matrix composites was summarized for subsequent researches through the following aspects. First, preparation methods as a prerequisite for studying Mg–Li matrix composites were briefly introduced. In the next two sections, matrix alloys and reinforcements as the research subjects were mainly reviewed, and structure and mechanical properties of typical Mg–Li matrix composites were shown. Then, the fifth section dealt with the interface chemistry in Mg–Li matrix composites as the research emphasis for improving interface bonding and mechanical properties of composites. Finally, the prospect section analyzed and discussed the most important problems need to be solved in Mg–Li matrix composites and the possible ways to solve these problems.

2 Preparation of Mg–Li matrix composites

Mg–Li alloy is a promising matrix of composite materials due to its low density and high specific strength. However, the active chemical character of Mg–Li alloy brings a certain difficulty in preparation of Mg–Li alloys and their composites. At present, the study on Mg–Li matrix composites is mainly inspired by other composites, and the preparation methods include powder metallurgy processing, pressure infiltration processing, stir-casting processing, foil metallurgy processing, and in-situ synthesis processing. Table 1 summarizes the advantages and disadvantages of these preparation methods for Mg–Li matrix composites.

2.1 Powder metallurgy processing

Powder metallurgy processing is a common technique for preparing composites, and its process schematic diagram is shown in Fig. 1(a). It is characterized by undemanding control over the type of matrix alloy, the type of reinforcement, and the volume fraction. The reinforcements can be distributed evenly in the matrix by powder mixing process. However, the equipment is complicated, the cost is high, and the hot-pressed composites must be extruded or rolled before use. This processing has successful application in fields of Al-matrix and Mg-matrix composites [9–11]. WHALEN et al [12] tried to prepare Mg–14Li–B composites by powder metallurgy processing with the following procedures: LA141 alloy bar was machined into chips (<25 μm) under dry argon atmosphere and boron powder (<75 μm) was used as reinforced phase. The Mg–Li chips and the boron powders were uniformly mixed, followed by cold-pressing and hot-pressing sintering, and then the compact was extruded at 450 $^{\circ}\text{C}$ with an extrusion ratio of 16:1. In order to obtain uniform microstructure, the extruded rod needs to be rolled into thin sheet, cut into small particles, then

pressed and extruded repeatedly. Though WHALEN et al [12] implied the applicability of metallurgy processing in Mg–Li composites, there are few researches on Mg–Li matrix composites. This is because that the entire preparation process must be executed under protective atmosphere owing to the active chemical properties of Mg–Li alloy. In addition, powder metallurgy processing cannot prepare complex-shaped parts, and ultrafine alloy powder is harmful to human body and has safety hazards.

2.2 Pressure infiltration processing

Pressure infiltration processing is one of the main preparation methods for Mg–Li matrix composites [13,14], and its process diagram is shown in Fig. 1(b). The specific process includes preparation of precast block and pressure infiltration. The reinforcement is dispersed evenly and shaped by compression molding, followed by drying and sintering, which endows the precast block with a certain compressive strength. The matrix alloy is melted and overheated to the proper temperature, and then poured into mold with precast block. At the same time, the pressure plug or inert gas is used to press the alloy melt into the pores distributed in the precast block with constant pressure for a period of time. The composites prepared by this process have excellent mechanical properties and stable quality.

However, there exist several problems to be solved [15]. First, binders (3–5 wt.%) need to be added into precast block of fiber or whisker for maintaining the block shape during infiltration process. SiO_2 is a common binder, but it easily reacts with Mg–Li matrix to form Mg_2Si , which leads to a decrease in strength and infiltrability of precast block. Second, although high temperature can improve infiltrability, the reinforcement is easily eroded by hyperthermal alloy melt, resulting in severe interface reaction and poor interface bonding. Third, the thickness of precast block is restricted to ensure complete infiltration. Finally, the combination of

Table 1 Advantages and disadvantages of preparation methods for Mg–Li matrix composites

Preparation method	Reinforcement type	Advantage	Disadvantage
Powder metallurgy processing	Particle	Mature technology; the volume fraction of reinforcement can be adjusted widely	Complicated process; high cost
Pressure infiltration processing	Particle, fiber, whisker	Wide range of application; excellent mechanical property; stable quality	Severe interface reaction and reinforcement erosion; limited size and shape
Stir-casting Processing	Particle, fiber, whisker	Good processing suitability; better mechanical property	Unevenly distributed reinforcement; severe interface reaction
Foil metallurgy processing	Particle, whisker	No interface reaction; preparation at low temperature	Complicated process; high cost; poor interface combination
In situ synthesis processing	Particle	No interface reaction; excellent interface combination; evenly distributed fine reinforcement	Limited reinforcement species

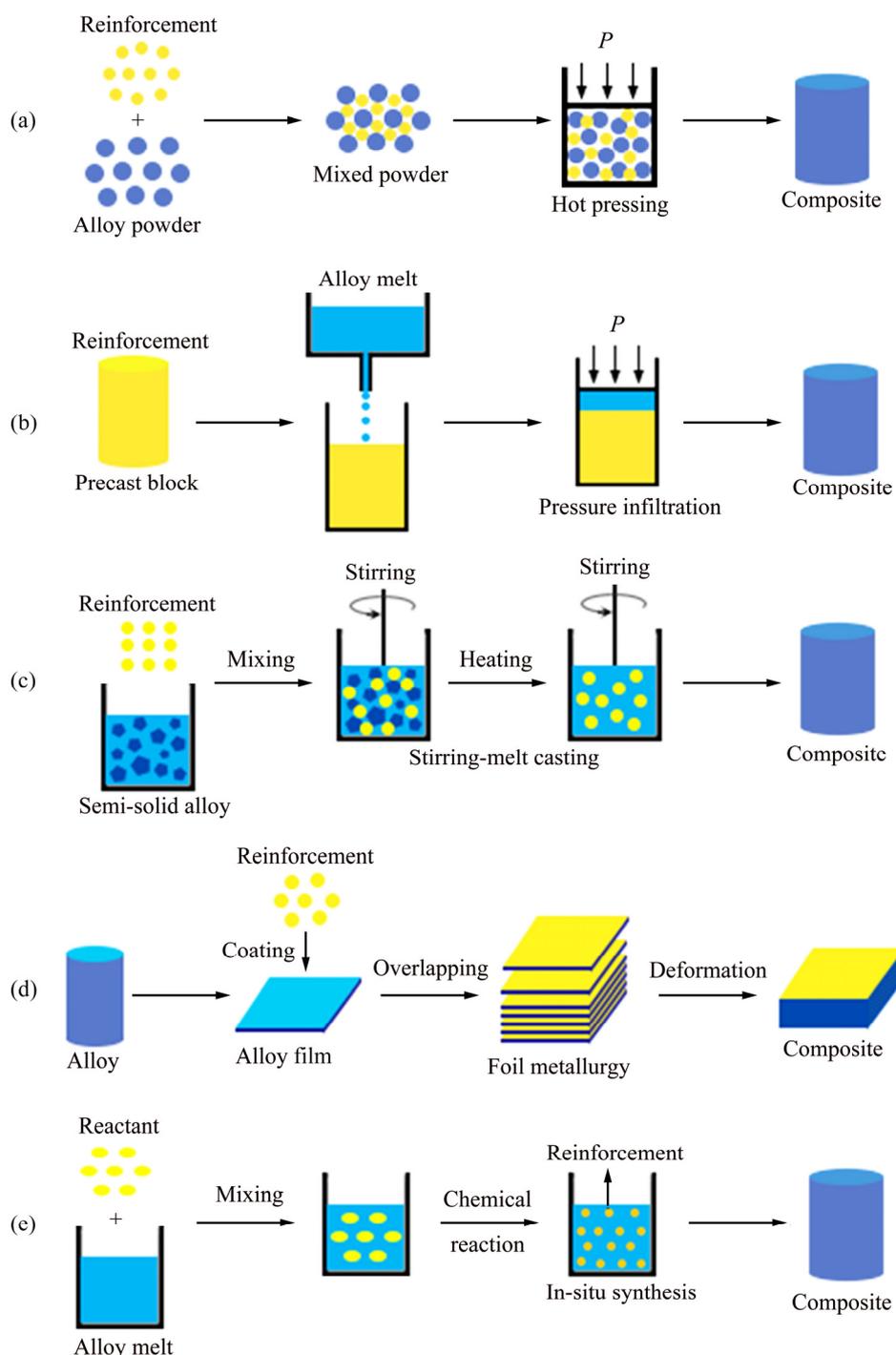


Fig. 1 Schematic diagram of preparation methods for Mg–Li matrix composites: (a) Powder metallurgy processing; (b) Pressure infiltration processing; (c) Stirring-melt casting processing; (d) Foil metallurgy processing; (e) In-situ synthesis processing

reinforcement and matrix is more difficult to control. Moreover, the whole preparation process of composites is under vacuum or inert gas protection.

2.3 Stir-casting processing

Stir-casting processing can be classified into three categories according to alloy state, namely liquid stir-casting, semi-solid stir-casting, and stirring-melt casting. In liquid stir-casting, the reinforcement is added

into liquid alloy melt and then the melt is stirred for a certain time followed by cooling down. While in semi-solid stir-casting the matrix alloy is at semi-solid state when the reinforcement is added. In case of stirring-melt casting (Fig. 1(c)), the reinforcement is also added into semi-solid alloy melt. After stirring for a period of time, the temperature is raised above the liquidus temperature of matrix alloy followed by stirring again and cooling down. Moreover, the composites

prepared by stirring-melt casting outperform those prepared by liquid and semi-solid casting on particle distribution and porosity. Stir-casting processing is widely used in Al-matrix composites and Mg-matrix composites [16,17], and it is also feasible to prepare ultralight Mg–Li matrix composites. WANG et al [18] prepared the Mg–Li matrix (Mg–14Li–3Al) composite reinforced by ultrafine Al_2Y particles (0.1–3 μm) via stir-casting processing at 650 °C in a resistance furnace under argon atmosphere. The prepared composites have the advantages of good interface bonding, fine grains, high strength and hardness. Whereas, the reinforcement with high density is difficult to distribute evenly due to the superlight feature of Mg–Li alloy. In addition, there also exist some shortcomings such as interface reaction, chemical incompatibility, and inhomogeneous composition.

2.4 Foil metallurgy processing

The schematic diagram of foil metallurgy processing is shown in Fig. 1(d). In foil metallurgy processing, the alloy matrix is made into thin foils by multiple cold deformation and intermediate annealing at low temperature (or hot rolling at low temperature), and then the reinforcement is coated evenly on thin foils of alloy matrix, finally the coated thin foils are overlapped and deformed (extrusion or rolling) at 180–230 °C to obtain composites [19]. The temperature is lower than 230 °C during the whole preparation process, resulting in no interface reaction between matrix and reinforcement. Moreover, cold deformation and recrystallization can refine matrix grains, and improve plasticity and strength. But the interface bonding is dominated by mechanical bonding with a low strength. Foil metallurgy processing, like powder metallurgy processing, has disadvantages in complicated preparation process, high requirement for equipment, and high cost.

2.5 In-situ synthesis processing

In-situ synthesis processing has been developed for preparing metal-matrix composites in recent years. Composites were prepared by this method using chemical reaction between matrix alloy and reactant to in-situ form required reinforcements in matrix, and its schematic diagram is shown in Fig. 1(e). Compared with the above preparation methods, in-situ synthesis can overcome some shortcomings, such as severe interface reaction, reinforcement segregation, incompatibility between matrix and reinforcement, and poor wetting property, which highly improves the mechanical property of composites.

WEI et al [7] obtained Mg–Li matrix composites reinforced with $\text{MgO}/\text{Mg}_2\text{Si}$. The SiO_2 powders were added into overheated Mg–Li alloy melt followed by

stirring and casting. During stirring and casting processes, the reinforcements $\text{MgO}/\text{Mg}_2\text{Si}$ were in-situ synthesized through the reaction of SiO_2 and Mg, and then uniformly distributed and well consolidated with matrix into submicron particles, resulting in an effective improvement in mechanical properties of composites.

3 Matrix alloys of Mg–Li matrix composites

In Mg–Li matrix composites, the matrix is Mg–Li binary alloy or multicomponent alloy with addition of proper elements. It is known that the crystal structure of Mg is hcp structure with few slip systems, leading to poor plasticity. The addition of Li with bcc structure could decrease c/a ratio of magnesium, resulting in more slip systems [20]. Moreover, the crystal structure of Mg–Li alloy transforms from hcp structure to bcc structure when Li content is over 5.7 wt.%, and achieves complete transformation when Li content is over 10.3 wt.% [21,22]. These changes not only lower the density, but improve the ductility and enhance the specific strength and stiffness of alloy.

Generally, the Mg–Li alloys with high Li content, such as Mg–8Li, Mg–12Li, and Mg–14Li [13,23,24], are chosen as matrix alloys for maintaining low density of Mg–Li matrix composites. Al is the most common alloying element in Mg–Li alloys, which can effectively strengthen alloys via solid solution and dispersion strengthening. MgLi_2Al and $\text{Mg}_{17}\text{Al}_{12}$ phases in Mg–Li–Al alloys can enhance the strength of alloys. Nevertheless, overaging is liable to take place in Mg–Li–Al alloys with high Li content owing to the transformation from metastable MgLi_2Al phase to MgLiAl or AlLi phase [25,26]. In addition, Zn, Ag, Zr, and rare-earth elements are also common alloying elements applied in Mg–Li alloys. Effect of Zn on Mg–Li alloys is similar to that of Al, but the strengthening effect of unit quality is not notable like that of Al [2]. Ag element can inhibit the overaging of Mg–Li alloys and improve the structure and performance stability [27]. Zr as a grain refiner can effectively refine the grains to improve the as-cast structure and enhance the strength of alloys [28]. Rare-earth elements increase the comprehensive performance of Mg–Li alloys by the solid solution of atoms and the formation of intermetallics. Furthermore, rare-earth elements improve the mechanical properties at elevated temperature via improving the thermostability of precipitated phases, and promote aging strengthening via increasing crystallization temperature of Mg–Li alloys [2]. The combination of strengthening elements (such as Al, Zn, and Cd) with elements that improve structure stability (such as Ag, Cu, and rare-earth elements) can ensure the high strength and structural stability of Mg–Li alloys.

4 Reinforcements of Mg–Li matrix composites

The reinforcements of Mg–Li matrix composites are divided into three categories, namely particle (e.g., B, B₄C, B₂O₃, MgO, Mg₂Si, and Al₂Y), whisker or short fiber (e.g., SiC whisker and δ -Al₂O₃ fiber), and continuous fiber (e.g., stainless steel wire, titanium alloy wire, and carbon fiber). The strengthening in particle-reinforced composites is based on the stress transfer between matrix and reinforcement, which involves an effect of particles on the modification of structure and the micromechanics of deformation, such as grain hardening, substructure hardening, and Orowan hardening [8]. The fiber-reinforced composites take full advantages of strength of fiber and ductility of matrix through sufficient bond between fiber and matrix. In these composites, the role of matrix is to distribute and transmit the load to the fibers, while the role of fibers is to carry significant proportion of the external load and prevent (or transfer) the propagation of crack [8,15].

4.1 Reinforcement with particles

4.1.1 B particle

B particle (B_p) is used as reinforced phase of composites due to its high melting point and low density. However, B can react with metal and nonmetallic at high temperature to form compound, so it is not ideal reinforcing particle. It has been reported that B_p with volume fraction ranging from 0 to 30 vol.% could reinforce LA141 (Mg–14Li–1.5Al) alloy [12,29]. Some physical and mechanical properties of Mg and Mg–Li matrix composites reinforced with B particles are shown in Table 2. Elastic modulus, strength, and specific stiffness of composites increased rapidly with increasing volume fraction of B particle. Good tensile ductility was achieved in Mg–Li composite with 10 vol.% B particles but was quite low for higher volume fractions.

4.1.2 B₄C particle

B₄C is often used as ceramic reinforced-phase in metal-matrix composites, due to its low density, high strength, good high-temperature stability and chemical stability. The Mg–9Li matrix composite containing B₄C

(5.0 wt.%) was prepared by press-bonding fine-grained Mg–Li foils with altering layers of B₄C powders [19]. In this process, the Mg–Li foils with a thickness of 0.2 mm were obtained after extensive cold-rolling with intermediate annealing steps at low temperature, and the B₄C powders (<20 μ m) were soaked in a mixture solution (5% HCl, 2% HF, 93% ethanol) to remove surface contamination. The dark Li₂O layer originated from the oxide layers on both surfaces of as-rolled foils prior to press-bonding, and was found between foils. The yield strength and elastic modulus of Mg–9Li–5B₄C (162 MPa and 49 GPa) were greater than those of Mg–9Li laminates (133 MPa and 47.7 GPa) and as-cast Mg–9Li alloy (110 MPa and 45.4 GPa), while their elongations were 13%, 55% and 22%, respectively. These results were attributed to the finer grain sizes of composites and the presence of B₄C and Li₂O compounds. The tension and compression behaviors of Mg–9Li–5B₄C were investigated [30]. The results revealed that the fine grained (2 μ m) particulate composite was superplastic at 150–200 °C and its superplastic flow occurred by grain boundary sliding controlled by grain boundary diffusion.

WU et al [31] prepared Mg–8Li–1Zn–2.4B₄C and Mg–8Li–1Al–1Y–2.34B₄C composites by foil metallurgy processing. Compared with as-cast and as-rolled Mg–Li matrix alloys, the strength of B₄C reinforced composites increased, while the elongation reduced. The Mg–8Li–1Al–1Y–2.34B₄C composite exhibited the highest strength (257.23 MPa), which was ascribed to work hardening, presence of zigzag interface and B₄C particles, and good interface bonding. DONG et al [32] used in-situ synthesis processing to prepare Mg–14Li–1Al–6B₄C composite and investigated the effect of hot extrusion on its microstructure and mechanical properties. The results showed that hot extrusion reduced the porosity of composite, improved the distribution uniformity of reinforced phase, and refined grains through dynamic recrystallization. Therefore, the mechanical properties of Mg–Li composite were effectively improved by hot extrusion. The composite extruded at 200 °C exhibited the optimal properties with tensile strength of 237.5 MPa, elongation of 21.7%, and specific strength of $167.8 \times 10^3 \text{ m}^2/\text{s}^2$.

Table 2 Some physical and mechanical properties of Mg–B and Mg–Li–B composites [12]

Matrix	$\phi/\%$	$\rho/(\text{g}\cdot\text{cm}^{-3})$	E/GPa	$\sigma_{0.2}(\text{compression})/\text{MPa}$	Specific stiffness/ $(\text{GPa}\cdot\text{cm}^3\cdot\text{g}^{-1})$
Mg	0	1.75	43.2	187	25.2
	25	1.87	79.2	240	43.2
Mg–14.1Li–1.5Al	0	1.53	50.2	180	33.5
	10	1.64	68.1	214	42.4
	20	1.75	79.3	220	46.2
	30	1.77	101.1	244	58.3

4.1.3 MgO/Mg₂Si particles

Preparing reinforcement by chemical reaction is a novel research field for Mg–Li matrix composites. For instance, B₂O₃, Si, and SiO₂ powders can react with Mg–Li alloy to in-situ form MgO/Mg₂Si particles, which could improve the properties of Mg–Li matrix [7]. Figure 2 shows the DTA curves of Mg–6Li alloy and its composites. There existed an endothermic peak at about 590 °C in all curves, which was caused by the melting of Mg–Li alloy. However, an exothermic peak appeared at temperature higher than melting point after adding particles (B₂O₃, SiO₂, and Si), indicating that the reaction between these particles and Mg–Li alloy occurred. The reaction temperatures of Mg–Li alloy and B₂O₃ (679.2 °C) or SiO₂ (739.8 °C) were closed to the smelting temperature of Mg–Li alloy, so these particles were suitable for in-situ synthesis of Mg–Li matrix composites [33].

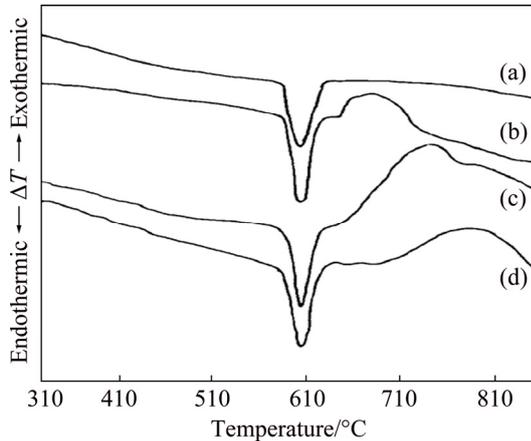
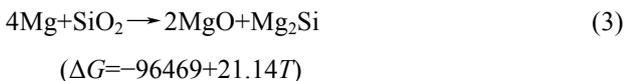
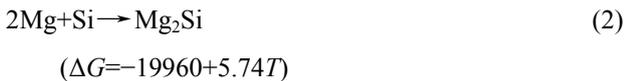
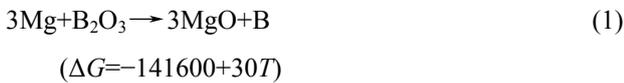


Fig. 2 DTA curves of Mg–Li alloy and Mg–Li matrix composites: (a) Mg–6Li; (b) Mg–6Li–5B₂O₃; (c) Mg–6Li–5SiO₂; (d) Mg–6Li–5Si [33]

In in-situ synthesis process, the reactions between particles and Mg–Li alloy and the corresponding Gibbs free energy equations are expressed as follows:



The reaction modes depended on the physical and chemical properties of particles. In Mg–Li–B₂O₃ system, the melting point of B₂O₃ particle was about 450 °C, which was lower than that of Mg–Li alloy, and the critical reaction temperature was 641 °C. Once B₂O₃ particles were added into Mg–Li melt, the temperature

around particles immediately decreased due to the melting endothermic of particles. After the particles melted and spread in Mg–Li alloy melt, the reaction began to occur. Therefore, this reaction was liquid–liquid reaction. The prepared MgO reinforced particles were fine (3–5 μm) and dispersed, but the generated particles might accumulate on pre-existing MgO particles and make them grow up. The reductive B particles existed in the form of tiny particle or solid solution in matrix, which performed auxiliary reinforcement. While in Mg–Li–Si/SiO₂ system the reaction was solid–liquid reaction due to the high melting points of Si and SiO₂ particles. Figure 3 shows the morphologies of MgO and Mg₂Si particles in Mg–Li matrix composite prepared by in-situ synthesis processing. The MgO particle was spherical particle with a size less than 1 μm, while the Mg₂Si particle was strip particle with a larger size. The interfaces between particles and matrix were clean and there were no obvious transition layer and reaction layer [34]. In addition, the in-situ formed particles could be crystallization nucleus to refine grains. Table 3 lists the mechanical properties of Mg–Li–MgO/Mg₂Si composites. The tensile strength and elongation of Mg–Li matrix composites reinforced with 5 wt.% particles reached the maximum, whereas further increase in particle content led to the degradation of strength and elongation, and the increase in elastic modulus and hardness.

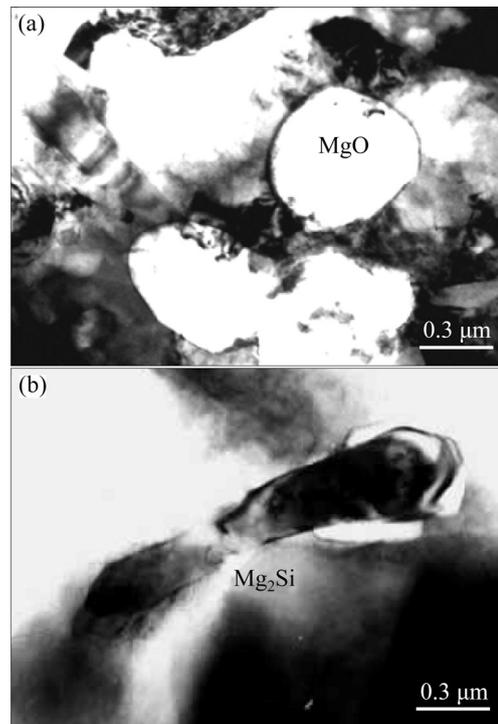


Fig. 3 Morphologies of MgO and Mg₂Si particles in Mg–Li matrix composite prepared by in-situ synthesis processing [34]

Table 3 Mechanical properties of Mg–Li–MgO/Mg₂Si composites [35]

Matrix	w/%	$\sigma_{0.2}$ / MPa	σ_b / MPa	δ / %	E/ GPa	HV
Mg–6.5Li	0	57.9	112.5	10	33.3	54.5
	5	82.5	137.4	5.6	44.6	68.9
	10	91.5	127.3	4.3	49.6	72.3
Mg–8Li	0	77.6	127.2	21	42.8	52.5
	10	97.8	157.4	9.5	49.5	63.6
Mg–11Li	0	66.3	104.6	16.4	35.4	46.2
	5	164.6	180.9	8.5	47.9	69.5
	10	142.6	162.1	5.3	62.0	77.1
	15	–	–	–	78.8	82.2

4.1.4 Al₂Y particle

Although the aforementioned ceramic reinforcements can enhance the strength and modulus of Mg–Li matrix alloy, there remain concerns about the chemical incompatibility between ceramic reinforcements and Mg–Li matrix, leading to undesired degradation in the mechanical properties of composites [36]. Since intermetallic compounds have become one of the most promising particulate reinforcements owing to their high specific strength, high specific stiffness, and high modulus [37,38]. Among these intermetallic compounds, Al₂Y compound has lower density (3.93 g/cm³), high hardness (HV=648), high modulus (158 GPa), and high melting point (1458 °C). Therefore, there are a large number of reports on Mg–Li matrix composites reinforced with Al₂Y particles [39]. Figure 4 indicates the micrographs of Al₂Y_p/Mg–Li composite and the interface between Al₂Y particle and Mg–Li matrix. The Al₂Y particles distributed homogeneously in matrix alloy, and there was no obvious agglomeration or segregation of particles in Mg–Li matrix. The clean interface indicated that Al₂Y particle bonded well with matrix without forming intermediate phases, and no porosities or reaction products were found at the interface. In Mg–Li composites reinforced with ceramic particles,

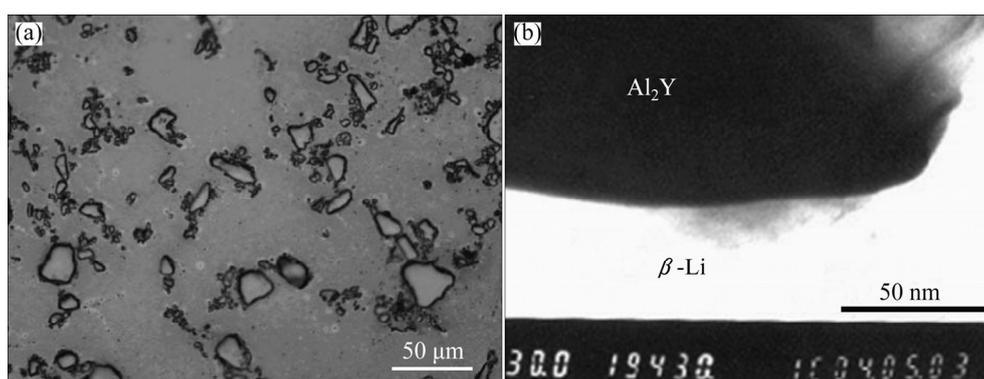
numerous micro-cracks initiated in the particles and interface during tensile deformation. While in Al₂Y_p/Mg–Li composite, the micro-cracks generated mainly initiated and propagated in the matrix that was close to the interface. The relatively “soft” intermetallic particles (such as Al₂Y) could be compatible with the deformation of matrix due to their good deformation capability, exhibiting a “soft” restriction to the matrix, and these “soft” particles could resist the propagation of cracks in micro-scale during the deformation of composites [39].

ZHANG et al [41] prepared Mg–14Li–3Al matrix composite reinforced with Al₂Y particles (20–25 μm) via stir-casting processing, and studied the effect of heat treatment at interface between Al₂Y and matrix. The results indicated that a diffusion zone rich in Y and Al elements was found in the vicinity of interface, and that these elements further diffused into matrix during heat treatment, resulting in a wider diffusion zone. Heat treatment improved the characteristics of transition interface layer, including the loadsharing function and the physical compatibility between matrix and particles, which benefitted the mechanical properties of Mg–Li matrix composite. WANG et al [18] prepared Al₂Y_p/Mg–Li composite by stir-casting and investigated the microstructural evolution of composite during warm-rolling. The rolling deformation was conducive to the uniform distribution and refinement of Al₂Y particles. Moreover, Y diffused from Al₂Y particles reacted with Al in the matrix to form AlY phase during warm-rolling, resulting in many nano-sized AlY particles produced and distributed in the matrix. Table 4 lists some mechanical properties of Al₂Y_p/Mg–Li composites. It could be seen that the strength and elastic modulus of Al₂Y_p/Mg–Li composites were much higher than that of matrix alloy, and the elongation got a good inheritance.

4.2 Reinforcement with whiskers or short fibers

4.2.1 SiC fiber or whisker

SiC fiber or whisker can effectively enhance the strength of Mg–Li matrix composites, and there are no

**Fig. 4** Micrographs of as-cast Al₂Y_p/Mg–Li composite (a) and interface between Al₂Y particle and Mg–Li matrix (b) [40]

any chemical reactions found at the interface between SiC and matrix. MASON et al [42] prepared Mg–Li matrix composites by squeeze infiltration using “Nicalon” multifilament, “Sigma” CVD monofilament, and “Tokawhisker” whisker as reinforcements with a volume fraction of 20 vol.%. In “Nicalon” reinforced Mg–Li matrix composite, the microstructure was unstable and the fibers tended to absorb lithium very rapidly during and after infiltration. The lithium penetration resulted in the damage of fibers to different degrees along the infiltration axis. In the case of “Sigma” reinforced Mg–Li matrix composite, no progressive chemical reaction took place. Nevertheless, the fibers tended to suffer pronounced degradation, especially at grain boundaries. The fiber cracking in form of intergranular failure was widespread in as-fabricated composite, and became more serious and universal after heat treatment. While “Tokawhisker” whisker did not appear to suffer any chemical reaction and degradation after contacting with Mg–Li matrix, even after prolonged exposure to high temperature. ZHANG et al [23] manufactured SiC_w reinforced Mg–Li matrix composites by liquid pressure infiltration in a vacuum furnace, and the mechanical properties of composites are shown in Table 5. The tensile strength and elastic modulus of composites were higher than those of corresponding matrix alloys, but the elongations were seriously degraded. The Mg–11Li–3Al matrix reinforced by 13 vol.% SiC whisker exhibited the best comprehensive performance. After extrusion, the tensile strength and elongation were enhanced due to the reduction of defects.

4.2.2 δ -Al₂O₃ fiber

Al₂O₃ fiber is one of common reinforcements, and Mg–Li matrix composites reinforced with Al₂O₃ fiber can exhibit high mechanical properties and other special properties. TROJANOVÁ et al [13] manufactured Mg–xLi (x=4, 8, 12) matrix composites reinforced with short δ -Al₂O₃ fibers by the pressure infiltration process, and the micrographs are shown in Fig. 5. It was observed that no significant fiber damage resulted from the Mg–Li melt attack. In Mg–4Li matrix composite, the composite was composed of α -phase and short δ -Al₂O₃ fibers, and the two-dimensional fiber structure was nearly perfect. The Mg–8Li matrix composite consisted of (α + β) structure and δ -Al₂O₃ fibers distributed randomly within α and β matrix regions. While δ -Al₂O₃ fibers distributed uniformly in Mg–12Li matrix composed of β phase and the etched grain boundaries were visible. It was reported that load transfer and enhanced dislocation density were regarded as the most important strengthening mechanisms in the fiber-reinforced Mg–Li matrix composites, and the mechanical properties decreased with increasing temperature [13,14,43]. KÚDELA et al [44–47] studied the fracture behavior of Mg–Li matrix composites reinforced with δ -Al₂O₃ fibers. The results revealed that poor interfacial bond in Saffil/Mg composites caused massive fiber pull-out and little fiber fragmentation, leading to premature failure. While in Saffil/Mg–Li composites, multiple fiber cross-breakage shown in Fig. 6 is indicative of strong interfacial bond in Mg–Li composites reinforced with Saffil fibers. This might be explained by the formation of ternary

Table 4 Mechanical properties of Al₂Y_p/Mg–Li composites at room temperature [39,40]

Matrix	Particle content	$\sigma_{0.2}$ /MPa	σ_b /MPa	δ /%	E /GPa	HB
Mg–14Li–1.5Al	0	–	130	12	34.6	67.9
	5 wt.%	–	189	7	50.1	107.4
Mg–14Li–3Al	0	94	115	26	34.5	–
	20 vol.%	161	225	9	73.2	

Table 5 Mechanical properties of SiC_w/Mg–Li–Al composites and matrix alloys [23]

Matrix	φ (SiC _w)/%	σ_b /MPa	E /GPa	δ /%	Specific stiffness/ (GPa·cm ³ ·g ⁻¹)	Specific strength/ (MPa·cm ³ ·g ⁻¹)	ρ /(g·cm ⁻³)
Mg–4Li–1Al	0	157.2	43.7	17	26.6	95.8	1.64
	13	190	58.7	4.5	31.9	103.2	1.84
	13(extruded)	246	58.7	5.6	31.6	132.3	1.86
Mg–8Li–1Al	0	131.5	44.7	35	29.4	86.5	1.52
	13	182.8	59.4	5.2	34.5	106.2	1.72
	13(extruded)	235	59.4	6.2	33.9	134.3	1.75
Mg–11Li–3Al	0	138.8	45.9	45	30.4	91.9	1.51
	13	196.6	60.5	5.8	36.2	117.7	1.67
	13(extruded)	250	60.5	7.2	36	148.8	1.68

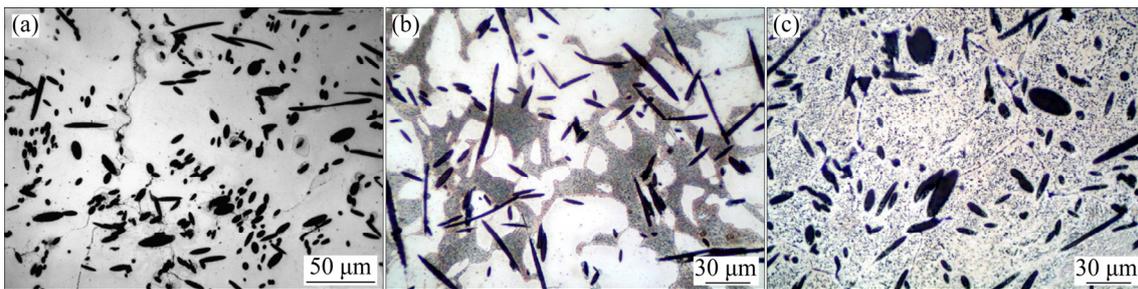


Fig. 5 Microstructures of δ - Al_2O_3 fiber reinforced Mg-4Li (a), Mg-8Li (b) and Mg-12Li matrix (c) composites [13]

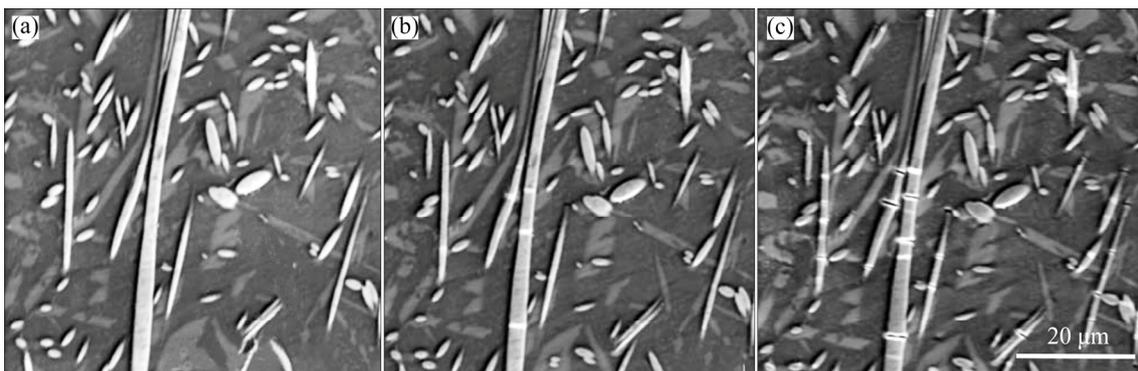


Fig. 6 In-situ SEM images (a-c) showing evolution of multiple cross-fracture in aligned fibers of bent δ - Al_2O_3 /Mg-8Li composite in vicinity of convex edge (The tensile axis (TA) is parallel to the convex sample edge) [45]

spinel-like Al-Li-O products in Saffil/Mg-Li composites. The products promoted the formation of strong interfaces via topotactic insertion of Li^+ ions into cation vacancies of δ - Al_2O_3 [48]. MASON et al [42] prepared Mg-Li matrix composites reinforced with Saffil fibers containing 5 wt.% SiO_2 and their mechanical properties are shown in Table 6. Both strength and strain of Mg-12Li matrix composite were higher than those of Mg-10.3Li-6Al-6Ag-4Cd matrix composite, due to the brittleness of latter composite leading to the load difficult to transfer to fibers. Furthermore, the binder SiO_2 could dissolve in Mg-Li matrix melt and form massive Mg_2Si precipitated particles, which degraded the mechanical properties of composites. In addition, Mg-Li matrix composite reinforced by short δ - Al_2O_3 fiber reveals strong anisotropy. The Mg-Li matrix composite reinforced by 10 vol.% δ - Al_2O_3 fibers were fabricated by melt infiltration under argon pressure to investigate the anisotropy of coefficient of thermal expansion (CTE) [49]. The results show that the cross-plane CTE is higher than in-plane ones, and the temperature dependence of cross-plane and in-plane CTE line is opposite.

4.3 Reinforcement with continuous fibers

4.3.1 Metal fibers

Metal fibers (such as steel wire and titanium wire) can effectively enhance the mechanical properties of Mg-Li matrix composites, due to their high strength and

elastic modulus. Table 7 lists some tensile data reported for Mg-Li matrix composites reinforced with steel wire and titanium wire. Compared with Mg-8Li alloy, the strengths (25 °C) of its composites reinforced with Y8A (15 vol.%), MC-200 (30 vol.%), and AT-3 (40 vol.%) increased by 5, 6, and 3 times, respectively. Moreover, the elevated temperature strengths maintained a high level. WILCOX and CLAUER [50] obtained AFC-77 reinforced Mg-14Li-1Al matrix composite with high tensile strengths of 758 MPa at 25 °C and 484 MPa at 200 °C. Although these metal wires can greatly improve the tensile strength of composites, they are not suitable for ultralight Mg-Li matrix composites due to large density.

4.3.2 Carbon fiber

Due to low density, high strength, and corrosion resistance, carbon fiber is supposed to be the optimal reinforcement for metal matrix composites. However, carbon fiber can react with Mg-Li alloy melt to form Li_2C_2 compound, which strongly degrades the strengthening effect. The key to prepare ultra-light C_f /Mg-Li matrix composite is to develop an effective coating of carbon fiber. KÚDELA et al [51,52] used pyrolytic carbon (100 nm) to coat T300 carbon fibers for preparing C_f /Mg-12Li matrix composite. The Li_2C_2 was found in composite and without displaying any significant radial concentration gradient. In the attack on carbon fiber, the role of Li element was dominant, while Mg element was marginal. Meanwhile, the Mg-8Li

Table 6 Mechanical properties for short δ -Al₂O₃ fiber reinforced Mg–Li matrix composites [42]

Matrix	φ (Fiber)/%	Tensile testing		Vickers hardness/(kg·mm ⁻²)	
		σ_b /MPa	δ /%	Macro	Micro
Mg–12Li	0	70	>6	35	42
		80	>8		
	12	200	3.5	96	77
		220	2.1		
		280	2.0		
24	105	0.6	113	81	
Mg–10.3Li–6Al–6Ag–4Cd	0	85	0.4	93	85
		95	0.4		
	12	170	0.3	103	85
		165	0.3		
	24	145	0.3	145	95
		140	0.5		

Table 7 Mechanical properties of Mg–8Li and Mg–14Li–1Al matrix composites reinforced with steel wires (Y8A, MC-200, AFC-77) and titanium wires (AT-3) [8,50]

w (Matrix)/%		Reinforcement		ρ (g·cm ⁻³)	E /GPa	σ_b /MPa	
Li	Al	Wire	φ /%			25 °C	200 °C
		–	–	1.61	42	100	20
8	0	Y8A	15	2.40	64	600	370
		MC-200	30	3.30	–	710	450
		AT-3	40	2.60	66	400	300
14	1	–	–	1.33	42	144	14
		AFC-77	32	3.50	–	758	484

matrix composites reinforced with T800H carbon fibers coated with pyrocarbon and pyrocarbon+SiC were manufactured. The carbon fibers coated with pyrocarbon layer exhibited a much higher morphological stability in contact with Mg–Li alloy melt than those coated with pyrocarbon+SiC layer, and the latter was seriously attacked during infiltration process. The pyrocarbon layer was conducive to inhibit the attack of carbon fiber, but led to a decrease in fiber strength [53].

5 Interface chemistry of Mg–Li matrix composites

Interface, a transition region from the matrix to the reinforcement, is an extremely significant microstructure for composites, and its structure and property mainly depend on the preparation process, surface treatment, characteristic of matrix and reinforcement. Interface can bond matrix and reinforcement together to form composite, and be responsible for transferring the load from matrix to reinforcement. Therefore, interface plays a key role in improving the properties of composites.

In various preparation processes for Mg–Li composites, the chemical reaction between matrix and reinforcement should be strictly controlled and avoided, except for in-situ synthesis processing where complete reaction between the added reactant and Mg–Li alloy melt is required. Solid-state preparation techniques (such as foil metallurgy processing) can effectively control the interface reaction, but result in inferior interface wettability and interface combination. Moreover, multi-pass deformation makes Li₂O compound present at certain locations along the interface between the alloy foils [19]. Among these preparation processes, in-situ synthesis processing is beneficial to fabricate composites with good wettability and clean interface.

Generally, the preparation of Mg–Li composites is conducted at high temperature above melting point of matrix alloy, which causes interface reaction between matrix and reinforcement. In addition, the segregation and diffusion of elements during the process of alloy solidification and heat treatment make the interfacial structure complicated. The chemical properties of B and B₄C particles are relatively stable, so these particles are

not sensitive to the attack of Mg and Li. The MgO/Mg₂Si reinforced particles in Mg–Li composites are usually prepared via in-situ synthesis processing. The interfaces between matrix and MgO/Mg₂Si particles were clean and no obvious transition layer and reaction layer were observed. Moreover, the crystallographic relationship between MgO particle and α -Mg was $[100]_{\text{MgO}}//[\bar{4}0\bar{4}3]_{\alpha}$ and $(011)_{\text{MgO}}//(\bar{1}2\bar{1}0)_{\alpha}$, and that between Mg₂Si particle and β -Li was $[310]_{\text{Mg}_2\text{Si}}//[411]_{\beta}$ and $(1\bar{3}\bar{1})_{\text{Mg}_2\text{Si}}//(\bar{0}01)_{\beta}$. Therefore, MgO and Mg₂Si particles had a coherent relationship with matrix at the interface [34]. In Al₂Y_p/Mg–Li matrix composites, the interface bonding was good, and there were no obvious chemical reaction and atom diffusion. However, the thickness of transition layer increased after heat treatment, and masses of Y and Al atoms from Al₂Y particles diffused into matrix farther away from the interface, which improved the mechanical properties of composites [41].

In Mg–Li composites reinforced with “Nicalon” fibers containing 15 wt.% C and 25 wt.% Si, the microstructure was highly unstable, and the fibers tended to absorb lithium after contacting with Mg–Li alloy melt and lithium vapor. The lithium depletion not only changed the matrix composition, but caused dramatic embrittlement of fibers [42]. There was no obvious chemical reaction in the case of “Sigma” fibers, but fibers still tended to suffer prominent degradation in the form of grain boundary attack, especially after heat treatment [42]. The intergranular cracking was caused by lithium penetration of grain boundaries, and the Y₂O₃ layer with a thickness of 1 μm could effectively provide protection against invade of lithium. “Tokawhisker” did not react with pure Mg, pure Li, and Mg–Li matrix, even after long exposure at high temperature [8]. The interface between Mg–Li matrix and “Tokawhisker” fiber was smooth and clean, without any reaction products, though the reactions between SiC whisker and Mg–Li matrix were possible according to the following thermodynamic calculation [23]:



$$(\Delta G_{(300-1100\text{K})}=-5.9\text{ to }-1.5\text{ kJ/mol})$$

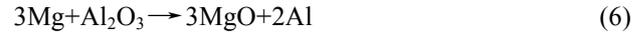


$$(\Delta G_{(300-1100\text{K})}=-34\text{ to }-23.4\text{ kJ/mol})$$

In case of SiC_w/Mg–8Li–1Al composite, the disordered atom-distributing region near the interface might result in the difference in thermal expansion coefficients between matrix and SiC whisker, which caused stress concentration at the interface of composites.

“Saffil” fibers are composed of δ -Al₂O₃ as a dominant phase and a small quantity of SiO₂. According

to thermodynamics, the following reactions might take place between fiber and Mg–Li alloy melt [23]:



$$(\Delta G_{(300-1100\text{K})}=-225.8\text{ to }-218.5\text{ kJ/mol})$$



$$(\Delta G_{(300-1100\text{K})}=-103.9\text{ to }-37.1\text{ kJ/mol})$$



$$(\Delta G_{(300-1100\text{K})}=-427.6\text{ to }-393.2\text{ kJ/mol})$$



$$(\Delta G_{(300-1100\text{K})}=-269.6\text{ to }-202.9\text{ kJ/mol})$$

Therefore, there existed a reaction zone between fiber and matrix possibly containing MgO, Li₂O, Mg₂Si, Al, and Si. The reaction of “Saffil” fibers with Mg–Li alloy melt during preparation process of composites was complicated and affected by alloy composition, temperature, time and the like. Some studies [42] suggested that δ -Al₂O₃ fibers were chemically stable, and hardly suffered the attack of Mg–Li alloy melt. While SiO₂ binder could rapidly dissolve into alloy melt, and a degree of interaction took place to form some precipitates (such as Mg₂Si, MgO, Li₂O). The embrittlement of fibers was mainly caused by the penetration of lithium along the grain boundaries. The other researches [47,54,55] considered that δ -Al₂O₃ fiber also participated in the interfacial reaction, and the scheme of interaction of “Saffil” fiber with Mg–Li alloys is shown in Fig. 7. Lithium quickly penetrated into fibers by diffusion, and Li⁺ cations were incorporated into δ -Al₂O₃ lattice to form intermediate metastable spinel-like compounds $(\text{Al}_9[\text{Al}_{(40-x)/3}\square_{(8-2x)/3}\text{Li}_x]\text{O}_{32})$ where “ \square ” represents octahedral cations or vacancies). Then the metastable spinel-like compounds further transformed into LiAl₅O₈ to achieve thermodynamic equilibrium after long-term annealing process. The Mg²⁺ cations could not compete with Li⁺ cations in incorporation into δ -Al₂O₃ lattice due to its lower efficiency in vacant occupation.

Both iron and titanium were relatively stable in Mg–Li alloy melt without the formation of intermetallic phases. However, some alloying elements from the highly alloyed maraging steel wire could react with Mg–Li alloy melt to some degree [50]. Carbon fibers were scarcely sensitive to the attack of pure magnesium, because magnesium carbides could not be formed quickly from elements. Nevertheless, carbon fibers immediately reacted with molten Mg–Li alloy to form Li₂C₂ compound during contacting with matrix melt, and the reaction was described as follows [56]:



$$(\Delta G_{(1000\text{K})}=-44.8\text{ kJ/mol})$$

The reaction damaged carbon fibers severely and consumed large quantity of lithium, leading to non-ideal strengthening effect. Therefore, it is imperative for preparing lightweight $C_f/Mg-Li$ matrix composites to develop the effective and lightweight coating for carbon fibers.

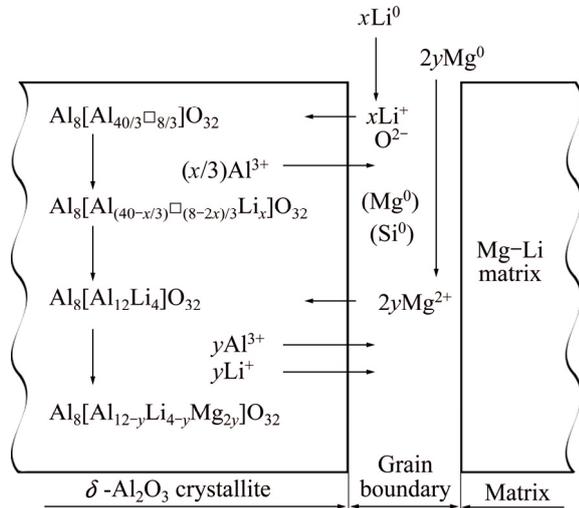


Fig. 7 Scheme of interaction of “Saffil” fibers with Mg–Li alloys [54]

6 Prospect

Composite approach provides an effective way to solve the low strength and worse structure stability of Mg–Li alloys. Mg–Li matrix composites, owing to low density, high strength and stiffness, and good creep resistance, have great potential in the fields of aerospace and military. However, there exist many problems that hinder the further development of Mg–Li matrix composites, and mainly focus on the following aspects.

(1) The control of interface reaction between matrix and reinforcement is an urgent problem in Mg–Li matrix composites to be resolved. Choosing proper reinforcement and developing effective surface coating can reduce and prevent the interface reaction to some extent. Some intermetallics (such as Al_2Y), which have high specific strength, good chemical compatibility with alloys, and no interface reaction with Mg–Li melt, are the promising reinforcements for Mg–Li matrix composites. Surface coating can prevent lithium penetration and inhibit interface reaction, its development is fundamental for reinforcements, especially for carbon fiber.

(2) The preparation processing of Mg–Li matrix composites is complicated and costly, due to high chemical activity of Mg and Li. In-situ synthesis processing, in which the reinforcements are formed in situ and have no interface reaction with substrate, might be a promising preparation way for Mg–Li matrix

composites.

(3) Although the strength of Mg–Li matrix composites is much higher than that of Mg–Li alloy, it is still far from the actual application. The properties of Mg–Li matrix composites need to be improved by selection of matrix and reinforcement, improvement of preparation method, and heat treatment and deformation. Comprehensively improving the properties of Mg–Li matrix composites is a systematic engineering, and requires a thorough design consideration.

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Mg–Li 基复合材料的研究进展

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摘要: Mg–Li 基复合材料由于具有高比强度和比刚度、良好的阻尼特性和耐磨性、以及较小的热膨胀系数, 成为航空航天和军事领域的理想结构材料之一。介绍了粉末冶金、压力浸渗、搅拌铸造、薄膜冶金以及原位生成等 Mg–Li 基复合材料常用的制备方法, 并对其优缺点进行比较。总结了 Mg–Li 基复合材料常用的基体合金和增强体, 以及典型 Mg–Li 基复合材料的组织与性能。概述了 Mg–Li 基体复合材料中基体和增强体的界面化学。最后, 针对目前存在的问题以及可能的解决途径进行了探讨。

关键词: Mg–Li 合金; 复合材料; 制备; 增强体; 界面化学

(Edited by Xiang-qun LI)