



Effects of solution treatment on microstructure and mechanical properties of thixoformed $Mg_2Si_p/AM60B$ composite



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ARTICLE INFO

Article history:

Received 1 December 2014
Received in revised form 17 February 2015
Accepted 18 February 2015
Available online 26 February 2015

Keywords:

Composite
Thixoforming
Solution treatment
Microstructure
Mechanical properties

ABSTRACT

The studies on heat treatment of thixoformed magnesium based composites are very scarce. To verify the heat treatment behaviors of this kind of materials, the effects of solution treatment on microstructure and mechanical properties of thixoformed $Mg_2Si_p/AM60B$ composite have been investigated. The results indicate that the microstructure evolution includes two stages during solutionizing at 415 °C. The first stage (0–2 h) refers to the rapid dissolution of eutectic β ($Mg_{17}Al_{12}$) phases and the resulted fast coarsening of both the primary α -Mg particles and secondarily primary α -Mg grains. The second stage (after 2 h) is regarded as the normal growth of the two kinds of grains through mergence. Due to the adding of Mg_2Si phase, the microstructure evolution is slowed. The tensile properties first increase as the time increases from 0 h to 6 h due to the decrease of β phase harmfulness, increased solid solution strengthening and composition homogenization, and then decrease because of the grain coarsening. Correspondingly, the fracture regime changes from intergranular regime in turn to transgranular and intergranular regime. The effects of solution temperature are similar to those with the solution time. After being solutionized at 430 °C for 6 h, the tensile properties are up to the highest values, UTS of 277 MPa and elongation of 18.5%, increased by about 20% and 135% respectively compared with those of the as-thixoformed composite.

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1. Introduction

Magnesium alloys have wide applications in the fields of automobiles, aerospace, electronic products, portable tools and sporting goods due to their specific properties such as high specific strength, good electrical conductivity, good dimensional stability and so on [1,2]. However, their high-temperature properties are relatively poor and the mechanical properties will drop rapidly when the working temperature exceeds 120 °C [3]. In addition, they also suffer from poor wear resistance. It is known that the introduction of Si element into AM60B alloy melt can generate a kind of Mg_2Si particle (Mg_2Si_p) reinforced AM60B ($Mg_2Si_p/AM60B$) in situ composite after solidification, which can improve the high-temperature properties and wear resistance of the AM60B alloy [4,5].

Thixoforming, a relatively new metal forming technology, has numerous advantages compared with conventional technologies. The most important advantage is that it can significantly reduce or eliminate porosities and gas pores. The decrease or elimination

of gas pores offers the thixoformed components to be heat treated to further improve mechanical properties [6,7]. Therefore, thixoforming is considered as an appropriate forming technology for magnesium alloys, of course, also for magnesium based composites.

As we all know, the performance of a component is determined by its microstructure, and the microstructure is determined by processing technology and subsequent heat treatment. Unfortunately, the current research of heat treatment on magnesium-based materials has been mainly focused on the traditional casting magnesium alloys. For example, the dissolution and precipitation of β phase for the conventional casting Mg–Al-based alloys and the related mechanical properties have been extensively investigated in previous works [8–13]. Only a few studies have involved the heat treatment of thixoformed magnesium alloys [14–18]. The research on thixoformed magnesium based composites has not been found.

According to previous investigation [19], there are large differences in the microstructures of the materials produced by traditional casting methods and thixoforming. The microstructure of a thixoformed alloy is consisted of small and spheroidal primary particles and intergranular secondarily solidified structures [6]. But the microstructure of a traditional casting alloy is composed of

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dendrites and interdendritic eutectics. So the heat treatment techniques for these two kinds of materials are different. It can be expected that the best performance of a thixoformed alloy cannot be obtained if the heat treatment techniques for conventional casting materials are used. Therefore, the investigation on the effects of heat treatment on microstructure and mechanical properties of thixoformed $Mg_2Si_p/AM60B$ composite has important theoretical significance and application values.

2. Experimental procedure

The materials used in this work are pure Mg, Al–35%Si and Mg–30%Sr master alloys and commercial AM60B alloy with a composition of Mg–5.98Al–0.42Mn–0.22Zn–0.024Si (in wt. (%)). Quantities of the raw materials were first remelted at 790 °C in a resistance furnace (using a specific covering agent for magnesium alloys, RJ-2 with a composition of 44MgCl₂–40KCl–7BaCl₂–9CaF₂ (in wt. (%)), to prevent oxidation), according to the composition of the 10wt.% $Mg_2Si_p/AM60B$ composite. 0.5wt.% Sr was then added into the melt in order to modify Mg_2Si phase. Subsequently, the melt was held for 20 min and 0.2 wt.% SiC particles (2 μm) were added in order to refine the primary α -Mg dendrites. After the SiC particles completely reacted with the melt, the melt was held for 5 min and degassed using 1.5 wt.% C₂Cl₆. After that, the melt was held for 10 min and finally poured into a steel mold with a cavity of $\Phi 50$ mm \times 500 mm at 790 °C.

The casting ingots were machined into some rods with dimensions of $\Phi 42$ mm \times 30 mm as the starting ingots for thixoforging. One rod was reheated in a resistance furnace at 600 °C for 60 min under protection of argon gas and then quickly handled into a forging mold with a cavity of 60 mm \times 60 mm \times 10 mm and forged at pressure of 160 MPa. The employed punch speed, pressure holding time

and mold temperature were 60 mm/s, 20 s and 200 °C respectively. Repeating the above experiment procedures, some thixoformed plates of $Mg_2Si_p/AM60B$ in situ composite were obtained.

The employed solution temperatures were 400, 415, 430, 445 and 460 °C, and the solution durations were ranged from 0 min to 36 h. All the plates were quickly water-quenched after solution treatment. Specimens with dimensions of 8 mm \times 8 mm \times 10 mm were cut from the plates for microstructure observation and hardness examination. The metallographic specimens were polished and etched using an aqueous solution contained 60 vol.% ethylene glycol, 20 vol.% acetic acid and 5 vol.% HNO₃ and then observed on an optical microscope (OM) and a scanning electron microscope (SEM). Subsequently, they were also analyzed using a D/MAX-2400 X-ray diffractometer (XRD). Three images with magnification of 100 times for each specimen were examined by the Image-Pro Plus 6.0 software and the average values were taken as grains size and Mg_2Si particles size. The hardness was examined using a HBRVU-187.5 Brinell–Rockwell–Vickers optical hardness tester. Tensile testing bars were machined from the center region of each plate and the dimensions of them were shown in Fig. 1. The tensile testing was carried out on a universal material testing machine in a nominal strain rate of 0.5×10^{-3} mm s⁻¹. The average of at least five testing values was taken as the mechanical properties.

3. Results and discussion

3.1. As-thixoformed microstructure

To clarify the microstructural evolution during solutionizing, the microstructure of the as-thixoformed $Mg_2Si_p/AM60B$ composite should be first verified. Fig. 2(a) shows that its microstructure is consisted of globular primary α -Mg particles (a solid solution of Al and Mn in Mg), Mg_2Si particles and net-like secondarily solidified structures (SSSs). The Mg_2Si particles distribute both in the net-like SSSs (marked by arrows in Fig. 2(a)) and the pool-like SSSs within the primary particles (marked by arrows A in Fig. 2(b)). In addition, the pool-like SSSs within the primary particles must generate from the original liquid pools. At the initial stage of partial remelting at 600 °C, many eutectic phases would not dissolve into the primary dendrites in time and been trapped within the dendrites. Subsequently, the entrapped eutectic phases melted and formed liquid pools within the primary particles. Moreover, the formation of liquid pools within solid primary particles is a common phenomenon for a semisolid non-dendritic ingot

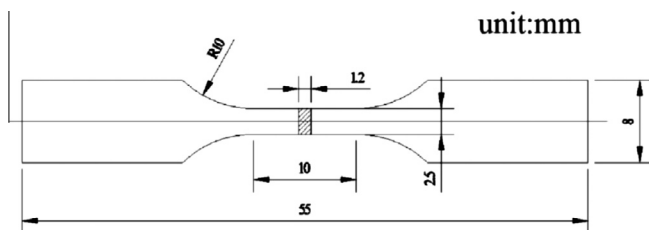


Fig. 1. Schematic of the used tensile testing bar.

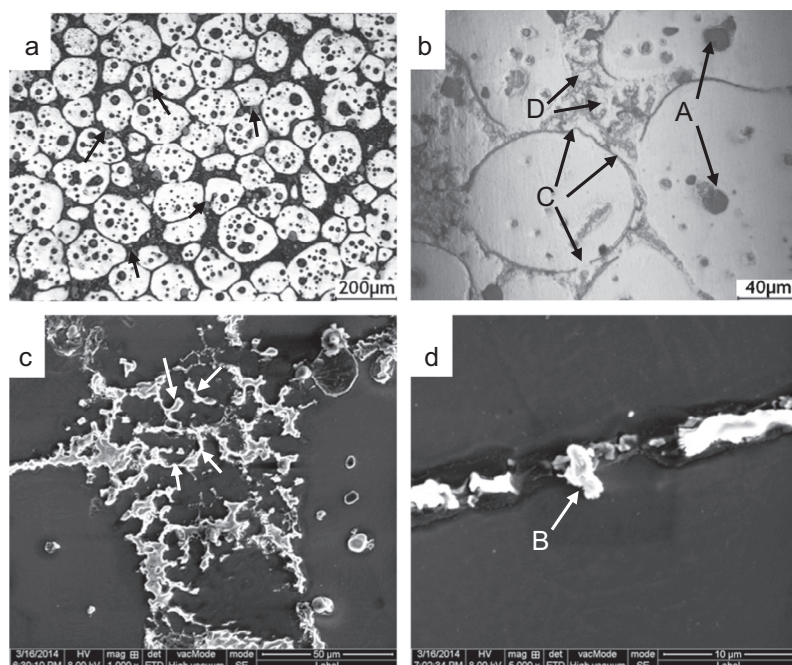


Fig. 2. (a and b) OM and (c and d) SEM images of the as-thixoformed $Mg_2Si_p/AM60B$ composite.

prepared by partial remelting [20–23]. The SSSs are composed of secondarily primary α phases (to differentiate from the primary α phases, the primary α phases solidified from the liquid phase in semisolid state are named as secondarily primary α -Mg phases) and eutectic structures (Fig. 2(b)). And the eutectics are consisted of eutectic α and β -Mg₁₇Al₁₂ phases. In the triangle regions with large area among the primary particles, the eutectic β phases are in skeleton shape in the gaps among the secondarily primary α -Mg dendrites (marked by arrows in Fig. 2(c)), while in the thin regions, the eutectic β phases are in particle or rod-like form (marked by arrows B in Fig. 2(d)). In view of the eutectic morphologies, they should belong to divorced eutectics.

Based on the phase constituents of the composite and the microstructures shown by Fig. 2, the secondary solidification process can be divided into three stages. First, due to the existence of the primary α -Mg particles, the secondarily primary α -Mg phases preferentially directly grow up on the primary α particle surfaces without nucleation, which in order to rapidly decrease the Mg content in the liquid phase close to the primary particles, forming the serrated or annular structures (marked by arrows C in Fig. 2(b)). Then, nucleation occurs and some of fine equiaxed α dendrites form (marked by arrows D in Fig. 2(b)) in the triangle regions with large area when the liquid supercooling degree is up to the requirement of heterogeneous nucleation. Finally, the residual liquid phase solidifies through the eutectic reaction of $L \rightarrow \alpha + \beta$, completing the solidification. It can be expected that the eutectic α -Mg phases also preferentially attach on the secondarily primary α -Mg phases without renucleation and only the eutectic β phases are left in the intergranular regions, forming the devoiced eutectic structures.

3.2. Effect of solution time on microstructure and mechanical properties

3.2.1. Effect on microstructure

Fig. 3 presents the OM images of the thixoformed Mg₂Si_p/AM60B composites solutionized at 415 °C for different time. It indicates that the microstructure changes dramatically during the initial stage of the solution treatment. A large number of fine particle grains have appeared within the original secondarily solidified regions and the outlines of the primary particles become indistinct after being heated for 0.5 h (Fig. 3(a)). That is to say, the fine particle grains originate from the SSSs. They are named secondarily primary grains to differentiate from the primary particles. As mentioned above, the SSSs are composed of fine equiaxed secondarily primary dendrites and interdendritic eutectics. The eutectics dissolve into the secondarily primary dendrites during solutionizing, which leads the secondarily primary dendrites to turn into small particle grains. As the time increases, both the primary particles and the secondarily primary grains gradually coarsen and connect with each other. The number of the fine secondarily primary grains sharply decreases (comparing Fig. 3(a) and (b)). With increasing the time to 2 h, the intergranular fine grains almost disappear. Simultaneously, the spherical morphology of the primary particles almost transform into polygonal form (Fig. 3(c)). As the time further increases, the polygonal grains gradually coarsen and it is difficult to distinguish these two kinds of grains (comparing Fig. 3(c), (d) and (e)). It can be expected that the driving force of the coarsening is the decreasing interfacial energy. According to the microstructural evolution mentioned above, the initial 2 h of the solution treatment is taken as the first

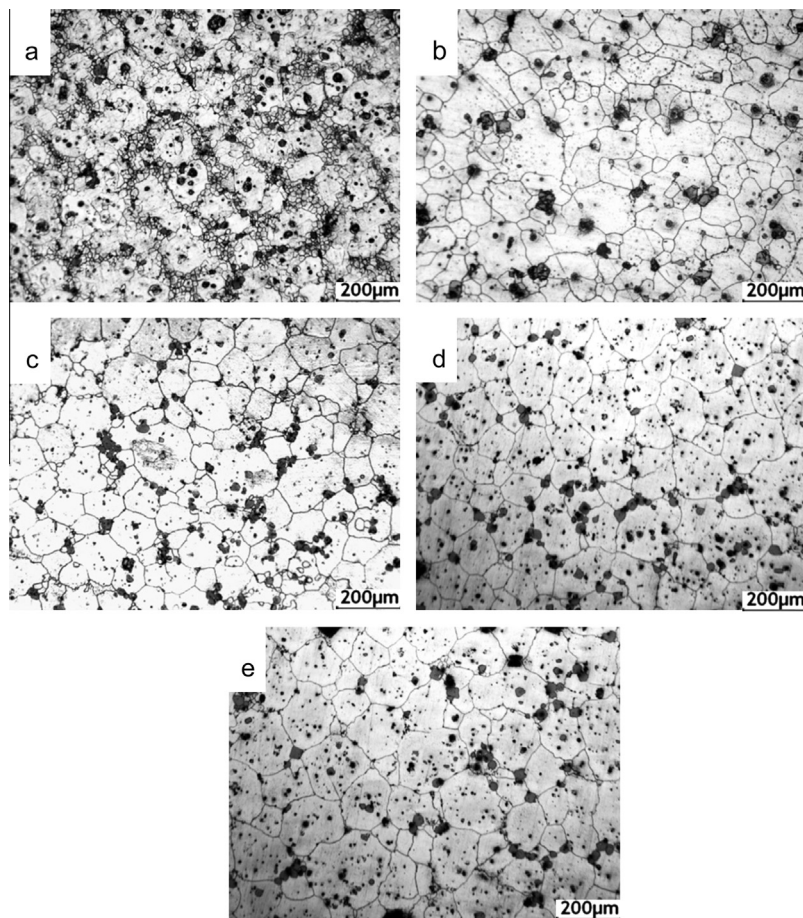


Fig. 3. OM images of the thixoformed Mg₂Si_p/AM60B composites solutionized at 415 °C for (a) 0.5 h, (b) 1 h, (c) 2 h, (d) 6 h and (e) 36 h respectively.

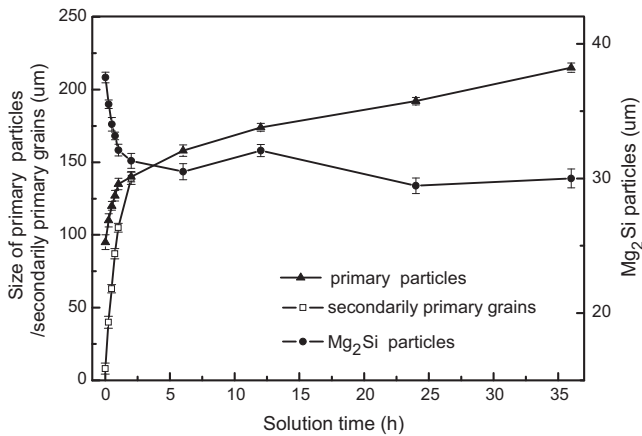


Fig. 4. Variations of the two kinds of α -Mg grains size and Mg_2Si particles size with the solution time at 415 °C.

stage in which the eutectic β phases rapidly dissolve and it results in the fast coarsening of primary α -Mg particles and secondarily primary α -Mg grains. The subsequent solution treatment is considered to be the second stage and the main phenomenon in this stage is the normal growth of the two kinds of grains driving by minimizing interfacial energy. In addition, some black particles always exist regardless of the solution progress (Fig. 3(a)–(e)). Considering the phase constituents of the composite, it can be suggested that these particles are Mg_2Si phases. They are relatively stable during the solution treatment and almost not dissolve.

The variations of the primary particles size, secondarily primary grains size and Mg_2Si particles size can be more clearly seen from the quantitative examinations as shown by Fig. 4. Based on the above discussion, the primary particles and the secondarily primary grains cannot be differentiated after being heated for 2 h. So both of them are taken as one kind of grains and named α -Mg grains after this. Fig. 4 shows that both the coarsening speeds of both the α -Mg grains are quite high during the stage of 0–2 h and then are obviously decreased, which further demonstrates the two stages of the microstructural evolution. However, Fig. 4 also shows that the Mg_2Si particles size relatively obviously decreases in the first stage and then basically keeps constant in the second stage.

According to the Mg–Al equilibrium binary phase diagram [24], β phases will dissolve into the primary α phases to form Al supersaturated α -Mg solid solution during solid solution treatment. As known from the above discussion, the small-sized Al-rich β phases distribute between the Mg-rich α -Mg phases (Fig. 2(c)). Therefore, large interfacial area of α/β exist in the composite. During solutionizing, the eutectic β phases in the SSSs will dissolve into their surrounding α -Mg phases (including the eutectic α -Mg phases, secondarily primary α -Mg grains and primary α -Mg particles)

and the dissolution speed is very rapid. The rapid dissolution leads the fine secondarily primary dendrites to evolve into the fine particle grains. More importantly, the fast coarsening of the primary particles and secondarily primary grains then occur also due to the rapid dissolution of the eutectic β phases between them. When the two kinds of gains, especially the fine particle grains, grow up to a degree, the normal growth with relatively low speed will dominate.

The present result indicates that there are three coarsening behaviors, namely the mergence between the secondarily primary grains, the mergence between the secondarily primary grains and primary particles and the mergence between the primary particles. Fig. 5(a) shows that a primary particle has swallowed the fine secondarily primary grains around it in region A and a relatively large-sized secondarily primary grain has also swallowed a small-sized one in region B. The result indicates that these two kinds of coarsening behaviors mainly occur in the first stage of the solution treatment. However, the mergence between the primary particles mainly operates in the second stage. Fig. 5(b) shows that the grain boundaries marked by arrows C and D will disappear during the subsequent heat treatment, which demonstrates that the mergence of the large-sized primary particles surely carry out during the long-time solution treatment. According to the Mg–Si binary equilibrium phase diagram [25], the solubility of Si element in the α -Mg phase slightly increases as the temperature rises. So it can be suggested that a little Si element will dissolve into the α -Mg matrix during solutionizing, resulting in the decreases of the Mg_2Si particles size. As the solid solution treatment continues, Si element reaches its saturability, and thus the Mg_2Si particles size basically keeps constant. More importantly, it can be found that the number of the Mg_2Si particles entrapped within the α -Mg grains increases as the time is prolonged (Fig. 3). This further demonstrates the grain coarsening through mergence.

Based on the above discussion, it can be concluded that the growth of the primary particles and secondarily primary grains are mainly controlled by the dissolution of β phases during the first stage and then their growth carry out through mergence of the neighboring grains. This result is consistent with the previous studies on thixoformed AM60B alloy [26,27].

In addition, it can be found that the solution process of the thixoformed composite is slower than that of the thixoformed AM60B alloy. The fast coarsening of the two kinds of grains of the thixoformed $Mg_2Si_p/AM60B$ composite have completed within 2 h, but that of the thixoformed AM60B alloy only need 1 h [26]. The difference between these two materials is that the composite has Mg_2Si particle reinforcements. As an intermetallic compound, the thermal conductivity of Mg_2Si phases is lower than that of magnesium alloy [27], and thus, the temperature rising of the composite is slower than that of the alloy. The result shows that it needs about 15 min for the composite to reach 415 °C while only 10 min for the alloy. So the solution speed of the composite is

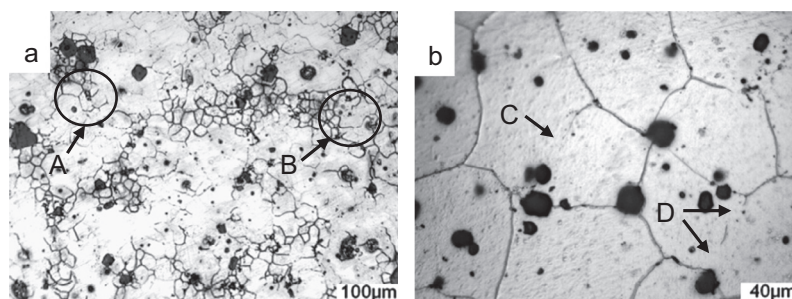


Fig. 5. OM images of the thixoformed $Mg_2Si_p/AM60B$ composites solutionized at 415 °C for (a) 0.5 h and (b) 12 h.

slowed. In addition, homogenization, which simply should be measured from the time when the temperature is reached is also slowed.

In order to have a deeper understanding of the grain coarsening, the size variation curves are fitted to estimate which function is obeyed as shown by Fig. 6. The results indicate that the growth models of the two kinds of grains well obey the traditional growth model in the first stage [28]:

$$\bar{d} = K(Dt)^n \quad (1)$$

where d is grain size, k is growth constant and D is diffusion coefficient. The D can be obtained by

$$D = \alpha \cdot D_0 \cdot \exp(-Q/RT) \quad (2)$$

where D_0 is diffusion constant related to temperature T , Q is activation energy and is 160.5 kJ/mol [29] and α is correction coefficient, which can be expressed as

$$\alpha = \frac{\bar{B} - \bar{B}_{\text{Mg}_2\text{Si}}}{\bar{B}} \quad (3)$$

where $\bar{B}_{\text{Mg}_2\text{Si}}$ is the volume of grain boundaries occupied by the Mg_2Si particles; \bar{B} is the total volume of grain boundaries. The fitting result shows that the α value is equal to 0.91 and the values of n for the secondarily primary grains and the primary particles are equal to 0.5 and 0.1 respectively. It is known that the coarsening is mainly result from phase transformation if the n value is less than 2 [25]. So it can be concluded that the growth of the two kinds of grains are mainly controlled by the dissolution of β phases at the first stage.

The results show that the growth in the second stage is quite different and it obeys the following equation:

$$\bar{d}_t^n - \bar{d}_0^n = kt \quad (4)$$

where \bar{d}_0 and \bar{d}_t are the initial and final grain size respectively, t is the solution time, k and n are constants related to diffusivity, interfacial energy and solute concentration. The present results indicate that the n value is 3.86, larger than 3, but lower than 4. The existing theory indicates if the coarsening is controlled by diffusion along grain boundaries, diffusion through the lattice and diffusion across a particle/matrix interface, n can be 4, 3, and 2 respectively [30]. So it can be concluded that the grain coarsening of the thixoformed composite obeys a mixture model including atom diffusion along grain boundaries and through crystal lattice. And the grain coarsening of the thixoformed alloy is similar to that of the thixoformed composite [27]. The fitting results show that the diffusion coefficient D is smaller than that of the thixoformed AM60B alloy. This further explains the reason why the solution process is slower than that of the alloy. Namely, the Mg_2Si particles play an obstacle role in the solution treatment of the thixoformed composite.

From the discussion above, it can be concluded that the microstructural evolution of the thixoformed $\text{Mg}_2\text{Si}_p/\text{AM60B}$ composite during solutionizing at 415 °C can be divided into two stages. The first stage (0–2 h) refers to the rapid dissolution of eutectic β phases and the resulted fast coarsening of the primary α -Mg particles and secondarily primary α -Mg grains. The second stage (after 2 h) is regarded as the normal growth of the two kinds of grains with relatively low speed. Due to the mergence, the number of the entrapped Mg_2Si particles within the α -Mg grains increases as the solution time prolongs. The fitting results indicate that the growth of the primary particles and secondarily primary grains are mainly controlled by the dissolution of β phases and obeys the traditional growth model during the first stage. But the coarsening obeys a mixture model including atom diffusion along grain boundaries and through crystal lattice in the second stage. During solutionizing, a little Si element will dissolve into the α -Mg matrix which resulting in the decreases of the Mg_2Si particles size. As the solid solution treatment continues, Si element reaches its saturability, and thus the Mg_2Si particles size basically keeps constant. In addition, the solution process of the thixoformed composite is slower than that of the thixoformed AM60B alloy.

3.2.2. Effect on mechanical properties

Fig. 7 gives the variations of mechanical properties of the thixoformed $\text{Mg}_2\text{Si}_p/\text{AM60B}$ composite with the solution time. It presents that the ultimate tensile strength (UTS) and elongation rapidly increase during the first stage and then slowly increase to peak values of 271 MPa and 16.7% respectively at 6 h (Fig. 7(a)). Subsequently, both the UTS and elongation continuously decrease. As the solution proceeds for 36 h, the UTS and elongation decrease to 178 MPa and 5.1% respectively. The hardness decreases quickly during the first stage and then slowly reduces (Fig. 7(b)). The result indicates that the solution process can also be divided into two stages similar to that from the above microstructural evolution. The mechanical properties change sharply in the first stage (0–2 h) and then the change rates become slow in second stage.

It is well known that the β phase is a hard and fragile metallic compound ($\text{Mg}_{17}\text{Al}_{12}$) phase [31]. So it can be expected that its dissolution into the α -Mg phases must cause the hardness to decrease. Based on this standpoint, it is easy to understand the rapid decreases of the hardness in the first stage. Subsequently, the main thing is the grain coarsening with relatively slow speed. This should be the only reason that leads the hardness to slowly decrease during the second stage.

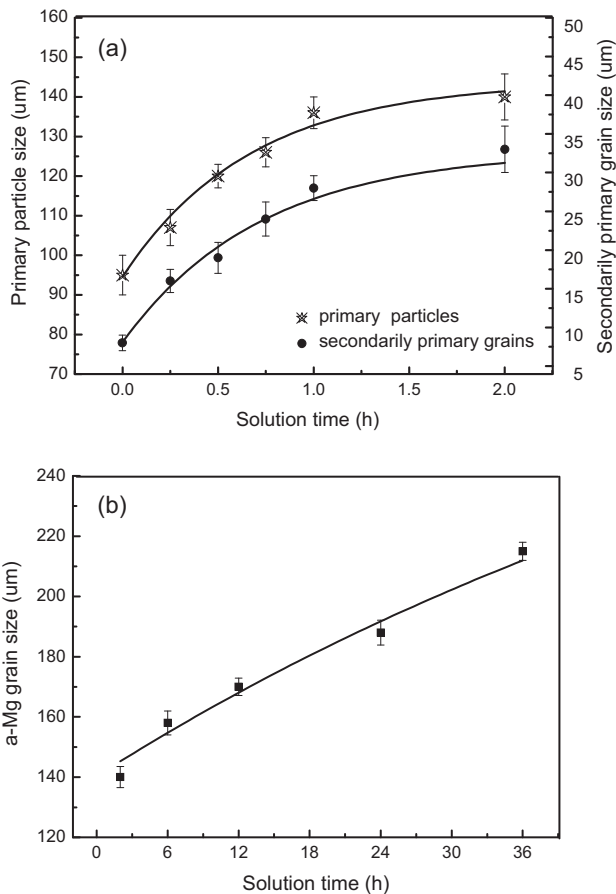


Fig. 6. Variations of the grains size with the solution time at 415 °C: (a) 0–2 h and (b) 2–36 h.

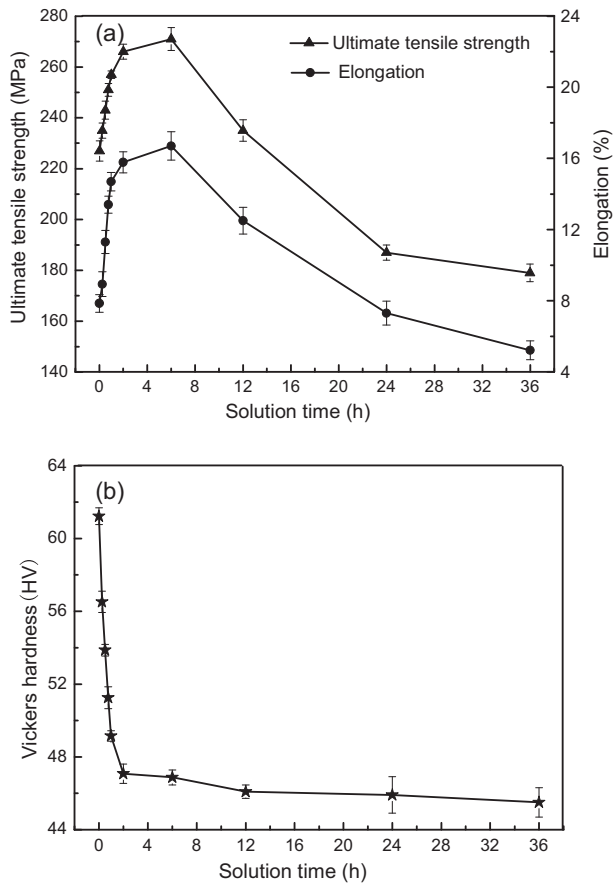


Fig. 7. Variations of (a) tensile properties and (b) hardness of the thixoformed $Mg_2Si_p/AM60B$ composites with the solution time at 415 °C.

Fig. 8 shows the fractographs of the thixoformed composite solutionized for different time. It indicates that the microstructure on the fracture surface of the as-thixoformed composite is quite compact and there are no apparent shrinkage porosities (Fig. 8(a)). It is well known that thixoforming can significantly reduce or eliminate porosities and gas pores [10,11]. The present result indicates that the mold filling ability and feeding ability to solidification shrinkage of the semisolid $Mg_2Si_p/AM60B$ composite ingot are relatively well under the employed forming conditions. Even if there are some small-sized porosities or gas pores, they should exist in the SSSs. In addition, oxide inclusions (if they exist) are also in these structures. As described above, the eutectic β phase in the SSSs (Fig. 2) is a hard and brittle phase. The large-sized β phases or large amount of β phases in the as-thixoformed composite are always harmful to tensile properties [32]. Namely, the SSSs between the primary particles are the weak points of this composite, and thus cracks preferentially initiate in and then develop along these structures during tensile testing (Fig. 9(a)). This phenomenon is quite common and can be frequently found in other thixoformed alloys [22,23,33,34]. Crack propagation across the primary particles can be occasionally seen, and cracks always develop across the pool-like SSSs within the α -Mg particles under this condition (marked by arrows in Fig. 9(a)). It can be expected that the shrinkage of the liquid pools within the primary particles cannot be feed during solidification and porosities always exist in the resulting pool-like SSSs. That is to say, the pool-like SSSs are also the weak points of this composite. So cracks will develop across these sites if they propagate across the primary particles. In general, the fracture of the as-thixoformed composite obeys intergranular mode (Fig. 9(a)).

The rapid dissolution of the eutectic β phases during the first stage must result in four effects. The first is the rapid decreases of the β phase harmfulness. The second is the increased solid solution strengthening. The third is the composition homogenization and the fourth is the coarsening of α -Mg grains. The former three effects must enhance the tensile properties (Fig. 7(a)). It must be noted that the enhancement of the elongation should be mainly attributed to the improvement of deformation coordination resulted from the first and third effects. But the grain rapid coarsening should decrease the tensile properties. The present result implies that the negative influence from the grain coarsening is smaller than the positive influence from the former three effects. The general fracture surface morphologies of the composites solutionized for different durations during this stage are quite similar. But careful observation shows that both the number and size of dimples (marked with arrows in Fig. 8(b)–(d)) increase and plastic deformation characteristics become more and more obvious as the solution time increases (comparing Fig. 8(b)–(d)). This is just consistent with the improvement of elongation with the solution time.

The SSSs rapidly evolve into fine polygonal particles with the rapid decrease of eutectic β phases during the initial stage of solution treatment, but the defects such as porosities and inclusions still exist in these regions. So cracks frequently propagate along the fine polygonal secondary primary grain regions, only occasionally develop across the large primary particles (marked by arrows in Fig. 9(b)). As the solution treatment proceeds, the difference between these two kinds of grains gradually becomes smaller and smaller due to the rapid coarsening of the fine secondary primary grains and both of them become into the large polygonal particles. More importantly, the grain bonding strength in the SSSs regions become strong due to the decrease of β phase amount and size. So the probability of crack propagation across the polygonal grains gradually becomes larger and larger (comparing Fig. 9(b) and (c)). Till to be solutionized for 2 h, cracks basically completely develop across the polygonal grains (Fig. 9(d)). That is the fracture regime changes from intergranular mode to transgranular mode after being solutionized for 2 h. The contributions of solid solution strengthening and composition homogenization to tensile properties are well known, so they are not necessary to be discussed in this work. Therefore, it can be concluded that the significant improvement of the tensile properties during the first stage should be attributed to the rapid decrease of β phase harmfulness, increased solid solution strengthening and composition homogenization. The β phase dissolution enhances the grain bonding strength and results in the change of the fracture regime from intergranular mode to transgranular mode.

When the composite is solutionized for 2 h, the XRD result indicates that the β phases have not completely dissolved (Fig. 10) although no obvious SSSs can be found (Fig. 3(c)). The residual β phases further decrease as the solution proceeds and almost disappear after being treated for 6 h (Fig. 10). So the tensile properties still continuously increase and reach the peak values at 6 h (Fig. 7(a)). Of course, the further increased solid solution strengthening and composition homogenization also play important roles to the tensile properties. The dimple size on the fracture surface further become large and plastic deformation characteristics evolve more obvious (comparing Fig. 8(d) and (e)). The fracture still maintains the transgranular regime (Fig. 9(e)). Compared with those of the as-thixoformed composite, the UTS and elongation of the solutionized composite are increased by about 20% and 115% respectively, being up to 271 MPa and 16.7%.

After this, the only phenomenon is the grain coarsening (Figs. 3 and 4) and the only positive effect on tensile properties is the continued composition homogenization. But this effect is very limited. So the tensile properties then continuously decrease after 6 h. In addition, the deformation coordination of grains themselves

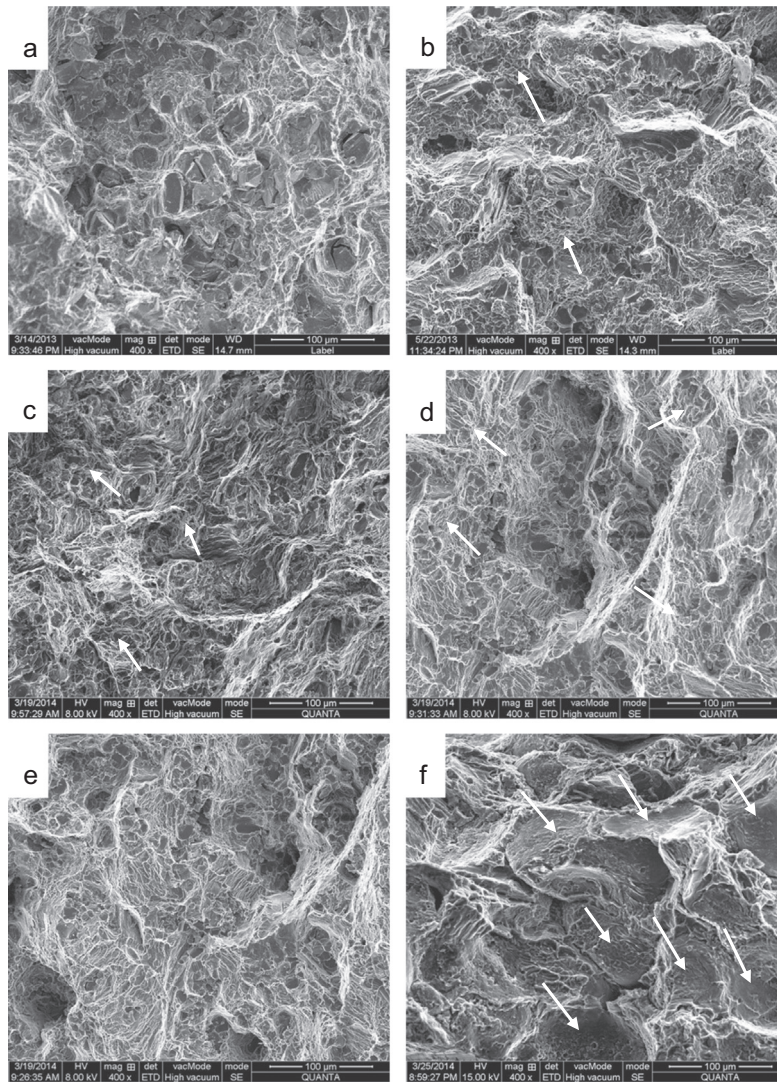


Fig. 8. Fractographs of the composites solutionized at 415 °C for (a) 0 min, (b) 0.5 h, (c) 1 h, (d) 2 h, (e) 6 h and (f) 36 h respectively.

should be improved due to the composition homogenization, but that of the whole composite may be deteriorated due to the grain coarsening. Furthermore, the differences in the different regions within a grain and in the different grains should become smaller and smaller as the composition homogenization continuously proceeds. The large difference should exist between the grain boundaries and grain insides, and the grain boundaries gradually turn into weak points again. So crack propagation path then gradually evolves from across grains into along grain boundaries. Fig. 9(f) shows that cracks develop along the grain boundaries in some local zones (marked by arrows) when the composite is solutionized for 36 h. It can be expected that the debonding of the neighboring polygonal grains will generate large smooth planes on the fracture surface as shown by arrows in Fig. 8(f). Fig. 8(f) shows that there are lots of large smooth planes and this means that the intergranular mode occupies a large portion for this composite. That is to say, there is a tendency that the fracture regime changes from transgranular mode to intergranular mode after being solutionized 6 h. In general, the mechanical properties of material fractured in transgranular mode are higher than those fractured in intergranular mode. Based on this standpoint, the variations of the tensile properties after 6 h can be well interpreted.

As the reinforcements of the composite, the behaviors of the Mg_2Si particles are necessary to be investigated during tensile testing. According to the existing investigation on traditional casting $Mg_2Si_p/AM60B$ in situ composite [35], the strengthening mechanisms of the Mg_2Si particles can be summarized as two aspects, the load transmission effect and the dislocation strengthening. Fig. 11 indicates that two kinds of behaviors can be concluded for the Mg_2Si particles. One is the interface debonding of Mg_2Si_p /matrix (Fig. 11(a) and (b)) and the other is the fracture of the Mg_2Si particles (Fig. 11(c) and (d)). As shown in Fig. 11(a), the Mg_2Si phase is a kind of polyhedral particle with sharp edges and corners. In addition, the interfaces of $Mg_2Si/\alpha-Mg$ are not coherent since their crystal structures and lattice constants are different [36]. During tensile testing, stress concentration will generate at the interfaces due to the load transmission effect. The existing investigation indicates that this stress is 2–4 times higher than that of the $\alpha-Mg$ matrix [37]. As the stress exceeds the interface bonding strength, the interface will debond. In addition, the matrix around the Mg_2Si particles is relatively softer and will plastically deform while the Mg_2Si particles do not deform. This can also generate high stress concentration in the $\alpha-Mg$ matrix around the Mg_2Si particles. It can be expected that the Mg_2Si particles will

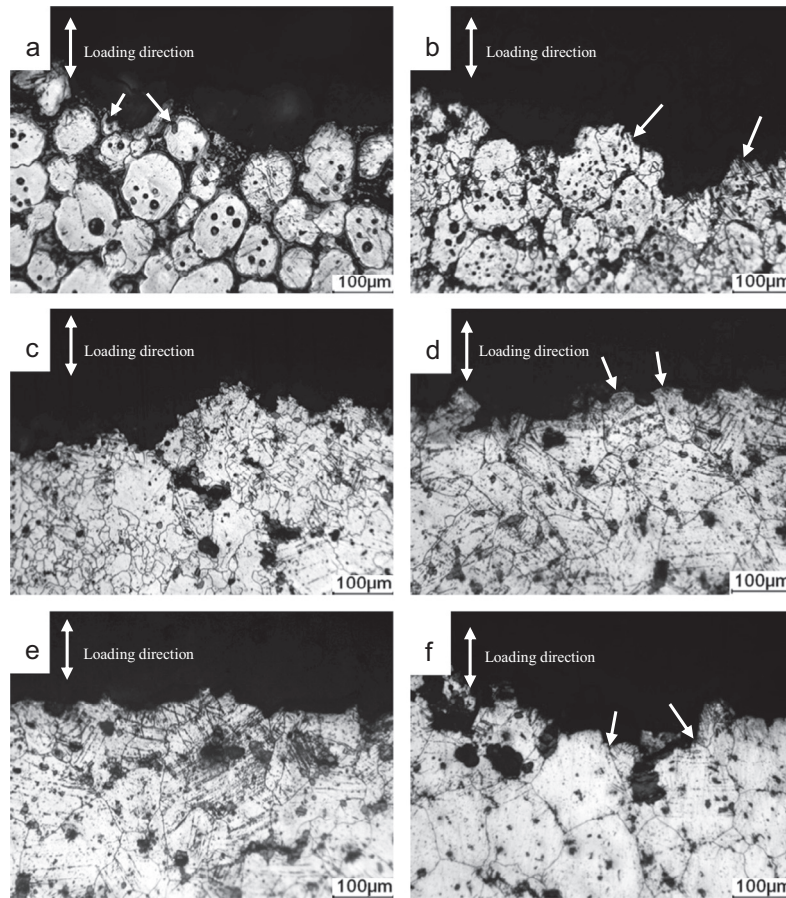


Fig. 9. Side-views of fracture surfaces of the composites solutionized at 415 °C for (a) 0 min, (b) 0.5 h, (c) 1 h, (d) 2 h, (e) 6 h and (f) 36 h respectively.

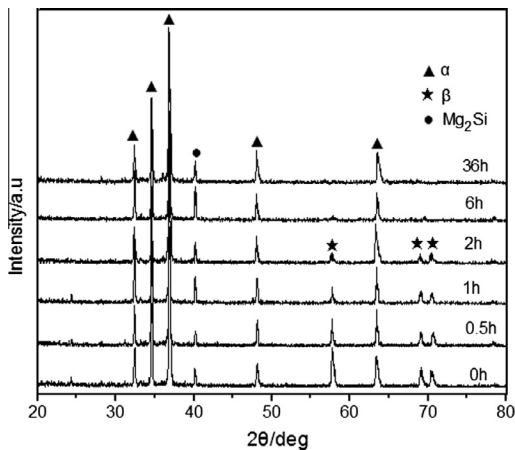


Fig. 10. X-ray diffractograms of the composites solutionized at 415 °C for different time.

break as the stress is up to a given degree. There are two broken ways for the Mg_2Si particles, either fractured parallel to the fracture surface of the whole specimen (Fig. 11(c)), or broke into several parts (Fig. 11(d)). The broken ways should be determined by the local stress conditions during tensile testing. As discussed above, the tensile properties of the preferred heat-treatment is higher than that of the as-thixoformed. And thus, the high stress concentration regions around the Mg_2Si particles are also higher than that of the as-thixoformed. And the result indicates that the broken behavior of Mg_2Si particles of heat-treatment is more and

the interface debonding of Mg_2Si_p /matrix of as-thixoformed is more.

The present result indicates that the debonded and broken Mg_2Si particles are always located in the bottom of dimple-like pits (Fig. 11). This implies that the failures such as the debonding and break are earlier than the fracture of the surrounding matrix. So the Mg_2Si particles are also the weak points of the composite. But their failures are later than those of the other sites, such as the defects of porosities, gas pores, inclusions and eutectic β phases, and thus the mechanical properties, especially the yield strength and hardness are always significantly improved compared with those of the matrix alloy [35].

In summary, the solution time has large effects on mechanical properties of the thixoformed Mg_2Si_p /AM60B composite. The hardness sharply decreases due to the rapid dissolution of the eutectic β phases during the first stage (0–2 h) and then slowly reduces during the second stage because of the grain coarsening. The tensile properties first increase as the time increases from 0 h to 6 h and then decrease. The increase of the tensile properties should be attributed to the decrease of β phase harmfulness, increased solid solution strengthening and composition homogenization. The grain bonding strength is continuously improved due to the decrease and final disappearance of the β phases and thus the fracture regime turns into complete transgranular form. The decrease of tensile properties after 6 h is contributed to the grain coarsening. Simultaneously, the features about the grain boundaries taken as weak points become obvious due to the composition homogenization and the fracture then changes from transgranular regime to intergranular regime. The highest tensile properties of UTS of 271 MPa and elongation of 16.7% are obtained at the

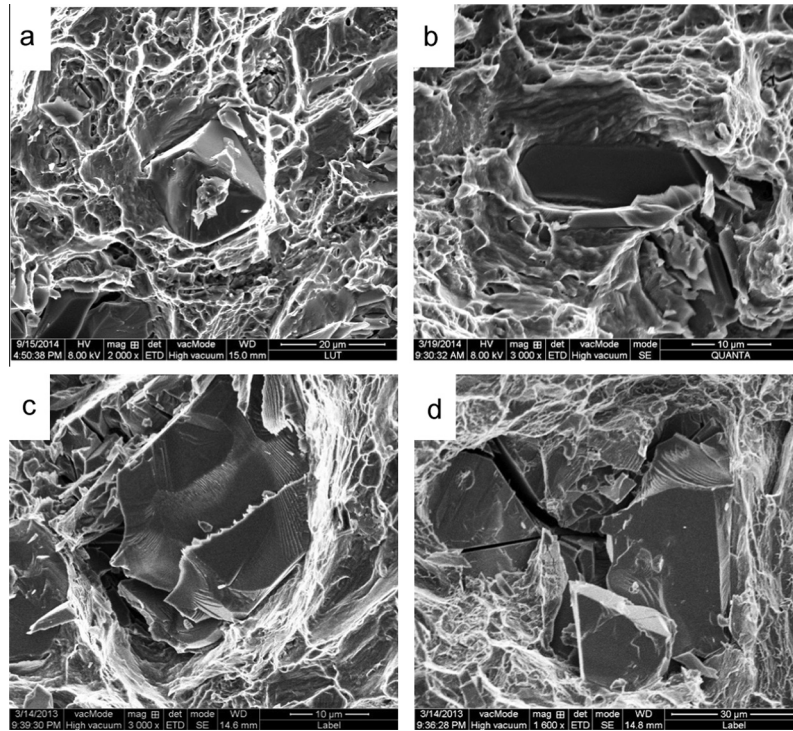


Fig. 11. Fractographs of (a and b) as-thixoformed $Mg_2Si_p/AM60B$ composite; (c and d) the thixoformed composite solutionized at $415\text{ }^\circ\text{C}$ for 6 h.

solution time of 6 h, increased by about 20% and 115% respectively compared with those of the as-thixoformed composite. Two kinds of behaviors can be found for the Mg_2Si particles in the composite. One is the interface debonding of $Mg_2Si/\alpha\text{-Mg}$ and the other is the break of Mg_2Si particles.

3.3. Effect of solution temperature on microstructure and mechanical properties

3.3.1. Effect on microstructure

The above section indicates that the thixoformed $Mg_2Si_p/AM60B$ composite under the solution time of 6 h has the highest tensile properties. Thus, this solution time was employed to investigate the effects of solution temperature on the microstructure and mechanical properties. Fig. 12 shows the optical microstructures of the composites solutionized at different solution temperatures. Together with Fig. 3(d), it presents that all of the microstructures are composed of relatively uniform polygonal particle grains and Mg_2Si particles, regardless of the solution temperature. That is to say, no SSSs or eutectic β phases can be seen in these composites and the grains originated from the primary particles and SSSs are not distinguished. So it can be suggested that all of the composites, even that solutionized at $400\text{ }^\circ\text{C}$, the lowest employed temperature in this work, have entered into the second stage. More importantly, it can be found that the polygonal grains obviously grow up as the temperature rises. Simultaneously, the size of Mg_2Si particles slightly decreases. The size changes of the polygonal grains and Mg_2Si particles in such ways can be more obviously seen in Fig. 13. In addition, the number of the Mg_2Si particles entrapped within the grains also increases.

It is well known that the solution temperature plays a direct effect on the atom diffusion. The higher the solution temperatures, the easier and faster the atoms diffuse. And thus, the faster the solution process and the more serious the grain coarsening. It is just the serious grain coarsening that the more interdendritic Mg_2Si particles are entrapped within the coarsened grains. This

result also indicates that the coarsening carries out through merger of the neighboring grains. In addition, the solubility of Si atoms in the $\alpha\text{-Mg}$ phases increase as the temperature rises according to the Mg–Si binary phase diagram [25]. So it can be expected that more amount of Si atoms will dissolve into the $\alpha\text{-Mg}$ grains, which results in the decrease of the Mg_2Si particles size.

3.3.2. Effect on mechanical properties

Fig. 14 gives the variations of mechanical properties of the thixoformed $Mg_2Si_p/AM60B$ composite with the solution temperature. It indicates that the tensile properties increase as the temperature rises, and reach the peak values under $430\text{ }^\circ\text{C}$, UTS of 277 MPa and elongation of 18.5%, increased by about 20% and 135% respectively compared with those of the as-thixoformed composite. Then the tensile properties continuously decrease. The hardness always decreases with the temperature.

As mentioned above, all of the composites, even that solutionized at $400\text{ }^\circ\text{C}$, have entered into the second stage and the main phenomenon in this stage is the coarsening of the $\alpha\text{-Mg}$ grains. In addition, composition homogenization should further proceed. The XRD result indicates that there are still a few β phases existing in the composite solutionized at $400\text{ }^\circ\text{C}$ although no SSSs can be seen, and these phases have completely disappeared till the temperature rises to $430\text{ }^\circ\text{C}$ (Fig. 15). So the solid solution strengthening is also enhanced as the temperature rises from $400\text{ }^\circ\text{C}$ to $430\text{ }^\circ\text{C}$. The present result indicates that the positive influence to tensile properties from the decrease of β phase harmfulness, enhanced solid solution strengthening and composition homogenization is larger than the negative influence from the grain coarsening. This should be the main reason that the tensile properties increase within the temperature range of $400\text{--}430\text{ }^\circ\text{C}$. When the temperature exceeds $430\text{ }^\circ\text{C}$, the positive influence from the composition homogenization is smaller than the negative influence from the grain coarsening. So the tensile properties then decrease. But all of the three changes (the decrease of β phases, composition

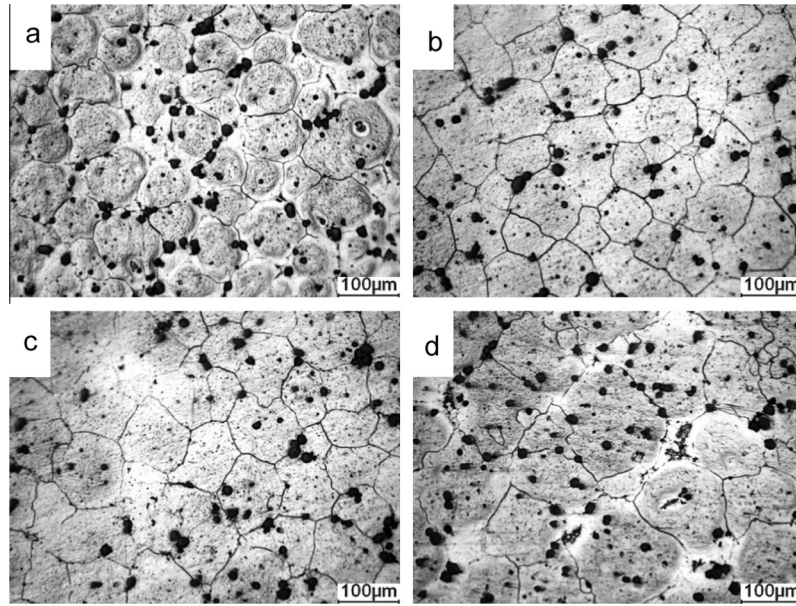


Fig. 12. OM images of the thixoformed $Mg_2Si_p/AM60B$ composites solutionized for 6 h at (a) 400 °C, (b) 430 °C, (c) 445 °C and (d) 460 °C respectively.

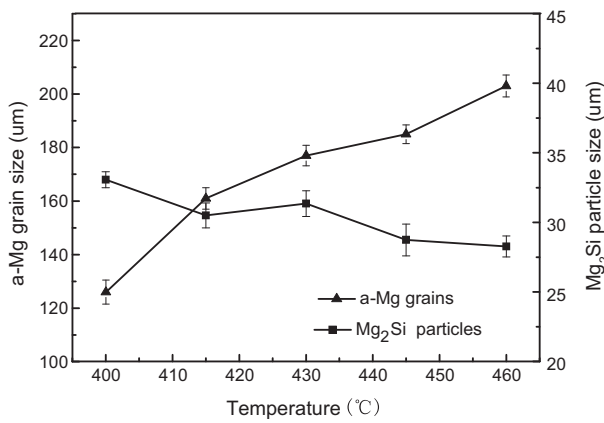


Fig. 13. Variations of the α -Mg grains size and Mg_2Si particles size with the solution temperature.

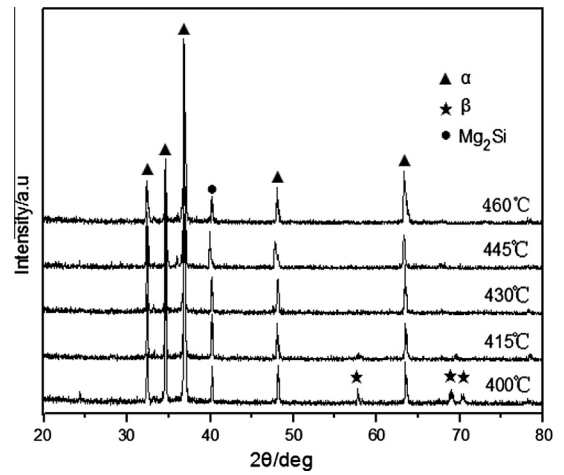


Fig. 15. X-ray diffractograms of the thixoformed composites solutionized for 6 h at different temperatures.

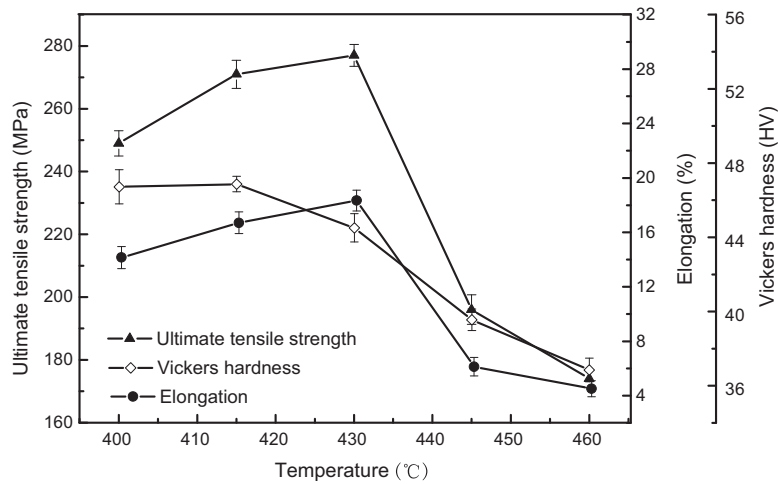


Fig. 14. Variations of mechanical properties of the $Mg_2Si_p/AM60B$ composites with the solution temperature.

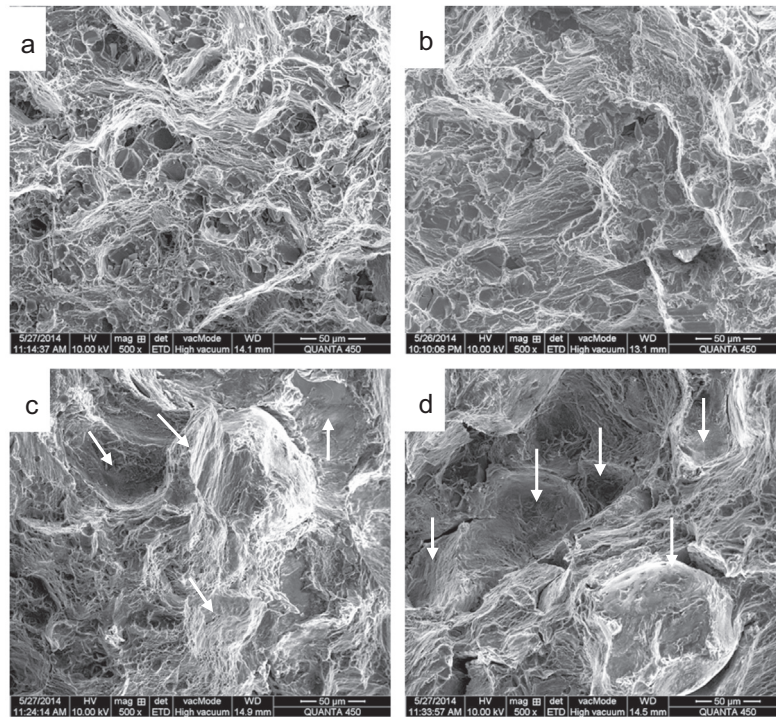


Fig. 16. Fractographs of the thixoformed composites solutionized for 6 h at (a) 400 °C, (b) 430 °C, (c) 445 °C and (d) 460 °C respectively.

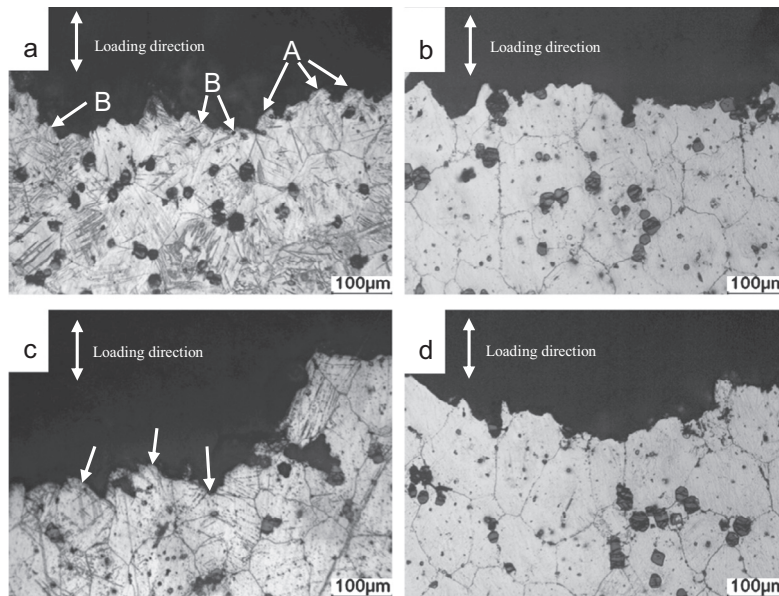


Fig. 17. Side-views of fracture surfaces of the thixoformed composites solutionized for 6 h at: (a) 400 °C, (b) 430 °C, (c) 445 °C and (d) 460 °C respectively.

homogenization and grain coarsening) have no benefit to improving hardness.

Fig. 16 shows the fracture surface of the composite solutionized at 400 °C is quite flat (Fig. 16(a)). It becomes more and more uneven as the temperature rises (comparing Fig. 16(a)–(c)) and some particle- or pit-like structures form on the surface marked by arrows in Fig. 16(c) and (d) when the temperature rises to 460 °C. The side view of the fracture surface of the composite solutionized at 400 °C shows that crack propagate either along the grain boundaries (marked by arrows A in Fig. 17(a)) or across the polygonal grains (marked by arrows B in Fig. 17(a)). These two regimes almost

occupy the same proportion. That is to say, the fracture regime of this composite obeys a mixture of intergranular and transgranular modes. Under this condition, crack propagation does not choose paths during tensile testing, resulting in the plate fracture surface. As the temperature rises, the grain bonding strength becomes stronger due to the decrease of β phases and composition homogenization. The probability of crack propagation across the grains thereby increases and the fracture regime changes from the mixture mode to a completely transgranular mode. Fig. 17(b) shows that the fracture of the composite solutionized at 430 °C almost belongs to completely transgranular mode. Correspondingly, the tensile

properties are also improved (Fig. 14). As the temperature further rises, the main phenomena are the grain coarsening and composition homogenization because the β phases have completely disappeared at 430 °C (Fig. 15). Similarly to the effect of solution time, the compositions, and thus the mechanical properties within a grain and between the grains become smaller. The large difference only exists between the boundaries and the grains. In addition, the deformation coordination becomes poor due to the grain coarsening. So the probability of crack propagation along the grain boundaries increases again (comparing Fig. 17(b)–(d)). The fracture regime turns from the completely transgranular mode (Fig. 17(b)) into a mixture of intergranular and transgranular modes (Fig. 17(c)), and finally into completely intergranular mode (Fig. 17(d)). That is crack propagation preferentially choose along grain boundaries. This selectivity must lead the fracture surface to become uneven. It can be expected that a pit- or a particle-like structure will form on the fracture surface if cracks propagate along a grain's boundary. So some pit- or particle-like structures appear on the fracture surfaces of the composites solutionized at 445 °C and 460 °C and this characteristic becomes more obvious as the temperature rises (comparing Fig. 16(c) and (d)). In view of this fracture regime change, the decrease in tensile properties above 430 °C can also be well interpreted.

Based on the above discussion, it can be summarized that the solution temperature, similar to the solution time, also has obvious effects on microstructure and mechanical properties of the thixoformed $Mg_2Si_p/AM60B$ composite. The α -Mg grains gradually coarsen through mergence as the temperature rises. Simultaneously, the number of the Mg_2Si particles entrapped within the grains increases due to the mergence. The tensile properties first increase as the temperature rises from 400 °C to 430 °C and then decrease. The highest tensile properties, UTS of 277 MPa and elongation of 18.5%, are achieved after solutionized at 430 °C, increased by about 20% and 135% respectively compared with those of the as-thixoformed composite. The increase of the tensile properties is attributed to the decrease of β phase harmfulness, increased solid solution strengthening and composition homogenization. The decrease is resulted from the grain coarsening. Correspondingly, the fracture regime changes from a mixture of intergranular and transgranular modes to transgranular and finally to intergranular again.

4. Conclusions

In this work, the microstructural evolution process of thixoformed $Mg_2Si_p/AM60B$ composite during solution treatment and the corresponding effects on its mechanical properties and fracture regime have been verified. Simultaneously, the effects of solution temperature on microstructure and resulting mechanical properties have also been clarified. The obtained results will play an important role for understanding the heat treatment behaviors and establishing heat treatment techniques of thixoformed $Mg_2Si_p/AM60B$ composite and other magnesium based composites or even other metal matrix composites. The main results obtained from this work are listed as followings.

- (1) The microstructure of the as-thixoformed $Mg_2Si_p/AM60B$ composite is consisted of globular primary α -Mg particles, Mg_2Si particles and secondarily solidified structures. The Mg_2Si particles distribute in both the net-like SSSs and the pool-like SSSs within the primary particles.
- (2) The microstructural evolution includes two stages during solutionizing at 415 °C. The first stage (0–2 h) refers to the rapid dissolution of eutectic β ($Mg_{17}Al_{12}$) phases and the resulted fast coarsening of both the primary α -Mg particles

and secondarily primary α -Mg grains. The coarsening is mainly controlled by the dissolution of β phases and obeys the traditional growth model. The second stage (after 2 h) is regarded as the normal growth of the two kinds of grains with relatively low speed. The growth obeys a mixture model including atom diffusion along grain boundaries and through crystal lattice in the second stage. The microstructure evolution is slower than that of the thixoformed AM60B alloy.

- (3) The Mg_2Si particles size slightly decreases in the first stage due to the dissolution of some Si atoms into the α -Mg grains and then maintain constant. The number of the Mg_2Si particles entrapped within the α -Mg grains continuously increases because of the grain mergence.
- (4) The tensile properties first increase as the time increases from 0 h to 6 h and then decrease. The increase is attributed to the decrease of β phase harmfulness, increased solid solution strengthening and composition homogenization. The decrease is contributed to the grain coarsening. Correspondingly, the fracture regime changes from intergranular regime in turn to transgranular and intergranular regime.
- (5) The hardness sharply decreases due to the rapid dissolution of the eutectic β phases during the first stage (0–2 h) and then slowly reduces during the second stage because of the grain slow coarsening.
- (6) Two kinds of behaviors can be found for the Mg_2Si particles in the composite. One is the interface debonding of Mg_2Si/α -Mg and the other is the broken of Mg_2Si particles.
- (7) The solution temperature also has obvious effects on microstructure and mechanical properties of the composite. As the temperature increases, the α -Mg grains grow up through the mergence of the neighboring grains while the size of Mg_2Si particles also slightly decreases due to the dissolution of more Si atoms. The mechanical properties, fracture regime and number of the Mg_2Si particles entrapped within α -Mg grains also have similar changes to those with the solution time and these changes can also be interpreted using the similar reasons.
- (8) After being solutionized at 430 °C for 6 h, the tensile properties are up to the highest values, UTS of 277 MPa and elongation of 18.5%, increased by about 20% and 135% respectively compared with those of the as-thixoformed composite.

Acknowledgments

The authors wish to express thanks to financial support from the National Basic Research Program of China (Grant No. G2010CB635106), the Program for New Century Excellent Talents in University of China (Grant No. NCET-10-0023) and the Program for Hongliu Outstanding Talents of Lanzhou University of Technology.

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