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Effect of ball milling on microstructural evolution during partial remelting of 6061 aluminum alloy prepared by cold-pressing of alloy powders

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Abstract: The effect of ball milling on the microstructural evolution was investigated during partial remelting of 6061 aluminum alloy prepared by cold-pressing of atomized alloy powders. The results indicate that the microstructural evolution of 6061 aluminum alloy can be divided into three stages, the dissolution of eutectic phases and the coarsening and growth behavior of the resulting grains, structural separation and spheroidization of primary particles, and the final coarsening behavior of the particles. Compared with the alloy without ball milling, ball milling accelerates the first stage of microstructural evolution due to the energy stored in the powders, but the latter two stages are slowed down because of the formation of large-sized powders. Moreover, the finer the as-cold-pressed microstructure is, the smaller and more spherical the primary particles in the final semisolid microstructure are. Furthermore, properly elevating the heating temperature is beneficial for obtaining small and spheroidal particles.

Key words: 6061 aluminum alloy; ball milling; powder thixoforming; partial remelting; microstructural evolution

1 Introduction

Particle-reinforced aluminum matrix composites (AMCs) have been widely used in aerospace and automotive fields because of their attractive properties such as low cost, low density, high stiffness and high strength [1−4]. Many methods have been developed to fabricate AMCs, such as squeeze-cast [5], stir casting [6], friction stir welding [7] and powder metallurgy (PM) [8]. It is known that PM process is popularly used because of the uniform distribution of reinforcements and the flexible design of constituents [1]. But the resultant composites generally have many voids besides high cost [8]. In addition, it is difficult to obtain large-sized components with complex shape [9]. However, thixoforming is not only a relatively simple process, but also can significantly decrease or eliminate voids. In addition, this technology is also suitable to fabricate large-sized parts with complex shape [10,11]. For PM technology, atomization technology has been widely used for preparing small and spheroidal alloy powders. The bulk alloy prepared by pressing of the atomized powders can be used as the feedstock of thixoforming [11]. Therefore, a novel technology named powder thixoforming which combines the advantages of powder metallurgy and thixoforming has been proposed. The blending and pressing steps of PM are applied to preparing the ingots with uniform distribution of reinforcing particles in the matrix, then the ingots are partially remelted and thixoformed. It can be expected that composite components with uniform distribution of reinforcing particles and low or without voids can be obtained by using this technology. Simultaneously, the process is simpler and thus the cost is lower compared with the PM technology.

Similar to the traditional thixoforming, the microstructural evolution during partial remelting of the feedstock is also a crucial topic for the powder thixoforming because this process has great influence on the resultant semisolid microstructure. And the resultant semisolid microstructure plays a decisive role in the mechanical properties of a component. In order to further study the microstructural evolution of composite during

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partial remelting, the microstructural evolution of the matrix alloy should be first clarified. The heat-treatable 6061 alloy has taken as the most commonly used matrix alloy of the AMCs [12,13]. And the author's previous investigation indicated that the 6061 alloy prepared by cold-pressing of the atomized alloy powders can be taken as the start ingots of thixoforming [13,14]. Furthermore, for preparing AMCs, high energy ball milling must be used to disperse the reinforcement particles to uniformly distribute in the matrix alloy powders [14−16]. Thus, a problem is then arising, how does the ball milling affect the semisolid microstructure? Unfortunately, most of the existing investigations focus on the effects of reinforcement size, morphology, distribution and volume fraction [17−19], and there is no investigation to involve this problem.

Therefore, in order to further study the microstructural evolution of the AMCs, the effect of ball milling on the microstructural evolution during partial remelting of 6061 aluminum alloy prepared by cold-pressing of alloy powder was studied in this work.

2 Experimental

The used 6061 aluminum alloy powder was produced by atomization and the average powder particle size was 18.376 μm. The nominal chemical composition (mass fraction) of 6061 alloy is as follows: 0.8%−1.2% Mg, 0.4%−0.8% Si, 0.7% Fe, 0.15%−0.40% Cu, 0.04%−0.35% Cr, 0.15% Mn, 0.25% Zn, 0.15% Ti and balance Al. The solidus and liquidus temperatures of this alloy were 610.2 and 674.6 °C, respectively, measured by a pyris diamond TG/DTA differential thermal analyzer (DTA). The powders were ball-milled using a planetary ball milling machine. The employed mass ratio of ball to powder, milling speed and milling time were 5:1, 100 r/min and 40 min, respectively. The diameter of balls was 9.5 mm. The ball milling parameters are the same as those which achieved homogenous distribution of SiC particles in the matrix powders. After being ball-milled, the average powder particle size was 16.282 μm, which was a little smaller than that of the un-milled powder.

The milled powders were cold-pressed into some specimens with dimensions of *d*22 mm × 5 mm using a pressure of 145 MPa on a jack. Some of them were heated at a semisolid temperature of 660 °C for different durations, ranging from 0 to 60 min, to study the microstructural evolution during partial remelting. The other specimens were heated for 20 min at various semisolid temperatures, 650, 655, and 665 °C, to investigate the effect of reheating temperature on semisolid microstructure.

All of the heated specimens were quickly quenched in a Bi-based low melting point alloy at about 50 °C. One cross-section of each specimen was ground and polished, followed by etching using NaOH aqueous solution. Subsequently, the metallographic microstructure was observed on an optical microscope (OM) and a scanning electron microscope (SEM). The compositions of the primary α(Al) phase of the specimens were examined by energy disperse spectroscopy (EDS). For comparison, the compositions of the specimens without milling were also examined.

3 Results and discussion

3.1 As-cold-pressed microstructure

In order to further study the microstructural evolution during partial remelting, it is necessary to first clarify the as-cold-pressed microstructure. It can be seen that after being cold pressed, both the ball-milled powders and un-milled ones are mechanically consolidated (Fig. 1). Furthermore, as shown by Fig. 1(a), the microstructure of the milled alloy can be divided into two regions: the first is the region of small-sized powers, and the second is the region of large powders surrounded by the long flat-shaped powders. But the microstructure of the un-milled one is regarded as large powders surrounded by small and nearly globular powders.

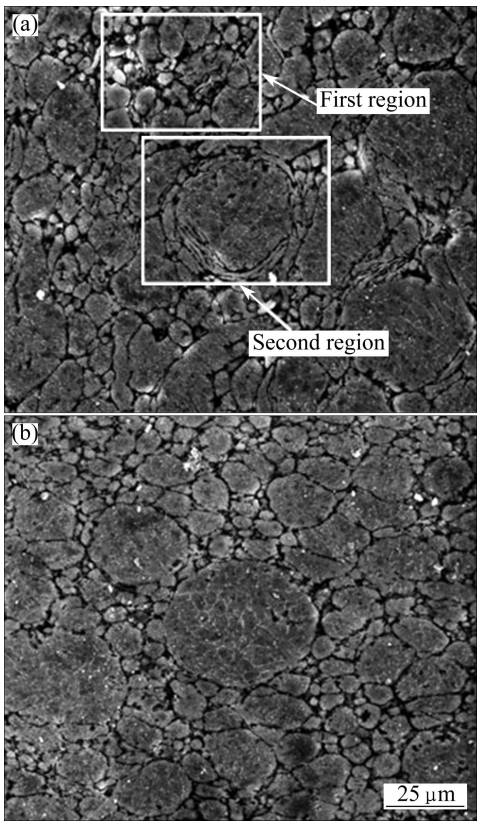

Fig. 1 SEM micrographs of 6061 alloy prepared by cold-pressing of ball-milled powders (a) and un-milled powders (b)

The deformation, fracture, and welding of powder happen during the ball milling process [20]. The deformation causes the powder morphologies to change from the original spheroidal form to plate-like shape. The deformed powders are welded together to form the long flat-shaped lamellar structures surrounded by the large powders (Fig. 1(a)). Simultaneously, the hardness of powders increases due to the extensive plastic deformation [20]. The powders may fracture in some local areas on the surface because of the work hardening, also leading to the formation of flat-shaped lamellar structures around the large-sized powder surface. Furthermore, some large powders, larger than 50 μm in width and 90 μm in length, can be seen in the ball-milled powders (Fig. 2(a)). But large powders cannot be found in the un-milled powders (Fig. 2(b)). So, it can be inferred that many small deformed powers are welded together to form the larger ones during ball milling. The sizes of both the ball-milled powders and un-milled ones are inhomogeneous (Figs. 2 and 3), and each powder experiences a unique processing history of deformation due to the statistical nature of ball milling [20]. In addition, ball milling causes the powder size to slightly decrease due to the fracture process (Fig. 3).

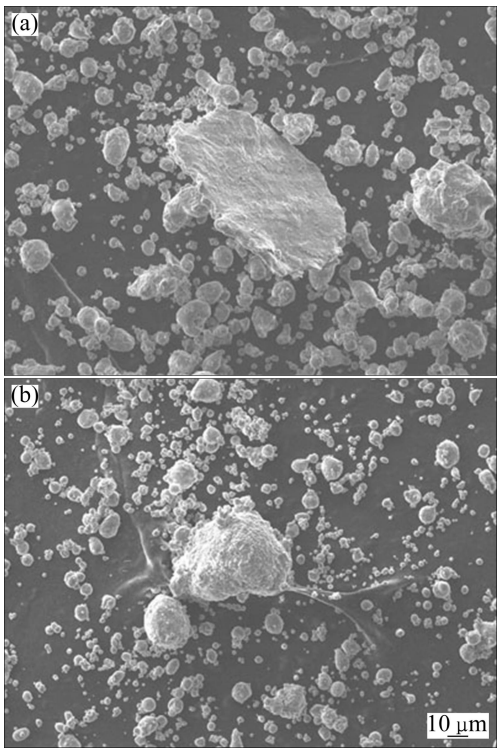

Fig. 2 SEM micrographs of ball-milled powders (a) and un-milled powders (b) of 6061 alloy

3.2 Microstructural evolution during partial remelting

Figure 4 shows the microstructures of the 6061 alloys heated for different durations at the semisolid temperature of 660 °C. During ball milling, the ball−ball

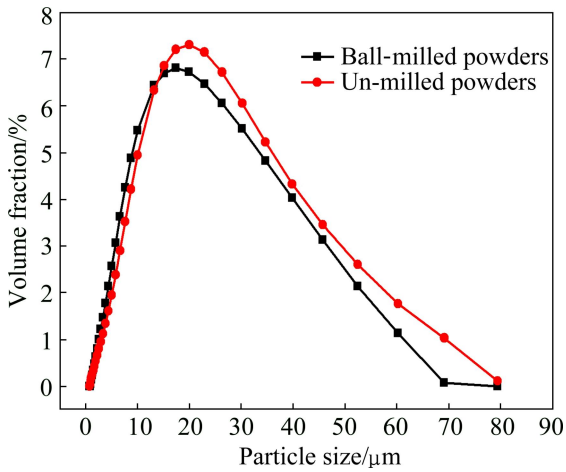

Fig. 3 Particle size distributions of both ball-milled powders and un-milled powders

sliding and ball−container wall collisions will generate large energy and causes the temperature to increase, which results in the solution of eutectic phases towards the primary α(Al) phase. So, the eutectic amount within the powders decreases after being ball-milled (the eutectic phases are in white in the SEM micrographs, compared with Fig. 4(a) and Fig. 5(a)). Similarly, during the heating period of 0−2 min at 660 °C, the eutectic phases further dissolve towards the primary α(Al) grains within the powders due to the temperature rise. Therefore, the amount of eutectic phases further decreases after 2 min duration (comparing Figs. 4(a) and (b)). It can be assumed that the grains may be coarsened and grow up through mergence of the neighboring grains when the eutectic phases between them have completely dissolved. Figure 4(c) shows that no eutectic phases can be found when the alloy is heated for 5 min and the powder has changed into a uniformly grey structure without grain boundaries, which implies that the grain coarsening and growth have actually occurred. Grain coarsening through mergence during the initial stage of partial remelting is commonly found in other casting alloys [21]. As described above, the average powder size is only about 16.3 μm, and the grains within the powder are smaller. It is well known that the smaller the grains are, the larger the growth tendency is. Furthermore, the boundaries between the laminar structures around the large powders also become indistinctive and even disappear (comparing Figs. 4(a), (b) and (c)). This indicates that the laminar structures also are coarsened. During the initial stage of partial remelting, the temperature rise of the specimen is very rapid and it is impossible that all of the eutectic phases completely dissolve when the temperature in the specimen is up to the eutectic point. The residual eutectic phases then melt to form liquid phase as the heating is further proceeded (Figs. 4(b) and (c)). Subsequently, the

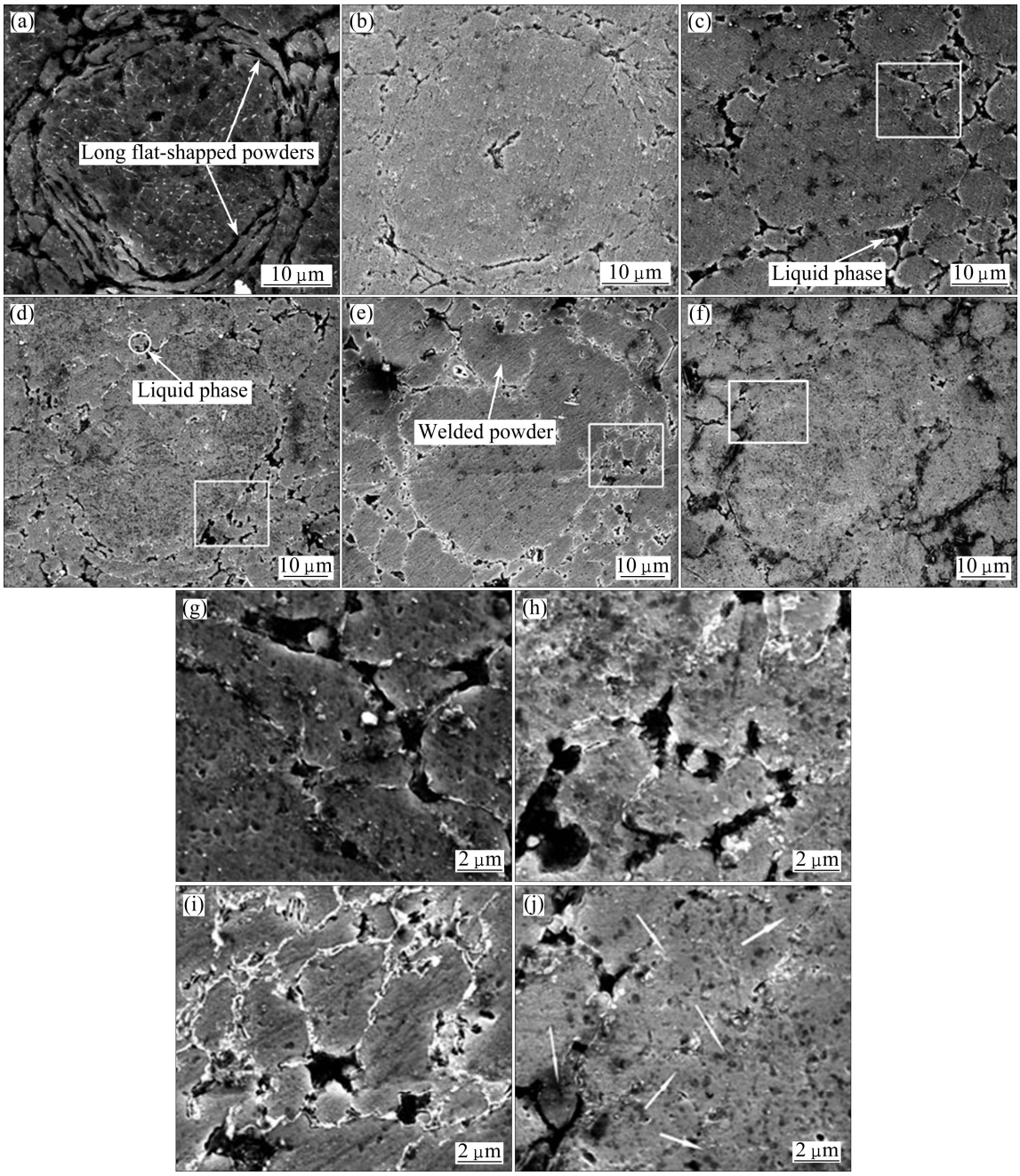

Fig. 4 SEM micrographs of 6061 alloys heated at 660 °C for 0 min (a), 2 min (b), 5 min (c), 20 min (d), 30 min (e) and 60 min (f), respectively; (g−j) Enlarged micrographs corresponding to rectangular regions in Figs. (c)−(f) respectively

powders (referred to the α(Al) phase) partially melt due to the further rise of temperature and the liquid phase increases. In addition, the welded powders generally store large energy due to ball milling and their sizes are also relatively small. So, the long welded flat-shaped powders can also partially melt and separate into short ones (Fig. 4(g)). For the un-milled one, the liquid phase does not form until 7 min duration (Fig. 5(b)). Therefore, it can be concluded that the microstructural evolution is accelerated, and the liquid phases formed earlier after the powders are ball-milled (comparing Fig. 4(c) with Fig. 5(b)). The acceleration is resulted from the energy stored in the powders. In addition, it can be proposed that the dissolution of eutectic phases and the resulted grain coarsening and growth are the main phenomena during the period of 0−5 min.

When being heated for 20 min, many long flat-shaped powders around the large-sized powders partially melt to form shorter ones (Fig. 4(h)). Some small powders among the large ones also melt to form liquid phase (Fig. 4(d)). And it can be seen that the distinction between the solid phase and liquid phase is not obvious. The reason should be that the content of the solutes in this alloy is low, so, the amount of eutectic

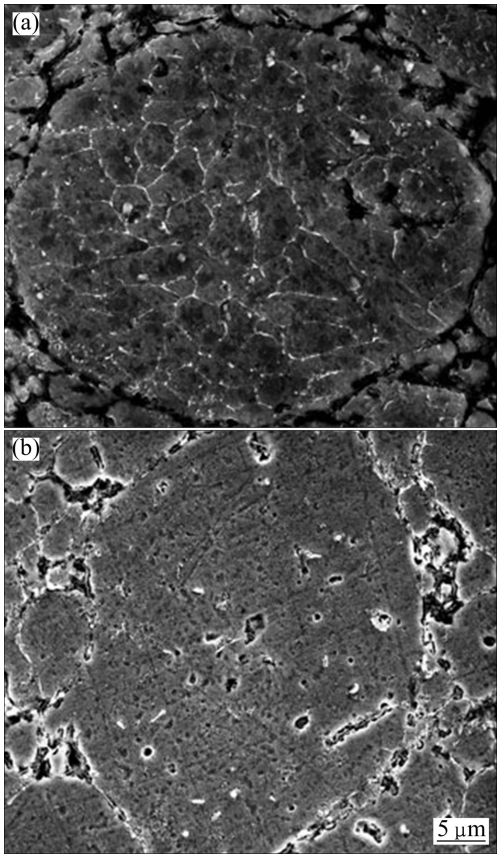

Fig. 5 SEM micrographs of 6061 alloys composed of un-milled powders heated at 660 °C for 0 (a) and 7 min (b)

phases is small in the as-cold-pressed state. The content of the solute atoms in the liquid phase further decreases because of the solid solution occurring during partial remelting. Therefore, most of the liquid phases are solidified into large amount of α(Al) grains during quenching, and only a small amount of eutectic phases are formed. In addition, the size of the formed α(Al) grains is also large, and they preferentially grow up on the solid α(Al) particle surfaces without nucleating due to their same crystal structure. Therefore, it is difficult to identify the liquid phase and solid phase. However, the solidification of the liquid phase is very rapid during quenching, so the secondarily solidified particles (solidified from the liquid phase in semisolid sate) are anyhow smaller than the primary α(Al) particles. Then, the relatively small particles in the quenched microstructures can be regarded as liquid phase. And it can be seen that the solid particles are uniformly suspended in the liquid phase (small particles) when the specimen is heated for 20 min (Fig. 4(d) and Fig. 6(b)). In addition, the liquid phase fraction nearly does not change after 20 min duration (comparing Figs. 4(d), (e) and (f)), which indicates that the system reaches the solid−liquid equilibrium state when the specimen is heated for 20 min.

Moreover, the boundaries between the welded powders should generally store energy although they coarsen during the initial stage. So, the boundaries may melt to form liquid phase, causing them to gradually separate from the large-sized powders (Fig. 4(e)). And more numbers of small and spheroidal particles which are mainly derived from the long flat-shaped powders are formed around the larger particles when they are heated for 30 min (Fig. 4(i)). Therefore, it can be suggested that the structural separation and spheroidization of primary particles are the main phenomena occurring from 5 min to 30 min. However, the separation process of the un-milled alloy is completed when it is heated for 20 min [14]. Compared with the microstructure evolution of un-milled one, the separation process is slowed down due to the formation of the welded larger size powders.

When the heating time is prolonged to 60 min, the particles coarsen to larger ones (Fig. 4(f)). It can be found that the particles grow mainly through mergence (marked by the arrows in Fig. 4(j)), driven by reducing the system's solid−liquid interfacial energy [22]. All of the particles coarsen to the interconnected ones. Then, it is reasonable to propose that the coarsening of particles is the main event during the period of 30−60 min. Compared with that of the un-milled one [14], this process is also slowed down because of the formed larger size powders.

Based on the above discussion, it can be concluded that the microstructural evolution of the 6061 alloy can be divided into three stages, the dissolution of eutectic phases and the resulted grain coarsening and growth, structural separation and spheroidization of primary particles due to their partial remelting, and the final coarsening of particles mainly owing to the mergence. In addition, compared with the un-milled alloy, the ball milling accelerates the first stage of microstructural evolution due to the energy stored in the powders, but the latter two stages are slowed down because of the formation of large-sized powders.

In addition, as described in the above section, the microstructure of the as-cold-pressed alloy can be divided into two regions: the region of small-sized powders and the region of large powders surrounded by the long flat-shaped powders. The present results indicate that the microstructural evolution processes are different for the two regions. Figure 6 gives the microstructures of the first region in the alloys heated at 660 °C for different time. After being heated for 10 min, the powders are separated by a small amount of liquid phases (Fig. 6(a)). As the heating time is further prolonged, the liquid phase fraction nearly does not change after 20 min duration (Figs. 6(b), (c) and (d)), and all the particle sizes are smaller than 50 μm. When the heating time is prolonged to 30 min, the particles

agglomerate and merge (marked by arrows in Fig. 6(c)), driven by reducing the system's solid−liquid interfacial energy [22]. Of course, the liquid phase also agglomerates. When the heating time is prolonged to 60 min, the particles coarsen to larger ones (Fig. 6(d)).

The microstructures of the second region in the alloys heated at 660 °C for different time are shown in Fig. 7. As discussed above, the two stages after 10 min duration are the structural separation and spheroidization of primary particles (Figs. 7(a), (b) and (c)), the final

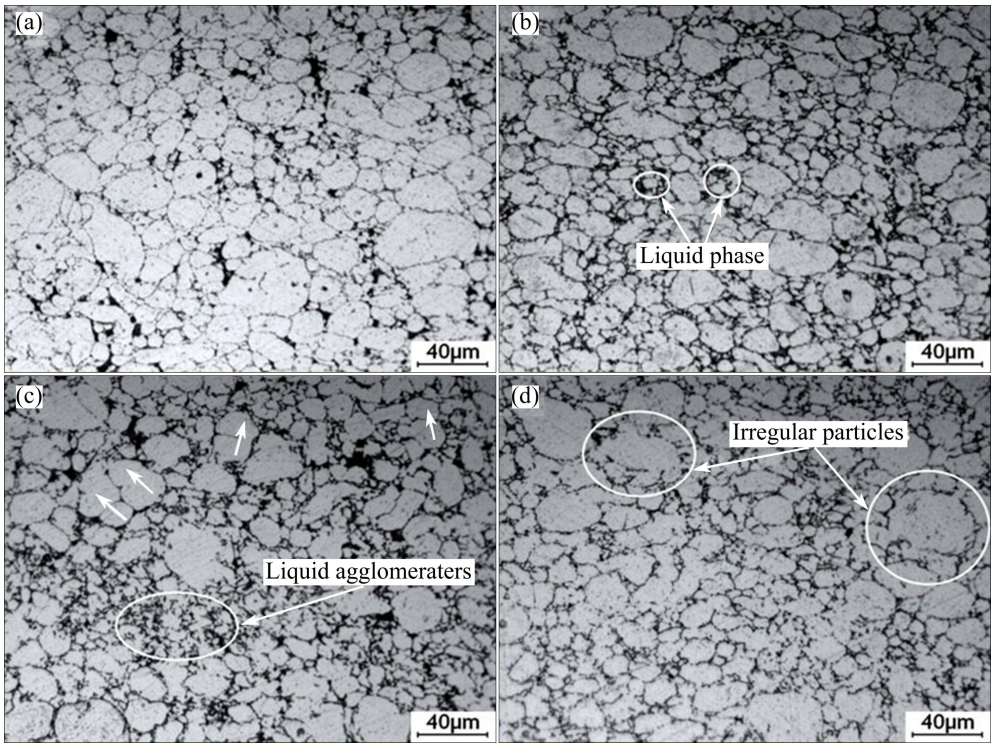

Fig. 6 OM micrographs of the first region (small-sized powders) of 6061 alloys heated at 660 °C for 10 min (a), 20 min (b), 30 min (c) and 60 min (d)

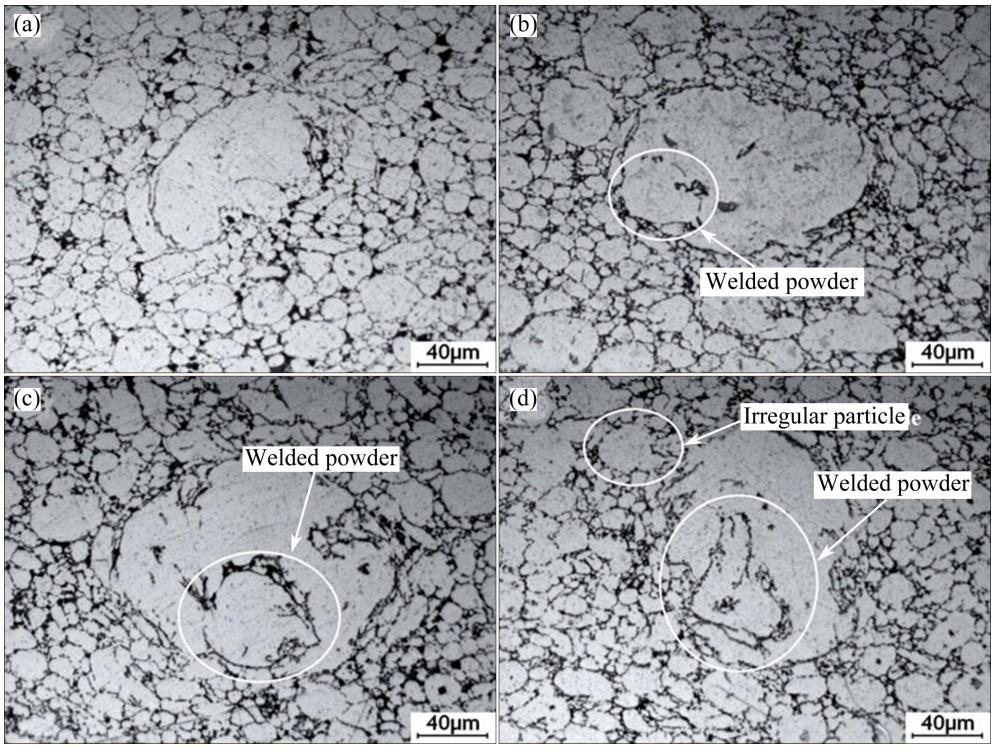

Fig. 7 OM micrographs of the second region (large powders surrounded by long flat-shaped powders) of 6061 alloys heated at 660 °C for 10 min (a), 20 min (b), 30 min (c) and 60 min (d)

coarsening of particles (comparing Figs. 7(c) with (d)). The difference between the first region and the second region lies in the morphology and particle size of the particles due to the different deformation degrees during ball milling. In the second region, there exist many irregular welded large-sized powders and the powder particle size reaches about 130 μm (Fig. 7(b)), which is much larger than that in the first region. The welded powders are gradually separated due to their partial remelting (Figs. 7(b), (c) and (d)). Therefore, a conclusion can be drawn that the initial microstructure in the cold-pressed state has a large effect on the final semisolid microstructure: a finer initial microstructure in the as-cold-pressed state results in a semisolid state microstructure with smaller and more spherical primary particles. Moreover, the average primary particle size in the semisolid microstructure (Fig. 6(b) and Fig. 7(b)) is about 34 μm.

Furthermore, it can be found that there still exist many irregular particles when the specimen is heated for 60 min (Fig. 6(d) and Fig. 7(d)). In order to explain this phenomenon, it can be suggested that there exists an oxide film on the surface of the atomized powders. The oxide film may thicken during partial remelting. As the heating time is prolonged, the amount of liquid phase increases. When a large amount of liquid phases are formed, the liquid should flow in the voids under the gravity. Furthermore, driven by reducing the system's solid−liquid interfacial energy, the oxide film may crack to form the connected channels of liquid pools between the neighboring powders. The formed liquid pools may solidify into secondarily particles with a small amount of eutectic phases. Moreover, as discussed above, secondarily particles would be prior to grow by clinging to the surface of primary particles without nucleating due to their same crystal structure. Therefore, the primary particles become larger and more irregular.

3.3 Microstructural evolution mechanism

As discussed in the above section, a main phenomenon is the decrease of eutectic amount during the initial stage of the partial remelting. It is supposed that the reason for this phenomenon is the dissolution of eutectics towards the α(Al) phase. From Table 1, it can be noted that both Mg and Si contents in the α(Al) phase increase in the heating time range from 0 to 2 min, which demonstrates that the solution actually occurs in this stage. The increment of Mg content is much higher than that of Si (Table 1). The reason should be that the diffusion coefficient of Mg in Al is higher than that of Si [23]. For comparison, the compositions of the un-milled specimens are also included in this table. It can be noted that the compositions of the α(Al) phase in the ball-milled specimen that is not heated are the same as those of the un-milled specimen that is heated for 2 min. This implies that the solid solution process has happened before partial remelting.

As the heating time is prolonged to 5 min, the temperature has reached the eutectic point [14], and the liquid phase has formed because of the melting of the small amount of residual eutectic phases. As the temperature rises, more liquid phases form from the partial remelting of the primary α(Al) phase, which leads to the structural separation and spheroidization of solid particles. According to the Al−Mg and Al−Si binary equilibrium phase diagrams [24,25], the solubility of the α(Al) phase will decrease with the rise of temperature in the solid−liquid intervals. Therefore, the contents of Mg and Si in the primary α phases decrease after 5 min duration (Table 1). Inversely, the solute contents in the liquid phase should increase. So, more eutectics form after being quenched as the heating time is prolonged to 20 min (Fig. 4(d)). The compositions do not change after being heated longer for 20 min (Table 1), which again demonstrates that the system reaches the solid−liquid equilibrium state. The subsequent main phenomenon with the increase of heating time should be the coarsening of primary particles. So, it can be concluded that the change of compositions in each period during partial remelting is corresponding to a specific stage of the microstructural evolution.

Table 1 Composition of primary α(Al) phase in 6061 alloys heated at 660 °C for different time including alloy prepared by un-milled powders

Heating time/min	w(Al)/%		w(Mg)/%		w(Si)/%	
	Not ball-milled	Ball-milled	Not ball-milled	Ball-milled	Not ball-milled	Ball-milled
0	98.4	98.3	0.9	0.9	0.7	0.8
2	98.3	97.8	0.9	1.3	0.8	0.9
5	98.0	98.3	1.2	1.0	0.8	0.7
10	98.6	98.9	0.8	0.7	0.6	0.4
20	99.3	99.6	0.4	0.2	0.3	0.2
30	99.3	99.6	0.4	0.2	0.3	0.2

3.4 Effect of reheating temperature on semisolid microstructure

A correct reheating temperature is of great importance for the thixo-formability of an alloy, because the temperature determines not only the liquid phase fraction of the final semisolid microstructure, but also the size and shape of the primary particles [21]. Figure 8 shows the microstructures of the first region of the 6061 alloy heated for 20 min at various temperatures. The first region is selected because the semisolid microstructure of the first region is finer than that of the second region, and the 20 min duration is employed because the system

reaches the solid−liquid equilibrium state. Together with Fig. 6(b), it can be seen that the number or the amount of the small particles increases with the rise of temperature. As discussed above, the small particles are originated from the solidification of the liquid phase. So, it can be considered that the liquid phase amount increases as the temperature rises.

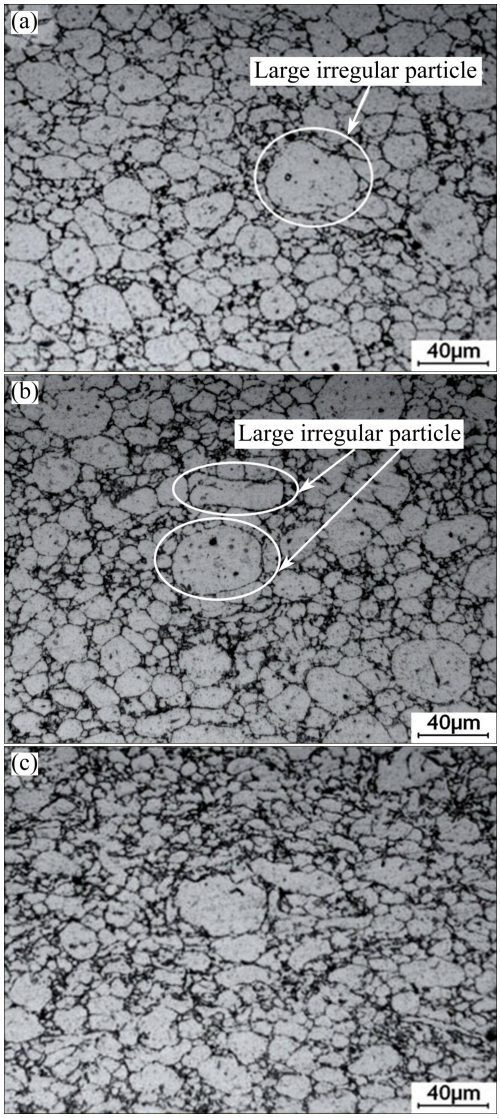

Fig. 8 OM micrographs of 6061 alloys heated for 20 min at 650 °C (a), 655 °C (b) and 665 °C (c)

The amount of liquid phase is relatively small at a low remelting temperature, and the distance between the neighboring primary particles is short. So, the merging possibility for the neighboring solid particles is also large, leading to the formation of large irregular particles (Figs. 8(a) and (b)). As the temperature rises, more and more amount of liquid phases should form and the distance between the primary particles gradually increases. So, the possibility of mergence becomes much small and the semisolid microstructure with small and spheroidal particles which are suspended in the liquid phase is achieved when the temperature is elevated to 660 °C (Fig. 6(b)). However, when the specimen is heated at 665 °C (Fig. 8(c)), too large amount of liquid phases are generated and the resulting forming behavior is similar to the full-liquid one, thus the advantages of thixoforming should not play enough.

Therefore, the effect of heating temperature on semisolid microstructure in this work is similar to that in the previous work [14]: properly elevating the heating temperature is beneficial to obtaining small and spheroidal particles, while too high temperature results in the loss of the advantages of thixoforming.

4 Conclusions

1) The microstructural evolution of 6061 aluminum alloy during partial remelting can be divided into three stages: the dissolution of eutectic phases and the resulted grain coarsening and growth, structural separation and spheroidization of primary particles due to their partial remelting, and the final coarsening of particles mainly owing to the mergence.

2) Compared with the microctructural evolution of the un-milled alloy, the ball milling accelerates the first stage due to the energy stored in the powders, but the latter two stages are slowed down because of the formation of large-sized powders.

3) The initial as-cold-pressed microstructure has a great influence on the semisolid microstructure: the finer the as-cold-pressed microstructure is, the smaller and more spherical the primary particles in the final semisolid microstructure are.

4) Properly elevating the heating temperature is beneficial to obtaining small and spheroidal particles, while too high temperature results in the loss of the advantages of thixoforming.

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球磨对 6061Al 合金粉末冷压块在部分重熔过程中组织演变的影响

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摘　要：研究球磨对 6061 粉末压块在部分重熔过程中组织演变的影响。结果表明，球磨 6061 铝合金在部分重熔过程中的组织演变可分为 3 个阶段: 初期共晶相的溶解及其引起的晶粒粗化和长大、初生相颗粒的分离与球状化以及最后颗粒的粗化。与未球磨合金的组织演变相比，球磨使粉末贮存能量，加快了初期演变进程；但大尺寸粉末颗粒的形成使得其后续分离和粗化过程减慢。此外，初始组织越细小、圆整，越有利于得到尺寸较小、更加圆整的半固态组织。适当提高重熔温度有利于获得细小、圆整的半固态组织。

关键词：6061 铝合金；球磨；粉末触变成形；部分重熔；组织演变

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