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ARTICLE

Effect of Ball Milling on Microstructure and Mechanical Properties of $Mg_2B_2O_5$w/6061Al Matrix Composites

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Abstract: $Mg_2B_2O_5$ wisker/6061 (Al$Mg_2B_2O_5$w/6061Al) matrix composites were fabricated through the powder metallurgy (PM) technique. The relationship among the parameters of ball milling, powder characteristics, and mechanical properties of the hot extruded composites was investigated. The results show that the reinforcement whiskers accelerate the milling process by increasing the matrix deformation and enhancing the welding and the fracture of particles. Furthermore, the mixing process is modified by a higher milling speed and suitable milling time, which result in reduced crystalline size, improved homogeneity of the whisker distribution, as well as enhanced tensile strength of the hot extruded composites.

Key words: Al matrix composite; $Mg_2B_2O_5$ whisker($Mg_2B_2O_5$w); ball milling; hot extrusion; mechanical property

Metal matrix composites (MMCs) with ceramic reinforcements have gradually attracted considerable attention due to their high strength, stiffness, fatigue and wear resistance in comparison with the unreinforced alloys[1,2]. Another attractive aspect of the MMCs is that they can be fabricated with conventional metal working process[3]. Powder metallurgy is an important technique for producing MMCs that can improve reinforcement segregation occurring in the casting metallurgy process. However, clustering may still occur on a smaller scale because of static charges acting on reinforcements, or due to geometrical reasons when there is a large difference between matrix and reinforcement sizes[4,5]. High energy ball milling has been used to improve reinforcement distribution throughout the matrix, because deformation, fracturing and cold welding of the powder occur, giving rise to the reinforcing particles being well embedded into every aluminum particle[6,7].

To obtain bulk composites from mechanically alloyed powders, several consolidation techniques have been employed, such as sintering, hot-pressing, hot-isostatic pressing, severe plastic deformation and hot extrusion of loose or prepressed powders. Uniaxial cold-pressing or die compaction is commonly used to promote the union and the densification of powder particles with varied geometries. Cold-pressed powder compacts can be consolidated by sintering or hot extrusion. The latter has the advantage of promoting full densification. Hot extrusion allows a high shear strain rate, providing a high-strength bonding between particles, and a microstructure very similar to that of wrought products[8,9]. In the case of aluminum and its alloys, hot extrusion can break the typical oxide layer coating the powder, providing better bonding of the particles. In MMCs, hot extrusion tends to eliminate the clustering of reinforcement and therefore a better distribution can be obtained through the metal matrix.

Although the improved mechanical response of ball milled MMCs is well reported in literatures, the final reasons for this improvement are not fully clear yet, especially for aluminum matrix composites reinforced with the whisker [10,11]. In fact, the present capability to predict their mechanical response is still poor. The present

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investigation was, therefore, focused on understanding the influence of a very specific parameter of ball milling on the microstructure and the mechanical properties in 6061Al matrix composites reinforced by magnesium borate whiskers. Meanwhile, the effects of whisker content on the tensile properties were also considered.

1 Experiment

The composites investigated were prepared by a powder metallurgical route. The rapidly solidified powder of 6061Al was used, whose average size was about 22.7 μm. The morphology of the powder is shown in Fig.1. It was seen that the Al particles were generally round with a rough, micro-cellular dendrite surface. The $Mg_2B_2O_5$ whisker supplied by Qinghai Citic Guoan Ltd Chengdu Company, with a diameter of 0.5~1 μm and a length of 10~30 μm was used as the reinforcement. The aspect ratio (length/diameter) was between 10 and 40. The morphology of the whiskers is shown in Fig.2. They were commonly long cylinders with smooth superficial aspect.

The mixture of 6061Al powder and $Mg_2B_2O_5$ whisker was dried for 10 h at 70 ℃, and then blended in a planetary ball mill at low and high milling speeds for different time, with a ratio of balls to mixed powder of 10:1. The blend was consolidated by extrusion at 723 K, with extrusion ratio of 25:1. No atmosphere control was used. For the high-speed milling, the powder was milled for various lengths of time as shown in Table 1. A monolith 6061 alloy bar was also processed from alloy powder for comparison purposes.

The microstructure of all materials were studied on sections (perpendicular to the extrusion direction). The samples were prepared by conventional metallographic techniques including finishing with silicon colloidal under very low pressure in an automatic polishing machine. The aspect ratio and distribution of $Mg_2B_2O_5$ whiskers and the fracture surfaces were observed by the scanning electron microscopy (SEM), and the composition analyses of materials were carried out using EDX facility. The crystalline size was measured with modified Scherrer equation. The apparent densities of the as-received powder and the mixed powder with different processes were measured. The apparent density is the mass per unit volume of a material including voids inherent in the tested material. For the powder, the apparent density expresses the density without the application of pressure. Tensile testing of the samples was carried out on an Instron1195 testing machine at a cross head speed of 2 mm/min.

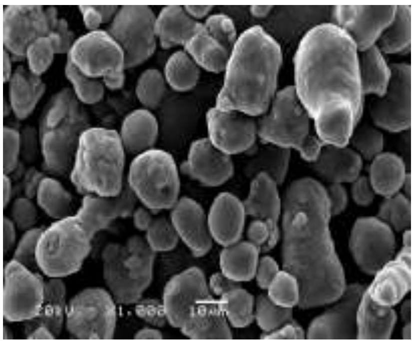

Fig.1 SEM image of 6061 powder

2 Results and Discussion

2.1 Results

The morphologies of the composite powders ball milled for different time are shown in Fig.3. For the as-received powder, the Al particles remain spheroidal and the $Mg_2B_2O_5$w are freely distributed at the periphery of the Al particles. The 8 h milled powder displays a flattened morphology, indicating that it undergoes severe deformation during mixing; however, for the 10 h milled powder, the Al particles show a more equiaxed morphology, and most of the whiskers are attached to the particles.

Fig.4 shows the effect of milling time on the apparent densities of different powders. The apparent densities decrease first with increasing of milling time; they reach their own minimum values and then start to increase. After longer milling time, the apparent densities reach steady value. By contrast, the addition of reinforcement whiskers obviously accelerates the evolution of the apparent density with milling time. In the present experiment, the stabilization of apparent density was taken as the main phenomenological aspect to determine the milling time for each composition.

Fig.5 shows the microstructure evolution of the aluminum alloy matrix expressed by the change of the mean crystallite size. It is shown that when the milling time increases, the

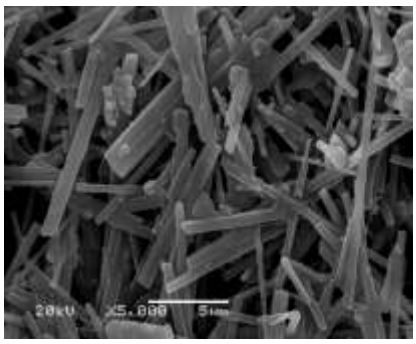

Fig.2 SEM image of $Mg_2B_2O_5$ whiskers

Table 1 Time of high-speed milling

Material	Milling time/h								
6061Al	0	2	4	6	8	10	14	18	22
$Mg_2B_2O_5$w/Al	0	2	4	6	8	10			

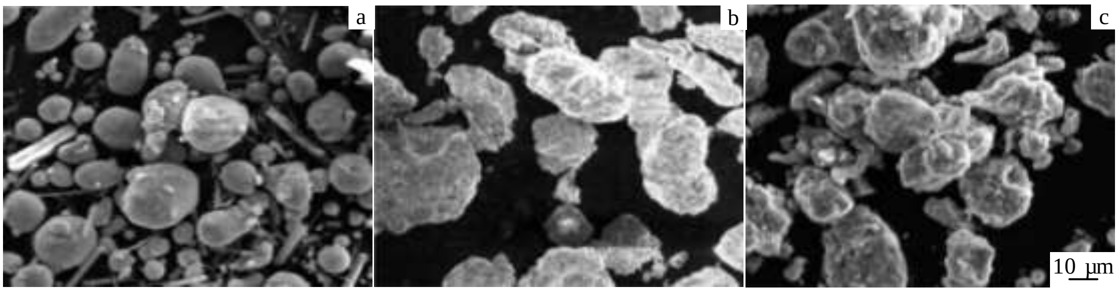

Fig.3 SEM images of mixed powders milled for different milling time: (a) as-received, (b) 6 h, and (c) 10 h

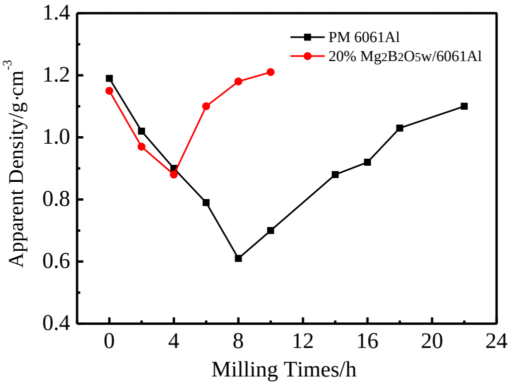

Fig.4 Effect of milling time on the apparent densities of 6061Al and 20vol% $Mg_2B_2O_5$w/6061Al composite powder

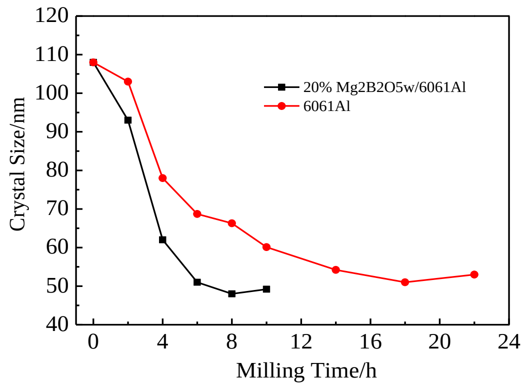

Fig.5 Effect of milling time on the crystalline size of 6061Al and 20vol% $Mg_2B_2O_5$w/6061Al

crystalline size of 6061Al powder reduces and then increases slightly in the latter stage of milling. For the mixed powder, it exhibits almost the same trend in a shorter milling time. The presence of reinforcement whiskers decreases the time which is necessary to produce the same level of refinement. The stabilization of crystalline sizes is almost concurrent with the time necessary to stabilize the apparent density.

Fig.6 shows the microstructures of the composites prepared from the mixed powders ball milled at different speeds. Fig.6a shows a large amount of clustered whiskers,

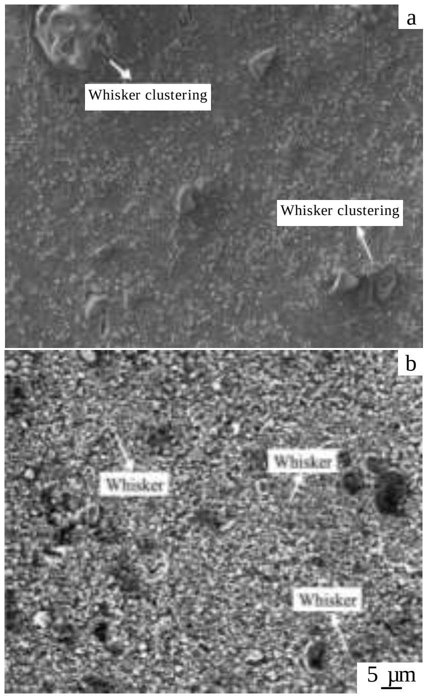

Fig.6 Distribution of the whiskers in 20vol% $Mg_2B_2O_5$w/6061Al composites prepared form powders milled at different speeds: (a) 100 r/s, and (b) 400 r/s

corresponding to the sample at ball milling speed of 100 r/s. Fig.6b shows the uniform distribution of the reinforcement pertaining to the sample at ball milling speed of 400 r/s corresponding to the no clustering zone. Meanwhile, the length of whiskers is smaller in the extruded composites than that in the as-received mixed powder. The consolidation process is able to produce full-consolidated products, and no appreciable porosity is found in SEM image. The relative densities calculated by the Archimedes method are no lower than (0.99±0.01%) those for all extruded materials.

Moreover, no oxidation and precipitates were detected by XRD analysis in the hot extruded composites as shown in Fig.7.

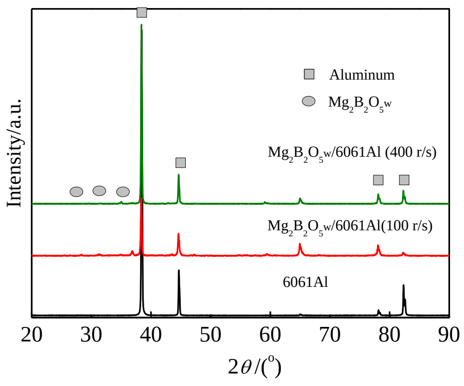

Fig.7 XRD patterns of 20vol% $Mg_2B_2O_5$w/6061Al prepared from the powders with different ball milling speeds

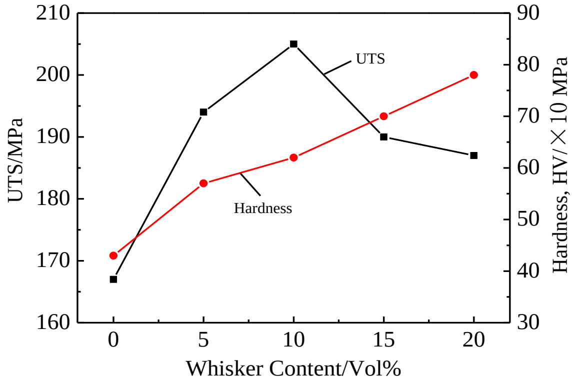

Fig.8 UTS and hardness of the hot extruded composites prepared from the powder milled at low speed of 100 r/s as a function of the volume fraction of $Mg_2B_2O_5$ whisker

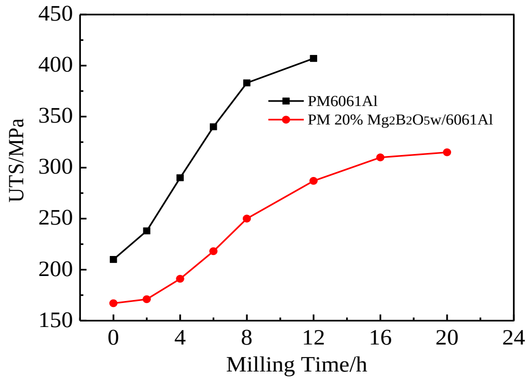

Fig.9 UTS of the hot extruded 6061Al and 20vol% $Mg_2B_2O_5$w/6061Al matrix composites at high speed milling of 400 r/s as a function of milling time

Fig.8 shows UTS and hardness of the hot extruded composites prepared from the powder milled at low speed of 100 r/s as a function of the reinforcement content. The UTS increases as the $Mg_2B_2O_5$w volume fraction is increased from 5% to 10%, and the higher whisker content results in the negative response. While the hardness increases with increasing of whisker content all the time.

Fig.9 shows the UTS of the hot extruded 6061Al and 20vol% $Mg_2B_2O_5$w/Al matrix composites prepared from the powders milled at high speed of 400 r/s as a function of milling time. For both of the two kinds of materials, the UTS increases obviously with increasing of milling time. The addition of reinforcement whiskers exhibits an obvious strengthening effect for the composite.

The best mechanical properties fabricated with two different methods are shown in Table 2. It is found that for the composite prepared at high milling speed its UTS, $\sigma_{0.2}$ and hardness are all significantly improved in comparison with that at low milling speed, and the strength improvements is associated with concomitant loss of ductility with 1.8%.

Table 3 illustrates the typical mechanical properties of some commercially available aluminum matrix composites; the combined mechanical properties of hot extruded $Mg_2B_2O_5$w/6061Al matrix composites are comparable with these applied composites, and in some cases the former is better. The composites hot extruded in the present experiment have good results, as demonstrated above, and the sample made is illustrated in Fig.10.

Table 2 Properties of $Mg_2B_2O_5$w/6061Al matrix composites under different states

Material	UTS /MPa	$\sigma_{0.2}$ /MPa	δ /%	HV/ MPa	Fabricated method
(10%)$Mg_2B_2O_5$w/Al	205	130	9	760	PM(100 r/s)
(20%)$Mg_2B_2O_5$w/Al	407	328	1.8	1400	PM(400 r/s)

Table 3 Typical mechanical properties of some commercially available aluminum matrix composites[12]

Material	UTS /MPa	δ/%	Fabricated method	Company
6061/SiC(25%)-T6	496	4.1	PM	DWA
6061/SiC(20%)-T6	498	6	PM	Ceracon
6061/Al_2O_3 (20%)	379	2.1	Cast	Duralcan

2.2 Discussions

For the mixed powder ball milled at high speed (Fig.3), the Al alloy particle morphology changes from the equiaxed to the flattened due to the initial preponderance of deformation. In the following stages, the flattened particles weld to each other, harden-fracture as shown in Fig.11. Simultaneously, the evolution of apparent density (Fig.4) with milling time is obviously due to morphological changes (Fig.3) imposed on the powder particles during the milling. Good powder packing and consequently a relatively high apparent density are typical of the equiaxial or quasi-spherical morphologies

Fig.10 Example of a component hot extruded composite

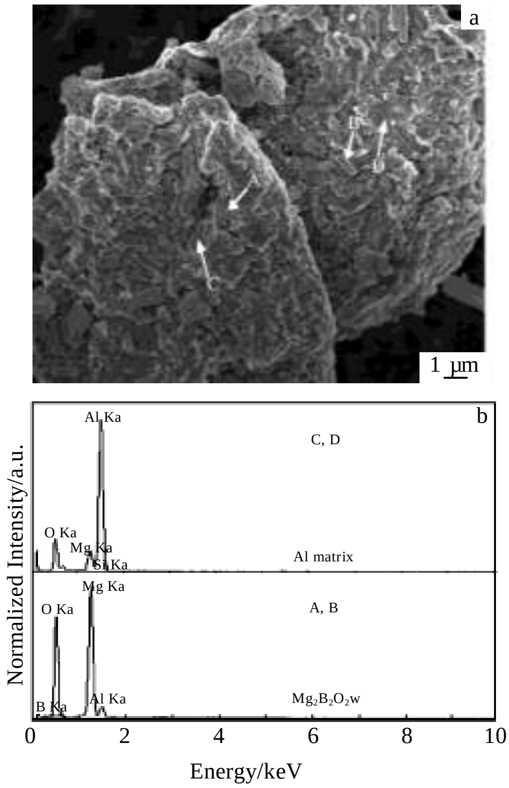

Fig.11 Harden-fracture morphology with an enlargement of the mixed powders ball milled with high speed (a) and EDS spectra of different locations (b)

of the as-received and long time-milled mixed powders. On the other hand, flatten aluminum particles fit randomly and thus do not provide good powder packing. So it could explain the lower apparent density for short time milled powder.

The hardening mechanisms of metal and alloys, promoted by deformation, grain size refinement and solid dispersion, are well known[13]. A high degree of deformation, due to high speed milling, reduces the grain size to nanometer level. An extremely fine distribution of oxides throughout the particles should also be produced: the aluminum oxide layer covers the aluminum particles and disperses into the aluminum matrix during the milling[14].

An important point to be considered in the process is the powder oxidation, as the process is carried out in air from the beginning to the end. XRD analyses (Fig.7) show that, despite the use of a non-protective atmosphere, no significant oxidation occurs during the milling. One possible reason for this is that the milling vessel is sealed before working, which partially protects the powder against excessive oxidation. On the other hand, a minor degree of oxidation is beneficial due to the oxide particle dispersion, as mentioned in the preceding paragraph.

It is known that the incorporation of particle reinforcements does not always bring an increase in tensile strength of the material. Problems such as reinforcement clustering, cracks in the reinforcement surface, or poor bonding between the matrix and reinforcements could also deteriorate the composite strength[15]. The UTS and hardness of the composites prepared from the mixed powder milled at low speed (Fig.8) confirm the poor improvement of these mechanical properties. Cracks presented in the whisker reinforcement and the reinforcement agglomeration constitute the defects which operate as the points of stress concentration in the composite. As can be seen in Fig. 12, the crack initiation site for the 10vol% $Mg_2B_2O_5$w/Al composite is the clustering of whiskers. The UTS is greatly influenced by the presence of such defects. And Fig.8 shows that the effects of whisker content on the mechanical properties are more obvious as the content is beyond 10vol%. The composites with 20vol% whiskers present non-uniform distribution and clustered whiskers in Fig.6a which may explain the lower UTS.

But for the hardness of the composites, it always increases with increasing of whisker content. As the extrusion parameters including extrusion ratio, rate and temperature are constant, and the whiskers do not undergo deformation except for the rearrangement along the extrusion direction and fracture to some extent, the large amount of reinforcement implies smaller volume fraction of the matrix material, which is the fraction that really is deformed during extrusion. It means that the higher amount of reinforcement produces greater deformation in the matrix during the extrusion. Unless the extruded material recrystallizes, the higher deformation produces a greater hardening effect, which explains the increase of hardness with the higher amount of reinforcement[16].

For the hot extruded 6061Al alloy milled at high speed, UTS increases with prolonging of milling time, as shown in Fig. 9, and its tensile strength is greatly improved in comparison with the alloy fabricated at low milling speed. As stated above, high-speed ball milling promotes a high degree of deformation and reduces the grain size to nanometer level (Fig.5). This claim could be proved through Hall-Petch equation which shows the relationship between the grain size

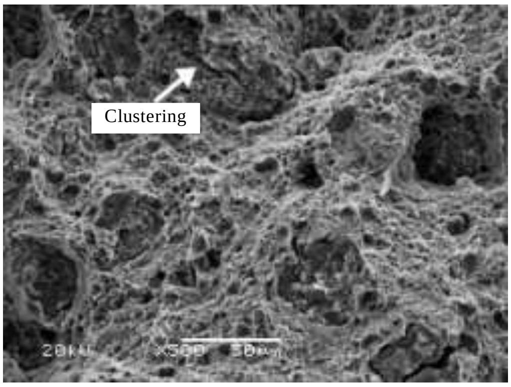

Fig.12 SEM image of fracture surface of the composite prepared from the powder ball milled at low speed containing 10vol% $Mg_2B_2O_5$w

and the flow stress at certain plastic strain up to ductile fracture[17].

$$\sigma_0=\sigma_i+K' D^{-1/2} \quad (1)$$

Where, σ_0 is the flow stress, σ_i is the stress opposing the movement of dislocations, K' is constant and D is the grain size. According to Eq.(1), as the grain size gets smaller, flow stress also increases, leading to high strength in the composites. Moreover, grain boundaries act just like obstacles against the movement of dislocations during plastic deformation. Thus, at low grain size, high grain boundaries which in turn act as obstacles against the movements of dislocations would be required and the composite strength increases. Finally, the dislocations density is inversely proportional to the grain size and directly proportional to flow stress. Therefore, as the size of the particles decreases, the dislocation density increases which in turn leads to higher flow stress.

Simultaneously, the higher UTS of the composites with 20vol% reinforcement whisker indicates that the improvement of its mechanical properties is attributed to the combined action of homogeneous whiskers (Fig.6b) and the refinement of grain size. However, as the addition of reinforcement, the elongation of the composites substantially decreases as shown in Table 2, especially for the hot extruded composites mode from the mixed powder milled at high-speed. One reason for this is that at high amount of reinforcement, the distance between the hard phases decreases; as a result, the dislocation movement is hindered. This is considered to be one of the most important factors in decreasing the flexibility and elongation [18]. Furthermore, addition of whiskers leads to less flexibility in the matrix itself. Therefore, according to the rule of mixtures, the elongation of the composites is reduced.

Through the above analyses, it is shown that the extrusion process in air is suitable for producing practically full density materials with no oxidation; meanwhile, it is available for getting the materials with enhanced composites properties.

3 Conclusions

1) During the process of high-speed ball milling, the difference of the mixed powder morphology with the milling time is subjected to the welding and fracture mechanism.

2) The apparent density and crystalline size could indirectly reflect the process of ball milling, which is more favorable to determine the specific parameters of ball milling.

3) By the process of high-speed ball milling, the crystalline size of Al matrix is reduced and the distribution of the whiskers is improved effectively, and the tensile strength of the hot extruded composites is enhanced significantly in comparison with the composites prepared from the mixed powder ball milled at low-speed.

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球磨工艺对 $Mg_2B_2O_5$w/6061Al 基复合材料微观组织演变及力学性能的影响

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摘　要：采用粉末热挤压法制备了硼酸镁晶须增强铝基复合材料，对球磨工艺参数、粉末特性及热挤压态复合材料间的相互关系进行了研究。研究结果表明，在球磨过程中，增强体晶须的添加促进了基体合金的变形，加快了铝粉颗粒的焊合与断裂过程的发生。在球磨过程中，经过适当时间的高速球磨后，铝合金基体晶粒尺寸减小，增强体晶须在基体中的分布得到显著改善，从而使热挤压态复合材料力学性能得到大幅度的提高。

关键词：铝基复合材料；硼酸镁晶须；球磨；热挤压；力学性能

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