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ARTICLE

Effect of Ball Milling Process on the in Situ Synthesis of Nano-TiB Whiskers

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Abstract: Ball milling process has an important effect on the microstructure and the morphology of final powder products and the corresponding as-sintered samples. In the present study, Ti-7Al-0.2B (wt%) mixed powder was ball-milled by low-energy and high-energy types of ball mills, and then the milled powder was sintered by hot-pressing. On this basis, the morphology evolution of the powder was analyzed, and the microstructures of the reinforcements in the as-sintered composite were investigated. Research results show that in the low-energy ball milling process, mechanical alloying occurs between the powder particles. TiB whiskers formed in sintering process are long and thin without linked coarse crystals bars or clusters. Under the high-energy milling, the average particle size of the powder is refined remarkably to 1 μm, and Ti(Al) supersaturated solid solution or even amorphous structure is formed during the milling process. In the following sintering process, nano-scale TiB whiskers are synthesized and they are uniformly distributed in the matrix.

Key words: ball milling; hot pressed sintering; TiB whisker

Titanium matrix composites (TMCs) are considered as potential materials for advanced automotive, military and aerospace applications because of their low density, high strength and superior elevated temperature properties. Compared with continuous fiber reinforced composites, discontinuous particles or whiskers reinforced titanium based composites show more promising prospects for practical applications since they are isotropic, cost-effective and easy to fabricate [1-3].

In the commonly used discontinuous reinforcements, TiB synthesized by in situ process is suggested to be an ideal reinforcement in titanium matrix due to their excellent capabilities, such as strong interface bonding with matrix, thermal stability at high temperature and similar coefficient of thermal expansion to titanium. TiB reinforced titanium based composites can be fabricated by several methods including casting, powder metallurgy and SHS (self propagation high temperature synthesis) technologies [4-6]. However, many undesired coarse particles and clusters sometimes appear in the composites, which may act as the source of cracks and dramatically lower the mechanical properties of the composites. Hence, it is very crucial to reduce the size of reinforcements for improving the mechanical properties of TMCs.

Ball milling is a powerful and cost-effective powder processing technique for grain size reduction and alloy synthesis. However, the final products of milling are significantly affected by various parameters of milling process. H. B. Feng et al.[7] investigated the influences of several factors including milling time, different powder mixtures, process control agent (PCA), ball size and rotating speed, but they ignored one important parameter of mechanical alloying (MA), i.e., the type of ball mill. In the present study, two different types of ball mills were used to perform ball milling processes on Ti-7Al-0.2B (wt%) mixed powder. The morphology evolution of powders during ball milling processes was analyzed and the characteristics of in situ synthesized reinforcements in the as-sintered composites were investigated.

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1 Experiment

In the present experiment, Ti powder with an average size of 25 μm (purity > 99.5%, irregular), Al powder with an average size of 75 μm (purity > 99.5%, spherical) and B powder with an average size of 5 μm (purity>99.5%, irregular) were mixed with a nominal composition of Ti-7Al-0.2B in wt%. The ball-to-powder mass ratio was 10:1, and the powders were ball milled in two different types of ball mills. One was the QM-ISP04 horizontal planetary ball mill with a speed of 160 r/min where the powders were milled for 40 h, hereafter referred to as low-energy milling. And the other was the double pendulum vibrating ball mill with a speed of 550 r/min where the powders were milled for 20 h, hereafter referred to as high-energy milling. During milling process, the ball mill was stopped every 5 h in order to take out a small quantity of powders for characterization and meanwhile to cool down the vial to prevent increasing temperature.

In above mixing and milling processes, the powders were protected by Ar to avoid contamination. Both milled powders were sintered by hot-pressing in vacuum at 1100 °C, 20 MPa for 1 h. As a reference, the powder blended for 1 h in the planetary ball mill without milling was also sintered.

The morphology evolution of the milled powders was studied by X-ray diffraction and DSC analysis. The microstructures of the powder particles and sintered composites were studied by scanning electron microscope (SEM) and transmission electron microscope (TEM).

2 Results and Discussion

2.1 Morphology evolution of the powders

Fig.1 shows the SEM images of milled powders. As can be seen from Fig.1a, after low-energy milling for 40 h, the morphology of powders is turned into an approximately equiaxed shape from the original irregular shape. The average particle size is 6~8 μm, which is less than that of starting powders (25 μm). Besides, the sharp edges and corners of the original powders are also polished. Generally, there are two main processes during MA, i.e., particles breaking and cold welding. In the present experiment, cold welding does not lead to an obvious aggregation owing to the low energy ball milling, as well as the limited change of the mixed powders size; only those thinner edges and corners or particles with bigger aspect ratio are fractured during the ball milling process.

Compared with the low-energy ball milled powder, the powder after high-energy ball milling for 20 h shows a sharp decline in particle size. As shown in Fig.1b, the powder particles are roughly spherical, and their average size is less than 1 μm. During the ball milling process, the particles sizes of powders experience a typical evolution, namely dramatic increase at the early stage, gradual decrease after a certain time and achieving a stable level at last. This result agrees well with the conclusions in previous reports[8,9]. At the early stage, high energy colliding and squeezing of ball/ball and ball/wall result in a severe plastic deformation of the powders and the deformed particles are cold-welded to large lamellar composite particles, which are usually much coarser than the original powders. With the extension of milling time, the composite particles suffer from continuously repeated process of deforming, cold-welding, agglomerating and fracturing caused by the colliding of high-energy balls, so that the powders are refined and gradually transformed to spherical shape. When the effects of cold-welding and fracturing achieve an equilibrium state, the particles size would not change further.

2.2 Structural evolution of the powders

Fig.2 shows the TEM images and selected area diffraction patterns of the powder milled for 40 h in low-energy mill. The selected area diffraction patterns confirm the existence of TiB and TiB_2 phases. That is to say, TiB and TiB_2 are mechanical alloyed during the milling process. From the chemical reaction Eqs. (1) to (3), it can be seen that TiB is formed by the reaction of Ti and B or by the decomposition of TiB_2. Turkdogan calculated the thermodynamic data of the reactions[10]:

$$\mathrm{Ti+B \rightarrow TiB} \qquad \Delta G = -163176+5.86T \tag{1}$$

$$\mathrm{1/2Ti+B \rightarrow 1/2TiB_2} \qquad \Delta G = -142256+10.25T \tag{2}$$

$$\mathrm{Ti+TiB_2 \rightarrow 2TiB} \qquad \Delta G = -41840-8.79T \tag{3}$$

According to thermodynamic theory, one prerequisite of a chemical reaction is that the Gibbs free energy (ΔG) is negative. The more negative ΔG means the reaction is easier to occur. Eqs. (1) to (3) show that the ΔG in the lower temperature range of ball milling process are all negative, indicating these three actions are all possible to occur in the present experiment.

The generation of TiB and TiB_2 reflects the characteristics of collision-induced reactions between elemental powders during

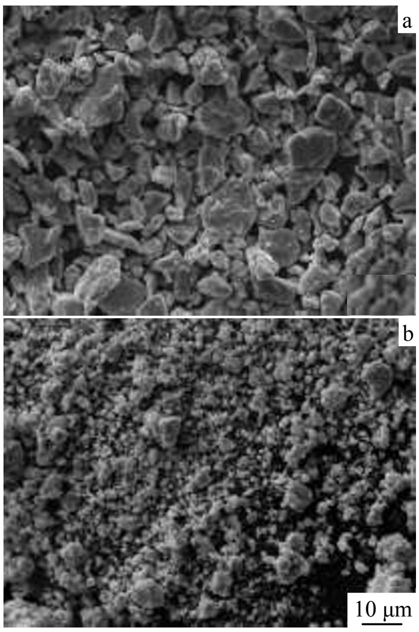

Fig.1 SEM images of the milled powders: (a) low-energy milled for 40 h and (b) high-energy milled for 20 h