CHARAC TERIZATION OF TiAl POWDER PRODUCED BY MECHANICAL ALLOYING ①

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ABSTRACT

The microstructure, alloying reaction and sintering behavior of the powder produced by Mechanical Alloying(MA) for 8 h from 64 wt.−% Ti powder and 36 wt.−% Al powder were studied by scanning electron microscopy, optical microscopy, X−ray diffractometry, differential scanning calorimetry(DSC) and dilatometry. The mechanically alloyed powder particles are Ti−Al composite particles. Thus, titanium aluminides can form easily in the powder through diffusion during heat treatment. It is shown that the sintering behavior of this powder, different from the behaviors of TiAl alloy powder and mixed powder of 64 wt.−% Ti powder and 36 wt.−% Al powder, changes from expansion at temperatures below 1 000℃ to shrinkage at temperatures above 1 000℃. Homogeneously alloyed TiAl material with a density over 96% of the theoretical density can be produced from the mechanically alloyed powder by compaction−sintering.

Keywords: TiAl alloy powder mechanical alloying SEM optical microscopy DSC dilatometry

1 INTRODUCTION

Powder metallurgy is a fabrication technique of near-net shaped bulk components. It is especially used for fabrication of brittle and hard−workable materials. Not only does arc−melted TiAl usually have a structure with coarse grains, but it also has a very inhomogeneous composition. These have severely affected the mechanical properties of TiAl alloys. The inherent low ductility of TiAl has led to the adoption of powder metallurgy as the preferred processing route. Since powder metallurgy techniques can effectively control the microstructures and composition homogeneity, TiAl alloys sintered from fine powders should have a fine grained structure with a relatively homogeneous composition. One method to get fine TiAl powders is rapid solidification technique(RST), another is mechanical alloying(MA), or attritor grinding[1−3].

The microstructure, alloying reaction and sintering behavior of the powder produced by mechanical alloying of 64 wt.−% Ti powder and 36 wt.−% Al powder(labeled MA powder)were studied. For comparison, the attritor−ground powder from crushed TiAl alloy powder (labeled TiAl alloy powder) and the mixed powder of 64 wt.−% Ti powder and 36 wt.−% Al powder (labeled mixed powder) were also studied.

2 EXPERIMENTAL

Mechanical alloying of MA powder and attritor grinding of TiAl alloy powder were carried out in argon for 8 h in the same milling vial. This vial was a stainless steel cylinder of dia 135mm× 150mm. The milling media was the argon atmosphere in the vial. The ball−to−powder weight ratio was 20: 1. The particles of Ti powder with a purity of 99% are smaller than 44

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μm. The particles of Al powder with a purity of 99% were smaller than 22 μm. The starting TiAl alloy powder used in this study was smaller than 175 μm. In order to control the mechanical alloying, 1.5 wt.−% stearic acid was added as a process control agent(PCA).

The microstructure and homogeneity of MA powder were observed using scanning electron microscope and optical microscope. Analysis of the alloying reaction between Ti and Al for MA powder and mixed powder was performed by X−ray diffraction and differential scanning calorimetry (DSC). The sintering behaviors of powders were studied by dilatometry. The dimensions of compacted samples with a density of 2.3 g/cm^3 were 5 mm × 5 mm × 15 mm. The dilatometer illustrated the length changes of sintering samples. Argon was used as the atmosphere. All samples were heated at a constant rate of 5 ℃ / min.

3 RESULTS AND DISSCUSION

3.1 *X−ray Diffraction Analysis*

Fig. 1 shows the X−ray diffraction patterns of mixed powder and MA powder. Comparing Fig. 1(a) with Fig. 1(b), there were no obvious titanium aluminides in MA powder, but the diffraction peaks of elemental Ti and Al were widened due to severe lattice distortion. From the X−ray diffraction analysis of heat−treated MA powder, the following results can be discovered: when MA powder was heat treated at 600℃ for 0. 5 h, the diffraction peaks of titanium aluminides appeared on its diffraction pattern and were more intense than those of elemental Ti and Al(Fig. 1(d)); when MA powder was heat treated at 600℃ for 3 h. The diffraction peaks of the titanium aluminides became much more intense(Fig. 1(e)). But there were only very weak diffraction peaks of titanium aluminides on the diffraction pattern of heat−treated mixed powder at 600℃ for 3 h (Fig. 1(c)).

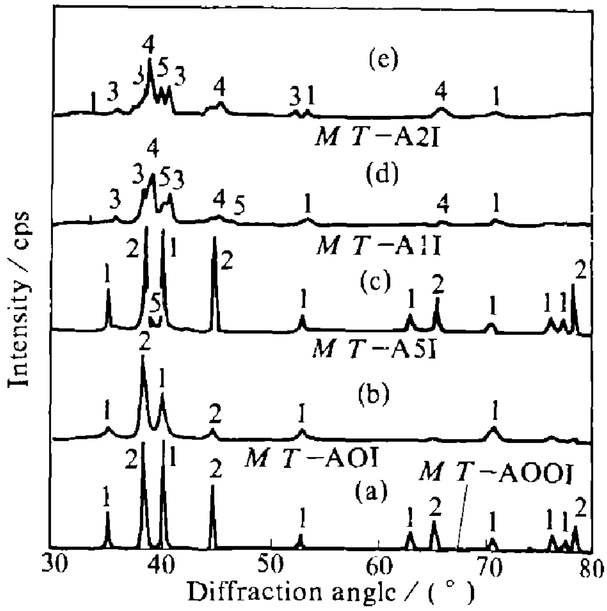

Fig. 1 X−ray diffraction patterns

(a)—mixed powder; (b)—MA powder; (c)—heat−treated mixed powder at 600℃ for 3 h; (d)—heat−treated MA powder at 600℃ for 0.5 h; (e)—heat−treated MA powder at 600℃ for 3 h.

It should be mentioned that the mechanical alloying process makes the alloying reaction between Ti and Al easier during heat treatment, although titanium aluminides do not appear in the powder produced by mechanical alloying for 8 h.

3.2 *DSC Analysis*

Fig. 2 shows the DSC curves of both mixed powder and MA powder. It is indicated in Fig. 2(a) that the exothermic alloying reaction between Ti and Al in mixed powder took place only after the melting of Al (There is the endothermic peak of Al melting on the curve before the exothermic alloying reaction peak). The fast exothermic reaction of self−propagation between Ti and Al made one sharp exothermic peak appear on the curve. The curve of MA powder(Fig. 2(b)) is different from that of mixed powder. Slow exothermic alloying reactions had taken place in MA powder before the melting point of pure Al (666℃).

The reaction, commenced at 245℃, and were completed at 600℃. Apparently, the exothermic reaction to form titanium aluminides was through the diffusion between Ti and Al.

Therefore, it can be concluded that titanium aluminides form easily in the powder produced by mechanical alloying through diffusion between Ti and Al during heat treatment.

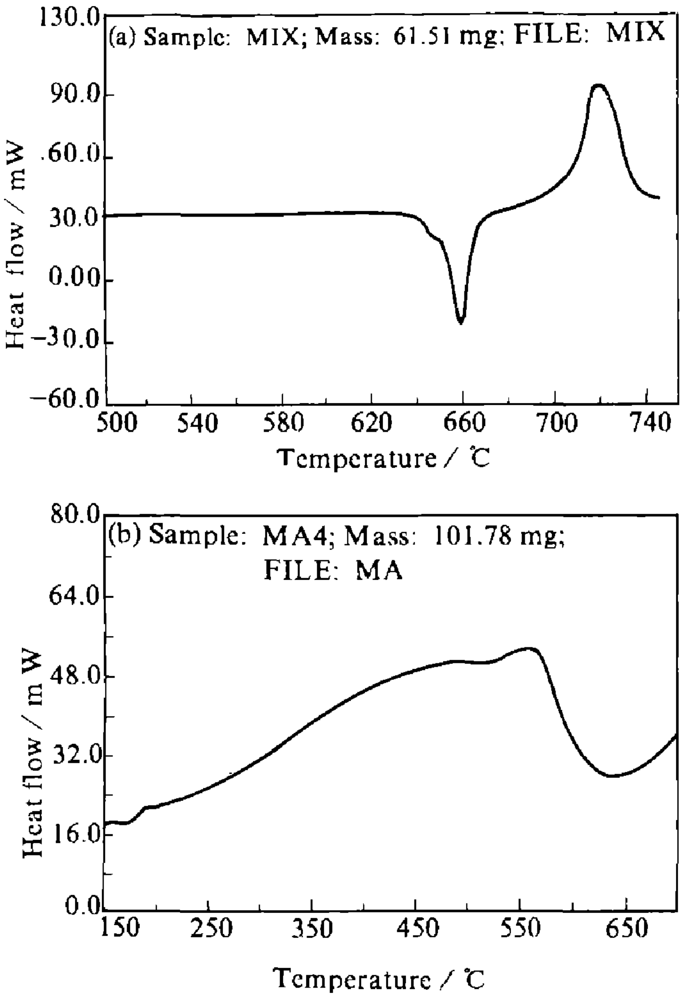

Fig. 2 DSC curves

(a)—mixed powder; (b)—MA powder

3.3 *Microstructures*

Fig. 3 shows the back-scattered electron micrographs of mixed powder and MA powder. There is an evident BSE image contrast between elemental Ti and Al in mixed powder (Fig. 3(a)). Ti powder particles are irregular. Al powder particles are spherical. In the BSE micrograph of MA powder (Fig. 3(b)), there is no evident contrast between the particles. Fine particles are equiaxial. Every powder particle is composed of many finer particles. Fig. 4 shows the metallographic structure of MA powder particles. Due to ball-powder-ball collision, elemental Ti and Al powder particles have become lamellar and have been cold-welded into larger MA powder particles with a lamellar structure. These indicate that the MA powder particles are Ti–Al composite particles of tiny Ti and Al particles, which were produced by cold-welding and fracturing of powders during mechanical alloying[4, 5].

The fact that the fine particles of MA powder are Ti–Al composite particles of finer Ti and Al particles indicates that the diffusion distance between Ti and Al was shortened, and the contact area between Ti and Al was increased greatly. In addition, the atomic activity was increased due to the severe lattice distortion caused by ball-powder-ball collisions. In this case, the alloying reaction through diffusion between Ti and Al could be enhanced greatly.

3.4 *Sintering Behaviours*

Fig. 5 shows the sintering curves of three kinds of powders–mixed powder, MA powder and TiAl alloy powder. This figure illustrates that the mixed powder compact expanded greatly at temperatures between 600 ℃ and 700 ℃. The expansion decreased only a little at temperatures above 1 000℃ and was never eliminated. Although the MA powder compact also expanded at temperatures between 400℃ and 500℃, its expansion was less than that of the mixed powder compact, and could be eliminated. When the temperature was over 1 000℃, the powder compact began shrinking quickly. The density of the sintered bulk at 1 400℃ for 1.5 h is 3.75 g/cm^3, i. e. 96. 2% of the theoretical density.

The sintering curve of TiAl powder is different from both of the former. The sintering of TiAl alloy powder is actually a common solid-state sintering procedure. Its compact did not expand during sintering (Here thermal expansion is not discussed) but only shrinked. The compact

Fig.3　Backscattered electron micrographs (× 600)

(a)—mixed powder; (b)—MA powder

shrank slowly at temperatures below 1 300℃ and shrank quickly at temperatures above 1 300℃ . The density of the sintered bulk at 1 400℃ for 1.5h was 3.76g / cm^3, i. e. 96.4% of the theoretical density.

During the sintering of the mixed powder compact, due to the Kirkendall's effect, Ti particles expanded and pores formed in Al particles after Al atoms in the particles had diffused into Ti particles. Thus, the mixed powder compact expanded during its sintering[6].

Fig. 4　Metallographic structure of MA powder particles (× 1 400)

For the sintering of MA powder from Ti and Al, the diffusion of Al into Ti must also cause the compact expand and micropores can form in the composite particles at the same time. But these micropores can shrink continuously under the action of greater surface tension during sintering. In this case, the sintering behaviour of this powder changed from sintering expansion at temperatures below 1 000℃ to sintering shrinkage at temperatures above 1 000℃, and before 1 300℃ it shrank much faster than TiAl alloy powder.

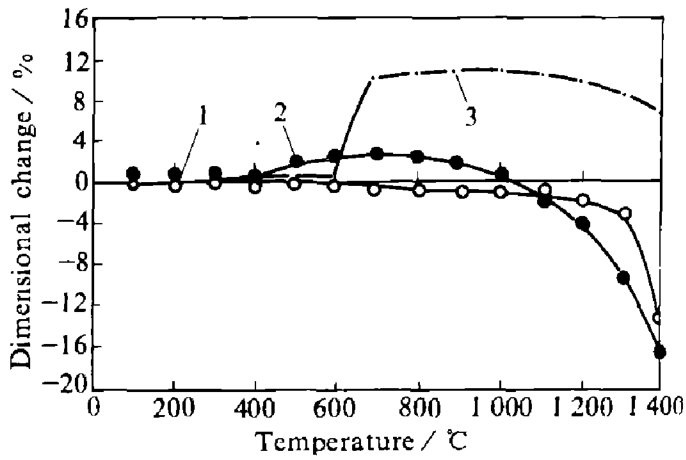

Fig. 5　Sintering curves of three kinds of powders

1—TiAl powder; 2—MA powder; 3—Mixed Powder

The X−ray diffraction results showed that the X−ray diffraction patterns of the as-sintered bulks of MA powder and TiAl powder at 1 300℃ for 1 h are essentially similar(Fig. 6). This indicates that homogeneously alloyed TiAl materials with densities over 96% of the theoretical density can be produced from both MA and TiAl powder by compaction–sintering.

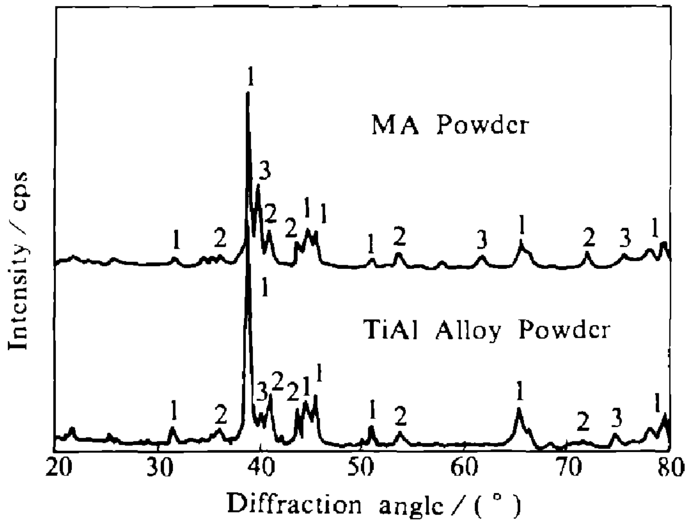

Fig.6 X–ray diffraction patterns of sintered bulks of MA powder and TiAl powder at 1300℃ for 1 h

1—TiAl; 2—Ti_3Al; 3—Al_3Ti

4 CONCLUSIONS

(1) The formation of Ti–Al composite particles in the powder produced by mechanical alloying greatly shorten the diffusion distance between Ti and Al. Thus titanium aluminides can form easily in this powder through diffusion during heat treatment;

(2) The sintering behavior of the mechanically alloyed powder changes from expansion at temperatures below 1 000℃ to shrinkage at temperatures above 1 000℃.

(3) Homogeneously alloyed TiAl materials with densities over 96% of the theoretical density can be produced from the mechanically alloyed powder by compaction–sintering.

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