EFFECT OF MILLING INTENSITY ON STRUCTURAL CHANGES OF MIXED Al-Fe-Ni POWDERS IN MECHANICAL ALLOYING PROCESS[①]

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ABSTRACT Mixed elemental aluminium, nickel and iron powders in the composition of Al-4.9 Fe-4.9Ni were mechanically alloyed under various milling intensities. The results showed that milling causes refinement of the microstructure. The powders with grain size(GS) of the nanometer order were produced after milling. The increasing of ball to powder weight ratio(BPR) and rotational velocity(RV) accelerates the refinement of the grain and the particle of iron and nickel in aluminium matrix, and an approximate formula has been put forward according to the experimental results to quantitatively describe the change of GS *versus* BPR and RV.

Key words Al-Fe-Ni powder structural change milling intensity mechanical alloying

1 INTRODUCTION

In recent years, the process of mechanical alloying(MA) has been widely used to synthesize amorphous alloys, nanocrystalline alloys, supersaturated solid solution and intermetallic compounds[1, 2]. Since the MA process involves repeated welding, fracturing and rewelding operations leading to microstructure refinement, the milling intensity is a critical parameter determining the constitution of the final product and microstructural evolution. It has been shown that the glass-formation range can be expanded in alloy systems, or the time required for amorphisation can be reduced by increasing the milling intensity[3, 4]. Variation in milling intensity leads to difference in results in the same system regarding the final phases produced. However, the effect of process variables is ill defined up to date. How to obtain desired powders of high quality and how to choose the process parameters are still problems to be solved.

The aim of the present work is to study the effects of the ball to powder weight ratio(BPR) and the rotational velocity(RV) on structural changes of the powders with milling time, and to put forward an approximate formula.

2 EXPERIMENTAL PROCEDURE

Elemental aluminium (99% pure, ~ 80 μm), iron(99.5% pure, ~ 4 μm) and nickel (99.9% pure, ~ 4 μm) powders were mixed in the composition of Al - 4.9Fe - 4.9Ni (in weight fraction), and subjected to attritor milling at room temperature controlled by cooling water under the protection of pure argon gas. The milling balls 6 mm in diameter were made of chrome hardened steel, 2% (in weight) stearic acid was added as a process control agent. At fixed time intervals, a small amount of powders were taken from the milling vial for analysis.

X-ray diffraction analyses were carried out on RIGAKU D/max-rB diffractometer with CuK_{α} radiation. The grain size was estimated from the line broadening of diffraction peak[5]. Lattice parameters were measured from the Al

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(311) diffraction peak.

Microstructures of powders were evaluated with scanning electron microscope (SEM). Samples for SEM observation were prepared by mounting the powders by epoxy resin, and after grinding and polishing golden film was coated. Random lines were drawn across the micrographs and the diameters of the particles intersecting these lines were measured. At least one hundred different particles were measured to determine the average size.

3 RESULTS AND DISCUSSION

Fig. 1 shows the X-ray diffraction patterns of Al-4.9Fe-4.9Ni powders milled for various times. All of the diffraction peaks broadened and their height decreased with increasing milling time. Increasing the ball to powder ratio (BPR) and rotational velocity (RV) accelerates the broadening of peaks and the reduction in the ratio of Al(311) peak intensity normalized to that before milling (Fig. 2). Evidently, the diffraction intensity decreased rapidly at the starting milling stage for the large BPR and RV values, but remained relatively stable after a certain period of milling. For small BPR and RV values, the diffraction peak ratio decreased slowly. The grain size of powders *versus* milling time for various BPR and RV values is shown in Fig. 3. The grain size decreased very rapidly at the initial period of processing. The increasing of BPR and RV accelerates the reduction rate of grain size. But no matter what milling intensity is used, the minimal grain size is not less than 12 nm. When RV = 450 r/min, the grain size decreased to 12 nm after 14 h milling, while it will take 30 h for grain size to diminish to the same value when RV = 300 r/min (see Fig. 3(b)).

According to the experimental results of both in this paper and in Ref[6], we put forward an approximate formula to describe the change of GS *versus* BPR and RV. For the composition of Al-xFe-yNi, with the increasing of Fe and Ni, the GS decreases, which is similar to the effect caused by increasing BPR, so we defined the equivalent BPR as B which is describe as

$$B = BPR(0.2 \times 1.1^{x+y} + 0.5) \quad (1)$$

and we have

$$GS = 4.4\left[17 - \left(\frac{RV}{100} - 1.1\right)^2\right] \times \left[0.36 + \left(\frac{B}{100} - 1.1\right)^2\right] \times \left(1 - \frac{RV}{2500}\right)^t \left(1.12 - \frac{9B}{2500}\right)^t + 200(0.983)^{RV} + 67(0.77)^B + 12 \quad (2)$$

The calculation results are in good agreements with the experimental ones, while the milling time is more than 2~3 h.

The premise for mechanical alloying is the formation of composite powders of different elements due to cold welding. Repeated plastic deformation, welding and fracturing in milling process refined the microstructure of the composite powders.

Paralleling with the refinement of

Fig. 1 XRD patterns of powders milled for various times (BPR = 35, RV = 370 r/min)

Fig. 3 Grain size of powders *vs* milling time

(a) —RV= 370 r/min; (b) —BPR= 35

microstructure, diffusion or chemical alloying occurs, the composite powders turn into alloy powders with homogeneous composition and microstructure. Because chemical alloying needs interdiffusion, which necessitates a fine microstructural scale, the microstructural refinement is essential to the enhancement of mechanical alloying rate.

The microstructural refinement of the composite powders is dependent on the plastic deformation of the powders collided by balls, thus it is related to: (a) the level of energy obtained during a single collision event; (b) the total energy transferred to the powder in unit time, which has relation to the number of collisions in unit time.

Previously, we have investigated the plastic deformation of powders sprayed on hard plate impacted by hard ball[7], it was shown that the plastic strain of powder aggregate entrapped between balls increased with increasing ball

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exceeding that value, it would seriously deteriorate the mechanical properties of the TiAl alloys. It is known from the analyses of Fig. 3 and Fig. 4, excessive Ce would form coarse particles and a lot of pores, which are detrimental to the TiAl alloys. The formation of pores results from vaporizing during melting because of the high vapourization pressure of Ce[5]. If the solid solubility of Ce in TiAl alloys is enlarged and a large amount of the pores is reduced by using powder metallurgy rapid solidification process, it can be expected that their properties be further improved.

5 CONCLUSIONS

(1) The deflecion and strength of the TiAl alloy with 0.002 mole fraction Ce were obviously improved. When Ce content exceeding that value, their properties deteriorate sharply.

(2) The property improvement in the TiAl alloys was caused by Ce due to refined microstructures and reduced oxygen content in γ-TiAl matrix. But excessive Ce is apt to form coarse particles and a lot of pores, thus seriously deteriorating their properties.

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velocity and with decreasing the initial height of powder aggregates.

Increasing the BPR and RV values results in high plastic strain of powders, thus, the refinement of microstructures speeds up, and the grain size diminishes faster.

In addition, the fine microstructures shorten the distance of inter-diffusion, and the grain boundary and defects provide many sites for low activation energy diffusion, this coupling effects accelerate the alloying rate for large BPR and RV values, thus the time for obtaining the powders with homogeneous composition is decreased.

4 CONCLUSION

Mechanical alloying has been used to synthesize Al-4.9Fe-4.9Ni alloy powders from the mixed elemental aluminium, iron and nickel powders. A supersaturated solid solution of iron and nickel in aluminium was formed, and the mechanical milling produced refinement of microstructures with nanocrystalline grain size. Milling intensity has great effects on alloying rate. The increasing of ball to powder ratio or rotational velocity accelerates the reduction of the grain size.

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