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Synthesis and microstructural evolution of nanocrystalline Al-Fe-V-Si-Nd alloy powder①

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[Abstract] The nanocrystallic $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) alloy powder was fabricated by mechanical alloying, and the microstructural evolution of alloy powder during mechanical alloying and annealing treatment were studied. It was found that under the condition of vibration ball-milling for 96 h, the microstructure of $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ alloy powder still consisted of single α(Al) solid solution phase without the trace of presence of intermetalic compound; after the appropriate heat treatment, the microstructure of $Al_{93.3-x}Fe_{4.3}V_{0.7}Nd_x$ alloy powder consisted mainly of nanocrystalline α(Al) phase and α-Al_{13}(Fe, V)$_3$Si phase; the addition of rare earth Nd enhanced the grain refining and amorphous inclination of Al solid solution during mechanical alloying, and resulted in new phases formed after heat treatment.

[Key words] rare earth Nd; mechanical alloying; Al-Fe-V-Si-Nd alloy; α(Al) nanocrystalline; α-Al_{13}(Fe, V)$_3$Si phase
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1 INTRODUCTION

Allied Signal Company in American first designed a series of rapidly solidified (RS) Al-Fe-V-Si alloys with high specific strength and excellent heat-resistance[1~6]. This type of materials were made by using melt spinning ribbons, mechanical grainding, vacum degassing, hot pressing of powder and extruding[7]. In order to obtain double-strengthened alloy containing nanoscale grain and dispersed particle, and to improve thermal stability of this nanocrystaline alloy, a new RS Al-Fe-V-Si-Nd thin ribbon was prepared[8], which consisted of nanocrystalline Al grains and Al_8Fe_4Nd particles. After heat treating at 673 K, the structure changed into consisting of nanoscale Al grains and α-Al_{13}(Fe, V)$_3$Si particles. In addition, under the same condition, the mechanical properties of new alloy exhibits one time higher than those of Al-Fe-V-Si microcrystaline alloys[9, 10]. However, whether to be able to obtain mechanical alloyed (called MAed hereafter) Al-Fe-V-Si-Nd alloy powders whose microstructure is approximate with that of RS Al-Fe-V-Si-Nd alloy hasn't been reported up to date. If possible, it can avoid melting and rapidly quenching process, simplify the procedure of material preparation, and reduce cost. Therefore, the mechanical alloying and the microstructural evolution of Al-Fe-V-Si-Nd alloy powder are investigated here.

2 EXPERIMENTAL

Elemental powders of Al, Fe, Si, Nd with a purity of 99.9%, and Fe 48.4% (mass fraction) with particles size of less than 63 μm. The composition of the powders was designed to be $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) and placed together with 304 stainless steel balls into a cylindrical 304 stainless steel pot. The radio of ball of powder was kept at 40:1 by mass. Powder and balls were sealed in an argon atmosphere in the pot. The alloy powders during mechanical alloying were investigated by X-ray diffraction analysis (XRD) and transmission electron microscopy (TEM).

3 RESULTS AND DISCUSSION

3.1 Microstructural evolution of mechanical alloyed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) powder

Fig. 1 and Fig. 2 shows the XRD spectra of the MA $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 3.0) powder mixtures after different duration of MA. It seems to be clear that the diffraction peaks of V, Si and Al (Fe) became broad and weak gradually, and the (311) peak position of Al shifted to high 2θ angle side with ball-milling time increasing. The changing rules of three MAed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) alloys were most approximate. With the time increasing to 96 h, the Al_8Fe_4Nd and α-Al_{13}(Fe, V)$_3$Si or other intermetallic phases was not observed, but ananoscale Al solid solution phase, which differed from the RS alloys[11]. In addition, with increasing ball-milling time, the lattice parameter or Al crystalline decreased, as shown in Fig. 3. The decrease in the lattice parameter indicates that V, Si

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and Fe, which have the smaller atomic radius, dissolved into Al gradually and formed Al solid solution[12, 13].

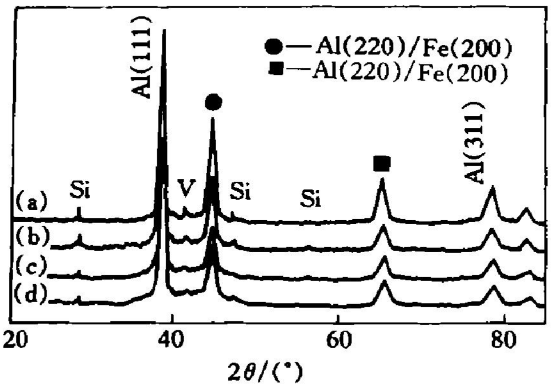

Fig. 1 XRD spectra of MAed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ ($x = 0$) powder mixture after different duration of MA
(a) —24 h; (b) —48 h; (c) —72 h; (d) —96 h

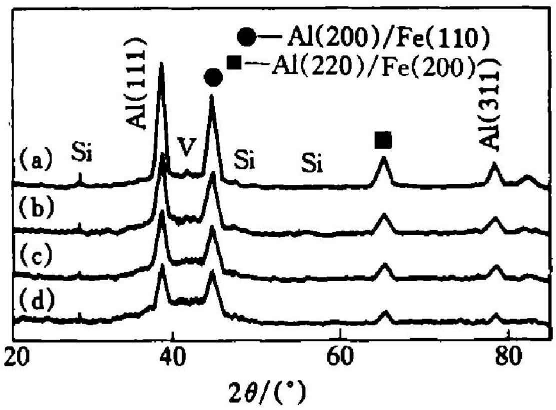

Fig. 2 XRD spectra of MAed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ ($x = 3.0$) powder mixture after different duration of MA
(a) —24 h; (b) —48 h; (c) —72 h; (d) —96 h

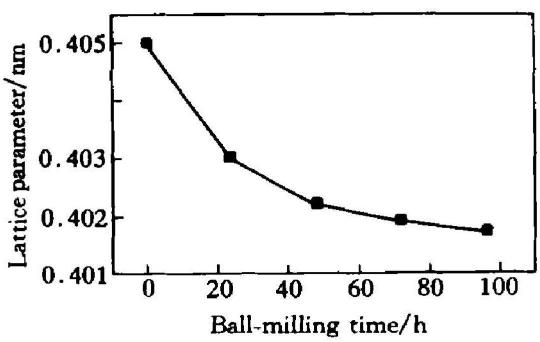

Fig. 3 Variation of lattice parameter of Al with ball-milling time

3.2 Heat treatment resulted in intermetallic precipitate phases

Fig. 4 shows the XRD spectra of the MAed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ ($x = 0, 1.0, 3.0$) alloy powders for 96 h after appropriate annealing treatment. It is seen that the microstructure of three kinds of $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ ($x = 0, 1.0, 3.0$) alloy powders mainly consisted of nanocrystalline α(Al) and α-Al_{13}(Fe, V)$_3$Si phase, which is just what we expected. Due to the refined α-Al_{13}(Fe, V)$_3$Si dispersoid with excellent thermal stability, which prevented nanocrystalline grains from growing up in the annealing. In addition, a new phase appeared in the alloy powder with the content of 1.0% Nd, the microstructure of which has not been indentified yet, while Al_3Nd phase formed in the alloy powder with the content of 3.0% Nd, whose effect on the alloy need further investigation.

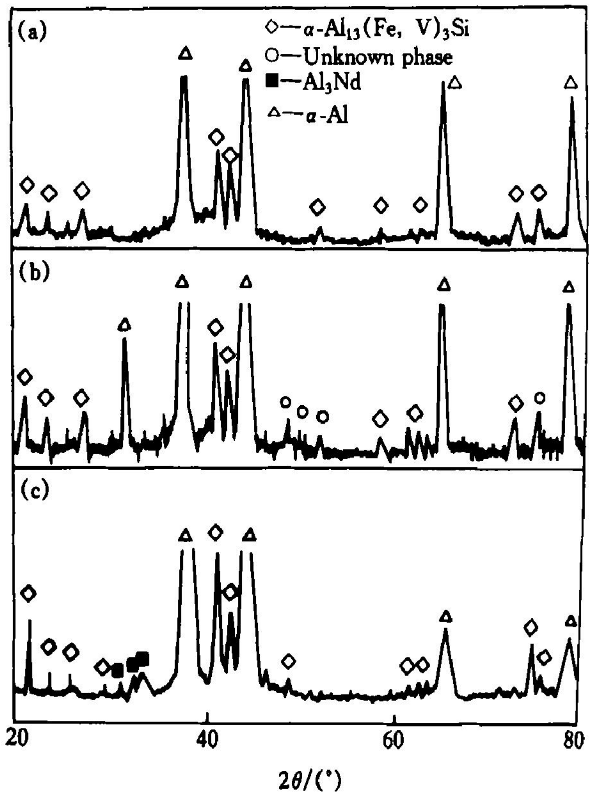

Fig. 4 XRD spectra of MAed $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ ($x = 0, 1.0, 3.0$) powder after appropriate annealing treatment of 96 h
(a) —640 K, 20 min; (b) —660 K, 20 min; (c) —630 K, 10 min

3.3 Rare earth Nd enhanced grain refining and inclination of amorphous of alloy

The refining effect of Nd on the alloy grains during the mechanical alloying is shown in Table 1. It is found that the average size of Al grains decreases with increasing ball-milling time. Moreover, upon the same ball-milling time, the average size of Al grains decreases with increasing content of Nd, as evidenced in dark field image of TEM (see Fig. 5).

At the meantime, with increasing amount of Nd, the amorphous inclination of Al solid solution also increased. It is seen from Fig. 6(a) that the (111) and (200) diffraction rings of Al couble be clearly identified in selected area diffraction pattern of the

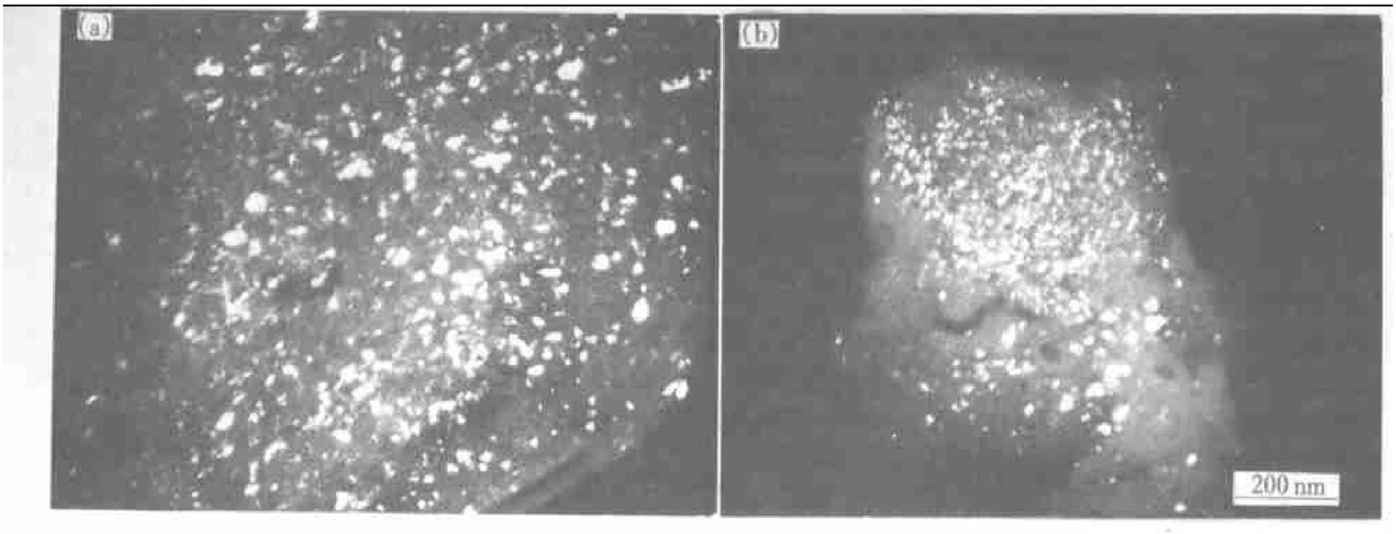

Fig. 5 Dark field images of TEM of MAed alloy powder of 96 h
(a) —$Al_{93.3}Fe_{4.3}V_{0.7}Si_{1.7}$($x$ = 0); (b) —$Al_{93.3}Fe_{4.3}V_{0.7}Si_{1.7}$($x$ = 3.0)

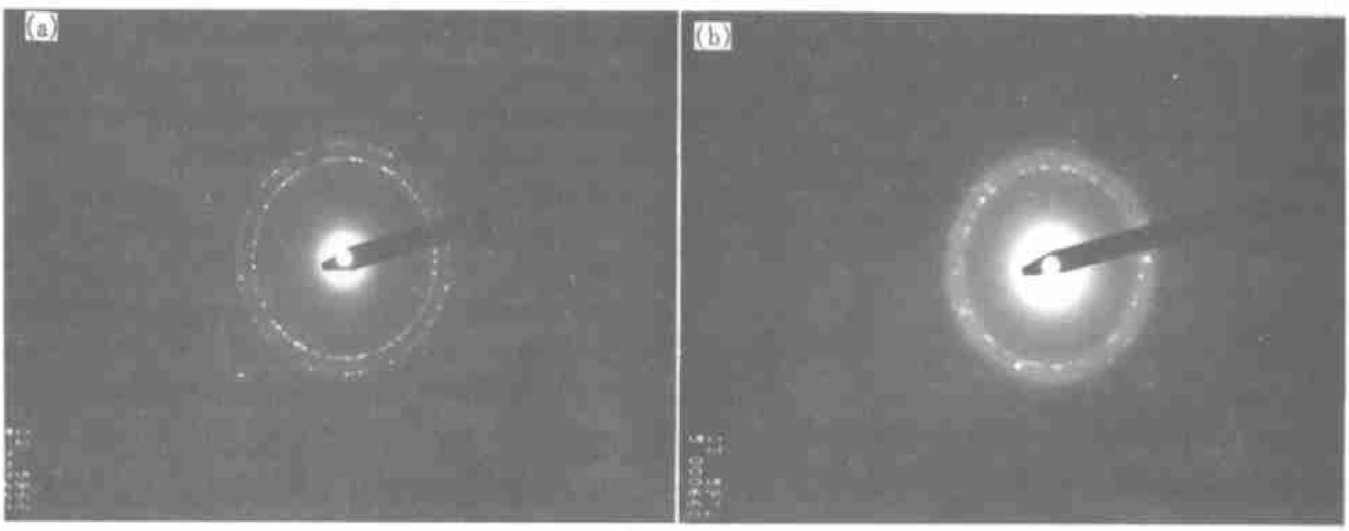

Fig. 6 Selected area diffraction patterns of MAed alloy powder of 96 h
(a) —$Al_{93.3}Fe_{4.3}V_{0.7}Si_{1.7}$($x$ = 0); (b) —$Al_{93.3}Fe_{4.3}V_{0.7}Si_{1.7}$($x$ = 3.0)

MAed $Al_{93.3}Fe_{4.3}V_{0.7}Si_{1.7}$ alloy powder without Nd content for 96 h. However, the pattern of the alloy powder with 1.0% Nd amount (see Fig. 6(b)), a broad halo ring appeared between the (111) and (200) diffraction rings of Al, which indicating some volume fraction of amorphous phase formed in the powder. That is to say, addition of Nd enhanced the grain refining and amorphous inclination of Al solid solution.

3 CONCLUSIONS

1) Till vibration ball-milling for 96 h the microstructure of $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) alloy powder still consisted of single nanocrystalline α(Al) solid solution, without the trace of presence of α-Al_{13}(Fe, V)$_3$Si and Al_8Fe_4Nd intermetallic compound.

2) After appropriate heat treatment, the microstructure of $Al_{93.3-x}Fe_{4.3}V_{0.7}Si_{1.7}Nd_x$ (x = 0, 1.0, 3.0) alloy powder consisted mainly of nanocrystalline α(Al) phase and α-Al_{13}(Fe, V)$_3$Si phase.

3) The addition of rare earth Nd enhanced the grain refining and amorphous inclination of Al solid solution during MA process, and resulted in new phase formed after heat treatment.

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