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Formation regularity of phases in nanometer powders of Al-Fe alloy prepared by gas evaporation①

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[Abstract] Nanometer powders of Al-Fe alloy were prepared by gas evaporation. The formation regularity of the phases in the as-prepared powders and the morphology of the particles were examined. The experimental results show that chemical composition of the master alloy is the key factor which controls the chemical composition of the compound phases in nanometer powders at given evaporating temperature, the compound phases with high Fe mole fraction will form with increasing of Fe content in master alloy. Only $Al_{13}Fe_4$, $FeAl_2$ and Al_2Fe compound phases form in nanometer powders in present experiment, changing of the pressure of Ar can only alter relative amounts of the compound phases in the powders. Nanometer particles with inhomogeneous tissue were obtained, which is very different from that of pure Al and Fe nanometer particles. When mole fraction of Fe in particles increases, the inhomogeneity is enhanced.

[Key words] gas evaporation; vapor condensation; nanometer powders; Al-Fe alloy
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1 INTRODUCTION

Nanometer alloy powders are expected to exhibit unique characteristics which can not be obtained for pure metal powders and conventional materials. More recently, a series of studies on the preparation technique and properties of nanometer powders were carried out, several preparation techniques were developed, such as gas evaporation, gas chemical reduction, alloy wire explosion. Gas evaporation technique is the most effective one, in which resistance with spiral or boat shape, laser beam or plasma flame can be chosen as the heating source[1~4].

It is of significance to investigate the formation regularity of the phases in nanometer alloy powders for controlling their phase composition. In this paper, nanometer powders of Al-Fe alloy were prepared by evaporating master alloys with different compositions under various Ar pressure using induction heating source and formation regularity of the phases in the powders as well as morphology of the particles are investigated.

2 EXPERIMENTAL

The experimental apparatus and procedure were the same as mentioned in a previous paper[5] with a powder collector of 800 mm in diameter and 900 mm in height. The Al-Fe master alloys were evaporated at 1 773 K in Ar with the purity about 99.995%, using an alumina crucible of 120 mm in diameter and 100 mm in height. Purities of the two metals were both about 99.95%. Temperature of the alloy melt was examined by an optical pyrometer and controlled by adjusting the input power of the equipment. A deactivating oxidation treatment was applied to the as-prepared powders before being exposed to air after the evaporation procedure. Samples were collected from the water cooled powder collector. There was no changing in Ar pressure and obvious temperature fluctuation during the experiment.

The phase composition and the lattice parameter a_0 of Al in the as-prepared nanometer powders were examined by X-ray diffraction (XRD). Transmission electron microscopy (TEM) was used to investigate the morphology and tissue of the nanometer particles.

3 RESULTS AND DISCUSSION

3.1 Formation of phases in powders under different conditions

The X-ray diffraction patterns of master alloy $Al_{1-x}Fe_x$ ($x = 0.33, 0.42, 0.53, 0.59, 0.66$) (mole fraction) powders prepared at 1 773 K in 1.0×10^2 Pa Ar are shown in Fig.1. When the master alloy $Al_{0.67}Fe_{0.33}$ is evaporated, α-Al phase forms, no examinable amount of a compound phase is found. When mole fraction of Fe in master alloy increases, the compound phase of $Al_{13}Fe_4$ forms, its relative amount increases rapidly when the mole fraction of Fe changes from 0.33 to 0.59. When the mole fraction of Fe increases to 0.66, a compound phase of $FeAl_2$ forms with a little of Al_2Fe phase which has the same chemical composition and different crystal lattice

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forms. It is obvious that the compound phases with higher Fe mole fraction form as the content of Fe in the master alloy increases. The formed phases and their relative amount are obviously affected by composition of the master alloy for given conditions.

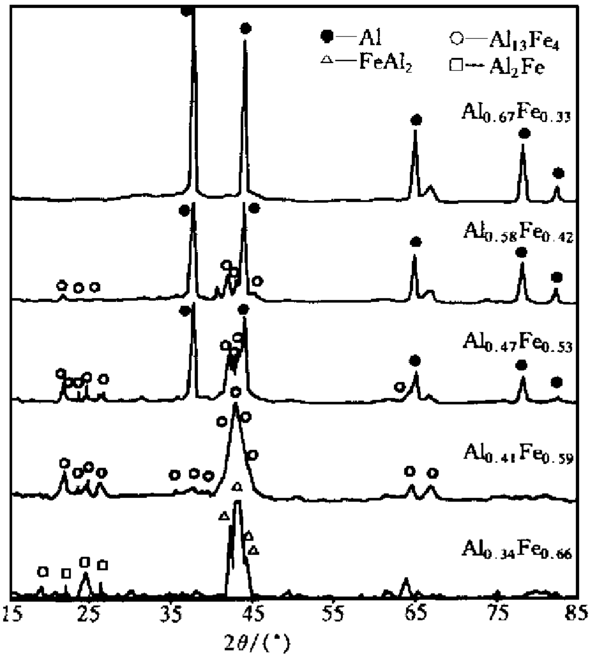

Fig.1 XRD patterns of prepared powders

From the XRD results in the as-prepared powders at different evaporating conditions, the lattice parameter a_0 of the α-Al phase is obtained. There is a little decrease of a_0 for master alloy of $Al_{0.67}Fe_{0.33}$ in contrast to that of pure Al, this means there is a little solid solubility of Fe atoms in the lattice of α-Al. But when the mole fraction of Fe in master alloy increases to 0.59, the a_0 is equal to that of pure Al within the scale of instrument error, this means no solid solution of Fe atoms in Al lattice in the particles in this case. It can be considered that most of the Fe atoms engage in the formation of compound phase.

When a master alloy is evaporated, the mole fraction of Al and Fe in the mixed vapor can be calculated by Eqns.(1) and (2):

$$x(\mathrm{Al}) = \frac{p(\mathrm{Al})}{p(\mathrm{Al}) + p(\mathrm{Fe})},$$
$$x(\mathrm{Fe}) = \frac{p(\mathrm{Fe})}{p(\mathrm{Al}) + p(\mathrm{Fe})} \qquad (1)$$
$$p(\mathrm{Al}) = a(\mathrm{Al})\, p^0(\mathrm{Al}),$$
$$p(\mathrm{Fe}) = a(\mathrm{Fe})\, p^0(\mathrm{Fe}) \qquad (2)$$

where $a(\mathrm{Al})$ and $a(\mathrm{Fe})$ are the activities of components of Al and Fe in an alloy melt, $p^0(\mathrm{Al})$ and $p^0(\mathrm{Fe})$ are the saturation vapor pressures at given temperature and can be calculated by Eqn.(3)[6].

$$\left.\begin{aligned} \lg p(\mathrm{Al}) &= -16\,450/T - 1.23 \lg T + 12.36 \\ \lg p(\mathrm{Fe}) &= -21\,080/T - 2.14 \lg T + 16.89 \end{aligned}\right\} \qquad (3)$$

When $T = 1\,773$ K, then $p^0(\mathrm{Al}) = 12.35$ Pa, $p^0(\mathrm{Fe}) = 1.13$ Pa. It can be concluded that the mole fraction of Fe in the mixed vapor increases with increasing Fe content of the master alloy. Table 1 gives the mole fraction of Fe in the mixed vapor vs the chemical composition of the master alloy under the condition of ideal evaporation.

Table 1 Fe contents in mixed vapor and chemical composition of master alloy

Chemical composition	a(Al)	a(Fe)	x(Al)	x(Fe)
$Al_{0.67}Fe_{0.33}$	0.824	0.0145	99.84	0.16
$Al_{0.58}Fe_{0.42}$	0.609	0.0721	98.93	1.07
$Al_{0.47}Fe_{0.53}$	0.3321	0.1134	98.07	1.93
$Al_{0.41}Fe_{0.59}$	0.2549	0.1556	94.73	5.27

For alloy $Al_{0.67}Fe_{0.33}$ evaporated at 1 773 K, x(Fe) is 0.16% which is higher than the maximum solid solubility of Fe atoms in Al lattice which is about 0.025% at 928 K and near to zero at room temperature[7]. But no examinable amount of compound phase forms in the powders according to Fig.1. The reason may be that x(Fe) is not high enough for a compound phase to form or the amount of the compound phase is less than the scale of instrument error. When the mole fraction of Fe in the mixed vapor is high enough, at least higher than 0.16% in this experiment, a compound phase appears in the as-prepared powders. It is obvious that the chemical composition of the compound phase is affected by the mole fraction of Fe in the mixed vapor. The higher the mole fraction of Fe in the mixed vapor, the higher the mole fraction of Fe in the compound phase, and the larger the amount of the compound phase referring to the changing in the strength of diffraction peak of the compound phase in Fig.1. It is complicated to accurately calculate the mole fraction of Fe in the mixed vapor and the amount of compound phases formed, because the evaporation behavior of the alloy melt and the formation dynamic behavior of a certain compound phases is obviously affected by the process parameters.

The master alloy of $Al_{0.34}Fe_{0.66}$ was chosen to investigate the formation regularity of the phases in the powders prepared at 1 773 K with Ar pressure changing from 1.0×10^2 Pa to 1.0×10^4 Pa, the X-ray diffraction patterns of the powders are given in Fig.2.

The phases formed in as-prepared powders are same as that in the powders prepared at 1.0×10^2 Pa as given in Fig.1. It is obvious that there is no notable change in kinds of the formed phases in the powders, but their relative amounts change according to the X-ray diffraction peak intensity of the phases. This regularity is same as that obtained in Ref.[5].

The compound phases Fe_3Al and Al_5Fe_2 in Al-Fe alloy diagram do not form in this experiment. It is hard for Fe_3Al phase to form in gas evaporation process because the cooling rate of the alloy vapor and

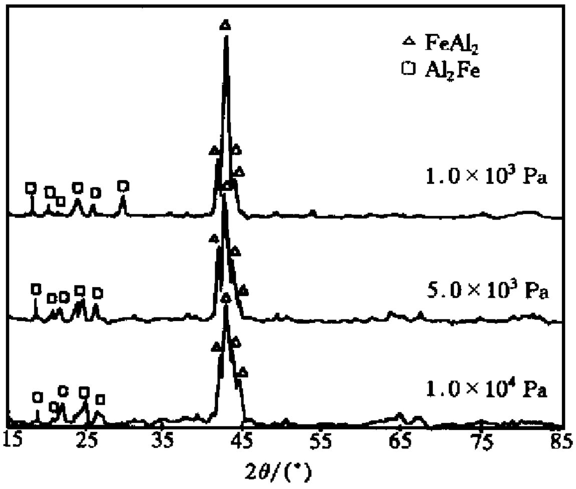

Fig.2 XRD patterns of prepared powders under different Ar pressure

particles is too high to satisfy the formation conditions of this phase. This phenomena were confirmed by other studies[5,8]. The reason why Al_5Fe_2 phase can not form during gas evaporation process may be a dynamic problem that waits for further study.

3.2 Particle morphology

Fig.3 shows the TEM micrographs and tissues of the particles under different experiment conditions.

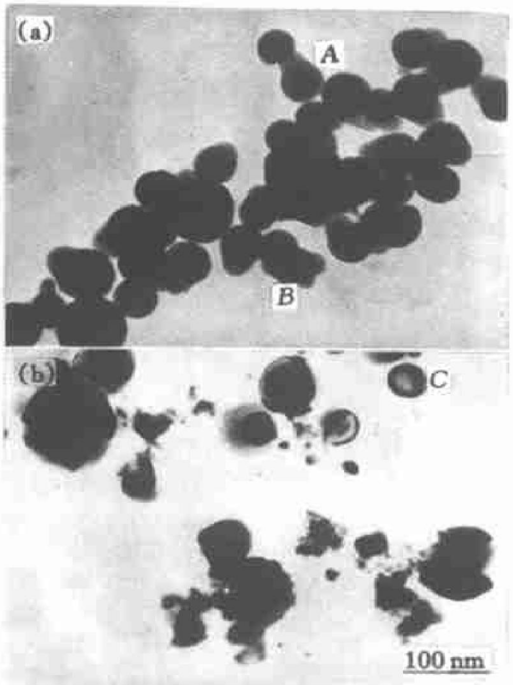

Fig.3 Micrographs of nanometer particles ($1\ 773$ K, 1.0×10^2 Pa)
(a) —$Al_{0.41}Fe_{0.59}$; (b) —$Al_{0.34}Fe_{0.66}$

The size of the particles in Fig.3(a) is 30 ~ 50 nm, most of the particles marked as *A* was ellipsoidal, a few of particles marked as *B* are inhomogeneous tissue which show different scattering contrast. According to Fig.1, it can be concluded that there may be $Al_{13}Fe_4$ phase in this particles.

The size of the particles in Fig.3(b) is 15 ~ 50 nm. Some particles marked as *A* show homogeneous tissue, but most particles marked as *B* and *C* show different scattering contrast, it can be concluded that particles *B* and *C* may be two-phase structures.

The morphology and tissue of the particles with compound phases in Fig.3 are very different from that of pure Al and Fe nanometer particles[9]. When mole fraction of Fe in particles increases, the inhomogeneity is enhanced.

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