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Phase formation regularities in Fe-based nanometer powders prepared by gas evaporation process①

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[Abstract] A serials of Fe-based nanometer powders were fabricated by reduced pressure gas evaporation process with induction current as the heating source. The formation regularities of the phases in as-prepared powders and the structures of the nanometer particles were investigated. Pure Fe nanometer powders with about 70% γ-Fe phase is prepared in present study by using the powder collector with good cooling effect. In the nanometer powders of Fe-Ni alloy, solid-solution phase γ-(Fe, Ni) and α-Fe phase form, but for Fe-Cr alloys only solid-solution phase α-(Fe, Cr) forms. In the nanometer powders of Fe-Cu alloy, only pure metal phases of γ-Fe and Cu form, and no compound or solid solution phase exists. The formation regularity of the phases in the nanometer powders of alloys obeys the common phase laws in bulk alloy state.

[Key words] gas evaporation; nanometer powder; Fe-based powder
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1 INTRODUCTION

Nanometer powders with particle size less than 100 nm present many novel properties absent in bulk materials and have been widely applied in metallurgy, material and chemistry fields[1~3]. It has been shown that Fe-based nanometer powder has unique magnetic properties, good catalytic characteristics in reactivity, selectivity and durability[4,5]. Several technologies have been developed for preparation of Fe-based nanometer powder, such as gas evaporation process in reduced pressure inert gas using induction electron current as heating source, liquid chemical reduction process and plasma evaporation technology[1~3]. A serials of experiments have been conducted to investigate alloy and composite nanometer powders[1,6~11].

It is of significance to investigate the formation regularities of the phases in nanometer alloy powder for controlling the kinds of the phases and their relative amount in as-prepared powders. In present study, two kinds of static powder collector were used to prepared pure Fe and Fe-M (M = Ni, Cr, Cu) nanometer alloy powders by gas evaporation technology using induction coil as the heating source. The formation regularity of the phases in as-prepared powder, the morphology and structures of the nanometer particles were investigated.

2 EXPERIMENTAL

The experimental devices for pure Fe and Fe-M nanometer powder were the same as mentioned in Refs. [10] and [12], respectively. Pure Fe and Fe-M (M = Ni, Cr, Cu) master alloys were evaporated at 1873 K in Ar atmosphere with high purity at 1.0×10^2 Pa in an alumina crucible with the dimension of d 120 mm × 100 mm. The purity of the metals were all about 99.95%. The temperature of the melt in the crucible was examined by an optical pyrometer and regulated by adjusting the input power of the device. A deactivating oxidation treatment was applied to the as-prepared powders before being exposed to air. There was no obvious changing in Ar gas pressure and temperature during the evaporation procedure.

X-ray diffraction meter was used to test the phases and their lattice parameters. Transmission electron microscope was used to examine the morphology and structures of the nanometer particles.

3 RESULTS AND DISCUSSION

3.1 Phases in pure Fe nanometer powder

Generally speaking, when pure Fe is evaporated in a device with poor cooling effect, α-Fe phase usually forms in as-prepared powder. But if the primary nanometer particles are immediately quenched or are reheated and then quenched in a special cooling device, γ-Fe phase will form[3]. In present study, two static collectors specially devised for nanometer powder with different cooling effect were used to prepare Fe nanometer powder, and the X-ray diffraction pattern of the Fe powder is shown in Fig. 1. The relative amount of γ-Fe phase is 70% according to the

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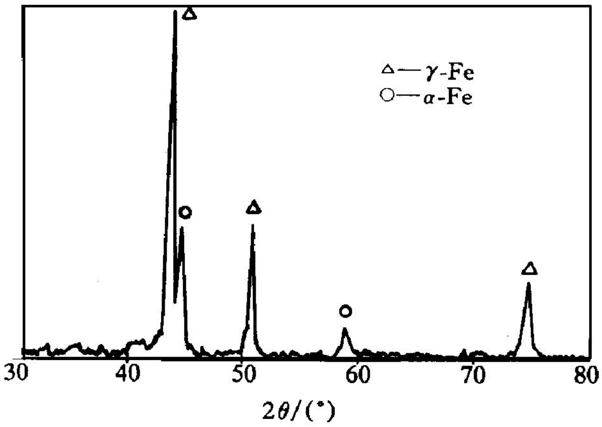

Fig. 1 X-ray diffraction patterns of pure Fe nanometer powders

semi-quantity calculation by a computer software, which means that the device has excellent cooling effect.

3.2 Phases in Fe-Ni and Fe-Cr alloy nanometer powders systems

Several Fe-Ni alloys with the composition of $Fe_{1-x}Ni_x$ (x = 4.77%, 10.52%, 24.08%, mole fraction) were respectively evaporated at 1 873 K in Ar atmosphere at 1.0×10^2 Pa. The X-ray diffraction patterns of as-prepared nanometer powders are shown in Fig. 2. It can be seen that the alloy powders are composed of α-Fe phase and γ-(Fe, Ni) phase which is an unlimited solid solution. Fig. 3 gives the change of the lattice parameter d_{200} of γ-(Fe, Ni) phase in the alloy powders with the composition of the master alloys. There is a continuous increasing of d_{200} with increasing mole fraction of Ni in the master alloys. There is no obvious changing in that of α-Fe phase.

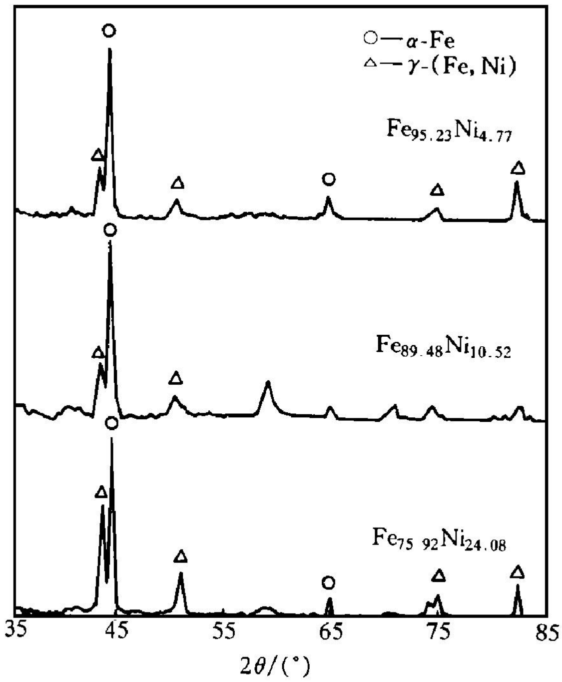

Fig. 2 X-ray diffraction patterns of Fe-Ni nanometer powders

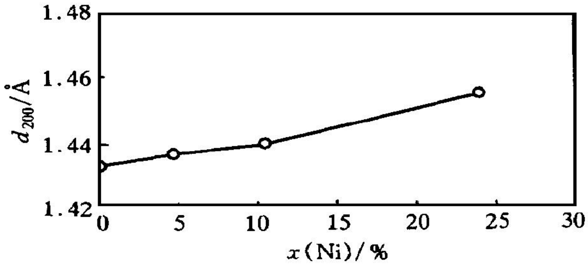

Fig. 3 Lattice parameter d_{200} of γ-(Fe, Ni) phase vs composition of master alloys

A serials of Fe-Cr alloys with composition of $Fe_{1-x}Cr_x$ (x = 10.7%, 15.9%, 21.2%, 26.4%, 31.5%, mole fraction) were respectively evaporated at 1 873 K in the argon atmosphere at 1.0×10^2 Pa. The X-ray diffraction patterns of the obtained nanometer powders are shown in Fig. 4. The results show that the alloy powders are mainly composed of α-(Fe, Cr) which is an unlimited solid solution phases. The tiny amount of $FeCr_2O_4$ phase is caused by the oxidation reaction of the powder in the air. Fig. 5 gives the change of the lattice parameter (a_0) of α-(Fe, Cr) phase with the composition of the master alloys. There is a continuous increasing of a_0 with increasing mole fraction of Cr in the master alloy.

Fe-Ni and Fe-Cr alloys belong to the unlimited solid-solution alloy system. From Fig. 4 and Fig. 5, it can be concluded that the formation regularities of the

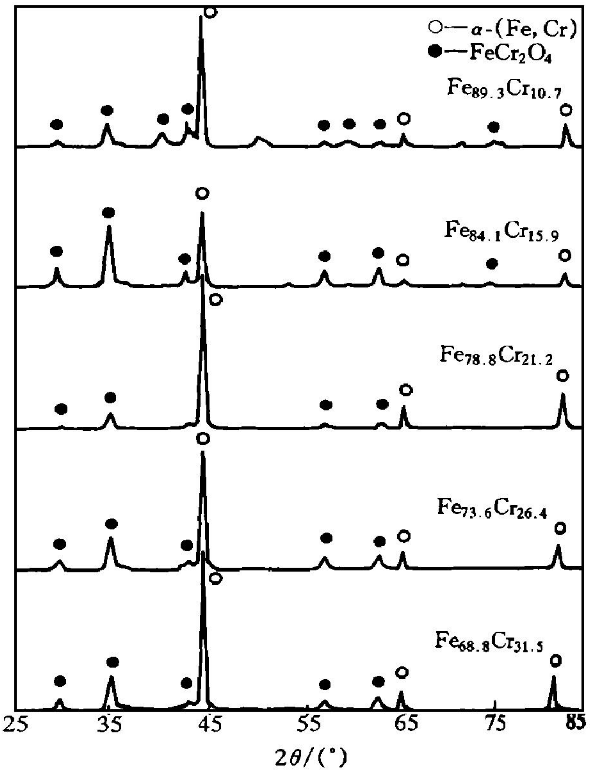

Fig. 4 X-ray diffraction patterns of Fe-Cr nanometer powders

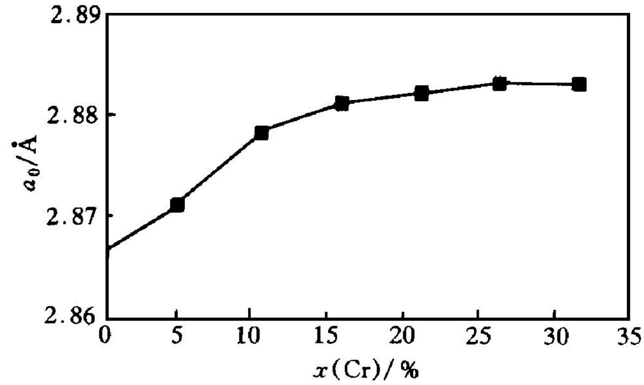

Fig. 5 Lattice parameter a_0 of α-(Fe, Cr) phase in powders

phases in nanometer powders obey the common phase laws in the bulk alloys.

3.3 Phases in Fe-Cu nanometer powders

Two master alloys with the composition of $Fe_{1-x}Cu_x$ (x = 3.1%, 11.2%, mole fraction) were respectively evaporated at 1 873 K in Ar gas at 1.0×10^2 Pa. The X-ray diffraction patterns of the nanometer powders are shown in Fig. 6. Only γ-Fe and pure Cu phases form in the as-prepared powders according to the automatic indexing of the diffraction peaks by a computer software. The diffraction peaks of γ-Fe phase and pure Cu phase overlap together because of their nearly equal diffraction angle and lattice index.

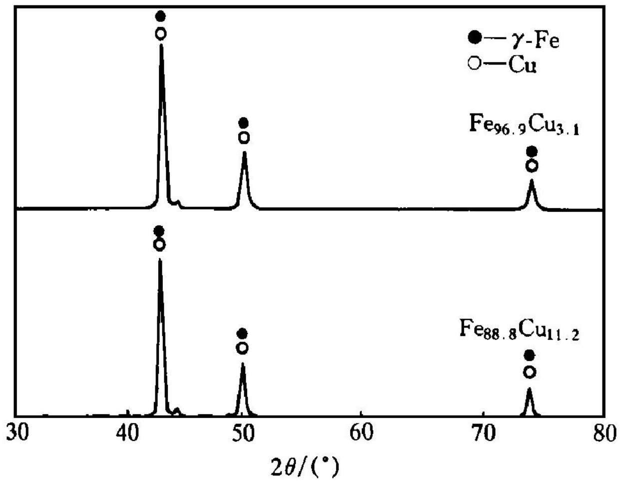

Fig. 6 X-ray diffraction patterns of Fe-Cu nanometer powders

3.4 Particle morphology and structure

Fig. 7 presents the TEM morphology of the Fe particles. The particle size in Fig. 7(a) ranges from 15 nm to 50 nm. The morphology shows the typical feature of the nanometer particles of α-Fe, and the particles arrange in chain-like because of their magnetic property. But the crystal feature of the particles in Fig. 7(b) is very different from the former. The average particle size is about 50 nm. There are hexagonal α-Fe particle (marked as A), truncated triangular γ-Fe plates (marked as B)[3] and irregular shape particles which may be result from the good cooling effect of the device.

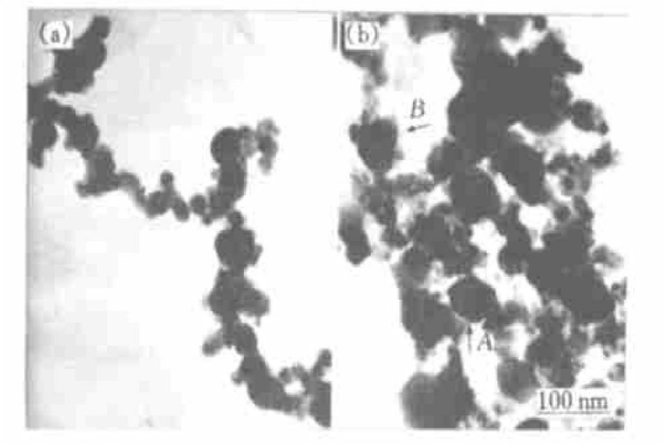

Fig. 7 TEM morphology of Fe particles
(a) —Pure Fe; (b) —Fe-M alloy

Fig. 8(a) shows the crystal feature of Fe-Ni alloy nanometer particles. The average particle size is below 80 nm. The shape of the particles are sphere and irregular. Obvious different scattering contrast exists in the particles, which means inhomogeneous structure. Referring to the results given in Fig. 2, it is clear that the particles are composed of two phases.

Fig. 8(b) shows the crystal feature of Fe-Cr nanometer particles. The average size of the particles

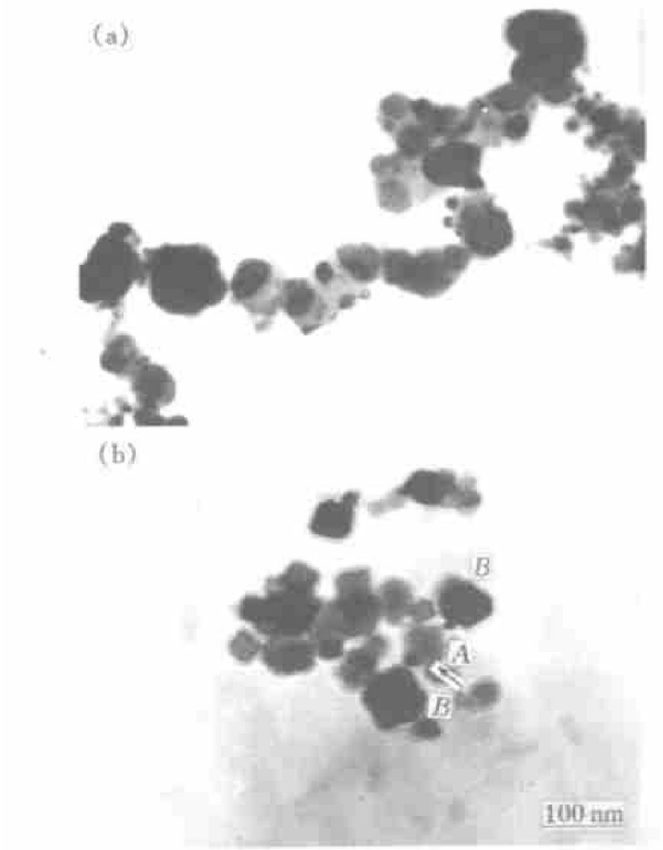

Fig. 8 TEM morphology of Fe-Ni and Fe-Cr alloy particles
(a) —$Fe_{75.92}Ni_{24.08}$; (b) —$Fe_{73.6}Cr_{26.4}$

is about 50 nm. Most of the particles show cubic crystal shape (marked as *A*), which is similar to that of α-Cr particles[3]. There are also a few truncated tetragonal particles (marked as *B*). There is no diffraction contrast among the particles, which means the particles are single phase. This is coincide with the X-ray diffraction result in Fig. 4.

The TEM morphology of Fe-Cu nanometer particles is shown in Fig. 9. Some particles are spherical in shape (marked as *A*), and main particles are irregular with rough surface. Twin interface (marked as *B*) and inhomogeneous tissue (marked as *C*) appear in many particles. According to the phase diagram of Fe-Cu alloy, it is clear that this inhomogeneous structure means different phase. The formation of these phases may be related to the thermodynamic property of Fe-Cu alloy and the growth mechanism of the particles.

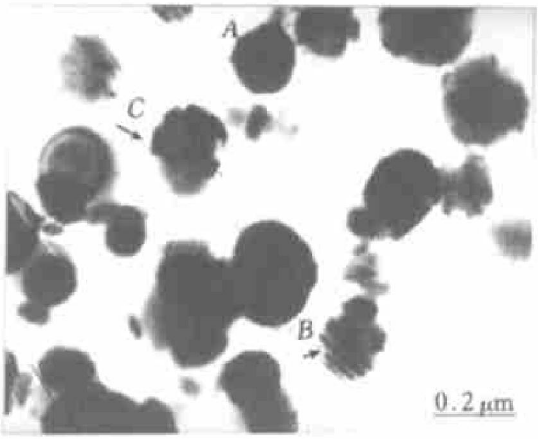

Fig. 9 TEM morphology of Fe-Cu alloy particles

5 CONCLUSIONS

1) Pure Fe nanometer powders with about 70% γ-Fe phase is prepared by using the powder collector with good cooling effect.

2) In the nanometer powders of Fe-Ni alloy, solid-solution phase γ-(Fe, Ni) and α-Fe phase form; but for Fe-Cr alloys, only solid-solution α-(Fe, Cr) forms. For Fe-Cu alloy nanometer powders, only γ-Fe phase and Cu phase form without any compound or solid solution phase.

3) The formation regularities of the phases in nanometer alloy powders obey the common phase laws in the bulk alloy.

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