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Magnetic properties of nanostructural γ-Ni-28Fe alloy

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Abstract: Nanostructural γ-Ni-28Fe alloy (nano γ-Ni-28Fe) was successfully prepared by mechanochemical alloying(MCA). The relationship between the microstructure and the synthesis conditions was investigated by using XRD, TEM, SEM as well as BET analyzer. The results show that nano γ-Ni-28Fe alloy is composed of a gamma phase (FCC structure). Its grain size is about 20 nm at reduction temperature below 600 ℃. The magnetic measurements indicate that the saturation magnetization of nano γ-Ni-28Fe alloy is 102.4 $A \cdot m^2/kg$, and the coercivity is much higher than that of conventional coarse-grained counterpart. The result may be attributed to its decrease of the grain size and chemical composition in nano γ-Ni-28Fe alloy.

Key words: nanostructural material; Ni-28Fe alloy; mechanochemical alloying; microstructure; magnetic property

1 Introduction

The nickel-iron alloys in the permalloy system, with about 10% to 65%(mass fraction) iron, are probably important soft magnetic alloys[1], which have been widely used in industry, such as recording heads, transformers or magnetic shielding materials. Among them, γ-Ni-22Fe alloy has been particularly emphasized because of its higher permeability, lower coercivity and relative high saturation magnetization. If much higher saturation magnetization is required for a certain application, one needs to increase the iron content in the permalloy system. However, this will lead to an increase in coercivity and a decrease in permeability due to the increase of the magnetocrystalline anisotropy with the rise of the iron content in Ni-Fe system[1], which will deteriorate its performance as a soft magnetic material. Recent experimental and theoretical studies have shown that nano-processing can effectively reduce the magnetocrystalline anisotropy constant of a material as long as the grain size is smaller than the ferromagnetic exchange length in this material[2, 3]. In other words, we can control its magnetic behavior by using the nano-processing, so one can obtain the permalloy of higher saturation magnetization without sacrificing other magnetic performances. Furthermore, the nano-processing of conventional materials has been proven to be very effective to their other properties[4−6]. In the case of Ni-Fe alloys, it leads to improvements of its properties, such as mechanical hardness, electrical resistivity, wear resistance and high-frequency performance due to the reduction of the eddy currents[7]. Therefore, the nano-processing seems to be a potential and promising measure to improve the magnetic properties of the Ni-Fe alloy system.

Recently, nanostructural γ-Ni-Fe (nano γ-Ni-Fe) alloy powders have been fabricated through various techniques such as elemental powders mechanical attrition[8,9], electrodeposition[10] and film deposition [11]. However, these processes induced one or all of the following disadvantages: internal strain, impurities, a low quantity or a high cost. To solve these problems, a mechanochemical alloying(MCA) was used to prepare nanostructural materials[12,13]. This method is suitable as the synthesis is scaled up easily and cheaply, and it can also avoid internal strain and contamination of nanosized powders. In the present work, the synthesis

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and magnetic properties of nano γ-Ni-Fe alloy by using MCA were investigated.

2 Experimental

The nano γ-Ni-28Fe alloy was prepared by using the mechanochemical alloying process. Namely, reagent grade NiO and Fe_2O_3 were weighed and sealed together with ethanol in the steel containers. The ball milling was performed in a planetary miller at a speed of 250 r/min for 24 h. The steel ball to powder mass ratio was 6:1. After ball milling, they were dried at 80 ℃ for 24 h. All the milled powders were reduced and in-situ alloyed for 30 min at 500, 600, 700 and 850 ℃, respectively.

The crystalline structure was examined by X-ray diffraction with Cu $K_{\alpha 1}$ radiation. The mean grain size was evaluated based on Scherrer formula. The main constituent elements were determined by energy dispersive X-ray spectroscopy(EDS) equipped with scanning electron microscope(SEM). The microstructural characteristics such as morphology, particle size and crystal structure were further confirmed by transmission electron microscopy(TEM). The specific surface area of the nano γ-Ni-28Fe powders was determined by the Brunauer-Emmett-Teller(BET) method with nitrogen adsorption. The specimens used for magnetic measurements were prepared by compacting the nanopowder into bar with a shape of 9.3 mm×4.7 mm×1.6 mm. The relative densities of the specimens were around 0.64. The magnetic properties were measured with vibrating sample magnetometer(VSM) made in Japan.

3 Results and discussion

NiO and Fe_2O_3 can be reduced into Ni and Fe elements by using hydrogen, and its reduction temperatures are around 310 ℃ and 450 ℃, respectively [14]. Fig.1 shows the X-ray diffraction patterns of nano γ-Ni-28Fe alloy reduced in H_2 atmosphere and in-situ alloyed at different temperatures for 30 min. All diffraction peaks are identified to be a face central cubic (fcc) crystalline structure (γ phase). Since no superlattice reflection peak is detected, it can be deduced that the obtained nano γ-Ni-28Fe alloy is random solid solution[15]. With the increase of temperature, the main features of XRD patterns do not change (Fig.1), except for the sharpening of the reflection peaks. The grain size *d* obtained from (111) and (200) reflection peaks of nano γ-Ni-28Fe powders is plotted in Fig.2 as a function of the temperature. It shows that at 600 ℃, the average grain size is around 20 nm estimated by means of the Scherrer formula.

The influence of in-situ alloying temperature on the formation of the nano γ-Ni-28Fe alloy is monitored by XRD patterns. One can find that an incomplete γ phase exists in the specimens reduced at 500 ℃ for 30 min (inset in Fig.1). With in-situ alloying temperature increasing, the residual nano-Fe element reflection peaks gradually disappear. It is conceivable that the stable γ-Ni-28Fe alloy is successfully synthesized at temperature of 600 ℃.

The morphology of nano γ-Ni-28Fe alloy powders is further characterized by SEM and TEM. It is seen that the size of Ni-Fe nanoparticles synthesized at 600 ℃ is around 30 nm (Fig.3), which is consistent with the XRD results. One can also see that fine particles can form intricate agglomerates in order to minimize the magnetostatic energy and surface energy. Fig.4 shows the specific surface areas of nano γ-Ni-28Fe alloy as a

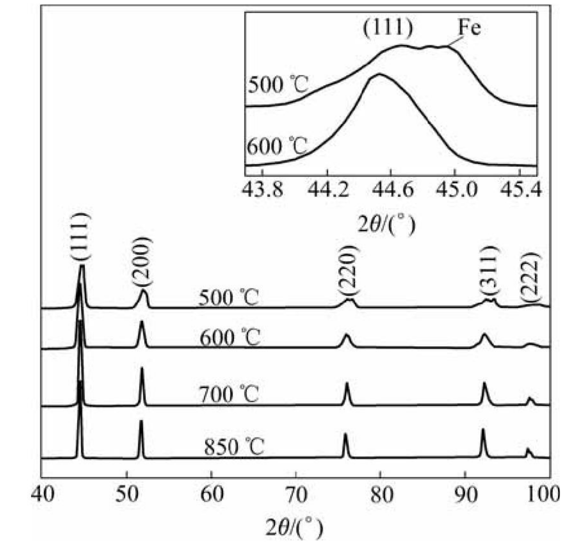

Fig.1 XRD patterns of nano γ-Ni-28Fe alloy by mechanochemical alloying at different temperatures

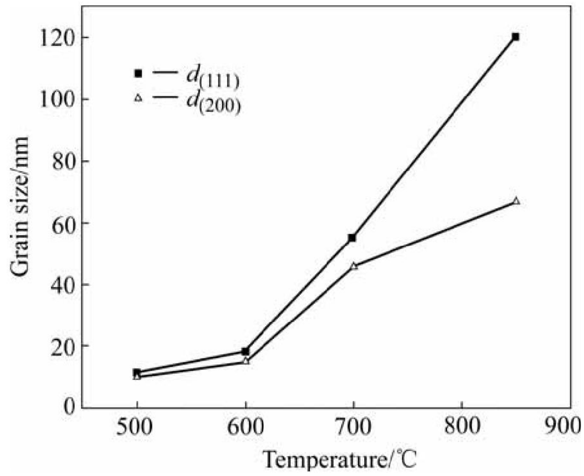

Fig.2 Mean grain dimensions in (111), (200) reflection peaks of nano γ-Ni-28Fe alloy as function of reduction temperature

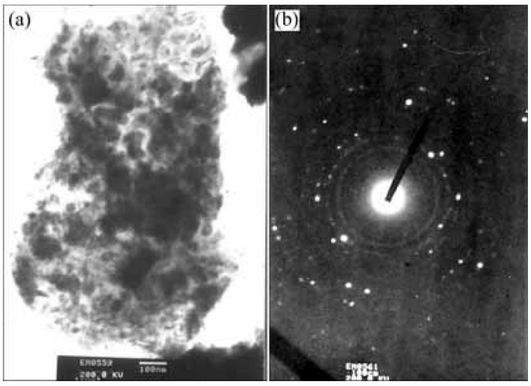

Fig.3 TEM micrograph (a) and selected area electron diffraction pattern (b) of nano γ-Ni-28Fe powders obtained at 600 ℃

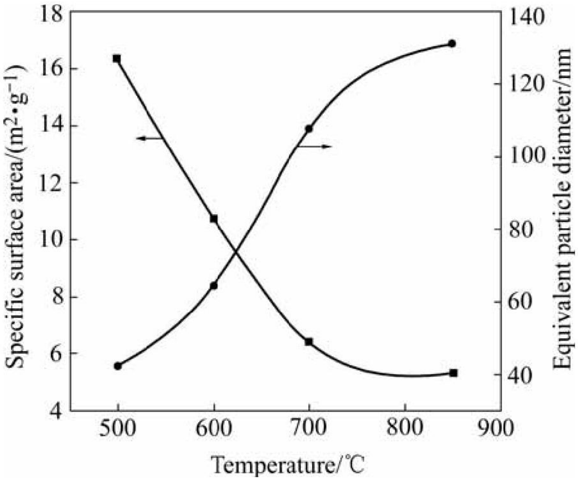

Fig.4 Relationship between specific surface area and equivalent particle size for nano γ-Ni-28Fe alloy with reduction temperatures

function of the temperature. It decreases from 16.35 m^2/g to 5.27 m^2/g as reduction temperature increasing from 500 ℃ to 850 ℃. Corresponding to this change, the agglomerate particle size increases rapidly from 42 nm to 131 nm by using BET analyzer.

Fig.5 shows the hysteresis loop of nano γ-Ni-28Fe alloy at room temperature. Magnetic measurements reveal that the specimens exhibit ferromagnetism, and no super-paramagnetic phenomenon is found in the specimens. It can be seen that the magnetization of nano γ-Ni-28Fe alloy reaches saturation at the external field about 800 kA/m. The saturation magnetization M_s is around 102.4 A·m^2/kg, and the coercivity is 9.87 kA/m at

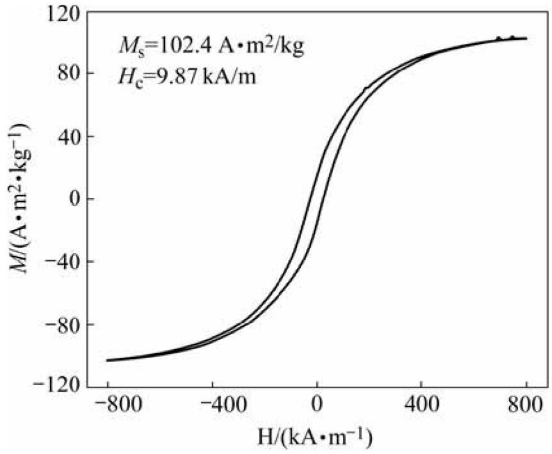

Fig.5 Hysteresis loop of nano γ-Ni-28Fe alloy at room temperature

It is well known that the magnetic properties of nanostructural materials depend on chemical composition and grain size[16]. Table 1 lists the compositions of nano γ-Ni-28Fe alloy. The results indicate that apart from Ni and Fe elements, there is about 1.7%Cr (mass fraction) in the specimens, which is induced by the ball milling process. In present experiments, the reason why there is such higher coercivity may originate from the decrease of grain size and low density of the specimens[3]. This inhibits the ferromagnetic exchange among nano γ-Ni-28Fe grains, which leads to higher coercivity. It can also be seen that the experimental data of saturation magnetization M_s is lower than the corresponding coarse-grained Ni-28Fe alloy. The decrease in M_s would be due to the decrease of grain size. In addition, the existence of Cr element would also be responsible for a partial decrease of M_s, as shown by other related reports[17, 18].

Table 1 Chemical compositions of nano γ-Ni-28Fe alloy (mass fraction, %)

Ni	Fe	Cr	Other elements
70.3	27.8	1.7	<0.2

4 Conclusions

The most important result of the present study is the successful synthesis of nano γ-Ni-28Fe alloy by using mechanochemical alloying(MCA) process. The nano γ-Ni-28Fe powders show gamma phase (FCC structure), and its average grain size is measured as 20 nm. Magnetic measurements indicate that nano γ-Ni-28Fe chemical compositions and the decrease of grain size.

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