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# Influence of annealing treatment on microstructure and soft magnetic properties of Fe-B-P nanoparticles prepared by aqueous chemical reduction

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**Abstract** Fe-B-P amorphous nanoparticles prepared by aqueous chemical reduction were annealed at 300 °C ∼ 500 °C for 30 min. The microstructure and the soft magnetic properties of the specimens were investigated by X-ray diffraction, Mössbauer spectroscopy, scanning electron microscopy and vibrating sample magnetometry, etc.. The results show that the particles are monodisperse Fe-B-P amorphous nanoparticles and the grain diameter distribution is from 200 to 300 nm with a very narrow size distribution. About 3 % of α-Fe appeared in the amorphous particles after annealing treatment at 300 °C for 30 min. After an annealing treatment at 400 °C and 500 °C for 30 min the samples are completely crystallized. The crystallization phases are α-Fe, $Fe_2B$ and $Fe_3B$. The soft magnetism after 300 °C annealing treatment is better than other specimens, while the full crystallization deteriorates the soft magnetic performance of the specimens after annealing at 400 °C and 500 °C.

**Keywords** Fe-B-P · Amorphous nanoparticles · Annealing · Mössbauer spectroscopy

## 1 Introduction

Soft magnetic nanoparticles have been paid much attention to in many fields such as high frequency microwave absorption, DC-DC conversion, catalysis, etc., because

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of their unique properties. There are many ways proposed to obtain soft magnetic nanoparticles, such as liquid phase chemical reduction, hydrothermal synthesis, polylol reflux, microemulsion, sol–gel, co-precipitation, etc. [1–3]. Among these the aqueous chemical reduction is a facile process with tunable size and shapes. The Fe base amorphous alloys, especially small sized particles, consisting of metal and metalloid, have low coercive field, high saturation magnetization and high initial magnetic permeability, and therefore have attracted increasing attention up to now in the field of high frequency electromagnetic noise absorption and DC-DC conversion. At the same time, the soft magnetic nanoparticles have also been proposed in the field of nanomedicine, such as cancer hyperthermal treatment, targeted drug delivery and magnetic resonance imaging agents(MRI) etc. More recently, we prepared Fe-B-P amorphous-like nanopartices [4] and nanobars [5] with good soft magnetic properties. However, the detailed microstructure of these nanomaterials is not well understood, especially concerning the effect of annealing on both the magnetic properties and the microstructure. In this work, a series of as-prepared Fe-B-P nanoparticles and annealed ones were examined by transmission Mössbauer spectroscopy, X-ray diffraction (XRD), scanning electron microscopy (SEM) and vibrating sample magnetometry (VSM).

## 2 Experimental

Fe-B-P nanoparticles were synthesized by aqueous chemical reduction. The detailed process was as follows. A 100 ml aqueous solution was prepared containing $FeCl_2$ 20g, $NH_4Cl$ 8g, $C_6H_5Na_3O_7{\cdot}2H_2O$ 24g and $NaH_2PO_2{\cdot}2H_2O$ 16g. NaOH solution was used to tune the pH value to be around 9.0, and $NaBH_4$ was used as reduction agent. The as-prepared Fe-B-P nanoparticles were further annealed at 300 °C, 400 °C and 500 °C for 30 min, respectively, then let to cool in the furnace to room temperature.

The microstructure of these nanoparticles was examined by XRD and MS. The morphology was checked by SEM and the magnetic properties by VSM. Before and after the annealing treatment Mössbauer spectra were measured at room temperature by a constant acceleration spectrometer. The $\gamma$-emission source was 57Co/Rh. The maximum count per channel in the spectrum was about $2.5 \times 10^5$. For calibration $\alpha$-Fe was used as a standard absorber. The analysis of each spectrum was carried out by a computer fitting program, which was written based on the improved Hesse method., and the fitting accuracy $\chi$ per channel was about unity.

## 3 Results and discussion

Figure 1 shows the SEM image of as-prepared particles. Figure 1 shows that the particles are monodisperse and that the grain diameter distribution is from 200 to 300 nm. SEM-EDS shows that the composition of the particles is $Fe_{84}B_{12}P_4$. Figure 2 is the XRD spectrum of an as made sample. According to the analysis of the experimental results of its diffuse scattered peak at $2\theta = 40^\circ \sim 50^\circ$ it is amorphous. In order to confirm its amorphous characteristics, Mössbauer spectra were recorded of as made samples. Figure 3a shows the Mössbauer spectrum of as-prepared Fe-B-P particles. It shows a broad spectrum which indicates the disordering of atomic

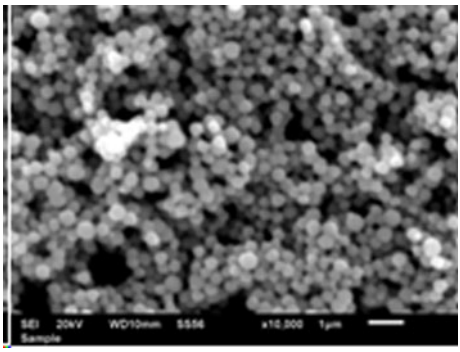

**Fig. 1** SEM images of as-prepared Fe-B-P particles

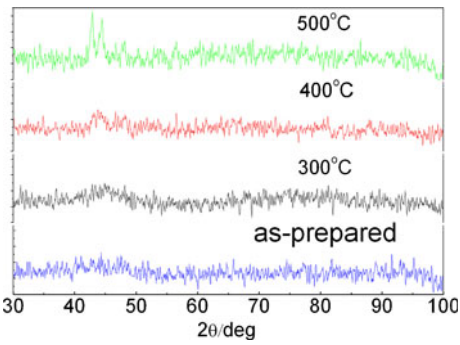


**Fig. 2** XRD of Fe-B-P particles before and after annealing treatmen

arrangements. The fitting result of the Mössbauer spectra of as-prepared Fe-B-P particles is listed in Table 1. We conclude that the Fe-B-P particles prepared by aqueous chemical reduction are amorphous nanoparticles. Figure 4 illustrates the hysteresis loops of as-prepared Fe-B-P particles. The saturation magnetization (Ms) of Fe-B-P is 129.3emu/g. The coercive field of as-prepared Fe-B-P particles is 11.5Oe.

The samples prepared by aqueous chemical reduction were then treated with isothermal annealing at 300 °C, 400 °C and 500 °C for 30 min in order to study the influence of an annealing treatment on the microstructure and soft magnetism of Fe-B-P nanoparticles.

Figure 2 shows the XRD spectra of the as-prepared samples and after annealing treatment. It shows that the diffuse scattered peaks gradually become sharper from room temperature to 500°C.

Figure 3b∼d are Mössbauer spectra after annealing treatment. Mössbauer spectra in Figure 3b exhibit sharper peaks. Figure 3c and d exhibit sets of sextets which indicate the formation of crystalline phases. The fitting results of the Mössbauer spectra after annealing treatment are listed in Table 1.

Table 1 shows that a small amount of a crystallized phase appears in the amorphous particles after annealing treatment at 300 °C. The volume fraction of the crystallized phase is around 3.0 %. Figure 2 shows that the crystallized phase is α-Fe. After the annealing treatment at 400 °C and 500 °C the samples are completely crystallized. The crystallization phases are α-Fe [6], $Fe_2B$ [7] and $Fe_3B$ [8].

It was found that the coercivity and the saturation inducted density of samples after treatment have increased compared with their original amorphous state as

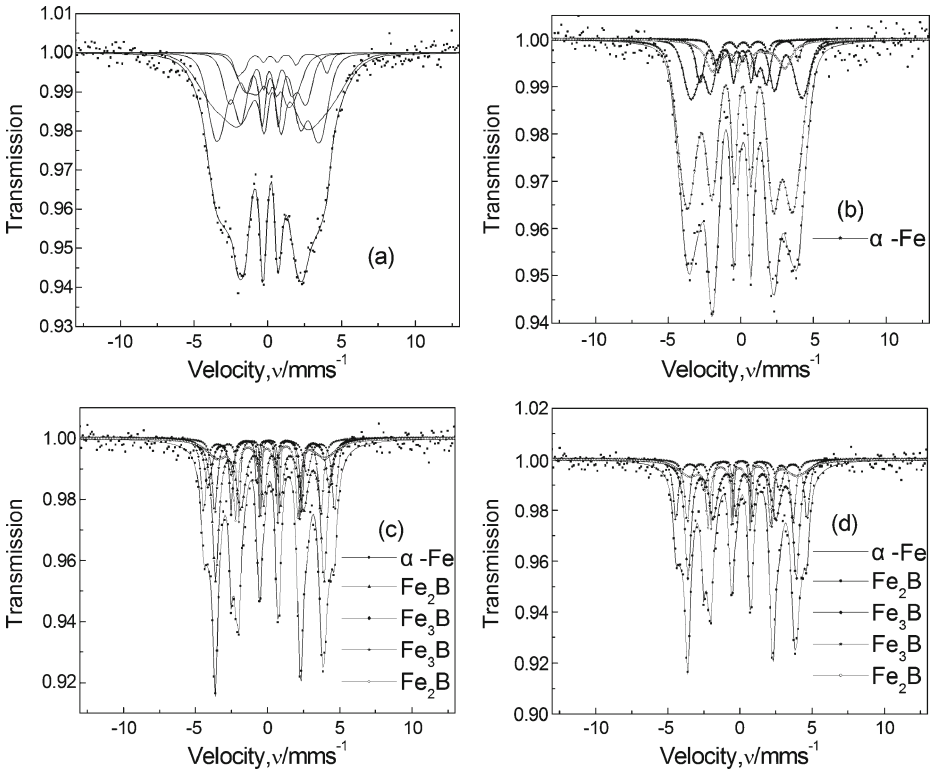


**Fig. 3** Mössbauer spec-tra of Fe-B-P particles (**a**) as-prepared, (**b**) annealed at 300°C, (**c**) 400°C, (**d**) 500°C

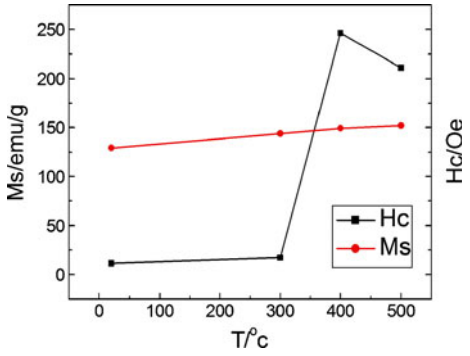


**Fig. 4** Magnetic hysteresis loop of specimens before and after annealing treatment

shown in Fig. 4. The coercivity of the sample after annealing at 400 °C and 500 °C has further increased and this leads to a deterioration of the magnetic properties of the samples because the crystallized phases block domain wall motion. The results show that the soft magnetism of samples with trace crystallization is slightly improved by the annealing treatment. But the soft magnetism of completely crystallized samples has deteriorated.

**Table 1** The fitting results of Mössbauer spectra of specimens before and after annealing treatment

| Annealing treatment/°C | IS/mms$^{-1}$ | QS/mms$^{-1}$ | Hhf/kOe | Area/% | Phase structure |
|---|---|---|---|---|---|
| As-prepared | 0.29 | −0.07 | 224.7 | 38.8 | Amorphous |
| | 0.06 | −0.11 | 159.6 | 13.3 | |
| | 0.58 | 0.80 | 188.1 | 4.1 | |
| | 0.10 | −0.16 | 214.7 | 30.6 | |
| | 0.24 | −0.18 | 105.3 | 13.2 | |
| 300 | 0.27 | 0.28 | 238.4 | 19.4 | Amorphous |
| | −0.38 | −0.27 | 142.4 | 8.2 | |
| | 0.60 | 0.84 | 182.0 | 3.3 | |
| | 0.04 | −0.18 | 226.1 | 66.1 | |
| | 0.40 | 0.38 | 255.9 | 3.0 | α−Fe |
| 400 | 0.14 | −0.02 | 284.0 | 17.5 | α−Fe |
| | 0.23 | −0.20 | 233.3 | 27.9 | $Fe_2B$ |
| | 0.01 | 0.08 | 262.0 | 18.9 | $Fe_3B$ |
| | 0.09 | −0.11 | 227.1 | 14.8 | $Fe_3B$ |
| | 0.13 | 0.19 | 232.3 | 20.8 | $Fe_2B$ |
| 500 | 0.12 | −0.01 | 282.7 | 15.1 | α−Fe |
| | 0.24 | −0.17 | 233.8 | 36.6 | $Fe_2B$ |
| | 0.01 | 0.17 | 262.1 | 14.8 | $Fe_3B$ |
| | 0.07 | −0.10 | 228.7 | 15.4 | $Fe_3B$ |
| | 0.11 | 0.31 | 232.8 | 18.1 | $Fe_2B$ |

## 4 Conclusion

1. The Fe-B-P particles prepared by aqueous chemical reduction are amorphous, and the grain diameter distribution is from 200 to 300 nm. The saturation magnetization is 129.3 emu/g. The coercive field is 11.5Oe.
2. The volume fraction of the crystallized phase is 3.0 % after annealing treatment at 300 °C for 30min and the soft magnetism is slightly improved.
3. After treatment at 400 °C and 500 °C for 30 min the samples are completely crystallized, and the soft magnetic properties of the specimens have strongly deteriorated.

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