



Structural-controlled chemical synthesis of nanosized amorphous Fe particles and their improved performances



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ABSTRACT

High-performance amorphous Fe nanoparticles with controlled structure and good thermal stability have been fabricated using an optimized chemical method by a direct reduction reaction of Fe^{2+} ions with NaBH_4 as reducing agent. The addition of polyvinylpyrrolidone (PVP) as a surfactant, the properties of solution acid/alkaline and the variation of reaction solvent compositions have shown great influences on the morphologies, surface compositions and magnetic properties of the products. The addition of surfactant PVP leads to the formation of fully amorphous Fe nanoparticles. Partial crystallization will occur in the samples prepared without PVP addition and result in their high saturation magnetization up to $168.0 \text{ A m}^2/\text{Kg}$. The alkali reaction solution with NaOH addition can facilitate the formation of amorphous Fe–B phases in the products instead of the surface B concentration in neutral solution, which promotes their good thermal stability. Reactions in the ethanol–water solvent favor the formation of smaller amorphous iron nanoparticles with about 20 nm in size. The relatively simple chemical synthesis and improved performances including controlled structure, good thermal stability and excellent intrinsic magnetic properties for these amorphous iron nanoparticles promise their potential applications in high-performance multifunctional magnetic devices.

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1. Introduction

Fe-based soft magnetic materials play an important role in many technical applications such as magnetic sensors, data recording and electric motors because of their overall high performances such as high saturation magnetization, low coercivity and good thermal stability [1–4]. As is known, Fe-based metallic glasses show much better mechanical and magnetic properties than those of their crystalline counterparts owing to the absence of grain boundaries and crystal magnetic anisotropy [5,6]. The traditional bulk Fe-based amorphous alloys have been usually prepared by melt-spinning technology or copper mold casting methods [7–9]. Nevertheless, their poor glass-forming ability and high cost restrict the actual applications for these Fe-based amorphous alloys in high-performance magnetic devices. It has been demonstrated that the nanosized magnetic materials exhibit better intrinsic mechanical and magnetic properties compared with their corresponding bulk alloys fabricated by traditional physical methods [10,11]. Various

controllable chemical methods have been widely used to fabricate magnetic metallic nanostructures with controlled morphology and particle size [12–15]. For further applications, these chemically synthesized magnetic particles with ideal structure can be densified via powder compacting molding process to full-density bulk soft magnetic composite materials. In our previous work, nearly-spherical FeCo particles with particle size ranging from 100 nm to 5 μm have been prepared by a direct chemical method and can be easily compacted into full-density magnetic devices [16,17].

Recently, much attention has been focused on amorphous Fe-based nanomaterials and their effective chemical fabrication due to their good soft magnetic properties, mechanical properties and low preparation cost. Many researches have been performed to synthesize varieties of amorphous magnetic nanoparticles by chemical method using borohydride (NaBH_4) as the reducing agent for compensating the limitation of traditional physical preparation methods [18–20]. In our recent work, nanosized amorphous Fe particles with narrow size distribution and good magnetic properties were prepared by a direct chemical reduction of Fe^{2+} ions with NaBH_4 as reducing agent at an optimal NaBH_4 addition rate of 4.0 mL/min [21]. In this work, high-performance amorphous Fe nanoparticles with combined ideal structure, high saturation

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magnetization and good thermal stability have been fabricated using an optimized chemical method. It has been found that synthesis processes such as the addition of PVP as a surfactant, the properties of solution acid/alkaline and the variation of reaction solvent compositions have shown great influences on the phase formation of these chemically synthesized nanosized iron particles. The influences of the preparation conditions on the morphologies, surface compositions and magnetic properties of these amorphous nanosized iron particles have also been systematically studied.

2. Experimental

In our previous work, we have reported a direct chemical method for preparing fully amorphous iron nanoparticles at room temperature by the reduction of Fe^{2+} with NaBH_4 as reducing agent in $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ aqueous solution at an optimal NaBH_4 addition rate of 4.0 mL/min [21]. In the present work, amorphous iron nanoparticles with controlled morphologies and modulated surface compositions have been prepared by the optimized chemical method according to a procedure previously reported [21] through adjusting the key reaction parameters including the addition of surfactant PVP, change of solution acid/alkaline and the variation of reaction solvent compositions. In the following typical procedures, four samples have been obtained by the modified chemical methods in four different reaction systems, respectively: (a) the only addition of PVP (0.5–1 g) in ferrous aqueous solution, (b) no addition of PVP in ferrous aqueous solution, (c) the combined addition of PVP (0.5–1 g) in ferrous aqueous solution and NaOH (0.06–0.1 g) in NaBH_4 solution and (d) the only addition of PVP (0.5–1 g) in the mixed ethanol-water solvent. All the samples in this work are fabricated at a fixed NaBH_4 addition rate of 4.0 mL/min and collected after drying the reaction products in a vacuum oven at room temperature for 6–8 h. Some of them were annealed at 475–600 °C for 10 min under high-purity Ar atmosphere to further investigate their crystallization characteristics.

The phase structure of the samples was characterized by X-ray diffraction (XRD) using a D/max 2500PC X-ray diffractometer with $\text{Cu K}\alpha$ radiation. The microstructure of the samples was identified by transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) using a JEOL JEM-2100 transmission electron microscope operated at 200 kV. The precise compositions of the samples were determined using an inductively coupled plasma optical emission spectroscopy (ICP-OES, Perkin–Elmer Optima 7000 DV). The thermal properties were analyzed by differential scanning calorimetry (DSC, STA 449 F3, NETZSCH) under an Ar atmosphere at a heating rate of 10 °C/min. The surface states of the samples were investigated by the X-ray photoelectron spectroscopy (XPS, ESCALAB 250 Xi) with an Al $\text{K}\alpha$ excitation source. Magnetic properties for the samples were measured with a vibrating sample magnetometer (VSM, Lakeshore 7307) under a maximum magnetic field of 10 kOe at room temperature.

3. Results and discussion

Fig. 1 shows the XRD patterns for the four samples prepared under different reaction conditions: (a) the only addition of PVP (1 g) in ferrous aqueous solution; (b) no PVP addition in ferrous aqueous solution; (c) the combined addition of PVP (1 g) in ferrous aqueous solution and NaOH (0.06 g) in NaBH_4 solution; (d) the only addition of PVP (1 g) in ethanol-water solvent with the volume ratio of 1:1. It can be seen from Fig. 1b that three broad peaks are observed with 2θ values of 44.8°, 65.2°, and 82.6°, which are in good agreement with characteristic peaks of the bcc-Fe phase. These broad peaks in the diffraction patterns also indicate partially crystallized states for the sample b prepared with no PVP addition,

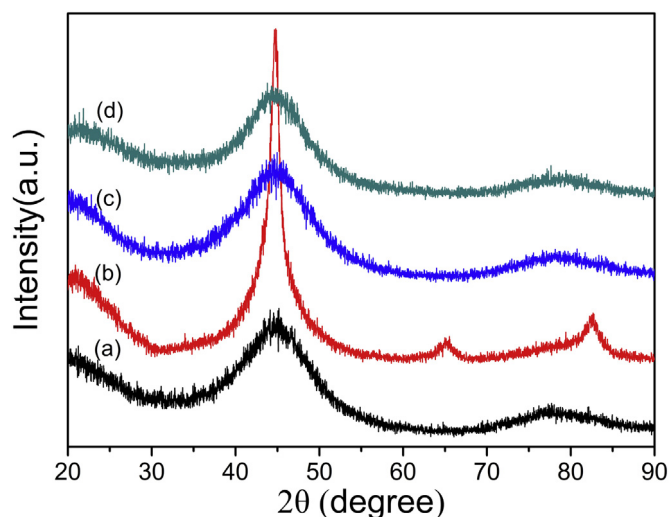


Fig. 1. XRD patterns of the samples prepared under different conditions: (a) the only addition of PVP (1 g) in ferrous aqueous solution; (b) no PVP addition in ferrous aqueous solution; (c) the combined addition of PVP (1 g) in ferrous aqueous solution and NaOH (0.06 g) in NaBH_4 solution; (d) the only addition of PVP (1 g) in ethanol-water solvent with the volume ratio of 1:1.

which can be confirmed by the SAED patterns of mixture phases and a relatively low crystallization transition temperature in the DSC curves discussed below. However, the other three samples with the addition of PVP exhibit the same diffraction characteristics with a broad peak around 2θ of 45°, which indicates the same fully amorphous states in these samples [22]. The further results show that the addition of PVP in any amount of 0.5–1 g with other reaction conditions fixed can facilitate the formation of fully amorphous samples, which clearly suggests that the surfactant PVP promotes the formation of amorphous nanosized iron particles fabricated by chemical reduction.

Fig. 2 shows the representative TEM micrographs and the corresponding SAED patterns of the four samples prepared under different conditions. It can be seen from the SAED patterns in Fig. 2b that besides the diffuse halo, there are a few single crystal diffraction spots, which indicates a mixture of crystalline and amorphous phases in the sample prepared with no PVP addition. The SAED patterns for the other three samples prepared with the addition of PVP (sample a, sample c and sample d) only show the diffuse scattering halos, which further demonstrates the fully amorphous phases in these samples. The reduction reactions of Fe^{2+} ions with NaBH_4 as strong reducing agent will have run quickly to generate Fe nucleuses, which may simultaneously form amorphous Fe nanograins with short-range order under room temperature and then grow into amorphous Fe nanoparticles. It has been reported that the short-range order of the nanograins can be observed in the boron-rich interfacial regions of the $\text{Fe}_{50}\text{B}_{50}$ nanoglass, which promotes the formation of their amorphous state [18]. The average particle sizes and their distribution of these samples estimated from more than 100 particles by selecting several TEM images for each sample are listed in Table 1. As can be seen from Fig. 2 and Table 1, the addition of PVP, the properties of solution acid/alkaline and the variation of reaction solvent compositions show an obvious effect on the morphologies of these products. The only addition of PVP in the reaction systems lead to the formation of uniform amorphous Fe nanoparticles with an average particle size of 90 nm (seen in Fig. 2a). While the sample prepared without any addition of PVP (sample b) exhibits a chain-like morphology with smaller particles and broader size distribution. It can be concluded that the

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