

Microstructural and Magnetic Characterization of Fe Nanosized Powder Synthesized by Pulsed Wire Evaporation

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We studied the microstructure and magnetic properties of Fe nanosized powder synthesized by the pulsed wire evaporation method. The x-ray diffraction spectrum confirmed that this powder had a pure α -Fe phase. Scanning electron microscope and transmission electron microscope measurements indicated that the prepared powder had uniform spherical shape with core-shell structure. The mean powder size was about 35 nm and the thickness of the surface passivation layer was about 5 nm. Energy dispersive X-ray spectroscopy measurement indicated that the surface passivation layer was iron oxide. Magnetic field dependent magnetization measurement at room temperature showed that the maximum magnetization of the prepared powder was 177.1 emu/g at 1 T.

Keywords : Fe nanosized powder, Pulsed Wire Evaporation, Core-shell structure, Microstructure analysis using TEM and EDX

1. Introduction

Nanosized magnetic materials are attractive for their potential applications to high performance permanent magnets, targeted drug delivery, ultrahigh density magnetic storage devices, and in various industrial areas [1-3]. Among various magnetic nanoparticles, Iron oxide (magnetite (γ -Fe₂O₃) and magnetite (Fe₃O₄)) nanoparticles have been widely investigated for practical applications [4-8]. Apart from the magnetic metal oxides, pure Fe and Fe based alloys are also used in various fields of magnetism because pure Fe has a much higher saturation magnetization ($M_s \sim 213$ emu/g) compared to other currently used iron oxide materials (M_s for bulk γ -Fe₂O₃ and Fe₃O₄ are 74 and 84.6 emu/g, respectively) [3, 9]. Despite good magnetic properties, Fe nanosized powder has a serious drawback for applications due to its inflammable property. Therefore iron oxide passivation layer covering surface of Fe powders is essential. Various synthesis methods have been used to make magnetic nanoparticles. Chemical methods such as precipitation technique, sol-gel process, and hydrothermal method have successfully synthesized

magnetic nanosized powders [10-12]. However, it has been reported that synthesized powders based on chemical method often undergo surface contamination or particle agglomeration [13]. Pulsed wire evaporation (PWE) is a dry synthesis method used as a one-step synthetic technique with high production efficiency. M. H. Lee *et al.* reported on the crystal structure of Fe nanosized powder synthesized in different ambient gas conditions during PWE [13]. However, to the best of our knowledge, the magnetic properties and chemical element analysis of Fe nanosized powder prepared by PWE have not yet been reported. In this work, we studied the microstructure and magnetic properties of Fe nanosized powder synthesized by PWE.

2. Experimental Methods

Fe nanosized powders were prepared using the PWE method. To form iron oxide layer on powder surface, we exposed the prepared powder to ambient air. The crystal structure of the prepared powder was characterized by X-ray diffraction (XRD) with Cu K α radiation ($\lambda=1.54046$ Å). The phase of the prepared powder was confirmed using XRD and particle size was estimated using the Debye Scherrer formula. The prepared powder microstructure and morphology were investigated by scanning

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electron microscope (SEM), transmission electron microscope (TEM) and energy dispersive X-ray spectroscopy (EDX) observations. The magnetic field dependent magnetization (M-H) at room temperature was performed using a vibration sample magnetometer (VSM) in a field range up to 1 T.

3. Results and Discussion

3.1. Crystal structural analysis

The XRD pattern of Fe nanosized powders is shown in Fig. 1 (a). On the XRD pattern, no distinct impurity phase was observed except for α -Fe phase, where all diffraction peaks can be indexed by ICDD card # 06-0696. The mean crystallite size was calculated using the Debye Scherrer equation. $D = 0.94\lambda/(\beta\cos\theta)$, where D (nm) is the mean crystallite size, λ (1.54046 Å) is the X-ray wavelength, β (radian) is the broadening of the peak, which can be estimated from the full width at half maximum (FWHM), and θ (radian) is the Bragg angle of the main diffraction peak. The FWHM values of observed peaks from 1st order to 4th order were 0.329°, 0.365°, 0.329° and 0.259°, respectively. Therefore, the estimated mean crystallite size was about 29 nm.

A SEM image is shown in Fig. 2 (a). Prepared Fe nanosized powders were uniformly spherical in shape. Considering the formation process of the particles, it can be expected that after the evaporation of the iron wire, the iron vapors are condensed into spherical shaped particles in order to minimize the surface energy [13]. The particle size distribution is shown in the inset of Fig. 2 (a). The observed particle size ranged from 20 nm to 200 nm and the average sizes were estimated to be about 35 nm. This indicated that the prepared Fe nanosized powder had a single crystal.

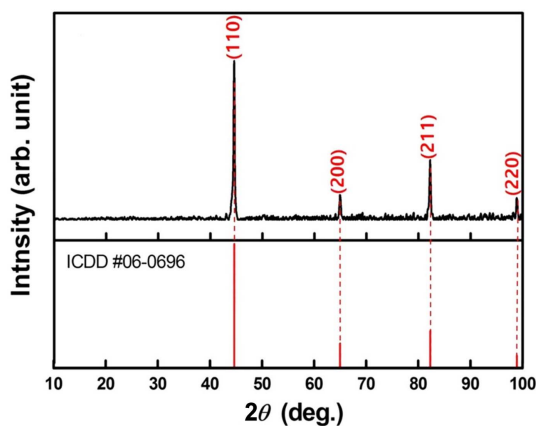


Fig. 1. (Color online) The x-ray diffraction patterns of the prepared Fe nanosized powder.

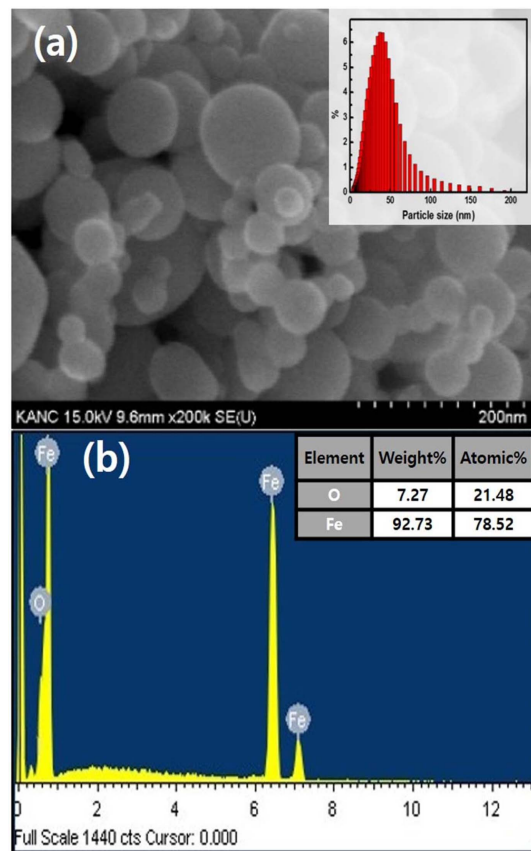


Fig. 2. (Color online) The SEM image (a) and EDX spectrum of the prepared Fe nanosized powder. Inset shows the particle size distribution estimated by SEM image.

To determine the chemical elements of the prepared powder, EDX was used [shown in Fig. 2 (b)]. The EDX spectrum revealed that the prepared powder was composed of the oxygen as well as iron atoms even though no Fe oxide phase was detected in XRD.

In order to investigate microstructural characterization at high resolution, the prepared powders were observed using TEM. The TEM image shown in Fig. 3 (a) revealed regions of two different contrasts (dark and light) in the morphology of almost all observed particles. This indicates that the prepared powder has a core-shell structure. Prepared powder EDX elemental mapping images shown in Fig. 2 (b) also clearly revealed that Fe (red dots) and O (green dots) elements were all distributed uniformly throughout the sample.

From the above observations, it can be assumed that the darker region corresponding to α -Fe core is surrounded by a light shell structure of the iron oxide passivation layer. Because this passivation layer is not thick enough (~5 nm) to be well crystallized, the diffraction peaks of the layer could not be detected [13].

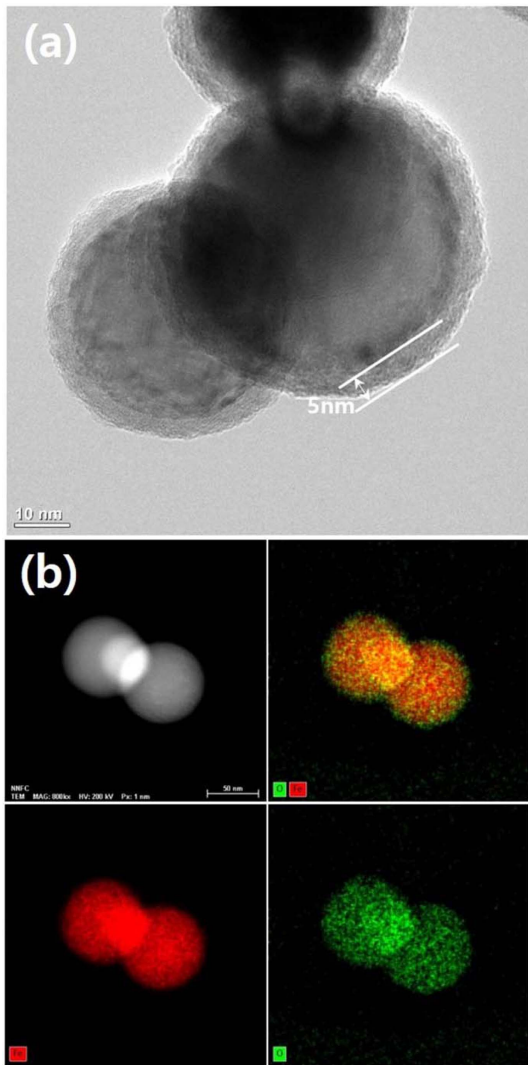


Fig. 3. (Color online) The TEM (a) and EDX elemental mapping (b) image.

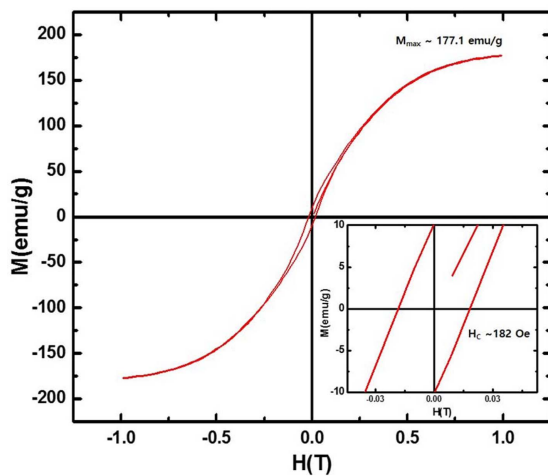


Fig. 4. (Color online) The plot of M-H loops of Fe nanosized powder.

Table 1. The M_s of various Fe based magnetic materials.

Material	M_s (emu/g)	Type [Ref.]
α -Fe	213	Bulk [3], nano [14]
Fe nanosized powder	177.1 >	Nano [In this work]
Fe_3O_4	84.6	Bulk [9]
$\gamma\text{-Fe}_2\text{O}_3$	74	Bulk [9]

3.2. Magnetic properties

The M-H at room temperature was measured by using VSM in the range of 0~1 T. Fig. 4 shows a typical soft magnetic hysteresis loop. The maximum magnetization at 1 T (M_{max}) is about 177.1 emu/g and coercivity is about 182 Oe. Table 1 shows the M_s of various Fe based magnetic materials reported in previous studies. This indicates that the M_{max} of the prepared Fe nanosized powder in this work is high compared to the magnetization value of bulk Fe.

4. Conclusions

We studied the microstructure and magnetic properties of Fe nanosized powder synthesized by PWE. The XRD spectrum confirmed that this powder had pure α -Fe phase. Microstructure characterization using SEM, TEM and EDX indicated that the prepared powder had spherical shape with a core-shell structure. The mean size of the powder was around 35 nm and the thickness of the surface passivation layer was about 5 nm. The M-H at room temperature showed that the maximum magnetization at 1 T is about 177.1 emu/g. These results indicate that Fe powder prepared by PWE is a high quality nanosized powder. Especially, this powder is assumed to be a good candidate to increase performance of soft magnetic composite for high frequency application because iron oxide passivation layer lowers inter-particle eddy current loss [15].

Acknowledgments

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References

- [1] H. Zeng, J. Li, J. P. Liu, and S. Sun, Nature **420**, 395 (2002).
- [2] S. R. Rudge, T. L. Kurtz, C. R. Vessely, L. G. Catteral, and D. L. Williamson, Biomaterials **21**, 1411 (2000).
- [3] E. P. Wohlfarth and K. H. J. Buschow, "Ferromagnetic Materials: A Handbook on the Properties of Magnetically Ordered Substance", North-Holland, Amsterdam (1988).

- [4] R. F. Ziolo, E. P. Giannelis, B. A. Weinstein, M. P. O'Horo, B. N. Ganguly, V. Mehrotra, M. W. Russell, and D. R. Huffman, *Science* **257**, 219 (1992).
- [5] Q. Liu and Z. Xu, *J. Appl. Phys.* **79**, 4702 (1996).
- [6] M. Allen, D. Willits, J. Mosolf, M. Young, and T. Douglas, *Adv. Mater.* **14**, 1562 (2002).
- [7] Y. F. Shen, J. Tang, Z. H. Nie, Y. D. Wang, Y. Ren, and L. Zuo, *Sep. Purif. Technol.* **68**, 312 (2009).
- [8] A. Dyal, K. Loos, M. Noto, S. W. Chang, C. Spagnoli, K. V. P. M. Shafi, A. Ulman, M. Cowman, and R. A. Gross, *J. Am. Chem. Soc.* **125**, 1684 (2003).
- [9] R. C. O'Handley, "Modern Magnetic Materials: Principles and Applications", Wiley, New York (1999) p. 125.
- [10] M. P. Morales, S. Veintemillas-Verdagure, M. I. Montero, and C. J. Serna, *Chem. Mat.* **11**, 3058 (1999).
- [11] M. Alagiri and Sharifah Bee Abdul Hamid, *J. Sol-Gel Sci. Technol.* **74**, 783 (2015).
- [12] K. R. Lestari, P. Yoo, D. H. Kim, C. Liu, and B. W. Lee, *J. Korean Phys. Soc.* **66**, 651 (2015).
- [13] H. M. Lee, Y. R. Uhm, and C. K. Rhee, *J. Alloys Compd.* **461**, 604 (2008).
- [14] Y. C. Han, Y. H. Kim, J. K. Heo, Y. C. Kang, and Y. S. Kang, *Proceeding, IEEE Nanotechnology Materials and Devices Conference 2006*, 650 (2006).
- [15] H. Kim and S. Y. An, *J. Magn.* **20**, 138 (2015).