HS-8 Investigation of thermal properties of Fe₃O₄ nanoparticles under bio-plasma treatment

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I. INTRODUCTION

Ferrites have been used in various industrial areas such as hyperthermia, MRI, bio-sensor, and drug delivery applications [1]. Especially, for the hyperthermia application, small particle size is required as well as low coercivity and high magnetization [2]. Recently, plasma treatment has been widely used in developing new materials synthesis and surface treatment processes, which can provide a unique way of obtaining material characteristics desired for many technological applications [3]. In this paper, we have studied the magnetic and thermal properties of Fe₃O₄ nanoparticles before and after the plasma treatment.

II. EXPERIMENT PROCEDURES

The Fe₃O₄ nanoparticles were synthesized by high temperature thermal decomposition (HTTD) method with starting materials of 6 mmol iron (III) acetylacetonate, 4 ml oleic acid, and 2 ml oleylamine. These were mixed with solvent of 30 ml benzyl ether. The mixture was heated at 200 °C for 30 min, and then reheated at 298 °C for different reheating times of 15, 30, 45, 60, and 120 minutes. Hereafter, these are referred to as S15, S30, S45, S60, and S120, depending on the reheating time. After cooling those down to room temperature, the nanoparticles were obtained via the centrifugation process. The crystal structure was analyzed by X-ray diffractometer (XRD) with Cu- $K\alpha$ radiation ($\lambda = 1.5406$ Å). The magnetic properties were investigated by vibrating sample magnetometer (VSM). Self-heating temperature was measured with magneTherm device. Also, a bio-plasma equipment was used with argon as the plasma generating gas and applied voltage of 100 volt for the plasma treatment. The Mössbauer spectra were recorded using transmission type with a ⁵⁷Co source in an Rh matrix for a constant acceleration mode.

III. RESULTS AND DISCUSSION

The crystal structure of Fe₃O₄ nanoparticles was determined to be cubic spinel with the space group of Pnma at room temperature. The average lattice constant of samples was a₀ = 8.373 Å. From the Scherrer equation for XRD pattern, the average particle size of samples was 10.6 nm, while S45 showed the largest size of 11.7 nm. The hysteresis curves were measured by VSM with a maximum applied field of 1.5 T at room temperature. The saturation magnetization of S45 was highest among the samples studied. We measured the self-heating temperature of the samples (20 mg of powders in air) under alternating magnetic field of 250 Oe at 112 kHz. The heating temperature increases quickly from the room temperature and becomes constant after 300s. The saturated temperature after 300 s agrees with the value from VSM measurement.

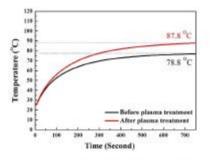
Since S45 has high saturation magnetization and low coercivity, S45 was treated with argon plasma and compared with the untreated sample, XRD pattern shows that there is no change in the crystal structure after plasma treatment. However, we observed slight decrease in the particle size after plasma treatment, Fig. 1 shows the self-heating temperature under time-varying magnetic field of 250 Oe at 112 kHz before and after plasma treatment. The self-heating temperature of the sample increases from 78.5 °C to 87.8 °C after the plasma treatment. In addition, we have obtained the hysteresis curve of S45 after treatment, showing the saturation magnetization increasing from 73.6 emu/g to 76.4 emu/g. Fig. 2 shows the Mössbauer spectra at room temperature before and after plasma treatment. These were analyzed by a least-squares method with one six-line for tetrahedral A site and two six-lines for octahedral B_1 and B_2 sites, resulting in the three six-line hyperfine pattern. The isomer shift (δ) values for the A, B₁, and B₂ sites were found to be 0.23 mm/s, 0.30 mm/s, and 0.59 mm/s, indicating that the valance state of Fe ion at A and B₁ site is ferric, and that at B₂ site is ferrous. The area ratios at A and B – including both B₁ and B₂ sites – sites were about 40:60. However, the hyperfine fields (Hhf) at A, B₁ and B₂ sites increased from 473 kOe to 482 kOe, 437 kOe to 445 kOe, and 388 kOe to 395 kOe after plasma treatment, respectively. Based on the Mössbauer spectra, we expect that the plasma treatment affects the magnetic properties of Fe₃O₄ nanoparticles with enhanced self-heating temperature.

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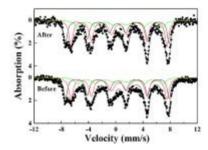


Fig. 1. Self-heating temperature under time varying magnetic field of 250 Oe at 112 kHz of before and after plasma treatment.

Fig. 2. Mössbauer spectra at room temperature of before and after plasma treatment.