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Novel synthesizing method of BaFe₁₂O₁₉ micro rod and its superior coercivity with shape anisotropy

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ABSTRACT

BaFe₁₂O₁₉ micro rod shapes (BMSs) for coercivity enhancement were synthesized by self-assembly method using diethyleneamine (DEA) with ultra-sonic treatment. The optimized condition for the elaborate submicron rod BaFe₁₂O₁₉ structure was 1.7 MHz frequency with pH > 12.2 DEA concentration. The coercivity of BMSs with 150 nm width and 700 nm length showed 7.5 kOe (iHc). These results revealed that the high coercive BMSs were successfully synthesized to micro size by triggering shape anisotropy during sonication process.

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Barium hexaferrite (BaFe₁₂O₁₉) is known as one of the common magnetic materials for various small D.C. motor appliances such as in blowers, fans, windscreen wipers and pumps due to its large magnetocrystalline anisotropy and high Curie temperature [1]. Even though its variety usages, the real coercivity of BaFe₁₂O₁₉ is lower than theoretical value (~17 kOe) [2]. This is because the imperfections of the real-structure give rise to incoherent magnetization reversal, even though the particle size is smaller than the critical single domain size [3]. In addition, there are incoherent curling or vortex modes, and irrespective of presence of imperfections for the particles larger than 20 nm in diameter limits the coercivity value [4]. If coercivity is improved, it is possible to size reduction of ferrite motor and enhanced thermal resistance which is critical weak point of ferrite magnet. To achieve higher coercivity; it is considered to turn magnetic anisotropy of the films by varying synthesis methods. Generally, magnetic anisotropy depends on two factors; the spin-orbit coupling; the magnetocrystalline dipole–dipole interaction. The former is related to magnetocrystalline anisotropy, the later is related to the shape anisotropy. Previously, there have been efforts to synthesize wire or rod-like shapes of BaFe₁₂O₁₉ to turn the shape anisotropy [5–7]. Since, homogeneous rods shapes of BaFe₁₂O₁₉ increase the shape anisotropy because of a reduced demagnetization factor along the long axis [8].

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Various chemical synthesis with a bottom-up approaches such as, co-precipitation, hydrothermal synthesis, sol–gel, low-temperature combustion and self-assembly methods can be used to from homogeneous rod shape [9–18]. Among them, the self-assembly method is most commonly used for synthesizing nanorods, nanofibers, hollow spheres, etc [19–22]. Previously, homogeneous hollow nano-structure BaFe₁₂O₁₉ spheres was synthesized using the self-assembly method by DEA addition as a surfactant to Ba(NO₃)₂ and Fe(NO₃)₃ [23]. It is also known that a strong basic surfactant of diethyleneamine (DEA) not only provides favorable site for the growth of the particulate assemblies but also influences the formation processes such as, nucleation, growth, and particle shapes [24]. In addition, the size controlling of BaFe₁₂O₁₉, especially for enhanced magnetic properties, has been widely studied [25,26]. However, the shape transform of BaFe₁₂O₁₉ has rarely been studied [27]. In this paper, we modified self-assembly method with ultra-sonication to study the relationship between BaFe₁₂O₁₉ micro rod shapes (BMSs) and magnetic properties such as demagnetization factor, magnetic anisotropy and coercivity.

For the synthesis, 1.1 mL (pH > 12.2) of DEA was dispersed into deionized water (100 mL) and stirred for 5 min prior to seal. While the aqueous solution was continuously stirred, 0.0 L/mol of Ba(NO₃)₂ and 0.12 mol of Fe(NO₃)₃ was added. Then the mixture solutions of BaFe₁₂O₁₉ compounds were treated by ultra-sonication for 1 min with various frequencies (0.5–2.0 MHz) and cleaned 5 times at 50 °C using deionized water and methyl alcohol with centrifugation by 10,000 RPM for 3 min. Finally the solutions

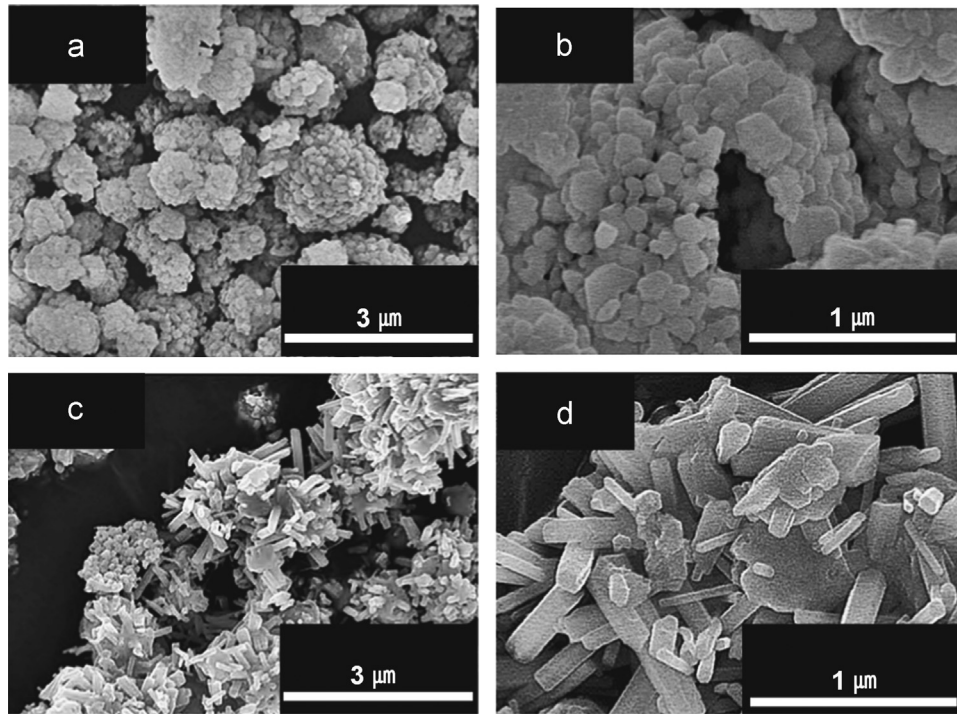


Fig. 1. FE-SEM images of the synthesized $\text{BaFe}_{12}\text{O}_{19}$ microstructures for different sonication treatments after heat treatment at $1000\text{ }^{\circ}\text{C}$ for 1 h: (a) and (b) 1.5 MHz and (c) and (d) 1.7 MHz.

were heated to $1000\text{ }^{\circ}\text{C}$ for an hour and quenched to room temperature by putting Ar gas. Magnetic properties were assessed using a vibrating sample magnetometer (VSM, Lakeshore-7400) at ambient temperature. Structural phase relations were analyzed by X-ray diffraction (XRD) with monochromatic $\text{Cu K}\alpha$ radiation. Field emission scanning electron microscopy (FE-SEM, JSM-6700F) was used for the morphology analysis.

Fig. 1 shows the detailed morphologies of the $1000\text{ }^{\circ}\text{C}$ heat-treated $\text{BaFe}_{12}\text{O}_{19}$ hollow spheres and rod shapes synthesized at different sonication frequencies. All the samples were aggregated. The hollow structure was formed for the samples, sonicated at 1.5 MHz (see **Fig. 1**(a) and (b)). It is also noticed that the beginning of micro rod shapes formation was also observed on the surface of the particles. On the other hand, the clear rod shapes with 100–150 nm width and 600–800 nm length were formed for the samples, sonicated at 1.7 MHz (see **Fig. 1**(c) and (d)). The measured r -factor, the ratio between length and width of rods ($r = \text{length}/\text{width}$) were 5.1 ± 1.3 . Therefore, the shape of $\text{BaFe}_{12}\text{O}_{19}$ can be turned from hollows to rods depending on sonication frequency. The physical origin of sonication frequency dependent structural formation can be explained by cavity resonance theory [28,29]. At lower frequency (in the region of ultrasonic frequency), the built-in high pressure in the colloidal will destroy the initial hollow-structured $\text{BaFe}_{12}\text{O}_{19}$. At higher frequency (1.7 MHz), the built-in pressure in the colloidal is lower. Therefore, the initial hollow structure remains and grows the rod-shapes at the from the surface of the $\text{BaFe}_{12}\text{O}_{19}$.

Room temperature X-ray diffraction patterns of $\text{BaFe}_{12}\text{O}_{19}$ powders, synthesized at different frequency showed in **Fig. 2**. For the samples treated at 1.5 MHz (**Fig. 2**(a)), the additional non-magnetic rhombohedral Fe_2O_3 (●) [JCPDS # 890599] and rhombohedral BaFeO_3 (□) [JCPDS # 740646] was exhibited. The origins of the secondary phase formations are related to (1) the presence of higher pressure inside solution during lower frequency sonication [26] and (2) the less optimized processing condition for the rod formation. Furthermore, the higher peak intensity appeared

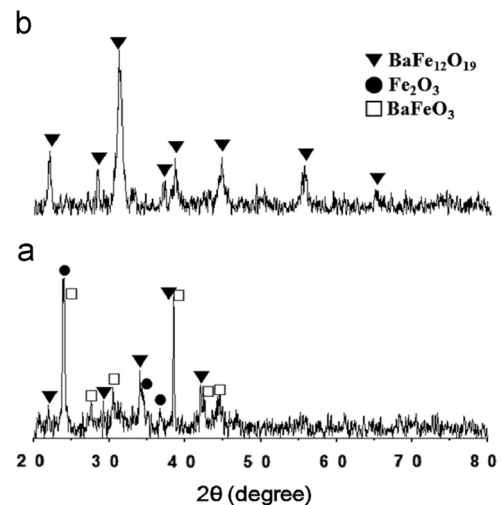


Fig. 2. Powder X-ray diffraction patterns of $\text{BaFe}_{12}\text{O}_{19}$ powders, synthesized at (a) 1.5 MHz and (b) 1.7 MHz.

around 24 degree, 34 degree and 40 degree is due to the overlap of other phases. In the case of the diffraction peak appeared 24 degree, the analysis revealed the overlap between (0 1 5) orientation of BaFeO_3 and (0 1 2) orientation of Fe_2O_3 peak. As a result the overlap intensity is larger than that of individual peak. For ~ 40 degree, the larger peak intensity is also due to the overlap between (2 0 3) orientation of $\text{BaFe}_{12}\text{O}_{19}$ and (2 0 5) orientation of BaFeO_3 . In the area of 34 degree, we also noticed the overlap of (1 0 4) orientation of Fe_2O_3 and (1 1 4) orientation of $\text{BaFe}_{12}\text{O}_{19}$. On the other hand, all the diffraction peaks could be indexed as hexagonal $\text{BaFe}_{12}\text{O}_{19}$ (JCPDS #840757) for the sample treated at 1.7 MHz (**Fig. 2**(b)) indicating that the optimal synthesis condition was achieved. Furthermore it is worth noting that the full width at half maximum of the secondary phase peaks are smaller than that

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