第 1論　文　の　要　旨

題　目: SYNTHESIS AND DISPERSION OF CORE-SHELL α”-$Fe_{16}N_2$ NANOPARTICLES FOR THEIR MAGNETIC APPLICATIONS

（コアシェル窒化鉄ナノ粒子の合成・分散と磁性材料への応用）

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Magnetic materials play a key role in modern life since these magnetic materials have been currently being applied in human life and industrial applications, such as magnetic recording devices, motor, biomedical, energy alternatives, etc. However, up to now, most of the high energy product of magnet materials contain rare-earth components, such as Nd, Sm, Dy, and Tb. Therefore, the high energy product of magnet materials without rare-earth components are highly desired for new generation of magnetic materials. α”-$Fe_{16}N_2$ invented as magnetic material with high saturation magnetization has a good potential as a candidate for new rare-earth-free magnetic material with high magnetic performance. Therefore, detailed understanding on the synthesis of α”-$Fe_{16}N_2$ particles and their magnetic performance are highly desired to make clear the application of this material for the formation of high performance of rare-earth-free magnetic material.

In this dissertation, synthesis of core-shell α”-$Fe_{16}N_2$ nanoparticles from plasma-synthesized core-shell α-Fe nanoparticles via gas phase process was studied in detail. Effects of particle sizes, shell compounds, and shell thickness of α-Fe on α”-$Fe_{16}N_2$ phase formation and magnetic performance were investigated. Enhancement on the α”-$Fe_{16}N_2$ yield, particularly on the nitridation of core-shell Fe/SiO_2 and large particles of core-shell Fe/Al_2O_3 nanoparticles were done by introduction of oxidation reaction prior to nitridation process. Since the prepared core-shell α”-$Fe_{16}N_2$ nanoparticles contained some agglomerates and aggregates, therefore dispersion process via low energy beads-mill dispersion were proceeded to prepare well-dispersed single domain α”-$Fe_{16}N_2$ nanoparticles slurry. The major contents of this dissertation are listed as follow.

Chapter 1 describes the background and the motives of the current research. Basic explanation and review of previous researches on the synthesis and application of α”-$Fe_{16}N_2$ nanoparticles of which magnetic moment is the highest among rare-earth free materials as well as an overview of beads-mill dispersion technology were provided.

Synthesis of core-shell typed α”-$Fe_{16}N_2$ from several kinds of core-shell α-Fe nanoparticles differed by particles size, shell thickness, and shell compounds and magnetic performance are discussed in **Chapter 2**. The results showed that the yield of α”-$Fe_{16}N_2$ decreased with an increase in particle size and shell thickness due to the limitation in nitrogen diffusion phenomena and no yield on the shell of SiO_2 was observed. The rate of nitridation was also calculated. By varying reduction and nitridation conditions, the best conditions to obtain high yield were found as; reduction at 300°C for 1-2 hours and nitridation at 145°C for 10 hours. The highest yield of 99% was obtained using particle with diameter of 43 nm and 20 wt% of Al_2O_3 shell, showing the best saturation magnetization of 190 emu/g.

In **Chapter 3,** the nitridation process of core-shell Fe nanoparticles with SiO_2 as shell was investigated in detail. It was found from X-ray diffraction, nitrogen adsorption and Mössbauer measurements that introduction of oxidation at 300°C for 3 hours before reduction by H_2 gas could produce α”-$Fe_{16}N_2$ phase up to 90%. The role of oxidation is confirmed as follows; (1) removing of the gap between the core and shell, (2) oxidizing to γ-Fe_2O_3 phase, and (3) forming of microporous structures of the Fe core phase by reduction. This finally enhances N atoms diffusion during nitridation.

According to the results in Chapter 3, the formation of α”-$Fe_{16}N_2$ phase from large-sized core-shell Fe/Al_2O_3 (62 nm in diameter) which was also found to be difficult for nitridation was investigated in detail as discussed in **Chapter 4**. The approach was similar to that in the case of SiO_2 shell, resulting in the yield of up to 90%. The saturation magnetization and magnetic coercivity of the core-shell α”-$Fe_{16}N_2$/Al_2O_3 magnetic nanoparticles were found to be 156 emu/g and 1450 Oe, respectively.

Since the dispersion process of magnetic nanoparticles to single domain size is the most important for their practical applications, beads-mill dispersion of agglomerated and aggregated α”-$Fe_{16}N_2$ nanoparticles was studied in detail in **Chapter 5**. Low energy dispersion of core-shell α”-$Fe_{16}N_2$ and α-Fe nanoparticles was performed in toluene by all separator type beads-mill machine with fine beads. The effects of bead size, rotation speed and dispersion time on the dispersibility were investigated. It was found that even a slight deviation from the optimum conditions (30μm, 20 Hz and 2-5 hours) would result in undispersed or broken particles due to fragile core−shell structure against stress or impact force of beads. The single domain α”-$Fe_{16}N_2$ nanoparticles showed unique magnetic properties due to the strong magnetic interaction among magnetic nanoparticles under the applied magnetic fields.

Chapter 6 contains the summary of all chapters and direction for further investigation.