論　文　の　要　旨

題　目：PREPARATION AND EVALUATION OF MAGNETIC NANOCOMPOSITE FILMS AND FIBERS CONTAINING α″-$Fe_{16}N_2$ NANOPARTICLES
（窒化鉄ナノ粒子を複合した磁性体ナノコンポシットフィルムおよびファイバーの合成と性能評価）

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Magnetic materials play a key role in modern life as they facilitate the conversion of electrical to mechanical energy, transmission and distribution of electric power, microwave communications, and data storage systems. Nowadays, magnetic materials are used in various advanced devices, such as motor, recorder data devices, biomedical, sensor, spintronic devices, ferrofluid-related devices, etc. Until now, permanent magnet has been continuously developed in response to the growing demand for higher volume-specific magnetic power to support the advancement of motors, generators, and other energy-related applications. These broad applications require a magnetic material with high energy product. Having the highest magnetic moment and high uniaxial magnetic anisotropy among any ferromagnetic material, α"-$Fe_{16}N_2$ nanoparticles (NPs) emerge as a potential rare-earth-free permanent magnet. However, these NPs have a high magnetic interaction among the NPs and a small magnetic coercivity. These problems can be solved by structuration of α″-$Fe_{16}N_2$ NPs. α″-$Fe_{16}N_2$ NPs were synthesized by nitridation and then followed by dispersion to break-up the agglomerates NPs, can be structurized in a nanocomposite form such as film and fiber. Magnetic field was applied during nanostructuration to align the magnetic moment of α"-$Fe_{16}N_2$ NPs. A magnetic field was applied during nanostructuration to align the magnetic moment of α"-$Fe_{16}N_2$ NPs for enhancing the magnetic properties. Therefore, detailed understanding on the preparation of α"-$Fe_{16}N_2$ nanocomposites under applied magnetic field and their magnetic performance are highly desired.

In this dissertation, preparation and evaluation of α″-$Fe_{16}N_2$ nanocomposite films via spin coating and fibers via electrospinning are systematically investigated. An external magnetic field was applied during the nanocomposites preparation to align the magnetic moment of the α″-$Fe_{16}N_2$ NPs. The effect of this applied magnetic field on the nanocomposite morphology, structure and magnetic properties was studied. The major contents of this dissertation are listed as follow.

Chapter 1 describes the background and the motivation of this research. Basic theoretical explanation and review of previous researches on the α″-$Fe_{16}N_2$ magnetic materials, which their magnetic moment is the highest among rare-earth free materials, as well as an overview of the magnetic nanocomposite materials were also provided.

Synthesis of α″-$Fe_{16}N_2$ NPs film via spin-coating under applied magnetic field as well as effects of these magnetic field on the magnetic performance are detail explained in **Chapter 2**. Single domain core–shell α″-$Fe_{16}N_2$/Al_2O_3 NPs with average size of 47 nm were synthesized by nitridation process. Due to the agglomeration, a low energy dispersion process was done to produce well-dispersed α″-$Fe_{16}N_2$/Al_2O_3 NPs in toluene solvent prior to film preparation. α″-$Fe_{16}N_2$ NPs film was prepared by spin coating of the well-dispersed NPs slurry on the Si substrate. An external magnetic field of 1.2 T was applied vertical to the substrate during the film formation to align the magnetic moment of NPs followed by fixation of the NPs with resin. The SEM and XRD results showed that densely packed assemblies of the NPs were formed and aligned perpendicularly in a ~1 μm film. Based on the magnetic hysteresis curve of the NPs film which is obtained from SQUID analysis, the application of the magnetic field during film formation increase the magnetic coercivity (Hc) and remanence (Mr) values of the resulted films by 23% and 55%, respectively.

In **Chapter 3** the effect applied magnetic field strength on the magnetic performance of α″-$Fe_{16}N_2$ magnetic film via spin-coating are discussed in detail. The film with thickness of 1 μm was prepared with same method as in the chapter 2. However, some external magnetic fields of strength 0, 0.6, 0.9, or 1.2 T were applied to examine the magnetic orientations of α″-$Fe_{16}N_2/Al_2O_3$ NPs films. X-ray diffraction and SQUID analyses showed the relationship between the aligned orientation of the NPs and their magnetic properties; magnetic coercivity, remanence, and maximum energy product enhanced by 24%, 66%, and 160%, respectively, with increase in magnetic orientation of 35%. The shape of hysteresis loops of the films approaches to rectangular by increasing the vertically applied magnetic field. These results were further verified by applying the magnetic field horizontally. The magnetic properties of the film were decreased by applying the magnetic field horizontal to substrate. These findings showed that control of the magnetic orientation of single domains of magnetic NPs improves the magnetic performances compared with that of agglomerated magnetic NPs.

According to the successfulness to synthesize well-dispersed single domain α″-$Fe_{16}N_2$ NPs film under magnetic field, the effect of applied magnetic field on diameter of nanocomposite fiber and its magnetic performance are investigated in **Chapter 4**. Magnetic nanocomposite fibers containing α″-$Fe_{16}N_2$ NPs, hard magnet, in polyvinylpyrrolidone (PVP) matrix were prepared via electrospinning with and without magnetic field applied to the spinning direction. The external magnetic field of 0.1 T was applied during process of electrospinning. α-Fe NPs which is soft magnet was also used to investigate the effect of NP magnetic strength on the fiber properties. These NPs were dispersed in toluene using beads-mill dispersion to prepare MNP slurry. The MNP slurry was mixed with a 15 wt % of PVP solution to make a ferrofluid precursor for electrospinning with two different loadings of 16.5wt% and 28.4 wt% of MNPs. The ferrofluid was then electrospun with a flow rate of 2–20 μl min^{-1} under an applied high voltage. The environment during electrospinning was kept at a temperature of (30±2) °C and a relative humidity of (30±5) %. SEM and TEM analyses results showed that the applying the magnetic field in the same direction as the electric field resulted in smaller and more uniform fiber diameters. Further, nanocomposite fibers containing α″-$Fe_{16}N_2$ had smaller diameters than those containing α-Fe NPs. These magnetic-field effects on the fiber formation were explained by referring to the kinetic energy of the moving jet in the electrospinning process. In addition, magnetic hysteresis curves showed an enhancement of the magnetic coercivity and remanence by 23% and 22%, respectively.

Chapter 5 contains the summary of all chapters and direction for further investigation.