主　論　文　の　要　約

論文題目　**High-performance Sm-Co permanent magnets with novel microstructures developed using low-oxygen powder metallurgy process**
(低酸素粉末冶金プロセスを用いた新規微細構造を有する高性能 Sm-Co 永久磁石の開発)

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論　文　内　容　の　要　約

While the development of eco-friendly energy transport means such as electric vehicles and electric airplanes is accelerating, high performance of rare-earth permanent magnets, a core material for motors, is required. Neodymium-iron-boron (Nd-Fe-B) magnets are the strongest existing rare-earth permanent magnets and are widely used throughout the industry, however, their performance is almost saturated to theoretical values. One way to develop post-Nd magnets is nanocomposite magnets with the exchange-coupled hard and soft magnetic phases. In this study, the microstructure analysis and magnetic measurement of magnetic nanopowders and bulk magnets prepared using a low-oxygen powder metallurgy process were discussed in detail.

Chapter 1 describes what a permanent magnet is and what physical properties of the permanent magnet are important. A high-performance permanent magnet is a fundamental material that converts electrical energy into kinetic energy and is a key component used in motors such as electric vehicles and power generators. In particular, in the case of an electric vehicle currently used, a permanent magnet with a high coercivity is essential at an operating temperature of about 473 K. Therefore, realizing a permanent magnet with high coercivity at high temperature is a task that mankind must solve in order to sustainable growth of the permanent magnet market and, ultimately, keep the green earth. As a result of investigating the realization of nanocomposite magnets for the development of high-performance bulk permanent magnets through references, it was suggested that nanoscale-controlled microstructures are essential. For achieving this requirement, in this study, the nano-sized magnetic precursor powders were prepared using the low-oxygen powder metallurgy process. The preparation of magnetic nanopowders is still challenging because of oxidation. This study first attempts to prepare the non-oxidized hard magnetic nanopowder using the low-oxygen thermal plasma (LO-ITP) process, which is a gas evaporation bottom-up process. The specific research plans for developing high-performance bulk permanent magnets using the magnetic nanopowder were stated.

In Chapter 2, the preparation of samarium-cobalt ($SmCo_5$)/Fe nanocomposite magnet by mixing of micro-sized $SmCo_5$ powder made by jet milling and Fe nanopowder made using the LO-ITP is presented. An anisotropic $SmCo_5$ powder with good squareness was prepared by jet milling, and its measured coercivity and magnetization under an external magnetic field of 7.16 MA/m were 1 MA/m and 102 A·m^2/kg, respectively. To achieve a nanoscale-controlled microstructure in NC magnets, non-oxidized Fe nanopowder was synthesized using the LO-ITP process. Magnetic measurements confirmed the anisotropic behavior and exchange-coupling effect of the $SmCo_5$/Fe magnet. Thin nano-sized Fe areas were created between $SmCo_5$ grains, resulting in the exchange-coupling in the $SmCo_5$/Fe magnet. The coercivity and magnetization under an external magnetic field of 7.16 MA/m of the $SmCo_5$/Fe magnet were measured as 0.38 MA/m and 120 A·m^2/kg, respectively. One approach for improving the magnetic properties of this bulk NC magnet, that is, the grain refinement and homogeneous distribution of the soft magnetic phase, exists. The LO-ITP process has potential in the synthesis of non-oxidized fine metal nanoparticles by controlling the cooling rate. When a quenching method was applied to an Fe-Co system with a higher magnetization than Fe to obtain Fe-Co nanopowders, further improvements in magnetization of a NC magnet could be realized.

In Chapter 3, the preparation of fine Sm raw powder by water-cooled Cu crucible gas atomization for use as a raw powder in the ITP process is presented. To prepare the Sm metal fine powder as a starting material for the ITP process, the HD process and water-cooled Cu crucible-gas atomization process were applied. Although the SmH_2 fine powder was obtained through the HD process, it was difficult to have Sm fine metal powders because of sintering during the hydrogen desorption process. On the other hand, the spherical Sm metal powder with D_{50} at 18 μm was prepared using the water-cooled Cu crucible-gas atomization process. Since this process has no quartz crucible, very pure Sm metal powder can be obtained. This Sm metal powder prepared using water-cooled Cu crucible-gas atomization was smoothly fed to the thermal plasma and uniformly evaporated.

In Chapter 4, the preparation of the anisotropic Sm-Co alloy nanopowder using the LO-ITP process is presented. Through this process, a mean particle size of 61 nm of non-oxidized Sm-Co alloy nanopowder was successfully prepared, which has not been reported so far. The metal magnetic compounds $SmCo_5$ and $SmCo_7$ were achieved without oxidation. Although a soft magnetic phase of Co was created, the measured coercivity was 0.45 MA/m. The anisotropic magnetic behavior of the Sm-Co alloy nanopowder was demonstrated by the crystallographic alignment in the X-ray diffraction (XRD) profile and magnetic measurements. From detailed microstructure analyses, nanoparticles with unique core/shell structures were observed, and the anisotropic magnetic behavior of Sm-Co alloy nanopowder was verified using a magnetic measurement. An exchange-coupling effect was also observed in the powder state, which may originate from the interaction between the soft magnetic phase of the Co-core and the hard magnetic phase of the Sm-Co-shell. In addition, the nanoparticle synthesis mechanism during the thermal plasma process was analyzed using numerical analyses, suggesting a plan for preparing Sm-Co alloy nanopowders with enhanced magnetic properties.

In Chapter 5, the Sm-Co bulk magnet prepared using Sm-Co alloy nanopowder as a precursor displays a giant coercivity of 4.1 MA/m at 300 K, which is the world's highest coercivity among the reported rare-earth permanent bulk magnets up to now. The single-crystal grains with an average size of approximately 0.63 μm and the sharp grain boundary were identified using a scanning transmission electron microscopic (STEM) observation and high crystallinity was estimated by XRD measurement, which contributed to the realization of a huge coercivity. Moreover, since the coercivity at room temperature is greatly high, it also exhibited a high coercivity of 1.6 MA/m even at a high temperature of 573 K and a good temperature coefficient of coercivity of -0.22%/K. The thermal stability of the Sm-Co magnet with fine grain size in this work is superior to that of the reported Nd-Fe-B magnets and Sm-Co magnets, suggesting that it has the advantage of being applicable to high-temperature magnets. The rare-earth fine alloy nanopowder prepared by a novel gas evaporation process is a promising candidate to consolidate the bulk permanent magnets with enhanced magnetic properties.

In Chapter 6, the conclusion of this study and future plans are summarized. this study is a preliminary experiment to prove the possibility of the LO-ITP process for preparing the magnetic nanopowder precursors. Especially, the success of preparing Sm-Co alloy nanopowder by the LO-ITP is of great significance. In the ITP process, when the elements, such as Sm and Co, with very different intrinsic properties such as vapor pressure and surface tension, are used, non-uniform composition distribution of the final product is caused. Despite the difficulty in synthesizing RE-alloy nanopowder, Sm-Co alloy nanopowders with relatively uniform composition distribution were successfully prepared. Furthermore, the experimental solution to improve the magnetic properties of Sm-Co alloy nanopowder was contrived by the numerical analysis. Moreover, this study proposed that the LO-ITP process was a new breakthrough strategy to prepare the different kinds of RE-alloy nanopowder with improved magnetic properties, and not only the Sm-Co system. The next task is to retain the stability of the synthesizing alloy metal nanopowder through the optimization of various parameters of the ITP process, such as the cooling rate, feeding condition, and vaporization condition, as well as proceed with the realization of an advanced magnetic material with nanoscale-controlled microstructures that cannot be achieved with conventional magnetic materials.