題 目: One-step Synthesis of Submicron-sized FeNi and SiO₂-coated FeNi Particles via Spray Pyrolysis Method and Their Magnetic Characteristics

(噴霧熱分解法によるサブミクロンサイズの FeNi および SiO2 コート FeNi 粒子のワンステップ合成 とその磁気特性)

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Advanced soft-magnetic particles are of paramount importance for the development of power converter components, particularly powder core inductors. These components play a crucial role in achieving sustainable development goals in the context of digital devices, electric machines, and renewable power generators. Incorporating advanced soft-magnetic particles as secondary materials offers significant improvements in the magnetic characteristics of powder core inductors, providing advantages for machine design. This dissertation presents a comprehensive investigation into the synthesis of submicron-sized spherical iron nickel (FeNi) and silica-coated iron nickel (FeNi@SiO₂) particles via the aerosol process, with the aim of utilizing them as secondary materials in powder core inductors. The research encompasses the exploration of various experimental parameters and equipment designs to evaluate the effectiveness of FeNi@SiO₂ particles for powder core inductors. Additionally, computational fluid dynamics (CFD) simulations are employed to gain a better understanding of the plausible mechanisms involved in particle formation. Each chapter of this dissertation is briefly described below.

Chapter 1 over-viewed the powder core inductor, fundamental magnetic characterization, recent studies in improving the magnetic characteristic of a powder core inductor, strategies in synthesizing FeNi and FeNi@SiO₂ particles, and a numerical approach based on computational fluid dynamic (CFD). This summary and problem statements lead to the motivation of the research covered within the scope of this dissertation.

Chapter 2 focuses on the synthesis of FeNi particles through spray pyrolysis, specifically addressing the challenge of using alternative reduction agents due to safety concerns associated with using 100% H_2 gas. The effects of different reduction agents, such as ethanol, ethylene glycol, and formic acid, and their concentrations in the precursor solution are investigated. Ethanol and ethylene glycol, at a concentration of 25 vol%, successfully yield spherical and submicron-sized FeNi particles with dense and hollow structures, respectively. Conversely, the use of formic acid results in the formation of segregated particles comprising FeNi and FeO phases. To explain the plausible mechanisms of FeNi particle synthesis using different reduction agents, CFD simulations are performed to examine temperature profiles and the formation of H_2 and H_2O gases in the spray pyrolysis reactor. It is concluded that the FeNi particle's formation of H_2 and H_2O gases derived from different reduction agents influences the resulting structure of FeNi particles, with ethanol emerging as the preferred reduction agent.

The use of ethanol in synthesizing FeNi particles presents a challenge in forming some hollow particles, which can limit the performance of inductors due to their low density. **Chapter 3** discusses strategies to control the structure and density of FeNi particles, transforming them from hollow (low density) to dense (high density), by manipulating

reactor temperature from 1200 to 1400 °C and carrier gas flow rate from 5 to 1.3 L/min. It is pointed that the temperatures above 1200 °C can promote the alteration of structures from hollow to dense, but it leads to the formation of undesired carbon nanoparticles. Therefore, the effect of carrier gas flow rates is investigated at 1200 °C. Decreasing the carrier gas flow rate prolongs particle residence time in the reactor, facilitating densification and reducing the formation of hollow particles. Consequently, the density of FeNi particles is significantly improved. Furthermore, magnetic characterization reveals that the synthesized FeNi particles exhibit the highest saturated magnetization value among other synthesis methods, which reach the theoretical value. In terms of inductor applications, FeNi particles with denser structures and higher densities offer improved DC bias characteristic of the inductor.

Chapter 4 is focused to the synthesis of FeNi@SiO₂ particles in a one-step process via spray pyrolysis, aiming a powder core with high DC bias characteristic and low energy loss. The strategy involves introducing the silica source to the intermediate product of FeNi aerosol. A preheater system is implemented to transform FeNi precursor droplets into their intermediate product. Hexamethyldisiloxane (HMDSO) is used as the silica source, and three types of developed connectors (T-shaped, Swirler-3, and Swirler-6) are compared. Based on the structure and morphology of particles obtained with different connector types, Swirler-6 is identified as the optimal choice, offering the highest coating ratio and the lowest generation of undesired nanoparticles. A CFD simulation is performed to assess the mixing performance between FeNi droplets and HMDSO vapor in the reactor, providing insight into the favorable distribution of FeNi droplets and HMDSO vapor until the end of the reactor, which promotes heterogeneous nucleation. The synthesized FeNi@SiO₂ particles are then compared with the synthesized FeNi particles in terms of inductor performance. The FeNi@SiO₂ particles not only enhance the DC bias characteristic (high saturation current) of the inductor but also reduce energy losses (core loss) between interparticles within the inductor.

Chapter 5 contains the summary of all chapters and directions for the future work.