Development of a method to observe demagnetization field in thin-foiled magnets using electron holography

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論 文 名：Development of a method to observe demagnetization field in thin-foiled magnets using electron holography
（電子線ホログラフィーを用いた磁性薄膜の反磁場観察技術の開発）

区　　分：甲

論文内容の要旨

Electron holography (EH), which is a method relevant to transmission electron microscopy (TEM), determines the phase shift of incident electron wave that has traversed a thin-foiled specimen. Since the phase shift is owing to the magnetic flux density B of the thin-foiled specimen, EH has been applied to magnetic imaging in various materials and devices. With reference to sintered magnets, including the Nd-Fe-B magnet which is widely used in traction motors in electric vehicles, a crucial problem is to disclose the demagnetization field H_d within individual crystal grains. This is because H_d plays an important role in determination of the engineering parameter "coercivity" which represents the critical magnetic field to induce undesired magnetization reversal. However, it remains a challenge to directly observe H_d, which is one component to express $B=\mu_0(M+H)$, where μ_0, M, and H stand for vacuum permeability, magnetization, and magnetic field, respectively. Here, H is regarded to be a summation of H_d inside specimen and the stray magnetic field H_s outside specimen: *i.e.*, $H=H_\mathrm{d}+H_\mathrm{s}$. In short, although EH allowed to determine the phase shift due to magnetic flux density ($\Delta\phi_B$), it has been difficult to deduce the phase shift due to demagnetization field ($\Delta\phi_{H_\mathrm{d}}$) from the observation of $\Delta\phi_B$. The lack of the technique to reveal H_d is common for other methods of TEM, such as Lorentz microscopy and differential phase contrast method, both of which measure the deflection of incident electrons by Lorentz force due to B.

Thus, the purpose of this study is to establish a method that allows for extracting the phase information related to H_d using EH observations. In order to separate $\Delta\phi_{H_\mathrm{d}}$ from the unwanted phase information, the phase shift due to magnetization ($\Delta\phi_M$) was determined by the surface integral with reference to $\mu_0 M$, which was performed over the cross-sectional area of specimen. The phase shift due to stray magnetic field ($\Delta\phi_{H_\mathrm{s}}$) was predicted based on the three-dimensional calculations of magnetic field for the area outside the specimen, whose shape, size, and crystal orientations could be determined accurately by TEM observations. As a result of the subtraction of $\Delta\phi_M$ and $\Delta\phi_{H_\mathrm{d}}$ from the EH observation of $\Delta\phi_B$, which was acquired from a $Nd_2Fe_{14}B$ crystal, the phase image representing $\Delta\phi_{H_\mathrm{d}}$was obtained to reveal the map of H_d. In addition, toward the precision improvement of this method, effectiveness of a noise-reduction technique referred to as wavelet hidden Markov model (WHMM) was assessed.

Chapter 1 described the background about Nd-Fe-B magnets, for which researchers made significant efforts to improve the coercivity in the previous three decades. Subsequently, a challenge to observe H_d using EH, which is essential for coercivity improvement was emphasized.

Chapter 2 reviewed the electron microscopy techniques that were employed in this study, including conventional TEM, EH, electron diffraction, and electron energy-loss spectroscopy (EELS). The basic principles to reveal the crystallographic microstructure, magnetic microstructure, crystal orientations with reference to the incident electrons, and thickness of thin-foiled specimens were explained.

Chapter 3 showed the outline of the method to figure out H_d information stored in EH observations. A process was proposed to obtain $\Delta\phi_{H_\mathrm{d}}$ through the subtraction of unwanted phase information $\Delta\phi_M$ and $\Delta\phi_{H_\mathrm{d}}$ from the experimental data of $\Delta\phi_B$. When the morphology of thin-foil specimen is uncovered by TEM and EELS, $\Delta\phi_M$ and $\Delta\phi_{H_\mathrm{d}}$ can be estimated based on the surface integral using the in-plane components (*i.e.*, the component vertical to incident electrons) of M and H_s multiplied by factors μ_0, e, and $\hbar$, where e and $\hbar$ stand for elementary charge and Planck's constant divided by 2π. In addition, this chapter remarked the issues of phase-shift calculations by using M and H_s, in place of the vector potential A which in principle exerts a force to electrons: that is, the offset of phase shift should be appropriately determined when the phase shift is calculated by the surface integral with reference to M and H_s.

Chapter 4 discussed the validity of this method, which was applied to the analysis using an artificial $Nd_2Fe_{14}B$ specimen having a simple rectangular shape. Following the process indicated in Chapter 3, $\Delta\phi_{H_\mathrm{d}}$ was extracted from the phase image representing $\Delta\phi_B$. The result of $\Delta\phi_{H_\mathrm{d}}$ was converted to a map of H_d within the rectangular specimen. The magnitude of H_d was maximized near the specimen edge, showing the maximum value of 0.67 tesla. The result was consistent with the predictions in previous simulation studies.

Chapter 5 examined the effectiveness of this method in greater detail using a real, thin-foiled specimen of $Nd_2Fe_{14}B$ which allowed for collecting the experimental data: that is, EH observations of $\Delta\phi_B$, TEM images, electron diffraction patterns, and thickness maps by EELS. The mapping of H_d, which was determined by the process in Chapter 3, showed an excellent agreement with the simulations (*i.e.*, Landau-Lifshitz-Gilbert calculations applied to this thin-foiled specimen) in terms of the magnitude and distribution of H_d. The results indicated that the method proposed could be applied to comprehensive studies about demagnetization field within real specimens.

Chapter 6 reported on the impact of noise reduction, which was carried out for improving the precision in the phase-shift analysis. The method described in Chapter 3 was comprised of complex image processing, including subtraction and differential of the phase images to obtain $\Delta\phi_{H_\mathrm{d}}$ and H_d. In order to reduce the noise that was amplified during this image processing, this study employed WHMM which allowed a reasonable separation of weak signal from noise. It was demonstrated that WHMM could be a promising tool for the noise reduction in EH observations, and accordingly beneficial to the process of extracting H_d.

Chapter 7 summarized the achievements in this study.