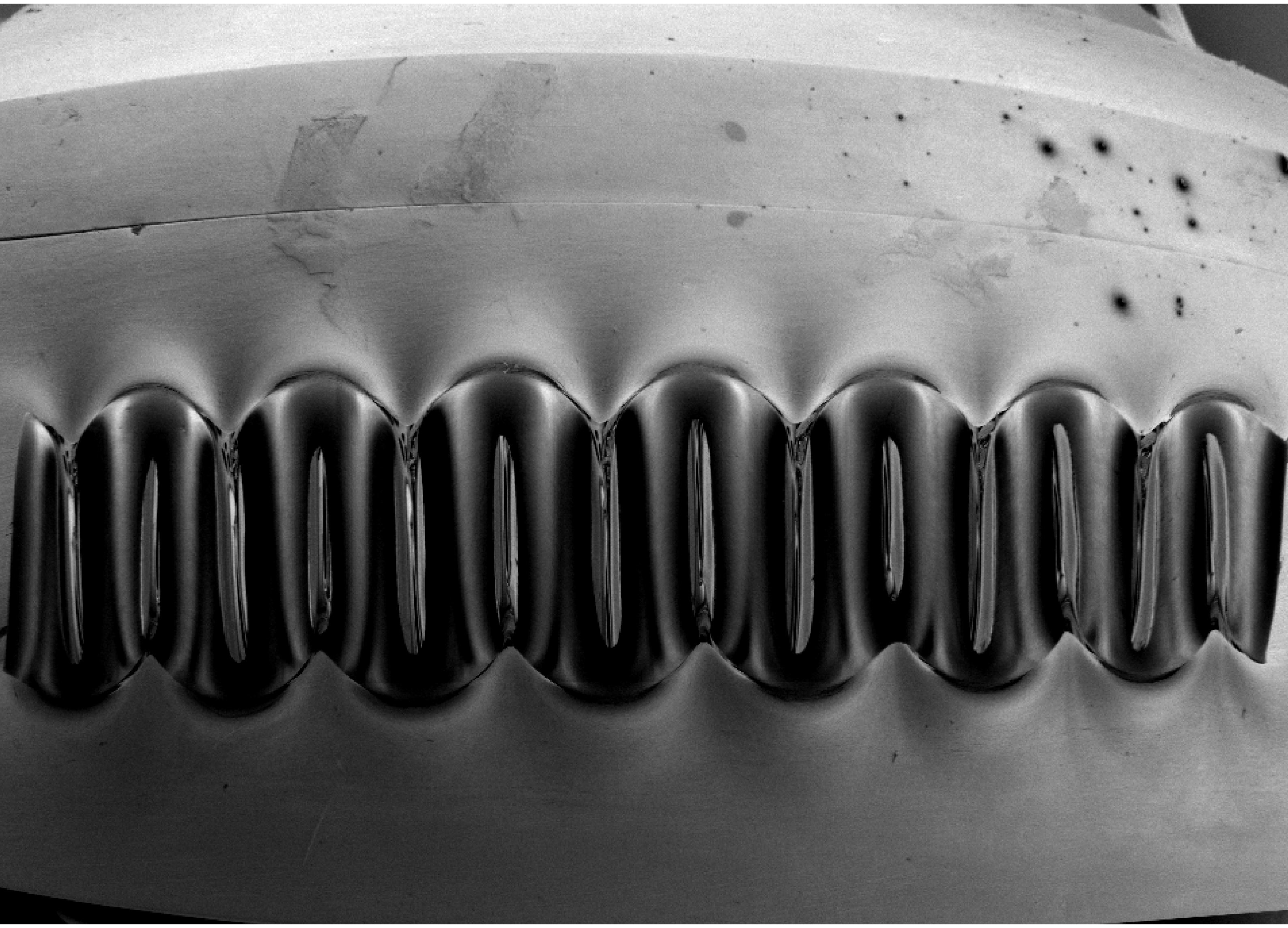
Application Note



How the Imaging of Magnetic Samples Benefit from Gemini Optics

ZEISS FE-SEMs

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“Can a scanning electron microscope image magnetic material?” This is a common question asked by many people. There are legitimate concerns when trying to put magnetic samples in an SEM. For example, will the magnetic field from the sample disturb the imaging? Or, will the sample be attracted into the lens and damage the SEM? In this application note, these questions will be answered and several examples of imaging magnetic samples using ZEISS FE-SEMs (Field Emission Scanning Electron Microscopes; ZEISS GeminiSEM Family and ZEISS Sigma Family) will be shown.

Introduction

“Magnetic material” is usually referred to as ferromagnetic or ferrimagnetic material, which has a spontaneous magnetic moment originating from the alignment of magnetic moments on a microscopic level [1]. On a macroscopic level, these aligned magnetic moments form magnetic poles on the sample surface which creates a stray field around the sample. In this case, the sample is polarized or magnetized. However, the net magnetic moment of one specimen can also be very small. The macroscopic specimen is usually composed of small regions called domains, within each of which the magnetic moments are all aligned. However, overall the domains form a closed magnetic loop, such that the net magnetization and the magnetic stray field from the sample is at minimum. In this case, the sample is demagnetized.

The presence of a stray field from the sample will deflect the primary electron beam in the SEM due to the Lorentz force. The stray field from a magnetized sample in an SEM not only causes distortion to the SEM image; it also gives a strong astigmatism and defocus (Figure 1a). Such an effect is especially strong if the energy of primary electron is low. On the other hand, a demagnetized sample (Figure 1b) does not generate a stray field, and the electron beam can be focused normally. In both situations, an objective lens with an axial gap is used where the magnetic field is contained within the lens (Figure 1a & 1b).

In order to reduce the lens aberration and enhance the resolution of the SEM, typical practice is to employ a magnetic immersion lens or single pole lens design where the complete sample is immersed in the magnetic field from the objective. This method, however, will induce a magnetic moment on a magnetic sample which strongly disrupts the intended magnetic field from the lens (Figure 1c) and causes the electrons to lose focus. Additionally, there would be a very strong attraction force between the sample and the lens which might cause catastrophic damage to the sample and the SEM.

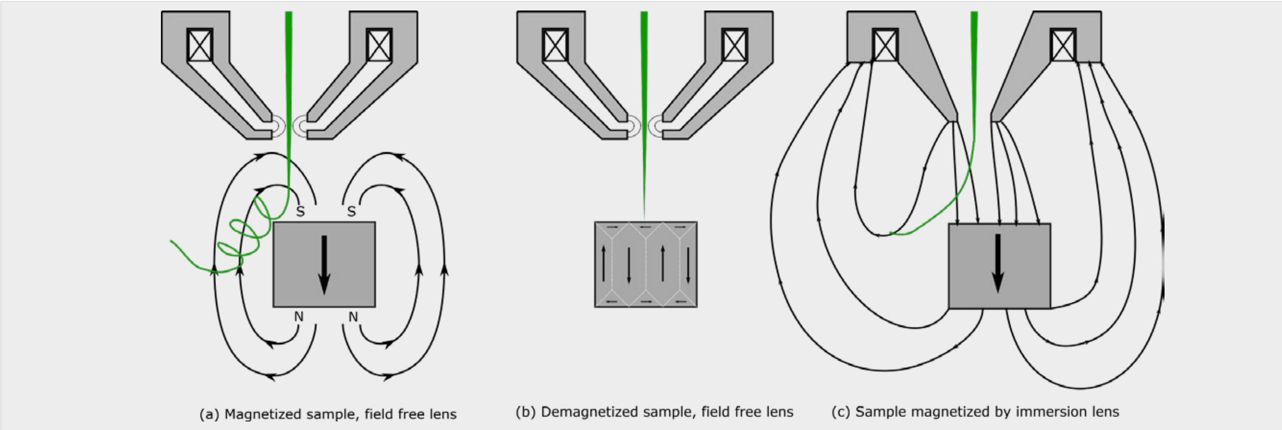

Figure 1 Different situations of magnetic material in SEM: (a) Magnetized sample under a field free lens. The stray field from the sample strongly influences the primary electron beam. (b) Demagnetized sample under a field free lens, the sample can be imaged properly. (c) The magnetic field from an immersion lens is strongly distorted by the presence of magnetic samples, thus focusing is impossible.

Conclusions

To image a magnetic sample in an SEM, two requirements must be fulfilled simultaneously:

- There should be no or a very weak magnetic field from the objective lens so that there is no induced magnetic moment on the sample
- The sample must be demagnetized so that there is no or only a very weak stray field around the sample

The Gemini lens design in ZEISS FE-SEMs utilizes the in-column beam deceleration of the beam booster to achieve reduced lens aberration and high resolution. Such design ensures that there is a minimum magnetic and electric field on the sample. Thus, for imaging magnetic samples using a ZEISS FE-SEM, there are generally no concerns about damaging the SEM or the samples. However, the user must pay attention to properly demagnetize the sample.

The stray field around the sample (caused by residue magnetization) can be easily seen with ZEISS FE-SEMs using the "fisheye" mode. In this special imaging mode, a low energy electron beam (with a landing energy of 50 eV) is scanned through a very large area across the complete SEM chamber. The low energy electron interacts strongly with the stray field of the sample. The presence of the stray field is usually observed around the edges of the sample where the background of the image is getting distorted. An extreme example of such distortion is shown by imaging a refrigerator magnet (Figure 2) where the alternating magnetic domains form the Halbach array [2]. A typical magnetic sample without proper demagnetization, will usually show mild distortion as it is imaged around the edge of a steel plate

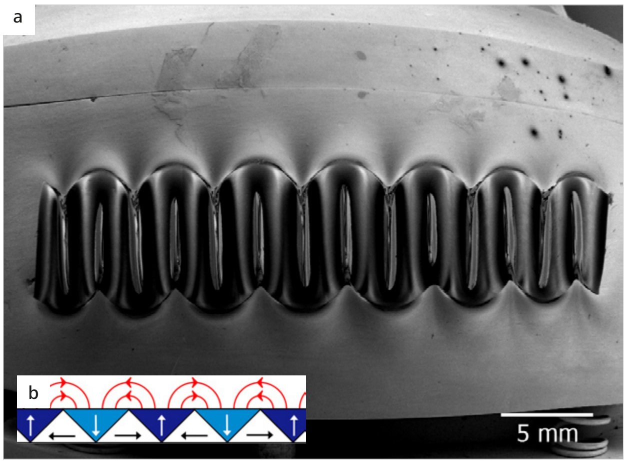

Figure 2 Refrigerator magnet imaged in "fisheye" mode, showing the arrangement of magnetic moments (a). Magnet flux distribution in the sample (b).

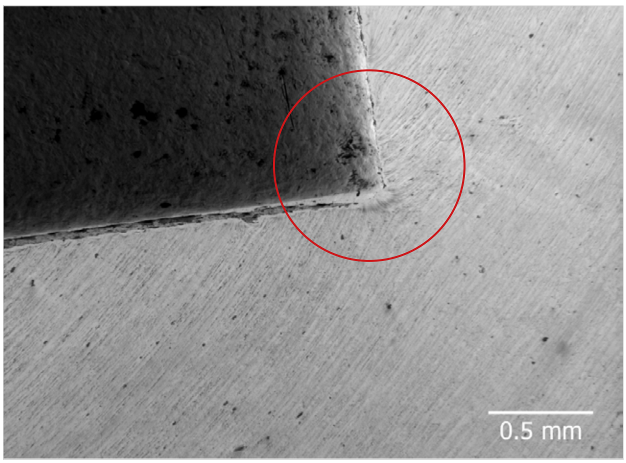

Figure 3 Steel plate imaged in "fisheye" mode. The residue magnetization from the sample causes distortion of the background of the image around the edge, indicated by the red circle.

(Figure 3). To perform high resolution imaging on such a sample requires a proper demagnetization. A magnetic sample can be demagnetized by placing it in a decaying alternating magnetic field, or if applicable, by heating the sample over its Curie temperature.

To demonstrate the importance of demagnetization, the fractured surface of a NbFeB magnet is imaged in an SEM. The NbFeB is a very strong magnet with a high magnetic moment. When imaged without demagnetization, even electron beams with a landing energy of 30 kV are deflected by the stray field from the sample - showing only the internal chamber of the SEM (Figure 4). This sample is then demagnetized by heating it up to its Curie temperature and cooled down again. It can then be imaged the same way as normal samples.

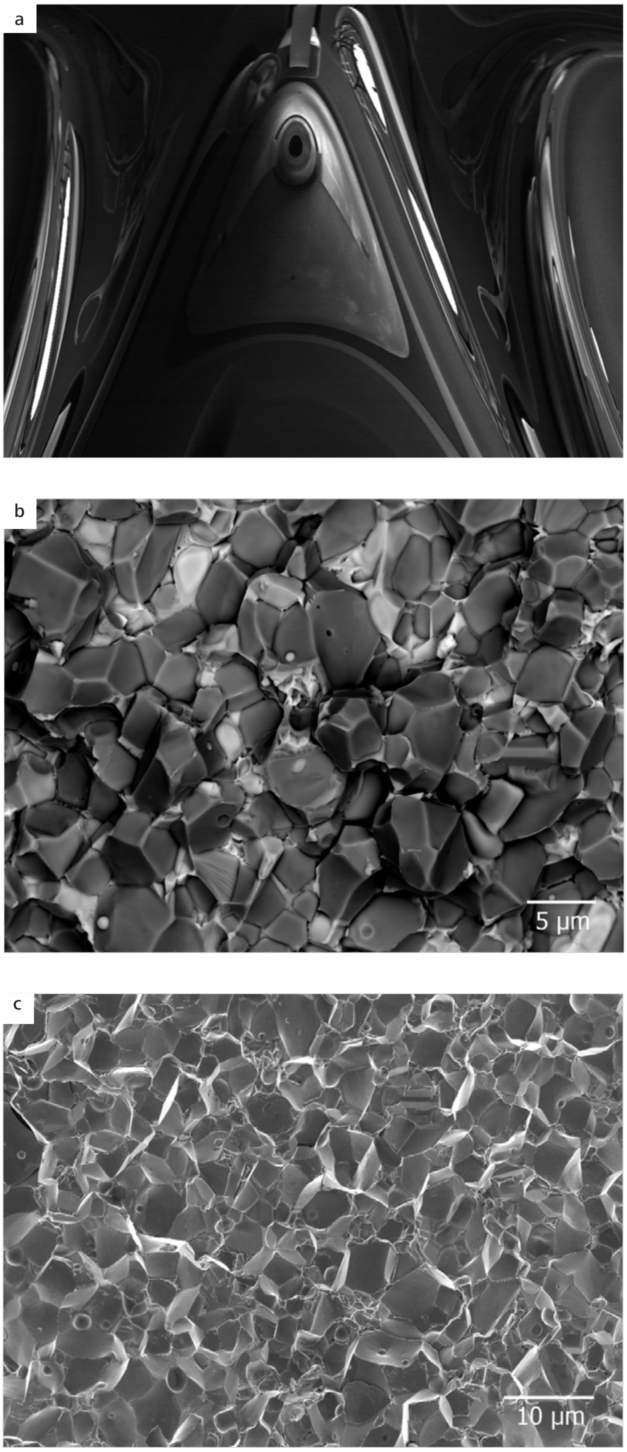

Figure 4 *The fractured surface of a NbFeB magnet imaged in magnetized state: (a) and demagnetized state (b) and (c). Image (b) is a BSE image obtained at 15 kV and shows the dark Fe-rich phase and bright Nb-rich phase. The SE image at 2 kV (c) shows the surface topography of the fractured surface.*

As long as the samples are demagnetized, they can be imaged using a ZEISS FE-SEM at high resolution in almost any condition. In the following examples, magnetic recording medium (Figure 5), bulk steel samples (Figure 6), and magnetic nanoparticles (Figure 7) are imaged at high resolution under various conditions.

Summary

- The magnetic samples must be demagnetized for SEM imaging
- The residue magnetic field from the sample can be checked using "fisheye" mode
- Demagnetized samples can be imaged just like non-magnetized samples in ZEISS GeminiSEM and ZEISS Sigma

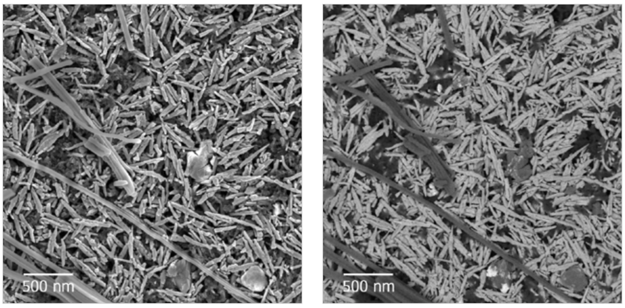

Figure 5 *Magnetic recording medium imaged at 2 kV and 1 mm working distance using Inlens SE (left) and BSE (right) detector. The needle like iron oxide particles are well resolved.*

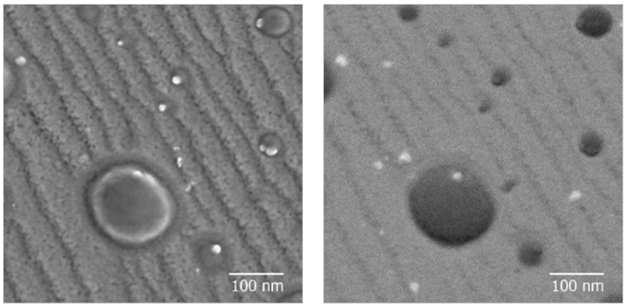

Figure 6 *Bulk steel imaged at 1 kV and 2 mm working distance using Inlens SE (left) and BSE (right) detector. The circular particles in the material are the non-metallic inclusions.*

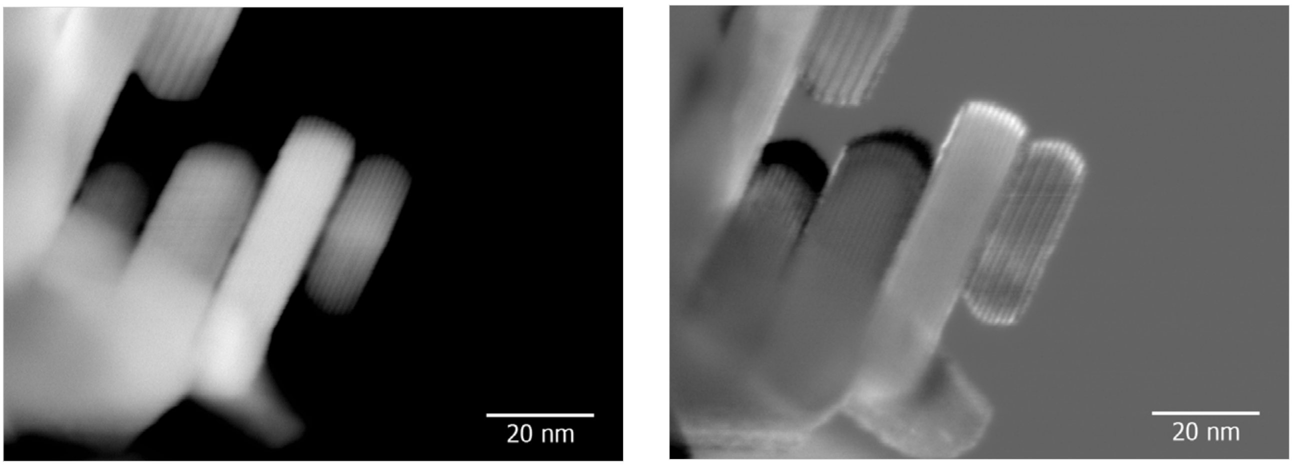

Figure 7 *Barium hexaferrites nanoparticles imaged at 22 kV and 1.5 mm working distance in STEM mode using high angular dark field (left) and oriented dark field (right). The lattice planes along the hexagonal axis are well resolved.*

References:

[1] C. Kittel, Introduction to Solid State Physics, 7th edition, John Wiley & Sons (1996)

[2] Refrigerator magnet (https://en.wikipedia.org/wiki/Refrigerator_magnet)

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