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Foucault imaging by using non-dedicated transmission electron microscope

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An electron optical system for observing Foucault images was constructed using a conventional transmission electron microscope without any special equipment for Lorentz microscopy. The objective lens was switched off and an electron beam was converged by a condenser optical system to the crossover on the selected area aperture plane. The selected area aperture was used as an objective aperture to select the deflected beam for Foucault mode, and the successive image-forming lenses were controlled for observation of the specimen images. The irradiation area on the specimen was controlled by selecting the appropriate diameter of the condenser aperture. © *2012 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4748124]

Lorentz microscopy[1] is a powerful tool for observing magnetic domain structures with high spatial resolution. The Foucault mode, which is one of the two typical kinds of Lorentz microscopy, has a particular advantage in which it can visualize the domain structure on the nano-meter scale using in-focus conditions, which the other Fresnel mode cannot. However, the Foucault mode is disadvantageous in which the optical system requires an off-centered objective aperture in order to select the deflected electron beam penetrating through the domain to be observed. One solution is using an electron biprism instead of an objective aperture,[2] but the necessity of an objective lens remains.

Most magnetic domain structures in ferromagnetic materials are affected by the external magnetic field generated by the objective lens. Many attempts have been carried out to reduce the magnetic field in which a specimen is immersed, for example, by switching off the objective lens,[3] by positioning the specimen above the objective lens pole-piece,[4] and by using specially developed lenses. The methods by switching-off the objective lens and off-positioning the specimen are effective for Fresnel method but not for Foucault method. As the Foucault method requires an opaque mask at the crossover plane (diffraction plane), positional correlation between the crossover plane and the plane where the mask is located is rather difficult for the electron optical system. In order to achieve the appropriate correlation, special objective lenses have been developed, for example, an electrostatic lens,[5] a magnetic-field shielding lens,[6] and a mini-lens[7] that is weakly exited for a long focal length. Although these attempts have succeeded to perform the Foucault method and to observe the magnetic domain structure, the pole-piece of the conventional objective lens has to be replaced by special one. As a result, the microscope is dedicated for the observation of magnetic domain structures in the ferromagnetic materials and then it is impossible to obtain the conventional high-resolution images with the altered pole-piece configuration. Therefore, the microscopes customized for observing magnetic domain structures are categorized as Lorentz microscopes. This specialization is one of the reasons why the Foucault mode has not been widely and practically utilized.

In order to extend the utilization of a conventional transmission electron microscope (TEM) to magnetic domain-structure observation, we have developed a Foucault mode that does not make use of any objective lens, similar to the Fresnel mode. Furthermore, it was confirmed that the developed optical system was effective for small angle diffraction with long camera lengths.[8]

Figure 1 shows the proposed optical systems for Foucault imaging mode in (a) and small angle diffraction mode in (b) by using a non-dedicated electron microscope. The objective

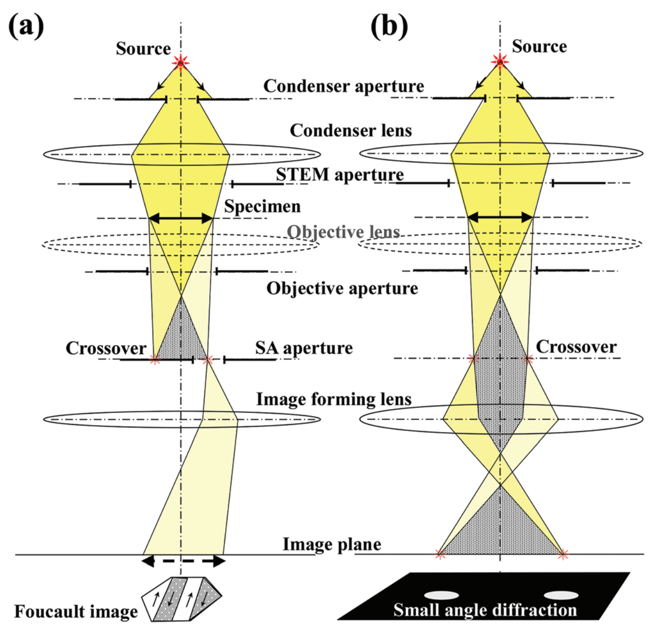

FIG. 1. Schematic diagrams of optical systems: (a) Foucault imaging mode, and (b) small angle diffraction mode.

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lens is switched off in order to obtain a field-free region around the specimen. The condenser lens is then used to control the electron beam such that after penetrating the specimen, it converges to the crossover on the plane where a selected area (SA) aperture is located. This SA aperture works as the angular selective aperture of the Foucault imaging method. The SA aperture and the crossover with diffraction/deflection patterns are imaged by the first image-forming lens which is set just below the objective lens (see Fig. 1(b)). When the magnetic domain structure in the specimen is observed, the focal length of the first image-forming lens is elongated, and the specimen image is focused on the image plane which is the object plane of the successive optical system (see Fig. 1(a)). The irradiation area and the convergent angle of the electron beam on the specimen are adjusted via the condenser optical system, which includes a condenser aperture and a specialized aperture for scanning transmission electron microscope (STEM) if the microscope has a STEM mode. Although the originally installed objective aperture is not exactly located at the specimen plane, distance between the objective aperture and the specimen plane is shorter than the focal length of the first image-forming lens. Then the objective aperture is possible to be used as the area selective aperture.

In order to eliminate the diffracted/deflected electron beams that are unnecessary for the Foucault mode, the SA aperture is set at the optimum position as a normal angular selective aperture. The proposed optical system is appropriate for this purpose because it is easy to obtain long camera lengths in diffraction mode. The directly propagated diffraction pattern of 100–200 mm camera length is composed on the SA aperture plane. Furthermore, a magnified diffraction camera length of over 5000 m is easy to achieve by using the successive image-forming lenses. The proposed optical system is also advantageous for experiments related to small angle diffraction (Fig. 1(b)). Switching between the Foucault mode and the small angle diffraction mode is done by controlling the first image-forming lens, shown in Fig. 1.

The optical system shown in Fig. 1 was constructed in the commercial high-resolution electron microscope, HF-3300 (300-kV acceleration voltage and field emission gun) by Hitachi High-Technologies Corp. without any modification to the optical system (i.e., no magnetic-field shielding lens and no mini-lens were installed). The specimen was set at the normal position after switching off the objective lens. The microscope HF-3300 which has two intermediate lenses and two projection lenses gave a maximum magnification factor of 9200 and a maximum camera length of 9700 m under the condition of the objective lens being turned off. Images and diffraction patterns were recorded on a charge-coupled device (CCD) camera (US1000 of Gatan Co.). It can be estimated that if an imaging-type post column energy filter, for example, GIF by Gatan Co., is equipped to the microscope, HF-3300, the magnification and the camera length would be increased about ten-times greater.

Confirmation of the Lorentz microscopy observation was demonstrated with 180° and 90°/180° domain structures of a manganite $La_{0.75}Sr_{0.25}MnO_3$ (LSMO). The LSMO is known to have unusual physical properties, such as a colossal magnetoresistance (CMR) and a metal-insulator transition. An approximately 100-nm-thick LSMO thin film of [111] orientation was prepared by conventional ion-thinning technique[9] and was then utilized to observe the magnetic domain structure of the ferromagnetic metallic state at room temperature.

Figure 2 shows the Fresnel images of the 180° domains of LSMO: (a) under-focused ($\Delta f = 1.6$ mm), (b) in-focus, and (c) over-focused ($\Delta f = -1.6$ mm) images. The in-focus and defocus conditions were controlled by the first image-forming lens. Crystalline twin boundaries for [110] orientation of rhombohedral structure of LSMO can be seen in all the panels in Fig. 2. Especially in the in-focus image in (b), twin boundaries are visible by the difference of Bragg diffraction between the crystalline domains. Ferromagnetic domain boundaries, such as domain walls, are visible as black and/or white lines in (a) and (c). It is clearly shown that the twin boundaries in (b) correspond to the position of the domain walls in (a) and (c). The ferromagnetic domain structure is correlated to the crystalline structure of LSMO.[9]

Figure 3(a) shows the small angle electron deflection pattern, which was recorded at a 300 m camera length, and

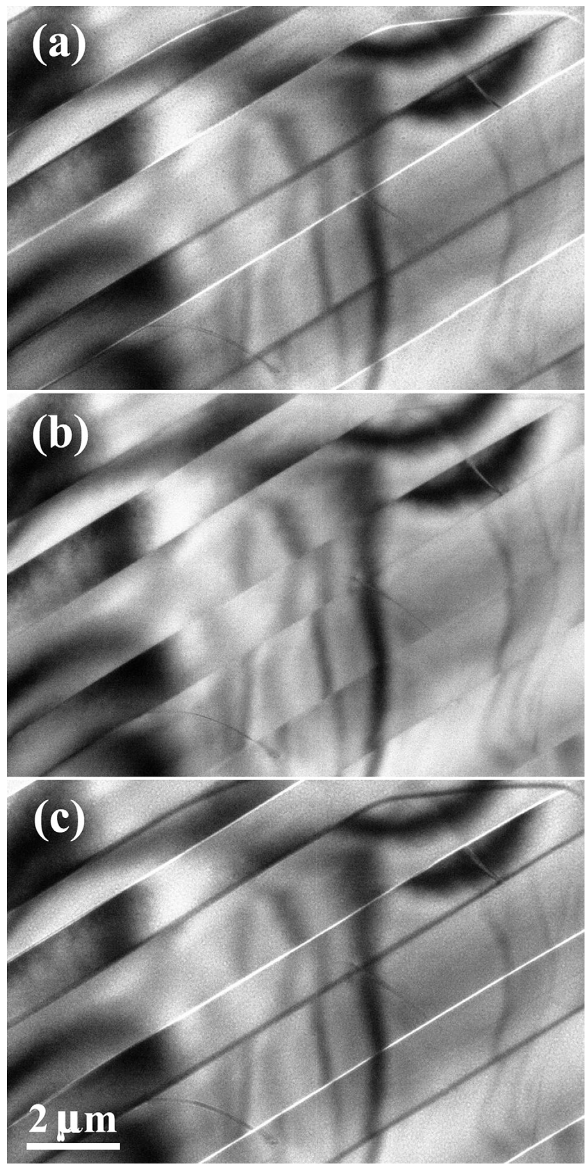

FIG. 2. Fresnel images of 180° domains of a manganite LSMO: (a) under-focus ($\Delta f = 1.6$ mm), (b) in-focus ($\Delta f = 0$ mm), and (c) over-focus ($\Delta f = -1.6$ mm).

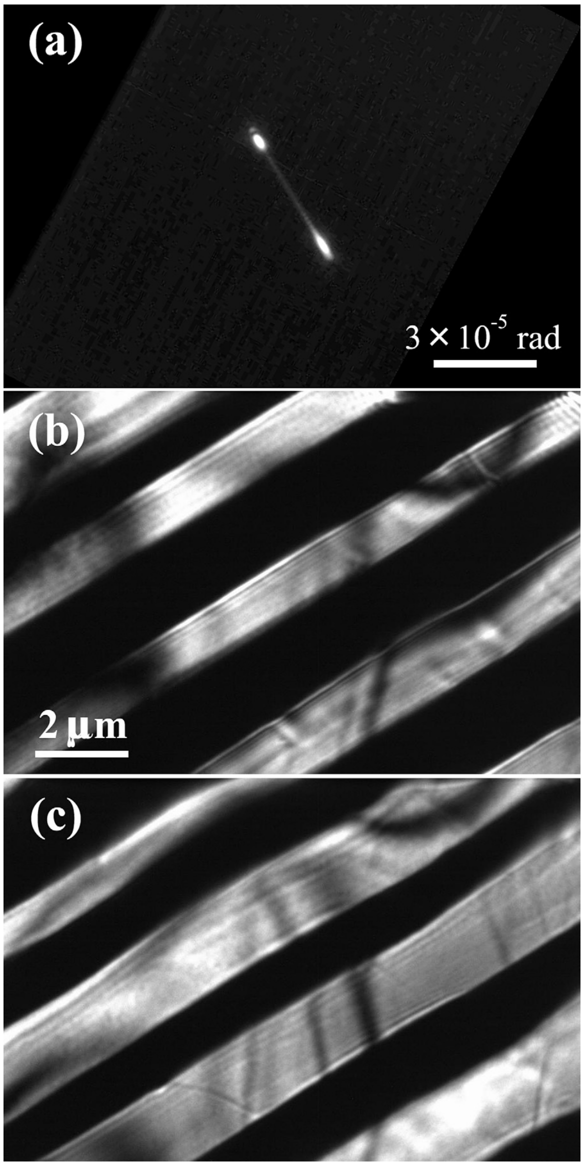

FIG. 3. (a) Small angle diffraction pattern, (b) Foucault image from left-hand spot in (a) of the same area as Fig. 2, and (c) Foucault image from right-hand spot in (a).

the Foucault images of 180° domains in (b) and (c), which are the same area as in Fig. 2. The two dispersed spots with the streak in Fig. 3(a) indicate that the material has 180° domains with the magnetic domain walls.[8,10] From the dispersed distance in the diffraction pattern, the magnetization of the LSMO can be evaluated at about 0.44 T at 300 K, when the specimen thickness is about 100 nm. The value is about half this at the low temperature (<100 K) magnetic state, but it is in good agreement with the evaluation result by superconducting quantum interference device magnetometer (SQUID).[11] It is considered that the continuously decreasing magnetic component across the domain wall produces weaker deflections resulting in the streak between the two spots.[12] The similar streak between the two spots is observed even at a single domain wall by using a small area selective aperture and an even thinner specimen. In the case of periodic domain-structure observation by using a larger area selective aperture, several Bragg diffraction spots corresponding to the periodicity are observed with the streak between the two main deflected spots.[8] So we consider the streak in Fig. 3(a) comes from Bloch walls, but further analysis is necessary before deciding the wall type.

A 90°/180° domain structure was observed as shown in Fig. 4(a). The Fresnel image in (a) shows black and white lines on the walls, and each magnetization direction in the domains is indicated by colored arrows. The 90° boundaries between the domains indicated by the red and green arrows (blue and purple arrows) and 180° domain boundaries between the red and blue arrows (green and purple arrows) seem to be of the same width. There might exist a difference of a factor of 1.4 between the boundary width of the 90° and the 180° domains; however, the resolution of the image was insufficient to observe it due to the low magnification factor of about 1000 and large defocus distance of −2.2 mm. On the other hand, the small angle diffraction with a camera length of 150 m from the 6-μm-diameter observation area shows four dispersed spots in Fig. 4(b). The deflection spots show us the magnetization direction in each of the domains directly. As the observation region is thinner than the region shown in Fig. 2 or 3, the dispersed distances among four spots in the deflection pattern is shorter when compared to the pattern in Fig. 3(a). The evaluated thickness is about 70 nm or less. The straight streak in the deflection pattern in Fig. 4(b) corresponds to the Bloch-type walls while the minor arc streak with weak contrast might correspond to azimuth rotation of the magnetization, such as Néel-type walls or Cross-tie type walls.[12] It remains for our future work to analyze the relation between the streak with weak contrast and the magnetization distribution. The precisely visualized contrast in diffraction/deflection patterns will also be extended to image in real space by using the diffraction microscopy technique.[13]

Each single dispersed deflection spot observed with a camera length of 150 m of Fig. 4(b) was selected by the SA aperture of 5 μm in diameter and the Foucault image from each spot was then observed as shown in Figs. 5(a)–5(d), respectively. The insets of Fig. 5 are corresponding to the deflection spot with the SA aperture. Foucault images have the advantage in showing both the domain structure and magnetization direction if a small angle diffraction can be observed within a single operation time. The proposed optical system made it possible by controlling the first image-forming lens only as shown in Fig. 1.

The 90°/180° ferromagnetic domains were colored one by one and reconstructed in one micrograph in Fig. 6, and the

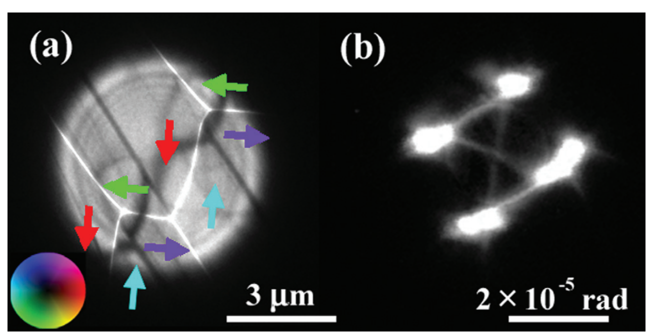

FIG. 4. (a) Fresnel image of −2.2 mm over-focused 90°/180° domains of LSMO, (b) small angle diffraction pattern of LSMO in (a). Arrows indicate magnetization direction of the domains.

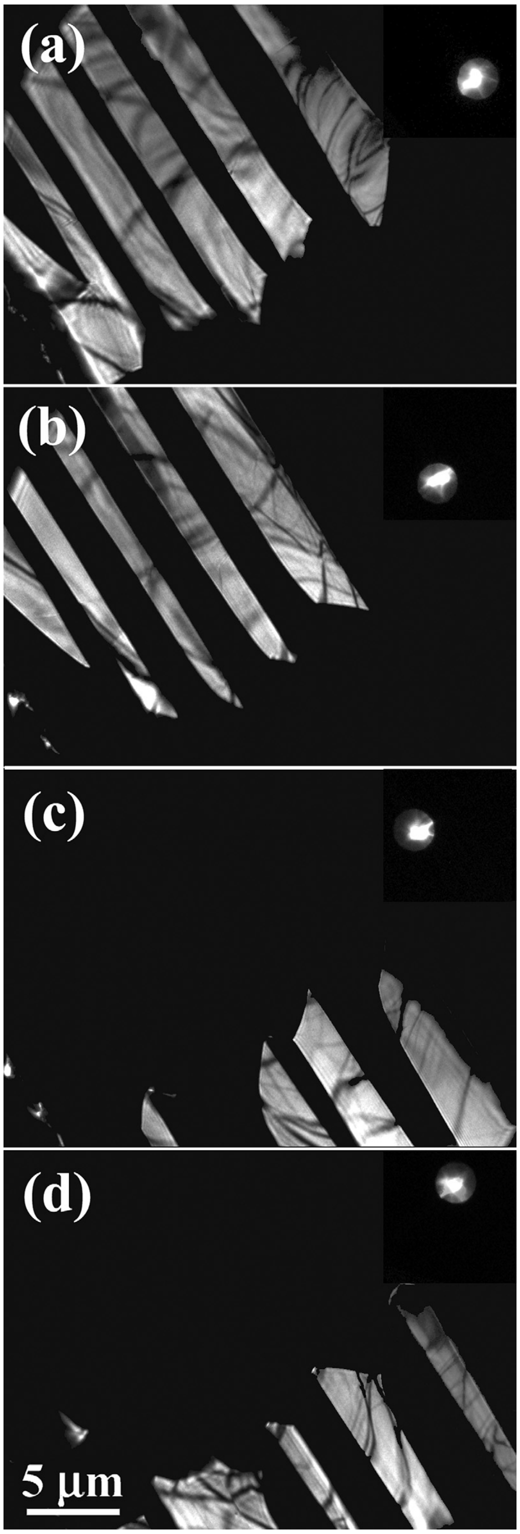

FIG. 5. Foucault images of LSMO from each single deflection spot in Fig. 4(b). Insets are selected single deflection spots shown with a SA aperture of 5 μm in diameter. Magnetic domains and spots in the insets correspond to each other.

inset of Fig. 6 also shows each corresponding deflection spot and the aperture size. The distribution of 90° domains is clearly visualized showing that the Foucault method has an advantage over Fersnel method in Fig. 4(a), i.e., the magnetization distribution among the domains is directly visualized. It should be noted that there is no boundary between the red and purple domains (green and blue domains), which might explain why there is no streak between these spots in the diffraction pattern in Fig. 4(b). Even if the domain boundaries between the red and purple domains (green and blue domains) might exist, the boundary length is too short to produce the streak in the diffraction/deflection pattern in Fig. 4(b).

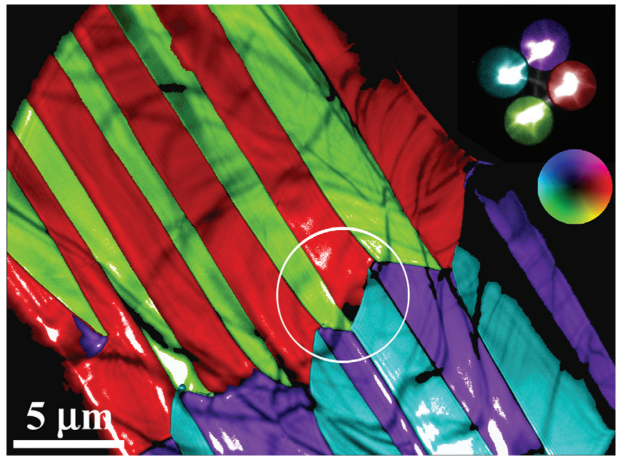

FIG. 6. Foucault image of LSMO reconstructed from Fig. 5. White circle shows the area in Fig. 4(a). Colors on domains correspond to each magnetization direction. Inset is a small angle diffraction corresponding to the colored Foucault image which is also reconstructed from insets of Fig. 5 and superimposed on diffraction of Fig. 4(b).

It is concluded that the proposed Foucault imaging method with switching off the objective lens can be practically performed within a non-dedicated high-resolution TEM. A condenser optical system was applied to the imaging system with the SA aperture being used for the angular selection of the deflected electron beam. The system was able to be utilized not only for the Foucault mode but also for the Fresnel mode. It is also shown that the optical system is advantageous for the observation of small angle diffraction by use of camera lengths of greater than 5000 m. We are confident that our proposed optical system will pave the way for another utilization of the conventional TEM.

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