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Lorentz microscopy methods for magnetic domain structure study

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Abstract. The capabilities of transmission electron microscope Hitachi HT7700 for magnetic domain structure (MDS) study of thin films using Lorentz transmission electron microscopy (LTEM) methods is discussed. Two methods of magnetic domain structure study (defocus method and aperture shift method, including low-angle electron diffraction (LAED)) in thin magnetic films were examined.

1. Introduction

Modern methods of transmission electron microscopy (TEM) can solve quite a wide range of research tasks in the field of solid state physics and materials science. Around TEM capabilities LTEM occupy a special place. Lorentz microscopy methods allow to obtain a direct MDS images of thin films magnetic materials. Nowadays, a number of researches describe both MDS study [1-6] and methodical reports on electron microscopes adaptation for LTEM [7-9]. Using specifically designed sample holders allows to *in situ* study of MDS evolution depending on temperature [1-3], external magnetic field [3-5], strain etc. The defocus method is usually used to MDS study, in which domain walls are observed as light and dark contrast on magnetic domains boundaries [10]. Nevertheless, there is the so-called 'aperture shift method'[10] (including LAED method), in which magnetic domains are directly observed instead the domain walls. The aperture shift method is the following: the electron beam transmission through the magnetized sample along the *y* axis, the electron beam is deflected by the Lorentz force along the *x* axis. The angle of electron beam deflection by the sample magnetic field is determined by the expression:

$$\varphi_x = \frac{4\pi\tau}{c} \left(\frac{e}{2Um} \right)^{\frac{1}{2}} M_y \quad (1)$$

where φ_x is the angle of electron beam deflection by Lorentz force along the *x* axis, τ is sample thickness, e is electron charge, m is electron mass, U is acceleration voltage along the *z* axis and M_y is the sample magnetization projection along the *y* axis.

Since the electron beam deflection has different directions in the adjacent magnetic domains, transmitted through the magnetized sample electron beam is split into several parts.



This split can be directly observed on the LAED patterns for both direct and diffracted beams. Aperture can close one of the split beams. Alternate domains look like light and dark areas. The dark domains correspond to the beam which is closed by aperture. Both methods in detail and the theory of the magnetic contrast origin are described in [10,11].

In this report we describe capabilities of conventional TEM Hitachi HT7700 (acceleration voltage up to 120 kV) for Lorentz microscopy methods (defocusing and aperture shift).

2. Methods

MDS images and LAED patterns were obtained by only changing of microscope lens currents. The major requirement for performing LTEM is external magnetic fields absence in the sample region. Therefore, the first stage is to close the current supply to the objective lens, and use the objective mini-lens instead. The objective mini-lens magnetic field does not influence on the sample magnetization [7]. Incident electron beam must be as possible more parallel for achieving clear MDS images in LTEM mode, thus condenser lens system is defocused for efficiently producing a point source at a large distance [10].

The second stage is achieving maximum possible magnification by maximum increase the current of projection lenses while maintaining sufficient brightness to obtain high-quality MDS images. The optimal combination of "magnification degree - brightness" is achieved in 'LOW MAG' mode and consist about 2kX (at 100 kV acceleration voltage) for Hitachi HT7700. The domain wall width image is about 100 nm in these conditions. It is much higher than the known magneto-optical MDS study methods. Intermediate lenses are used to focus MDS image.

The third step is adjusting electron beam and sample image on the optical axis of the microscope by deflecting coils of intermediate lens and column. Intermediate lens deflecting coils shift the sample image in X-Y plane, objective mini-lens rotate image around z axial. This step is necessary to overlap LTEM and TEM images that to study correlations between domain walls and lattice defects location in thin magnetic foils or epitaxial films. As a result, magnetic contrast [10] can be observed with magnification from 100 to 2kX.

Observation LAED patterns also occurs when the current supply of the objective lens is closed. Splitting diffraction peaks (satellites) expedient to study on central undiffracted beam because it has the greatest intensity than diffracted beams intensity. Satellites will be observed only if the selected area contains at least two adjacent magnetic domains. Satellites are quite small (about 10^{-4} rad) depends mainly on the magnetization magnitude and film thickness (at the same accelerating voltage). Effective camera length must be increased as far as possible (about 20m) by adjusting projection lens that satellites are observed. Shifting the aperture can close one of two split beams. Then the alternate magnetic domains look like light and dark areas, at that, the dark domains correspond to the beam which is closed by aperture. Magnetic domain images thus are similar dark-field TEM images. Objective aperture used as selective aperture but selective aperture used to close one of split diffraction peaks.

The advantage of this method lies in obtaining images of magnetic domains and crystalline structure in a precise focus. The aperture shift method allows study as magnetic domains as focused lattice defects at the same time.

3. Materials

Fe and Ni magnetic thin films were chosen for Lorentz microscopy methods study. Starting materials (Fe and Ni) purity was better than 99.95%. Fe thin films were obtained by the thermal deposition at a of 300 °C onto NaCl (001) substrate in a vacuum at a residual pressure of 10^{-6} Torr. Ni thin films were obtained by magnetron sputtering at room temperature onto NaCl (001) substrate. The base chamber pressure and total sputtering pressure were 10^{-6} Torr

and 6×10^{-3} Torr respectively. Fe and Ni film thickness was about 30 nm. Then films were separated from the substrates in distilled water and deposited onto the supporting copper TEM grid. Magnetic domain structures were studied at 100 kV accelerating voltage.

4. Results and discussion

4.1. Defocus method

Fig. 1 shows MDS (domain walls) observation scheme by defocus method [10]. The electron beam is deflected at the opposite directions with divergence angle 2ϕ (see Eq. 1) in adjacent magnetic domains. Intensity excess is observed in region 1'', intensity lack is observed in region 2'', when sample image is observed in A'' - B'' plane (under-focus condition). Reverse contrast (region 1' is intensity lack, region 2' is intensity excess) is observed in A' - B' plane (over-focus condition). The contrast is alternating light and dark bands onto magnetic domain boundaries (so-called domain walls). Domain wall contrast disappears at in-focus condition (A - B plane). It should be noted that the width of the domain wall image is dependent not only on the defocus distance, but also on the film thickness and the magnetization distribution within the domain wall [10].

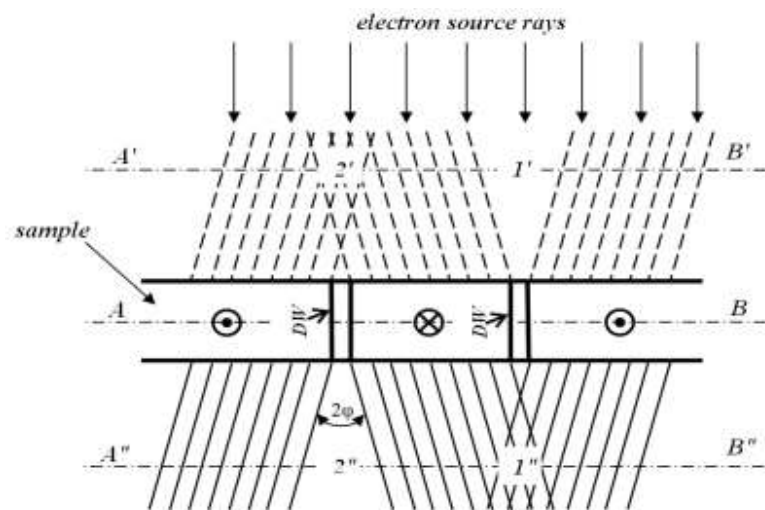


Figure 1. The Observation scheme of magnetic domains by defocus method (DW - domain wall)

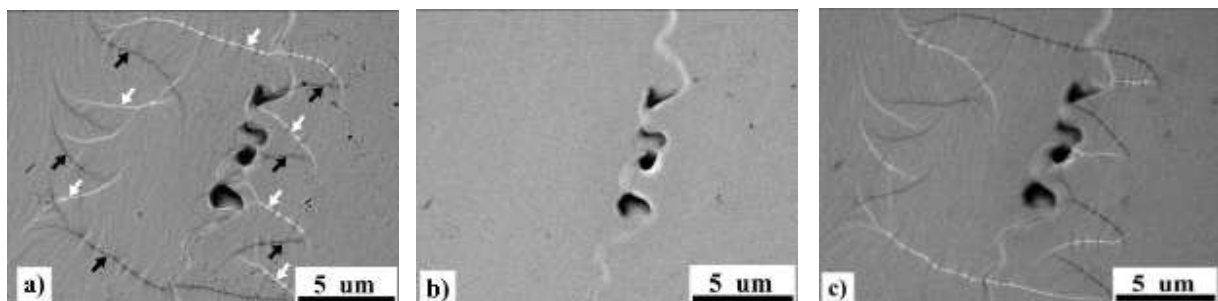


Figure 2. Magnetic domain images of Ni thin film was obtained at different observation conditions: a - over-focus, b - in-focus and c - under-focus.

Fig. 2 shows magnetic domain images of Ni thin film at over-focus (A' - B' plane (see fig. 1)), in-focus (A - B plane) and under-focus (A'' - B'' plane) conditions.

White and black arrows (see. Fig. 2a) show magnetic domain boundaries, where excess (collecting domain walls) and lack (divergent domain walls) of intensity are formed respectively. Figure 2b shows that magnetic contrast absent at in-focus condition. Figure 2c shows the same area image of Ni thin film with a reverse magnetic contrast (A'' - B'' plane fig. 1).

Fig. 3 shows classic example of vortex magnetic structure formation in Fe thin film upon objective lens external magnetic field application. External magnetic field is perpendicular to the film plane. The "fibration" or magnetization ripples is clearly visible besides the main domain walls. "Fibration" is characterized for magnetic films having magnetic anisotropy dispersion, including the normal direction to the film plane [12]. Red arrows indicate the magnetic poles - the areas in which the magnetization vectors perpendicular to the sample plane but opposite on direction.

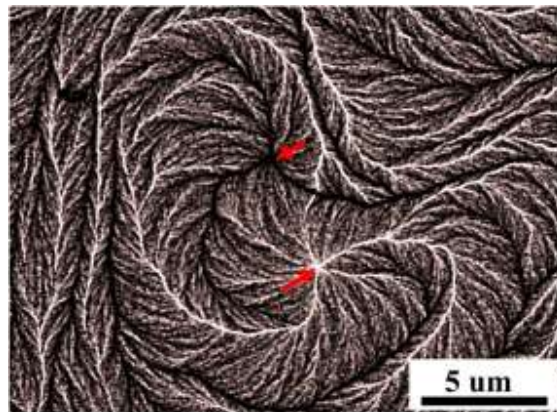


Figure 3. The vortex magnetic structure image of Fe thin film (under-focus condition).

The main advantage of defocus method is the capability to determine the local magnetization vector direction in the film plane by the "fibration" direction. However, this method does not allow to observe magnetic contrast and focused lattice defects image at the same time.

4.2. Aperture shift method

Fig. 4 shows MDS observation scheme by aperture shift method. Since current supply of main objective lens is switched off, diffraction image is formed in selective aperture region. Selective aperture used to isolate one of the split LAED peaks. This is analogically operation with objective lens aperture in TEM mode. The objective lens aperture used to select the sample region, which corresponds to the LAED pattern. Minimum aperture size of objective lens Hitachi HT7700 is 10 μm in diameter. Consequently, the sample area size corresponding LAED is about 10 μm .

Fig. 5a shows magnetic domain image of Fe thin film obtained by defocus method. LAED pattern (insert of Fig. 5a) allow to determine magnetization direction of adjacent magnetic domains, if zero diffraction peak corresponding un-split reflex (e.g. LAED pattern was obtained from blank area) was superposed with LAED pattern of adjacent magnetic domains.

Straight-line tension bar between double diffraction peaks mean that domain walls are Bloch walls. Tension bar have arcuate shape if domain walls are Néel walls.

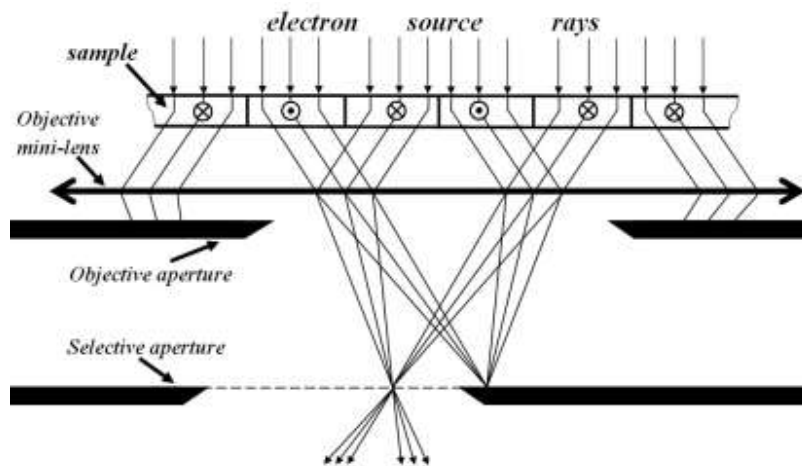


Figure 4. The Observation scheme of magnetic domains by aperture shift method.

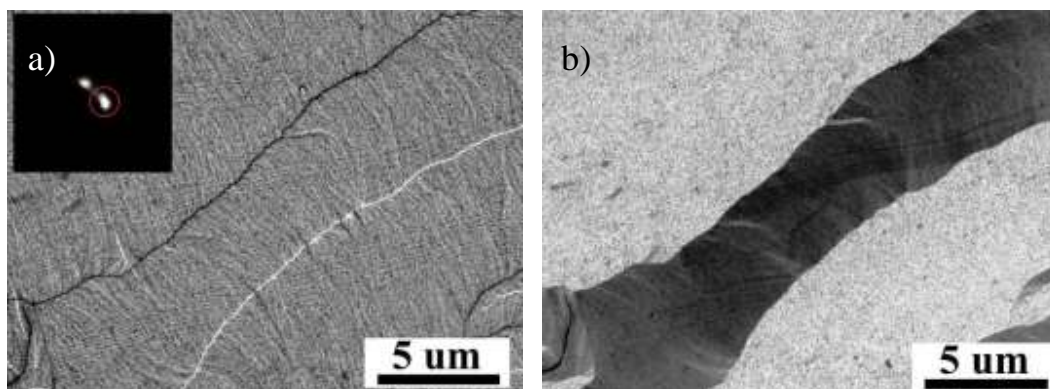


Figure 5. Magnetic domain images of Fe thin film was obtained by (a) defocus method (The insert shows the corresponding LAED pattern) and (b) aperture shift method.

However, Neel walls are formed at low film thickness (about 10 nm), therefore deviations are quite small to determine the domain wall type by the tension bar shape. For 180-degree magnetic domains value of local film magnetization possible to determine by distance between double diffraction peaks if the film thickness is known and vice versa [10]. Fig. 5b shows image of the same film area obtained by aperture shift method. Domain corresponding to the closed beam (circled in red on insert of Fig. 5a) has become dark. Magnetic inhomogeneity are visible inside dark domain. In addition there is an inversion in the phase banded contrast, the nature of which requires further study.

Fig. 6 shows magnetic domain images of one and the same area of Ni thin film. Fig. 6a is magnetic domain image obtained by defocus method, the inset shows the corresponding

LAED pattern. The distance between the split diffraction peaks of Ni thin film is 3.5 times less than Fe thin film which is consistent with the published data on the magnetization measurement for Fe and Ni [10, 11]. Fig. 6b and fig. 6c are magnetic domain images obtained by aperture shift method and correspond to the closed upper and lower diffraction peaks (see insert of fig. 6a), respectively.

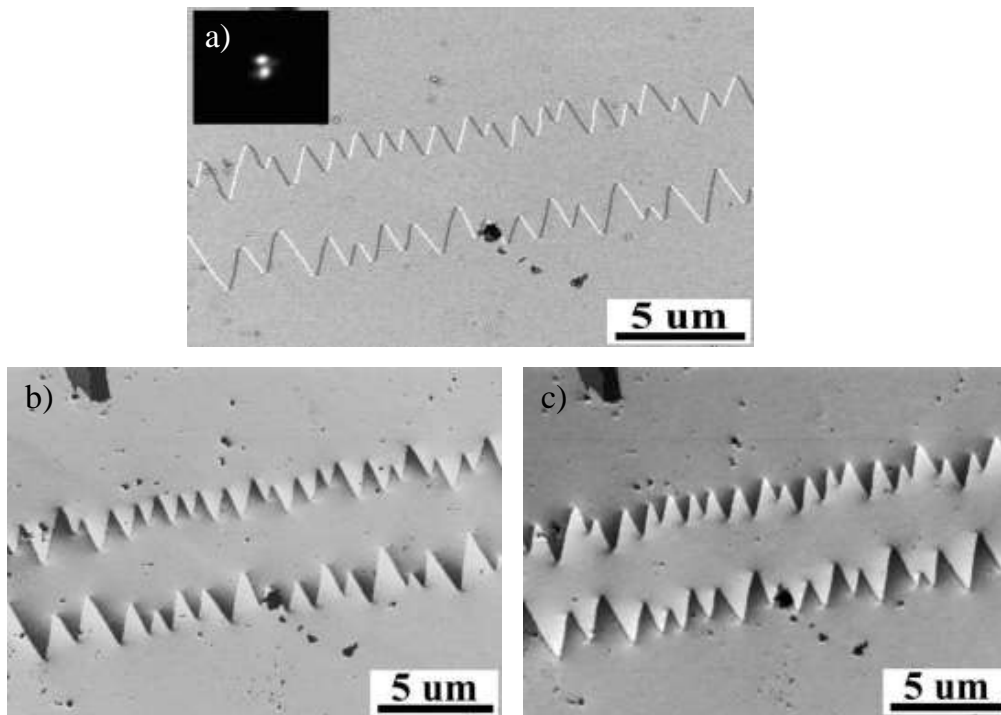


Figure 6. MDS images of Ni thin film obtained by (a) defocus method (the insert shows the corresponding LAED pattern) and (b, c) aperture shift method.

LAED method is most preferred to determine the magnetization vector direction in thin foils and epitaxial films (due to lack of "fibration" which characterized for polycrystalline and amorphous films).

5. Conclusion

We demonstrated capabilities of Hitachi HT7700 for MDS study of thin films. The combination of defocus, LAED and aperture shift methods can provide full information not only about the size and shape of the magnetic domains as well as the local distribution of the magnetization vector direction in the film plane. The features of LAED method can establish the correlation between domain walls and crystal lattice defects.

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