Characterization of the JEM-2100F-LM TEM for Electron Holography and Magnetic Imaging

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The holography performance and magnetic imaging capabilities of the JEM-2100F-LM electron microscope are reported here. Measurements of the fringe spacing, contrast, and hologram width obtained as a function of applied biprism voltage are presented. Measurements of the spherical aberration coefficient Cs = (108.7 ± 2.2) mm and minimum achievable focal step Δf = (87.6 ± 1.4) mm for the specially designed objective lens of this microscope are also presented. We characterize the magnetic field present in the sample region of the JEM-2100F-LM and find a remnant field about 4 Oe under normal operating conditions. Additionally, we characterize the objective lens remnant field along two orthogonal directions, the spatial characteristics of the field profile during specimen entry into the column, and the field conditions at the sample region as a function of objective lens excitation. Finally, we provide experimental holography results illustrating some of the more unique capabilities of the microscope.

Introduction

Several experimental techniques (each having distinct advantages and disadvantages, or applicability) are available to study magnetic structure and properties of materials [1, 2]. As the characteristic size in device technologies utilizing magnetic phenomena decreases, quantitative magnetic materials research with ever increasing spatial resolution and sensitivity becomes critical. For example, understanding reversal dynamics, the effect of neighboring magnetic elements, or the fine details of magnetic structures is crucial to continued technological development, and methods capable of quantitative magnetization measurement with high sensitivity and spatial resolution are required.

Of the available techniques to carry out quantitative magnetization measurement, off-axis electron holography in a transmission electron microscope (TEM) [3, 4] has significant potential with regard to spatial resolution and sensitivity compared to many other techniques. By recovering the phase shift of electrons passing through the sample, a direct relation to the electrostatic and magnetostatic potentials associated with the sample is obtained [5]. A critical aspect of holography is to characterize the fringe spacing and contrast that can be obtained with the holography setup for a given instrument. The fringe spacing is directly related to the spatial resolution that can be obtained [6], while the fringe contrast directly determines the phase sensitivity of the experiment [7]. Along with the width of the interference patch, these parameters specify the limits of what features are meaningful in the recovered electron phase shift.

In studies of magnetic materials, electron holography is certainly not the only useful TEM technique. Indeed, important information of the magnetic structure and domain configuration down to the nanometer scale can be obtained through Lorentz and Foucault modes of operation [8, 9]. In situ magnetization dynamics can also be studied in the TEM, for example, with a specialized sample holder capable of applying a small field in the plane of the specimen [10-13]. Typically, the main objective lens of the microscope is switched off during these studies, and imaging is performed with the objective mini-lens, similar to the low-mag-mode in conventional TEM imaging. The reason for this is to avoid penetrating the sample with the strong field ~30 kOe (for a 300kV high-resolution TEM) of the main objective lens. Nevertheless, the remnant field from the objective, even while switched off, can produce fields at the sample region on the order of a few hundred Oersted. Certainly studies have exploited this fact to carry out in situ magnetization experiments by tilting the sample in this remnant field [14], however, one needs to know the field in order to calibrate the “applied field”.

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The JEM-2100F-LM at Brookhaven National Laboratory, Fig. 1(a), is specially designed and dedicated to quantitative magnetic imaging and holography, and is the first of its kind in the United States. The instrument is based on the JEM-2010F, a 200 kV TEM with a thermally assisted field-emission source. The unique feature of the JEM-2100F-LM is its long focal length (17 mm) objective lens shown in Fig. 1(b). Since the OL is weakly excited and the specimen is located above the pole piece gap, the magnetic field at the sample area is significantly reduced to a few Oe when the lens is on (3-4 orders of magnitude smaller than a standard objective lens). A similar prototype objective lens was first installed and tested on a JEM-3000F microscope at Prof. Shindo’s lab at Tohoku University in Japan. Although the pole-piece gap of our instrument is about 8 mm, the tilt angle of the sample holder is still limited to about 15° due to the narrow entrance of the pole-piece to reduce the leakage field. Since the objective lens is weakly excited, the lattice resolution of the microscope in its specification is 0.7 nm, although we have obtained test results better than 0.5 nm. The JEOL specification for the spherical aberration Cs of the machine is 95 mm, while for the chromatic aberration Cc is 16 mm. The other performance of the instrument is quite the same as a standard JEM-2010F, including a wide range of diffraction capabilities. This is particularly important for magnetic domain imaging, where we can easily observe the splitting of the central beam due to the variation in local magnetization, which is extremely useful for quantitative Foucault imaging. Our instrument is equipped with a 2 k × 2 k 16-bit CCD camera (14 µm pixel size) and a JEOL biprism (0.6 µm diameter, 180° rotation) for electron holography.

In the following section we present measurements of the fringe spacing, hologram width and fringe contrast as a function of biprism voltage for the HOLO-M mode setup of our JEM-2100F-LM. In the section of “Lorentz Imaging Characteristics”, we present measurements of the objective lens spherical aberration and the minimum focal step that can be obtained with the microscope. In addition to the holography measurements, we describe in the section of “Objective Lens Field Measurements” a relatively simple modification to adopt a Hall probe to a standard analytic TEM holder that allows us to easily and accurately measure the OL remnant field in our JEM-2100F-LM. We characterize the field along two orthogonal directions, one perpendicular to the specimen plane and the other along the axis of the holder, and the spatial characteristics of the field profile for all stages of specimen entry into the column. We also measure the OL field in the sample region as a function of operating OL voltage, which is important for in situ magnetization experiments.

Holography Characteristics

In order to characterize the holography capabilities of the JEM-2100F-LM, the fringe-spacing, contrast, and hologram width were measured as a function of biprism voltage [15]. Here, “hologram width” refers to the FWHM of the interference patch, which differs from the well-defined concept of “interference distance” [16]. The results are shown in Fig. 2 where data were collected in the HOLO-M mode of the microscope, and lens program determined and installed by the factory. The results in Fig. 2 show the basic capabilities of the microscope where moderate improvement to the contrast might be expected with special attention paid to the room environment. All data were acquired using the now standard procedure of stretching the illumination along the perpendicular to the biprism [6].

For measurement of fringe spacing and contrast, holograms were recorded at a nominal 48 kX magnification, which corresponds to a calibrated pixel size of 0.22 nm on the 2 k × 2 k CCD camera. This corresponds to a sampling of about 6 pixels per fringe for the finest spacing. In this case, the transfer function of the CCD camera is likely to have an adverse influence on the measured contrast, although the detection properties of the CCD camera have not been measured. Regardless, Fig. 2(a) and (c) show that fringe spacing less than 1.5 nm can be easily achieved with a moderate contrast around 10-15%. The data shown in Fig. 2 were recorded with 2 seconds exposure times during normal working hours. Longer exposures often resulted in significantly reduced contrast, and it is clear that the principle limitations to the holography performance are currently due to vibration and the room environment in general. This point, although far from being made for...
the first time, accentuates the importance of the laboratory environment in carrying out the most demanding experiments.

Figure 2(b) shows the measured hologram width as a function of biprism voltage. With the highest voltage where the fringe contrast is still reasonable, the hologram width is easily 1 μm. While a large field-of-view is potentially desirable, especially for magnetic studies where fringing fields can be quite long ranged, in this case the full 1 μm field-of-view cannot be completely utilized. This is due to limitations of the CCD camera. That is to say, a minimum magnification is needed to sample the fringes with enough pixels and simultaneously, enough total pixels on the camera are needed to cover the whole hologram. In our case, the magnification of the microscope is sufficient to sample the fringes with about 6 pixels per fringe, but then the field of the camera is only about 500 nm. This translates to a less-than- optimum setup of the recording conditions for biprism voltages larger than about 75 V for our microscope. Installation of a 4 k × 4 k CCD camera could provide better terms of sampling, utilizing the full field of the hologram.

Finally, consider the regime where the hologram width is roughly 1.5 times the CCD field-of-view for the highest available magnification. This corresponds to a situation where the Fresnel fringes from the biprism edge are largely outside of the recording region, and represents a regime where the sampling is roughly optimum given the constraints of the CCD size and holography geometry. For our setup, this corresponds to a biprism voltage around 70-75 V and, consequently, fringe spacing around 1.7 nm. Under these conditions, 5 nm spatial resolution may be easily obtained with roughly a 500 nm field-of-view. Further, the fringe contrast is 20-25%, which provides suitable phase sensitivity in general. For this microscope, these conditions would be considered characteristic of its median performance where higher resolution, higher sensitivity, or larger field-of-view cannot all be simultaneously obtained with any sort of ease. The interplay between spatial resolution and sensitivity (embodied by the holographic fringe contrast) is summarized in Fig. 2(d) where the fringe contrast (sensitivity) is plotted vs. the fringe spacing (resolution) for our JEM-2100F-LM. For comparison, similar measurements made on our JEM-3000F (a 300 kV high-resolution TEM) in Lorentz mode where the main OL is turned off and an objective mini-lens is used for image formation, are also shown in the figure. There is an order of magnitude improvement in holography performance between the two machines.

Lorentz Imaging Characteristics

Although the holography performance of the JEM-2100F-LM is generally satisfactory for a wide range of potential applications, one desires complementary capabilities for quantitative magnetic imaging. In particular, one may use through-focus techniques [17,18] to obtain quantitative magnetic information, but it is necessary to know the focal step between recorded images. Additionally, knowledge of the spherical aberration Cs of the imaging lens is required, either directly in the case of through-focus reconstruction, or indirectly for proper interpretation of results in the case of transport-of-intensity methods [19]. Knowledge of defocus and Cs is also important for holography applications involving high resolution since the image wave is reconstructed rather than the object wave. For this reason, we have measured Cs and the minimum focal step δf for our microscope.

A focal series of images were recorded at 200 kx magnification of holey carbon covering the focal range from ~36 to +36 focal steps around zero defocus. For each image, numerical diffractograms were calculated, and all visible ring positions were measured. Between 9 and 35 ring measurements were obtained per image depending on the focus. Following well-documented methods, Cs and the absolute defocus for each image were obtained [8,20-21]. Figure 3(a) shows the results of Cs measurement for each of the 73 images recorded in the series. During calculation of Cs for each image, which requires a linear fit to the ring positions, the R² value of the fit was noted and used as a weight when calculating the overall average Cs value. The result, as shown in Fig. 3(a) by the dashed line, is Cs = (108.7 ± 2.2) mm. This compares reasonably well with the value of 95 mm cited by the manufacturer JEOL.

Figure 3(b) shows results of the absolute focus obtained for each of the 73 through-focus images. The focus is linear over the range of ±3 μm with a focal step δf = (87.6 ± 1.4) nm. For quantitative Lorentz imaging, these characteristics of the focal behavior are satisfactory. The measured value of Cs = 109 mm gives a point-to-point resolution r_wid = 0.667 μm Cs1/2 =0.77 nm [21]. In terms of holography applica-
tions, this does not impose any limitations for the HOLO-M mode since the minimum fringe spacing that can be achieved only approaches 1 nm and, therefore, a resolution larger than 3 nm in the reconstructed wave.

As an example of the magnetic imaging capabilities of the JEM-2100F-LM, Figs. 4(a) and (b) are Lorentz images of magnetic domains in square elements of patterned Permalloy films grown by UHV e-beam deposition on SiN substrate using shadow-mask techniques. A line-scan of the image intensity, inset of Fig. 4(a), across the divergent domain-wall (white line features in the image) clearly show oscillations in the Fresnel contrast, indicating the suitability of the image formation capabilities for possible quantitative studies. Figure 4(b) shows a closure domain structure in a similar Permalloy element where splitting of the transmitted beam due to the magnetic domain configuration is clearly seen in the diffraction pattern, Fig. 4(d). Figure 4(c) indicates the area of the sample selected by the diffraction aperture, and this example illustrates some of the possible diffraction capabilities of the microscope. Combined with the Lorentz imaging capabilities and holography performance, several experimental techniques are available with the JEM-2100F-LM suitable for magnetic studies.

**Objective Lens Field Measurements**

The TEM holder that we adapted for the Hall probe modification was the Gatan half-finished heating holder without heating elements (Model 646), which is commercially available, and is a high-resolution double-tilt TEM holder (for a 2 mm pole-piece gap) with electrical through-put providing four terminals near the sample position for electrical contacts. The carriage that normally holds the TEM sample was removed for our modification to allow additional space to mount the Hall sensor accurately at the normal specimen position within the holder. Figure 5 is a picture of our modified holder with the Hall sensor mounted at the sample position; additional details can be found in reference [22].

The objective lens of the JEM-2100F-LM has a long focal length that allows the sample to be situated above the pole-piece gap, in a “field-free” region, during normal operation. The manufacturer provides specification that the remnant field at the sample is ≤ 8 Oe. The pole-piece for this microscope is designed such that the strong field from the lens is routed around the region where the sample sits by metal shielding. During insertion of the sample into the TEM column, the sample passes through an 8-mm gap in the shielding and, therefore, may be subjected to significant magneticizing effects. This is one of the features we wished to investigate with our adapted Hall probe, in addition to the field characteristics at the sample region during normal operation.

**Measurements perpendicular to specimen plane**

The first measurements were made with the Hall probe oriented in the modified holder so as to measure the field along the optic axis of the TEM, i.e., perpendicular to specimen plane. This is expected to be the principle orientation of the OL remnant field. We measured the field along various positions of sample insertion into the column with the OL on and as well as off, and Fig. 6 shows the results of our measurements.

As evident in the figure, there is a significant difference for the 2100F-LM depending upon whether the OL is switched on or off. When the lens is excited to normal operating conditions, the sample is subjected to about 350 Oe field during insertion, presumably as it traverses the gap in the upper pole piece of the lens. With the OL off, however, only the remnant field of the lens is directed along the shielding and the sample is subjected to significantly reduced field about 18 Oe. This indicates that normal practice for loading a magnetic sample in this microscope should involve switching off the OL prior to insertion. With the sample fully inserted into the column, the measured field is 4 Oe when the OL is on, and 0 Oe when the OL is off. In this case, the manufacturer’s specification of less than 8 Oe residual field at the sample position is confirmed.

We performed additional measurements with the Hall probe situated at the normal working position of the holder in the TEM column, and excited every lens in the microscope (except the OL, which we describe below) to their fullest extent in order to determine whether nearby lenses affected the field at the sample region. We did not measure any difference in the field at the probe position, regardless of lens excitation. Nor did we measure any difference as a function of sample position over the full extent of stage translation. As a function of sample tilt we measured a slight reduc-
tion of the field (from 4 Oe to 3.8 Oe) for high tilt above 20°. This is consistent with the remnant field aligned mainly along the optic axis of the microscope.

In terms of OL excitation, we indeed measure a field dependence on the lens potential used to excite the lens. Figure 7 shows results of measurements made as a function of lens excitation. The standard lens excitation used for normal operation of the microscope corresponds to a lens potential of 0.76 V as read from the lens-data display of the TEM. With moderate lens excitation, we measure a roughly linear increase in the field generated at the sample area. While these excited OL conditions are far enough from normal operating conditions that one does not have to worry about generating above 4 Oe on the sample during conventional imaging and diffraction, it does allow for the possibility to carry out in situ magnetization experiments of the sort described in the introduction.

**Measurements in the specimen plane**

By rotating 90° the aluminum support holding the Hall probe, we were additionally able to measure the component of remnant OL field along the axis of the holder, i.e., one of the directions in the plane of the specimen. Figure 8 summarizes the measurements made, and there is again a significant difference depending whether the OL is on or off. As seen in the figure, when the OL is switched off, the measurement is about 18 Oe and switches from an initial negative polarity (directed away from the column center) when the sample insertion begins, to a positive polarity (directed toward the column center) when the sample passes through the mu-metal shielding. Still, the field component is relatively small, although the same magnitude as the perpendicular component. This further supports the conclusion that the OL lens should be turned off prior to sample insertion in order to minimize magnetizing effects from the OL field.

With the OL turned on, the field component along the holder axis is quite large on average, switching polarity relative to the probe during the insertion process. The best fit to the right-most measurement (at 7 cm distance in Fig. 8) corresponds to the probe located in the loadlock of the specimen chamber, which explains why the measured field is not zero. Nevertheless, our results indicate that this in-plane component is just as significant as the out-of-plane component during sample insertion regardless of whether the OL lens is on or off, albeit the net field is much smaller with the lens turned off. When the sample is in the normal operating position, i.e., fully inserted, we measure about 1.6 Oe for the in-plane field component. Comparing this to the 4 Oe vertical component, it appears that the residual field on the sample cannot be considered essentially vertical, even if the main component is indeed along the optic axis.

**Holography Examples**

The unique capabilities of our JEM-2100F-LM allow us to investigate magnetic structures in comparatively low field, about 4 Oe vertical residual field compared to ~200 Oe in a conventional microscope when the objective lens is switched off. While the principle utility of the JEM-2100F-LM is for application to magnetic studies, we illustrate the comparatively high resolution and sensitivity of the holography capabilities of this microscope with two examples. The first example, while purely an electrostatic problem involving measurement of charged latex spheres, nicely illustrates nevertheless the sensitivity of the microscope.

**Figure 9** shows holography results from a 94 nm latex sphere dispersed on holey carbon substrate. The cosine map of the reconstructed phase, Fig. 9(a), is amplified 16× to show the fringing field associated with the charged sphere. The phase was reconstructed from a hologram recorded with 1.97 nm interference fringes and 18% contrast in the sample. A profile of the circularly averaged phase shift across the sphere is shown in Fig. 9(b), along with the calculated phase shift shown as a red line. The best-fit parameters of the calculated phase shift gives a mean inner potential of 5.4 V and 65 electrons of charge distributed on the surface of the latex sphere. In this experiment, a much smaller charge than 65 electrons could have been detected (at least by a factor of 2), but serves in any case to showcase the sensitivity of the microscope when combined with theoretical modeling and interpretation.

With the low-field characteristics of the specially designed pole-piece of the JEM-2100F-LM, combined with the capability to carry out holography experiments at comparatively high spatial resolution, we have initiated study of the structure of vortex cores in magnetic nanodisks. The vortex core is a nanometer-sized region of vertical magnetization that forms at the center of the vortex closure-domain, and is typical of nanodisks in a certain size range. While this phenomenon was experimentally observed using surface-sensitive spin polarized scanning tunneling microscopy [2], a great deal remains unknown, including the degree of extension of the vortex core as a function of the anisotropy of external parameters and disk size, as well as the transition of spin moments from in-plane to out-of-plane near the core due to quantum mechanic exchange.

Preliminary holography experiments involving measurement of the vortex core structure in thin Permalloy disks are summarized in Fig. 10. Samples consisted of 1 μm patterned Permalloy disks, about 20 nm thick, deposited on holey carbon substrate using shadow mask techniques by UHV e-beam deposition. Holography experiments were carried out with a biprism voltage of 110 V giving 1 nm interference fringes and about 5-10% contrast in the sample. In Fig. 10, the phase profile retrieved from holography (black line) is obtained after circular averaging about the core center, and is compared with a simulated profile where the core structure is neglected (red). In the inset, a more physical phase profile (blue) is matched against the data showing that near the core the signal drops due to the associated vertical component of the magnetization, m<sub>v</sub>. In modeling the vertical component of the vortex core we took the ansatz m<sub>v</sub> = [(1 - r<sup>2</sup>)<sup>2</sup>]<sup>-1/2</sup> where r is the distance from the core center. Best fit to the holography data gives r<sub>0</sub> = 28 nm and α = 7/3. The FWHM of m<sub>v</sub> with these parameters is 33 nm providing a characteristic size of the vortex core region in these particular Permalloy elements.
Summary and Conclusion

While we continue with experiments and analysis of vortex core structure, the simple examples described above, including charge measurement on latex spheres, illustrate the unique holography capabilities of the JEM-2100F-LM for both electrostatic and magnetostatic studies. In characterizing the holography performance of the JEM-2100F-LM, we have found it easily suited for magnetic studies requiring a large field-of-view (about 500 nm) while maintaining high spatial resolution (about 5 nm) in the reconstructed phase. Measurement of the spherical aberration coefficient of the specially designed objective lens, which agrees with the manufacturer’s claim, and calibration of the minimum focal step allows for the possibility for quantitative Fresnel imaging techniques, including through-focus techniques, that can supplement electron holography studies. During measurement of the objective lens field of the microscope we found that several hundred Oersted can be applied to the sample, if desired, which depends linearly on the moderate lens excitations we measured. Overall, the residual field on the sample due to the specially designed pole-piece is about 4 Oe under normal operating conditions, suitable for a wide range of studies involving magnetic materials. Combined with theoretical developments, such experimental capabilities will offer an opportunity to gain new essential scientific knowledge on technologically important magnetic structures at the nanoscale.

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