

TEM imaging and application of thin-film magnetic rings for phase plates

C J Edgcombe¹ and J C Loudon²

¹TFMM Group, Dept of Physics, University of Cambridge, CB3 0HE, UK

²Electron Microscopy Group, Dept of Materials Science, University of Cambridge, CB3 0FS, UK

email: cje1@cam.ac.uk

Abstract. The maximum out-of-plane field that some thin-film cobalt rings resisted while remaining in the vortex state was found experimentally to be lower than expected from simulation, possibly due to tilt. Analysis of a Zernike phase plate shows that the observed bright outlines around large objects result from the low-spatial-frequency limit of correction imposed by the beam hole. The maximum size of phase object that can be imaged accurately with a Zernike plate can be raised by increasing the effective focal length of the objective lens.

1. Introduction

The use of phase plates in transmission microscopy (TEM) offers the benefits of (1) increased contrast when the image is in focus, without contrast reversal over a wide range of spatial frequency, and (2) reduced electron dose to the specimen, of particular value for cryomicroscopy.

Of the various devices that have been built or proposed for phase plates, ferromagnetic rings are of interest because (1) the radial extent over which they intercept scattered electrons is expected to be smaller than for other plates; (2) all electrons scattered beyond the minimum effective angle experience the same phase shift. Previous work [1] demonstrated that the magnetization of thin-film rings of cobalt can be switched between two stable states, one of which has the continuous loop or vortex configuration of flux desirable for use as a phase plate in TEM. Further work on the fabrication of these rings is in progress. Here we report first some observations on the effect of axial (out-of-plane, OOP) field on rings in the vortex state, then some more general calculations on the imaging of extended objects by phase plates. These results show why bright outlines have been observed around some objects when phase plates are used, and suggest how to increase the size of objects that can be imaged without exciting these bright outlines.

2. Toleration of axial field in TEM

The most obvious location at which to place a phase plate is close to the back focal plane (BFP) of the objective lens. At this position, the axial field due to the objective lens may be as large as 2T in some TEMs, but appreciably smaller in others designed for biological applications. Simulations suggest that both circular and semicircular cobalt rings in the vortex state may tolerate an out-of-plane (OOP) field of ~1T. Thus it seemed possible that a ring of cobalt might be able to maintain the vortex state of magnetization in the presence of the axial field of the objective lens of a biological TEM.



Semicircular rings were deposited on SiN as described in [1]. The lift-off procedure succeeded in removing the centres of two of the rings, of which the narrower had width 30 nm. The rings were magnetized in the vortex state, inserted in the specimen position of a TEM and inspected by holography. Initially both were found to switch to the onion (or dipole) state when an OOP field as small as 0.1 T was applied. After remagnetisation in the vortex state, both were found to remain in that state when the OOP field was increased to 0.3T, but switched to the onion state when the field was raised to 0.5T (Fig. 1). This value of switching field was substantially lower than that expected from simulation. The reason for this low experimental switching field and its variability has not been

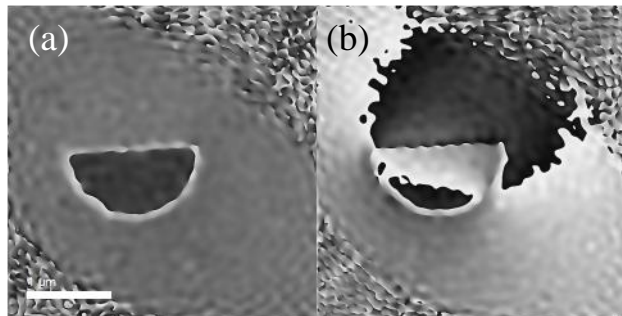


Fig. 1 Wrapped phase distributions (including jumps of 2π) near one Co ring after exposure to out-of-plane fields of (a) 0.3T and (b) 0.5T. Scale bar: 1 micrometre.

identified but may be related to initial tilt of the specimen on insertion in the column.

Even if the alignment of the magnetic ring in the region of the objective aperture is controlled sufficiently precisely for a ring to remain in the vortex state in an axial field of 1T, its sensitivity to OOP field is obviously inconvenient for use in practice. However, while a phase plate at the BFP is known to increase the contrast at focus, it has a further effect on imaging which suggests that modifying the TEM lens system may have advantages.

3. Effect of a phase plate on imaging

The type of Zernike phase plate that has been most widely reported (eg [2]) consists of a plain disc of material such as carbon, of controlled thickness, with a central hole to pass the direct beam. Images of biological specimens made with this type of plate are observed to show bright outlines or halos around certain features [3, 4]. Analysis of imaging has recently shown how these halos occur. It is necessary to consider the response not just to a single frequency but to the distribution of frequencies present in a typical object. In principle this can be done straightforwardly by Fourier transforming the object phase to find its spatial frequency distribution at the BFP, multiplying by the response of the phase plate and further transforming to find the resulting spatial distribution. In practice, for most objects it is difficult to do the second transform analytically for an arbitrary diameter of the central aperture of the phase plate. Some results of analysis are presented here for an object that produces a uniform phase shift within a circular disc; further details are given elsewhere [5].

An object wave with a small phase advance ($A\phi$) relative to free space is represented here approximately by

$$f \approx 1 + i A \phi(r_o, \theta_o)$$

where r_o and θ_o are transverse coordinates at the object and the propagation factor $\exp i(kz - \omega t)$ has been omitted. Here A is the amplitude of phase variation, assumed $\ll 1$. The plate is assumed to advance the phase of scattered wave components by α at spatial angular frequencies that are greater than a value q_0 . This q_0 is 2π times the quantity described elsewhere as the ‘cut-on’ frequency. Detailed analysis for an ideal lens shows [5] that for some typical forms of ϕ the image can be expressed as

$$g = 1 + i A \phi(r_i, \theta_i) - i (1 - e^{i\alpha}) A \eta(r_i, \theta_i, q_0)$$

where r_i and θ_i are transverse coordinates at the image. The intensity at the detector is then

$$|g|^2 = 1 - 2 A \eta \sin \alpha + O(A^2)$$

showing that where $(\eta \sin \alpha)$ is non-zero, the variation of image intensity from the mean is proportional to A . The ratio $\eta(r_i, \theta_i, q_0) / \phi(r_o, \theta_o)$ may vary over the image. At frequencies below

q_0 , the phase contrast transfer function is much less than unity, so the plate behaves as a high-pass filter.

The uniform phase shift produced by a circular disc of radius b is defined here without dependence on θ_0 as

$$\phi(r) = \begin{cases} 1, & 0 < r < b \\ 0, & b < r \end{cases}$$

and the result for η with unit magnification can be written with similar independence of θ_i as

$$\eta(r_i, q_0) = \phi(r_i) - B \int_0^{r_i/b} \frac{1}{(\rho^2 - 1)} [\rho J_0(\rho B) J_1(B) - J_0(B) J_1(\rho B)] d\rho$$

where $\rho = r_i / b$ and $B = q_0 b$. The resulting intensities are shown in figure 2 for object discs of unit amplitude and a range of values of B . The uniform object distributions are imaged with little overshoot when B is less than about 1. In Fig. 1(d) of [6] the image intensity was found to follow the object phase variation well when the cut-on periodicity was 50 nm, while there was appreciable overshoot for periodicity 25 nm. The corresponding values of B are estimated as 0.96 and 1.91.

As shown in [5], the magnitudes of steps at $r_i = b$ are always imaged fully but as B increases beyond 1, the low-frequency components are progressively lost from the image and the *mean* value at a step falls to the background value ($\eta = 0$). Since the unit range of the step is maintained, η then has to be $+0.5$ at radii just less than b and -0.5 just outside the step. Consideration of $|g|^2$ with $0 < \alpha < \pi$ shows that as η becomes more negative, the magnitude of g and the image intensity increase. Thus a bright halo or outline is produced just outside the boundary $r = b$, for objects with $B > \sim 1$.

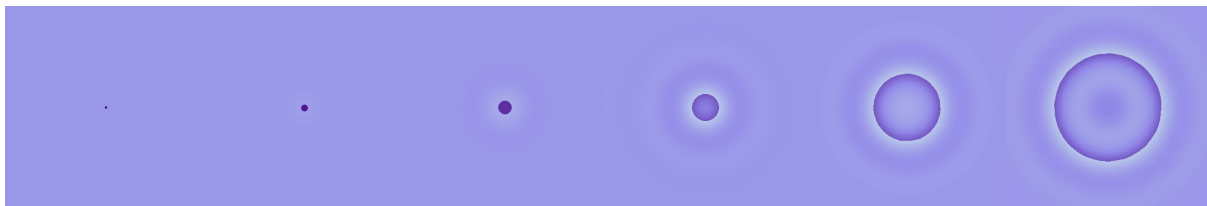


Figure 2. Image intensities produced by a $\pi/2$ phase plate with fixed q_0 for uniform disk objects with a range of diameters $2b$. Responses are shown for values of $B = q_0 b$ (increasing from the left) of 0.2, 0.5, 1, 2, 5 and 8.

On the axis, $\eta(0, q_0)$ is given by the Bessel function $J_0(B)$ and so the intensity on axis (and elsewhere) oscillates as B varies. This effect can be seen in Fig. 1(b) of [4] where the distribution of intensity in the body of phage T4 appears to correspond to the distribution of η for $B = 8$ in figure 2.

The minimum radius s_2 at which a simple Zernike plate can be effective is that of the hole needed to pass the direct beam. The corresponding minimum scattering angle at which phase will be corrected, θ_{\min} , is given approximately by geometry as

$$\theta_{\min} \approx s_2 / f$$

The same angle equals the ratio of transverse and axial propagation constants q_0 (at cut-on) and k , where $k = 2\pi / \lambda$ and λ is the wavelength of electrons in the beam. Hence the maximum diameter corresponding to the maximum B for accurate imaging is

$$2b_{\max} = 2B_{\max} / q_0 \approx B_{\max} \lambda f / \pi s_2$$

When $f = 3$ mm, $s_2 = 0.5$ μm and $\lambda = 3.7$ pm (100kV), then $\theta_{\min} = 0.167$ mrad, $q_0 = 0.283$ rad (nm) $^{-1}$ and with $B_{\max} = 1$, $2b_{\max} \sim 7$ nm. This b_{\max} is inversely proportional to θ_{\min} . To increase the size of object that can be imaged accurately, it will be necessary to reduce θ_{\min} by reducing s_2 or increasing f , the focal length of the objective lens.

4. Location of phase plate in TEM column

The most obvious location for a phase plate is the objective aperture, nominally at the BFP of the objective lens. This location in the conventional TEM has some disadvantages. The space available for a traverse mechanism, heating arrangements or thermal insulation for cryogenic specimens is very limited. If the phase change is provided by a ferromagnetic ring, there is a question whether the ring can be aligned accurately enough to withstand the axial field of the lens. Also the maximum size of object that will be imaged accurately (as shown above) may be smaller than is desired for biological applications.

As deduced above, the maximum object size can be increased by increasing the effective focal length of the lens used as objective. Some instruments have been reported in which additional transfer optics are used. In many cases, however, the most practical alternative location for a phase plate is the selected-area (SA) position. Since this must still be the BFP of the lens that is to act as the objective, it suggests the use where available of a Lorentz lens, for which the BFP is at the SA height. Such lenses are liable to show large spherical aberration, but it has been reported [7] that this has been successfully reduced by using an existing aberration corrector.

5. Implications for biological microscopy

Published results (eg [2]) for Zernike phase plates show clearly that they provide the expected gain in contrast at focus. The theory outlined above suggests that accurate information transfer from the object phase to the image intensity is obtainable for object dimensions satisfying $q_0 b < \sim 1$. For a conventional TEM with $f = 3\text{mm}$ and beam energy 100kV, the maximum object diameter for accurate transfer is thus about 7 nm. Broader variations within the object will be attenuated, and sharp phase changes within such broad features are likely to produce overshoots which show up in the image as bright lines. As described above, if the effective focal length of the objective lens can be increased by use of a Lorentz lens or otherwise, this should allow proportionately larger object features to be imaged accurately.

6. Summary

The white outlines or halos seen around some phase objects when a Zernike plate is used have been shown [5] to be due to the high-pass response of the phase plate, defined by its central hole.

The disadvantages of placing a Zernike phase plate at the BFP of the objective lens can be reduced by placing it at the SA location and modifying the objective lens to have greater effective focal length, for instance by using a Lorentz lens where this is available. Doing so will increase the largest diameter of object features whose phase will be imaged accurately.

Acknowledgment

The authors thank A. M. Blackburn for ebeam lithography of the semicircular rings and R. M. Glaeser for stimulating discussions. Fig. 2 was produced by use of MATHEMATICA.

References

- [1] Edgcombe C J and Loudon J C 2012 *J. Phys.: Conf. Ser.* 371 ed G Moebus *et al* (Bristol: IoP) p 012001
- [2] Danev R and Nagayama K 2001 *Ultramicroscopy* **88** 243-52
- [3] Fukuda Y, Fukazawa Y, Danev R, Shigemoto R and Nagayama K 2009 *J Struct Biol* **168** 476-84
- [4] Danev R and Nagayama K 2011 *Ultramicroscopy* **111** 1305-15
- [5] Edgcombe C J 2014 *Ultramicroscopy* **136** 154-9,
<http://dx.doi.org/10.1016/j.ultramic.2013.09.004>
- [6] Hall R J, Nogales E and Glaeser R M 2011 *J Struct Biol* **174** 468-75
- [7] Freitag B, Bischoff M, Mueller H, Hartel P and von Harrach H S 2009 *Microscopy and Microanalysis*, **15** (Suppl. 2) pp 184-5