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AN INVESTIGATION OF THE MAXIMUM SPECIMEN THICKNESS FOR DIFFERENTIAL PHASE CONTRAST LORENTZ MICROSCOPY

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ABSTRACT

Examination of magnetic domain structure in the transmission electron microscope is generally confined to very thin foils, where the specimen approximates to a pure phase object, and is achieved by the long established methods of Fresnel or Foucault contrast Lorentz microscopy, or by differential phase contrast (DPC) imaging in a scanning transmission electron microscope (STEM).

If no quantitative interpretation of the image is required then magnetic contrast can be observed from thicker foils, and in this paper we describe an attempt to determine experimentally the range of foil thickness over which this is possible. To this end we have examined electropolished foils of single crystal Incoloy using an extended VG HB501 STEM to produce both DPC and Fresnel contrast images of the same area. The foil thickness at points along the domain walls was measured from the change in the Lorentz deflection angle as the STEM probe was moved across the domain wall, and this led to an estimate of ~ 700nm for the limiting thickness at which domain contrast was still visible in the DPC images.

This value is obviously influenced by a number of factors, including the degree of inelastic scattering and the saturation magnetisation of the material, but it is sufficiently high that there might exist a range of thickness over which both transmission and scanning electron microscopes could be used to study the domain structure in the same areas of specimen.

Keywords: magnetic contrast, differential phase contrast, maximum specimen thickness, Lorentz microscopy, Fresnel contrast, Foucault contrast, magnetic domain walls.

INTRODUCTION

The use of the conventional transmission electron microscope (CTEM) to study the magnetic domain and domain wall structures in thin specimens of ferromagnetic elements or alloys is dependent on the fact that an electron wave passing through the region of magnetic flux suffers a phase shift proportional to the flux linked [Aharanov and Bohm (1959)]. Thus in the vicinity of the domain wall in Fig. 1a, the phase change $\phi(x)$ is given by

$$\phi(x) = \frac{e}{\hbar} \int_{0}^{x} B_{\phi}(x)dx$$

(1)

where $e$, $\hbar$ have their usual meaning, $t$ is the specimen thickness, $B_{\phi}(x)$ is the average in-plane component of magnetic induction and it is assumed that there is no magnetic field above or below the specimen. Thus a normal in-focus image in the CTEM will show no magnetic contrast. To reveal such contrast one of the phase contrast modes of image formation must be used; for magnetic specimens these modes are referred to collectively as Lorentz microscopy.

The most common method of examining magnetic structure is the Defocus or Fresnel Mode [See Fig. 1a] in which the phase change is translated into an intensity change and the domain walls are revealed as dark or bright bands on a uniform background. Unfortunately, if quantitative information is sought, e.g. the domain wall profile, this technique and the equivalent mode in the scanning transmission electron microscope (STEM) [Chapman et al (1977)] suffer from a number of disadvantages. The most serious of these lies in the interpretation of the data since for most magnetic specimens the intensity distribution in the image is not related linearly to the specimen transmittance [for a discussion see for example, Chapman et al. (1978)]. Another technique which has been applied in the CTEM is Foucault Contrast, where an opaque aperture is inserted to obstruct one half of the back focal plane of the objective lens so that only those electrons which pass through the other half contribute to the image. In this way (Fig. 1a) domains which lie alternately parallel and anti-parallel to the y axis appear alternately bright and dark. The extraction of quantitative information from Foucault micrographs is even more difficult than is the case with Fresnel imaging. Not only is it impossible to invert the intensity data directly, but also the intensity profile of a wall region is very sensitive to the exact positioning of the aperture.

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SYMBOLS AND ABBREVIATIONS

\[ B_0, B_y = \text{magnetic induction (Tesla)} \]
\[ e = \text{electronic charge (C)} \]
\[ h = \text{Planck's constant (Js)} \]
\[ t = \text{specimen thickness (m)} \]
\[ \alpha_D = \text{semi-angle subtended by the detector at the specimen plane (radians)} \]
\[ \alpha_0 = \text{semi-angle subtended by the probe forming aperture at the specimen plane (radians)} \]
\[ \beta_L = \text{the Lorentz deflection angle (radians)} \]
\[ \phi(x) = \text{phase shift of the electron wave (radians)} \]
\[ \text{BSE} = \text{backscattered electrons} \]
\[ \text{CTEM} = \text{conventional transmission electron microscope} \]
\[ \text{DPC} = \text{differential phase contrast} \]
\[ \text{PSL} = \text{post-specimen lens} \]
\[ \text{STEM} = \text{scanning transmission electron microscope} \]
\[ \text{VOA} = \text{virtual objective aperture} \]

Dekkders and de Lang (1974) have shown that in the STEM the difference signals from two semicircular detectors (See Fig.1b) are related to one component of the derivative of the phase variation of the specimen transmittance. For magnetic specimens an examination of equation (1) shows that differential phase contrast should yield directly information on the spatial variation of the magnetic induction in the specimen. Hence for the last few years our group has been carrying out an experimental and theoretical investigation of the application of the split detector and the related quadrant detector system (see for example, Chapman et al (1978) loc. cit., Waddell (1978), Waddell and Chapman (1979), Morrison and Chapman (1981), Morrison (1981)). It has been shown that these differential phase contrast (DPC) systems can tolerate relatively large phase excursions at low spatial frequencies and still image linearly and it is this which makes them particularly well suited to quantitative Lorentz microscopy and in particular to the determination of accurate domain wall profiles.

If quantitative information is not required in Lorentz microscopy, the question arises as to whether or not the DPC imaging mode in the STEM would be suitable to detect the presence of magnetic domain structure in thick specimens. As the specimen thickness is increased the angular distribution of scattered electrons will broaden and the peak height will diminish; in the absence of phase gradients in the specimen, the distribution will however remain symmetric about the optic axis. Since in the DPC imaging mode it is the asymmetry in the scattering distribution due to the magnetic induction which is responsible for the magnetic contrast it can be argued that the method should be relatively insensitive to increasing the specimen thickness at least until the difference signal is comparable to the noise in the detector system. In this paper we report the results of an experimental determination of the maximum thickness at which magnetic contrast was visible in DPC images.

EXPERIMENTAL DETAILS AND RESULTS

The microscope [see Fig. 2] used in this investigation was the V.G. Microscopes HB501 FEGSTEM (field emission scanning transmission electron microscope) extended by the inclusion of a second condenser lens and a set of three post specimen lenses (PSL) [Craven et al (1980)]. For DPC imaging the quadrant detector system consisted of a windowless version of the Centronic QD-100 quadrant photodiode detector mounted on a retractable carriage and positioned in the column below the annular dark field detector [Morrison and Chapman (1981) loc. cit.]. To use this type of detector it is important that the condition

\[ \alpha_D > \alpha_0 \]

is satisfied [Morrison (1981) loc. cit.] where \( \alpha_D \) and \( \alpha_0 \) are respectively the effective semi-angles subtended at the specimen by the detector and the aperture defining the probe angle. The probe-forming conditions suitable for this type of Lorentz microscopy have been discussed by Chapman et al (1980), whilst the operation of the PSL system has been considered in detail by Craven and Buggy (1981). When using a detector which is sensitive to an intensity distribution which is asymmetric about the optic axis, it is important that any systematic movement of the distribution in the detector plane as the probe is scanned over the specimen is cancelled out. This motion, which arises because the detector is in the far field relative to the specimen, rather than a true Fraunhofer plane, can be corrected by feeding an appropriate descanning signal to the Grigson scan coils situated above the objective lens.

When studying magnetic contrast in the STEM it is necessary to avoid saturation effects in the specimen from the field of the objective lens. In this case both the objective and the second condenser lenses were switched off, the electron probe being formed by the first condenser lens. Since the purpose of the experiment was to determine the limiting thickness at which domain contrast was visible, it was essential to have a high current in the probe and hence a 500µm virtual objective aperture (VOA) giving a probe semi-angle of \( 1.2 \times 10^{-3} \) radian was selected; under these conditions the probe current was \( 1.5 \times 10^{-5} \) A and the coherent and incoherent probe sizes were respectively \( \sim 100\)nm and \( \sim 15\)nm. The inequality of equation (2) was satisfied by using camera lengths between 1.5m and 4m giving values of \( \alpha_D \leq 1.4 \times 10^{-3} \) radian.

An electropolished single crystal specimen of a commercial magnetic alloy known as Incalloy, which has a composition of 33.8 Ni, 51.0 Fe, 14.0 Co, 1.2 Ti wt %, was used in this investigation. It was selected because it exhibited a series of straight 180° domain walls running approximately radially into the bulk of the specimen from the edge of the thinned area. Fig. 3a is a bright field image showing the area of foil around the hole which was visible under normal operating conditions. By greatly increasing the electronic amplification of the detected signal and using the maximum possible probe current, magnetic contrast was visible in the DPC image (Fig. 3b) from a considerably greater area of the foil. It should be noted that, although the DPC image was normalised by the sum signal from all four quadrants to minimise the effects of
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Fig. 1a) A schematic diagram illustrating how Fresnel and Foucault contrast can arise in the CTEM.

Fig. 1b) Differential phase contrast can be generated in the STEM by using a split or quadrant detector system which is sensitive to any deflection of the bright-field cone across the detector plane.

varying specimen thickness, all contrast from the thinner regions of the specimen has been lost due to the effects of amplifier saturation. Fig. 3c shows a Fresnel contrast image of the same area, again taken with a probe semi-angle of $1.2 \times 10^{-3}$ radian, but with the camera length reduced so that a 500µm bright field collector aperture subtended a semi-angle of $4 \times 10^{-4}$ radian.

To estimate the specimen thickness at points along a domain wall, the movement of the zero order diffraction disc was recorded from the diffraction screen as the probe was moved across the domain wall. The movement of the disc is directly proportional to twice the Lorentz angle ($\beta_L$) and since the probe angle $2\alpha_0$ is known, $\beta_L$ may be determined from a double exposure such as that shown in Fig. 4. The foil thickness can be determined directly from the formula

$$t = \frac{h\beta_L}{eB_0\lambda}$$

Fig. 2. The electron optical column of the extended HBS01 electron microscope.
Fig. 3a) Bright field image showing the thin area around the edge of a hole in an Incalloy foil.

Fig. 3b) DPC image of the same area of foil taken with the probe current and electronic amplification increased to reveal magnetic contrast from thicker regions of foil. The signals from the quadrant detector were combined in the manner \( \frac{(A + D) - (B + C)}{(A + B + C + D)} \), and the detector oriented with respect to the specimen as shown in the diagram.

Fig. 3c) Fresnel contrast image.

Fig. 4. A double exposure illustrating the deflection of the bright field cone which can be observed as the STEM probe is moved across a 180° domain wall.
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where $\lambda$ is the relativistic electron wavelength. Unfortunately, due to the rapid fading of the central disc in the diffraction pattern, arising from the increasing number of electrons scattered inelastically and the consequent increased angular width of the scattering distribution, it was found impossible to measure thicknesses above ~ 400nm using this technique; this thickness is less than that at which domain contrast was still visible in the DPC images. Another possible method of thickness measurement, via the thickness fringes, was unsuitable since their contrast diminished rapidly above about 350nm. Hence it was decided to estimate the maximum thickness by assuming that there was a linear relationship between specimen thickness and distance along the domain wall from the edge of the hole in the specimen. On this basis the limiting thickness to which magnetic contrast remained visible in the DPC image was estimated to be ~ 700nm for 100 keV incident electrons. In the case of the Fresnel contrast STEM image in Fig. 3c, the limiting thickness was estimated to be ~ 500nm.

DISCUSSION

The purpose of these experiments was to obtain an estimate of the maximum specimen thickness for the observation of magnetic contrast in the DPC imaging mode of the FEG-STEM. When operating at the limit of domain visibility there is clearly no possibility of obtaining domain wall profiles; the low signal to noise ratio means that insufficient grey levels are available in the image for an accurate profile, the large spherically-aberrated probe associated with the choice of the largest probe forming aperture inevitably degrades the resolution beyond the point where a reliable wall profile could be obtained and this effect would be compounded by the beam spreading which will occur for a specimen of this thickness.

Although the experiments carried out to date are only preliminary we believe that for Inconel they provide an approximate lower limit for the usable thickness. A significant potential source of error lies in the assumption that the crystal has a uniform wedge shape radially outward from the edge to the point at which magnetic contrast disappears. Nonetheless, examination of thickness fringes over the range where they are visible suggests that the assumption is well founded and it is unlikely that our estimate of 700nm is in error by more than 10% from this source.

A factor which may lead to an increase in the usable thickness would be the use of smaller camera lengths, leading to a larger fraction of the inelastic scattering distribution being utilised. With the camera lengths cited, the condition $\alpha_D > \alpha_0$ was always easily satisfied, but for the thickest regions of specimen investigated both angles were considerably less than the half-angle of the emergent electron distribution. Finally we should note that with the experimental conditions as defined, the observed thickness limit is probably set by the noise performance of the quadrant detector and its associated electronics [Morrison (1981) loc. cit.]. The detector is certainly incapable of detecting single electrons and so the noise level in the images considerably exceeded that due to intrinsic beam shot noise. Hence, the quoted usable thickness is unlikely to represent a fundamental limit.

The estimate for 100 keV electrons of ~ 700 nm for the limiting thickness for magnetic contrast in the Inconel specimen could only be applied to other single crystal materials if they possessed the same inelastic scattering distribution as a function of specimen thickness and they had approximately the same value of saturation magnetisation. Given the composition of Inconel its inelastic scattering distribution should not be vastly different from those of the individual ferromagnetic elements Ni, Fe and Co, and hence the figures of ~ 700nm should also serve as a realistic lower limit for the maximum thickness in Fe and Co, although it may overestimate the magnitude for Ni. It should be stressed at this point that the situation will be quite different if the specimens are micropoly-crystalline rather than single crystal. The substantial increase in incoherent scattering for the polycrystalline case will reduce the limiting thickness for films of Fe, Ni and Co well below the figure of ~ 700nm.

For the case of Fresnel imaging it is much more difficult to assess how the maximum usable thickness will depend on the experimental parameters. Hence the figure of ~ 500nm must be accepted as relevant only to the particular set of parameters used in the experiment. However, the arguments given above concerning the relevance of the figure of ~ 500nm to other single crystal specimens of Fe, Ni and Co and their ferromagnetic alloys should still apply in this case.

The figure obtained in this investigation for the limiting thickness in DPC imaging is surprisingly high and leads us to speculate on the exciting possibility that there may exist an overlap between the DPC method as applied to ‘thin’ films of cubic materials in the STEM and the observation of type II magnetic contrast in the backscattered electron (BSE) images of bulk specimens of the same materials in the scanning electron microscope [for a review of magnetic imaging in bulk materials see Wells and Shimizu (1982)].

Recently the ease of observation of type II contrast has been greatly simplified by the pioneering work of Wells in the development of lock-in amplifier techniques [Wells and Savoy (1981)]. If it is possible to provide an a.c. magnetic field to drive the domain walls, then this technique largely eliminates interference from topographic or atomic number contrast. For the transition metal elements Fe, Ni and Co the extrapolated range for 100 keV electrons is ~ 10µm and the maximum escape depth will be half of this. Data on the energy distribution of the BSE that contribute to type II contrast when operating at normal incidence is not available. For oblique incidence the BSE in the top 30% of the energy spectrum contribute to the magnetic contrast [Jakubovics and Wells (1980)]. The question is therefore whether or not there will be sufficient type II contrast from a 700nm single crystal specimen of Fe, Ni or Co. This we intend to explore as part of our experimental programme. We hope that others skilled in the application of Monte Carlo techniques will explore the possibility theoretically.

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REFERENCES


