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LETTER TO THE EDITOR

Magnetic x-ray diffraction from ferromagnetic iron

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Abstract. Non-resonant magnetic diffraction of synchrotron radiation from ferromagnetic iron has been measured. We have used, for the first time, a white beam and single crystal sample. It is demonstrated that this combination can yield magnetic flipping ratios with small statistical and systematic errors. The technique has been used to investigate the polarization dependence of magnetic scattering.

Although it has been known for some time that diffraction of elliptically polarized x-rays from ferromagnets is weakly spin-dependent (Lovesey 1987, Blume and Gibbs 1988), experiments which exploit this effect have proved very difficult since such scattering amounts to a very small fraction of the total intensity. Unlike pure magnetic diffraction peaks from antiferromagnets which can occur at positions separated in reciprocal space from the charge peaks (de Bergevin and Brunel 1972), spin-dependent interference scattering appears only as a small change in Bragg peak intensity on reversing the magnetizing field direction or 'handedness' of the circular polarization of the incident x-ray beam.

Spin-dependent scattering can be enhanced if the energy of the incident x-ray beam is close to an absorption edge and several interesting experiments of this type have been carried out (e.g. de Bergevin and Brunel 1981, Namikawa *et al* 1985, Vettier *et al* 1986). Interpretation of the data is complicated, however, by the need for a precise understanding of the resonance involved. For this reason it is advantageous to measure the smaller non-resonant magnetic diffraction. Synchrotron radiation sources provide highly polarized radiation which can be used for these measurements. Elliptically polarized radiation can be extracted at a small angle above or below the synchrotron orbital plane and the magnetic contribution to the scattering can be isolated by reversing the sample magnetization direction (Cooper *et al* 1986).

For the special case in which the scattering is horizontal at an angle of 90° and the magnetization direction is aligned with the scattered beam (a geometry very close to optimum for such measurements and to that adopted for this work) the magnetic flipping ratio is given by the following simple formula:

$$\frac{\Delta I}{I} = \frac{g P_{\mathrm{c}} \mu(k)}{(1 - P_{\mathrm{l}}) n(k)} \tag{1}$$

where g is the ratio of the x-ray energy to the electron rest energy, $\mu(k) = 2S(k) + L(k)$, $S(k)$, $L(k)$ and $n(k)$ are the spin and orbital angular momentum

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and charge form factors (Lovesey 1987), and P_c and P_l are the degrees of circular and linear polarization.

The first non-resonant measurements were made by scattering monochromatic radiation from powder samples. The technique was adopted by Brunel *et al* (1983) and more recently by Kaiser *et al* (1989) and clearly demonstrated the feasibility of measuring such small flipping ratios ($\Delta I/I \sim 0.1\%$). The errors obtained by this technique, however, remain quite large.

The present work is the first magnetic diffraction measurement to use white radiation. There are a number of major advantages in using white radiation over monochromatic. First, since small changes in sample orientation do not cause a departure from the Bragg condition, then as long as the detector aperture is larger than the Bragg spot, the technique is insensitive to the small sample movements which may occur as the magnetizing field is reversed. Secondly, the need to scan mechanically over reflections to obtain integrated intensities is removed, allowing a fixed scattering geometry to be employed. A final point in favour of white radiation is that by elliminating the monochromator, the beam incident on the sample has a higher degree of circular polarization, and the polarization is more easily calculated. Using a single-crystal sample (as opposed to a powder) gave high diffraction count rates and reduced the relative strength of the fluorescence signal.

The sample was a single crystal of pure α-iron. The scattered x-rays were counted by a solid state germanium detector, and the energy discrimination of the detector enabled the harmonics of each reflection to be resolved and measured simultaneously. The sample (an 8 mm diameter disc with a $\{111\}$ face normal) was mounted across the poles of a small electromagnet, with the field direction along $\{11\bar{2}\}$. A schematic diagram of the experiment is shown in figure 1. A scattering angle of 95° was chosen to optimize the ratio of magnetic to charge scattering, with the magnetization direction $\sim 12°$ from the scattered beam. An important potential source of error that is not removed by using white radiation arises from fluctuations in the incident beam intensity. The slow decay of the synchrotron beam did not present a problem as the electron beam life-time was in excess of 24 h. However, any small movement in the beam orbit can produce a sudden change in the x-ray intensity. We therefore recorded the Bragg peak intensities after every half-second period in the asynchronous field reversal cycle. The data could therefore be checked after the experiment for any sudden changes in beam intensity, and a point-by-point statistical analysis was carried out.

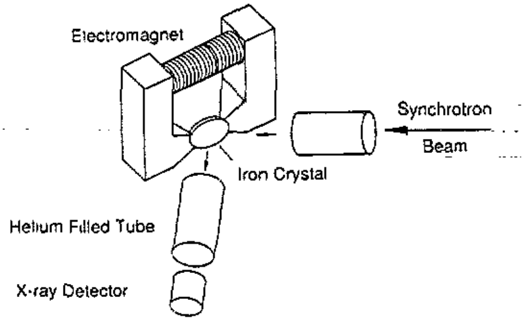

Figure 1. A schematic diagram of the experiment. Helium-filled tubes reduced the total air path to around 10 cm, enabling the $\{110\}$ reflection at around 4.2 keV to be observed.

Measurements were made on station 7.6 of the Daresbury synchrotron radiation source (SRS) with the incident beam taken at various small angles from the synchrotron orbital plane (see Holt and Cooper 1983), each providing a different degree of linear and circular polarization. The horizontal linear polarization is maximum in the synchrotron orbital plane and decreases away from it, while the converse is true for the circular polarization which changes sign on crossing the orbital plane. One would therefore expect, from equation 1, that the magnetization flipping ratio should peak for some optimum combination of P_c and P_l. Due to time limitations only the $\{hh0\}$ reflections were measured, with statistically significant magnetic effects obtained for $\{110\}$ and $\{220\}$. Data were collected for one hour at each beam angle, with the count rates in each of the two main lines kept to around $2.5 \times 10^4\ \mathrm{s}^{-1}$ by making fine adjustments to the beam width (the beam cross sections were typically 1 mm^2).

The results of the measurements are shown in figure 2 along with the predicted ratios. A statistical analysis of the point-by-point data showed that beam instabilities and sample movements had a negligible effect on the measured flipping ratios, and the symmetry between measurements made above and below the orbital plane shows that the data were unaffected by sample movements correlated with the magnetizing field direction. The measured curves show the same qualitative features as the predictions, peaking at around 0.06 mrad, but it is clear that there is some discrepancy between the measurements and calculations. These could in part be due to dead-time effects in the counting chain. Such effects would have caused a scaling down of the measured flipping ratios by a few percent at most. The main source of the discrepancy is likely to be in the calculation of the beam polarization, and in particular the linear component. This is presently being investigated.

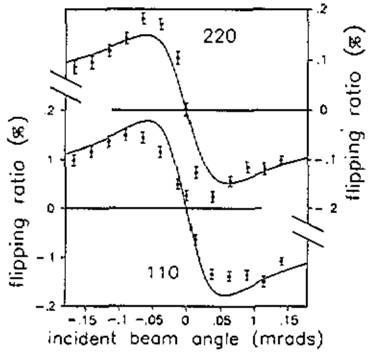

Figure 2. The measured magnetization flipping ratio ($\Delta I/I$) for $\{110\}$ and $\{220\}$ reflections at various angles above and below the synchrotron plane. The full curves are calculations based on the cross section of Lovesey (1987) and form factors from Tawil and Callaway (1972). The beam polarization was calculated from measured SRS beam parameters (Laundy 1990).

Even with the uncertainties in beam polarization, the measured flipping ratios are close to the predictions. This experiment demonstrates that x-rays can be used to measure magnetic form factors with low statistical and systematic errors, and future work should yield data complementary to neutron studies.

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