EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** ***Dimensional and single-atom effects on critical fluctuations of magnetic surfaces and thin films***	**Experiment number**: HE623
Beamline:	**Date of experiment**: from: September 1999 to: September 2001	**Date of report**: 28-2-2001
Shifts:	**Local contact(s)**: N.B. Brookes	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this project was to develop a new technique for the study of the dynamics in magnetic systems, called Magnetic X-ray Intensity Fluctuation Spectroscopy. This technique uses time correlation of speckle intensities to probe the slow dynamics of magnetic systems. Magnetic sensitivity is obtained by using the strong magneto-optical effects found at x-ray absorption edges where a core hole resonates with a magnetic valence shell.
The project was allocated a long term beam time grant which runs until July 2001. This interim report covers the 30 shifts allocated on ID10A and 42 shifts on ID12B.

The research goals of the proposal were:
1. Coherent magnetic scattering experiments on ID10A
2. Development of detector systems
3. Characterization of the coherent flux and the stability of ID12B
4. Production of magnetic speckle patterns
5. Actual M-XIFS measurements

The first goal has not been attained. Points 2-4 have been very successful, and a preliminary report has been given in the ESRF newsletter (September 2000). Formal publications will be submitted shortly. The last goal will be started with in the remaining week of beam time on ID8.

Coherent magnetic scattering experiments on ID10A

Circular polarization adds extra information to magnetic scattering, as is exemplified by the results described below. In view of this a QWP system was build by the Troika team (C.D.). The commissioning and characterization of this system was the subject of a separate proposal (see report MI 290). As described there, while ultimately very

successful, this project took longer than expected, and the commissioning continued in the beam allocation of HE-623.
The circularly polarized beam was subsequently used in an attempt to obtain magnetic diffraction patterns both in reflection and transmission of self-organized stripe domains in GdFe films at the Gd L_3 edge. These films, which were also used in the other experiments described here were grown and characterized using MOKE and Magnetic Force Microscopy (see below) at the van der Waals-Zeeman Institute, Amsterdam. The dichroic signal on the Gd L_3 edge of these samples is in the order of 5%, and weak magnetic satellites are to be expected. Unfortunately, in the remaining time it turned out to be impossible to surmount the technical problems and no magnetic diffraction patterns were obtained. However, the cross fertilization between a scattering group and a spectroscopy group turned out to be extremely useful and greatly helped the more successful soft x-ray program described below.

Development of detector systems

The development of speckle methods on ID10A greatly benefited from the availability of 2D detector systems. For soft x-rays such systems were not readily available at ESRF, which led us to develop a UHV compatible scintillator-type small-angle scattering camera. This system consists of a scintillator, coupling optics and a CCD camera. The scintillator layer used presently is a 5 µm thick P46 layer, grain size 1 µm, deposited on a 18 mm diameter, 3 mm thick vacuum window. For the coupling optics we used a 4x microscope objective and several commercial lens systems. The CCD camera was a Sensicam CCD camera from the ESRF loan pool.

Although the overall efficiency of this system is less than 1%, due mainly to the low efficiency of the scintillator, it has the huge advantage of allowing one to use the full dynamic range of the CCD. Furthermore it allows rapid trading of q-resolution versus q-range. With the microscope objective the highest pixel resolution is 5 µm, sufficient for high resolution imaging of soft x-ray speckle images with the screen at 0.3 m behind the sample. We are still working on improving this system with better coupling optics and scintillator materials.

For soft x-ray intensity fluctuation experiments we require a reliable photon counting system with a high dynamic range, good linearity and a quantum efficiency of 1. We have acquired an avalanche photodiode that will go under test beginning 2001. Other possibilities are channeltrons in combination with photocathodes. These detectors will give a asynchronous signal that will be fed to a digital autocorrelator.

Characterization of the coherent flux of ID12B

The undulator radiation of third generation sources is highly transversely coherent, due to the small emittance. This coherence is partially destroyed by the beam line optics, and spatial filtering has to be applied to discard incoherent fractions of the beam at sample. For our spatial filter we employed high quality laser drilled pinholes of 5 to 25 µm diameter in combination with vertical and horizontal slits upstream from the pinholes.
The coherence was analyzed by imaging the Fraunhofer diffraction of the pinholes (see fig. 1). By tuning slits upstream from the pinhole, nearly perfectly circular patterns could be obtained. The intensity of such Fraunhofer patterns turned out to depend strongly of the position of the analyzing pinhole along the beam direction, and the position of the upstream slits along the beam line. The highest intensity is obtained by post-optics spatial filtering at the end of the beamline. However, extra slits can be omitted altogether, because we found that the intrinsic coherence length is at least 20 µm in the focus of the beam. The coherent flux through a 10 µm pinhole is ~6×10^8 ph/s at ~1200 eV (1 nm).

Static magnetic diffraction: the 3D structure of stripe domains

As an intermediate step towards coherent speckle measurements we studied the incoherent diffraction of magnetic stripe domains in amorphous GdFe thin films (Fig.2). We used both transmission and reflection geometries and compared the Gd M_{45} and Fe L_{23} edges. An in-situ field of 0.5 Tesla could be applied to magnetically saturate the sample. Most experiments were done at 20 K in order to enhance the magnetic signal.
Fig. 3 shows the diffracted intensity at the Gd M_5 edge of disordered and ordered stripe patterns. The diagram on the left shows two 'powder' diffraction rings from the disordered magnetic structure, shown in real space in the MFM image. After in-plane magnetization the stripes are aligned along the direction of the field and the diffraction

pattern becomes the satellite structure produced by the magnetic grating. The intensity on the first order satellites was strong enough to be observable by eye. Higher orders were falling off the scintillator window in this image. Fig. 4a shows the logarithm of the integrated diffracted intensity recorded for a number of values of a perpendicularly applied field. Up to 5 diffraction orders are visible, showing a pronounced circular dichroism. These data clearly show an increase of the stripe periodicity as the field is increased, while at high fields the diffraction pattern reduces to a broad structure which corresponds to the form factor of very narrow reverse domains.
The 2^{nd} and 4^{th} diffraction peaks show a pronounced circular dichroism at zero fields, which can be traced back to an interference between a scattering amplitude proportional to M_z (z//surface normal) in the domains and one proportional to the square of the in-plane magnetization component M_x in the domain walls. The dichroism follows the broad form factor feature, showing that it arises from the domain-walls connected with the reverse domains. Similar data were taken at different angles of incidence in transmission, as well as in reflection geometry, for both in-plane and perpendicularly applied fields. This huge amount of data is being analyzed and will be the basis of future papers and a Ph.D. thesis (J.F. Peters).
The interest of these data is that they allow one to obtain the 3-dimensional magnetic structure on the length scale of the domain wall dimensions. This is a unique feature in magnetic domain imaging, since all other techniques measure the surface magnetization or the fringe fields outside the sample. With future improvements in flux and detector technology we expect a spatial resolution in the order of 5 nm can be reached.

Production of magnetic speckle patterns

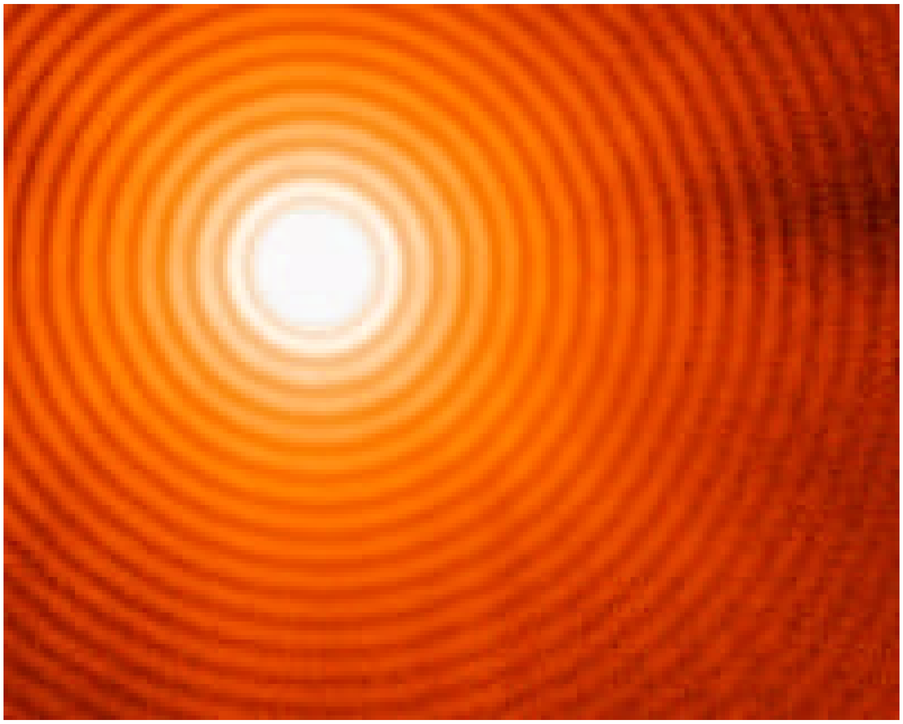
Already in the very first data set that we obtained, the diffraction ring of the disordered stripes showed a graininess due to the coherence of the beam. In following runs, the quality of the speckle data was improved mainly by avoiding vibration of the beam-defining pinhole and the sample. Fig. 5 shows the transmitted beam and the +/-1 diffraction orders of the ordered stripe lattice. The coherence of the beam is witnessed by the Airy pattern of the primary beam. This is reflected in the speckle pattern on the diffracted beams, which have an angular width close to the central spot of the Airy pattern. Other proof of the magnetic origin of these speckle patterns lies in the fact that it disappears on saturating the sample, and reappears afterwards in a different configuration. This shows that this speckle pattern indeed reflects the local magnetic structure on length scales between 180 nm and 10 μm.

Furthermore, the speckle pattern can be seen to be central-symmetric with a correlation coefficient of 0.94. Note that the detector had to be realigned for these two images, showing the stability of the speckle and the reproducibility of the measurement over periods of hours. This symmetry justifies Fourier transforming the speckle intensity in order to obtain the magnetic correlation function (Fig. 6). This function shows the size of the beam, the stripe period and the details of the correlated regions of the structure. It compares very well with the equivalent MFM correlation function obtained from a different part of the same structure.

This proves conclusively that the method employed here should be sensitive to magnetic fluctuations on a length scale between 180 nm and 10 micron. The intensity per speckle is about 5000 cts, which given a detector quantum efficiency of 1 and ten times higher flux as can be obtained on ID8 should make it possible to acquire data at video rate.

M-XIFS measurements

The final step to be taken involves critical scattering experiments. This will be started in June 2001, in the last week of allocated beam time.



Fg. 1. Airy diffraction pattern of a coherently illuminated 10 micron pinhole at λ=1 nm. On this logarithmic intensity plot, 24 rings can be observed.

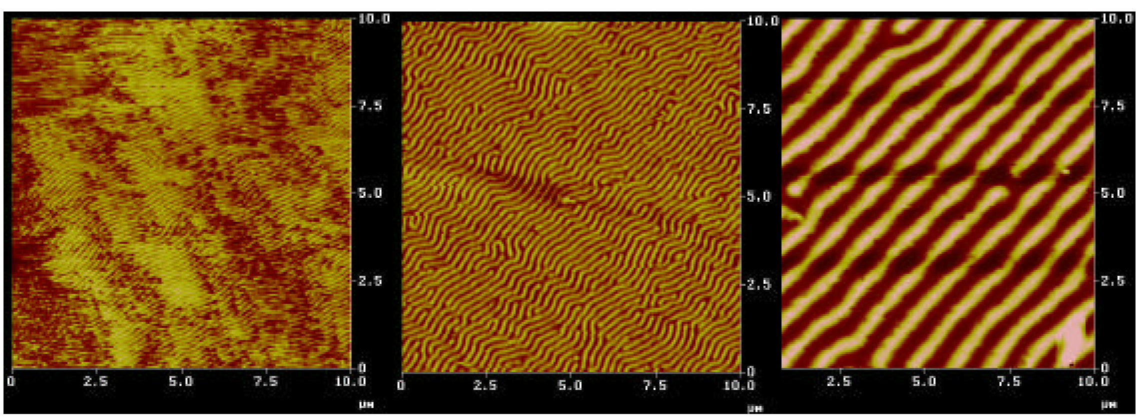


Fig. 2. Magnetic Force Microscopy image of the stripe domains in GdFe films of various thickness, showing the tunability of the stripe period. The same can be achieved by varying the composition.

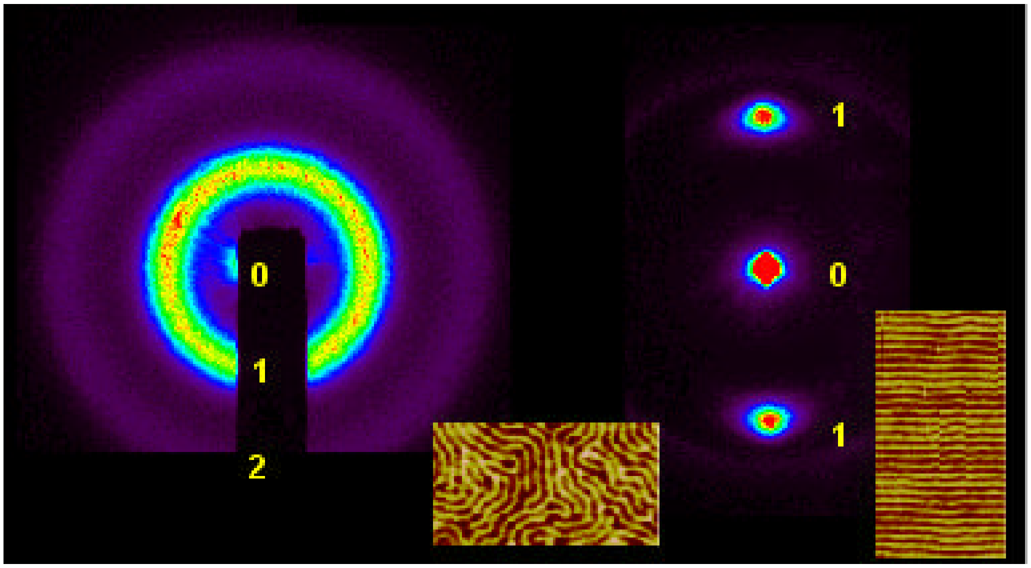


Fig. 3. Diffraction patterns taken taken in transmission at the Gd M_5 edge of GdFe stripe domains (T=20 K). Left: disordered stripes give rise to a "magnetic powder" ring. Right: Magnetically aligned stripes act as a grating and produce a series of maxima.

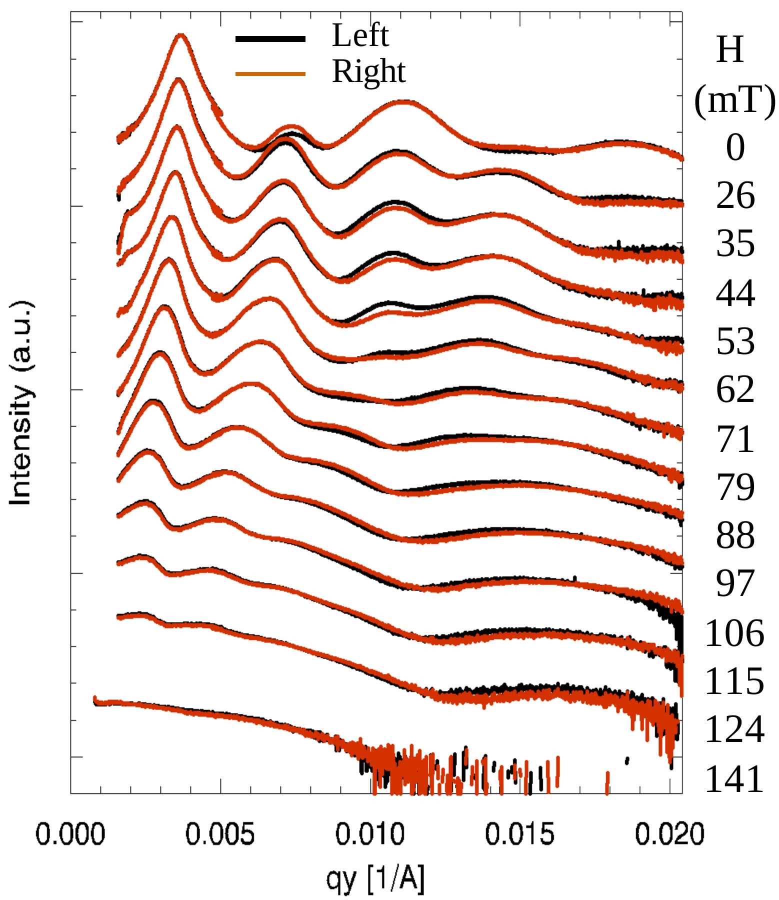


Fig. 4. The scattered intensity profiles as a function of applied field. Red and black denote left and right circularly polarized intensities.

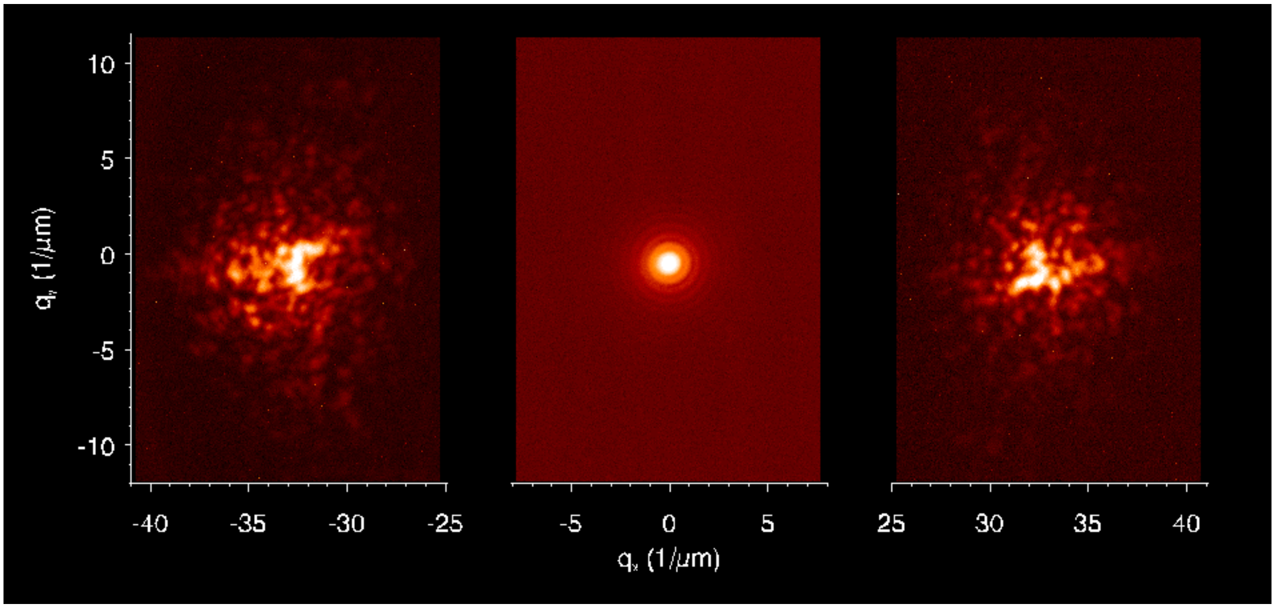


Fig. 5. 1st order speckle intensity of aligned stripes around the transmitted 10 μm primary beam. The latter shows clear Airy fringes, demonstrating the coherence of the light. The speckle images are centralsymmetric and are correlated for 94%.

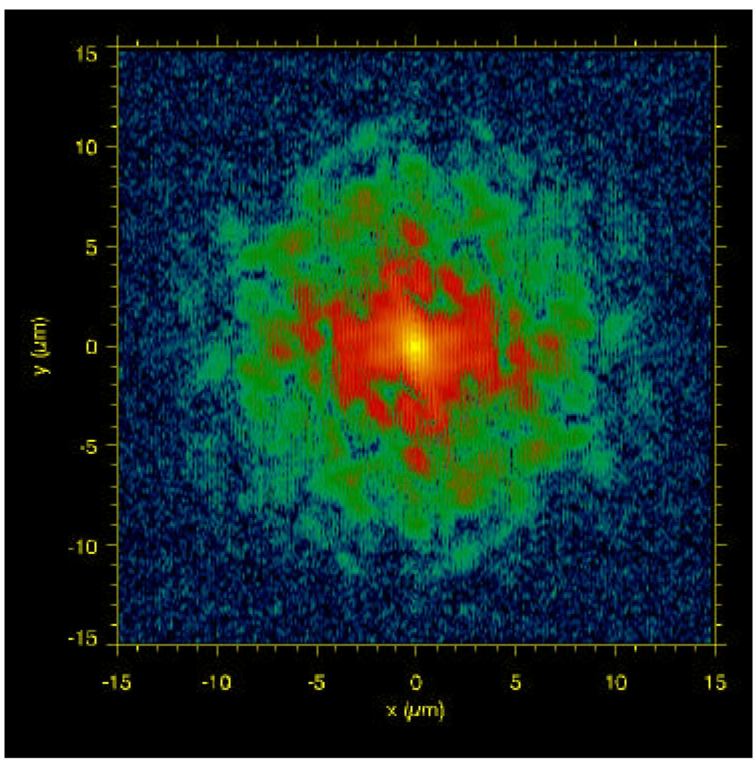


Fig. 6. Magnetic correlation function obtained by Fourier transforming the speckle intensities of Fig. 5.