XMaS the UK-CRG

Newsletter

May 2000

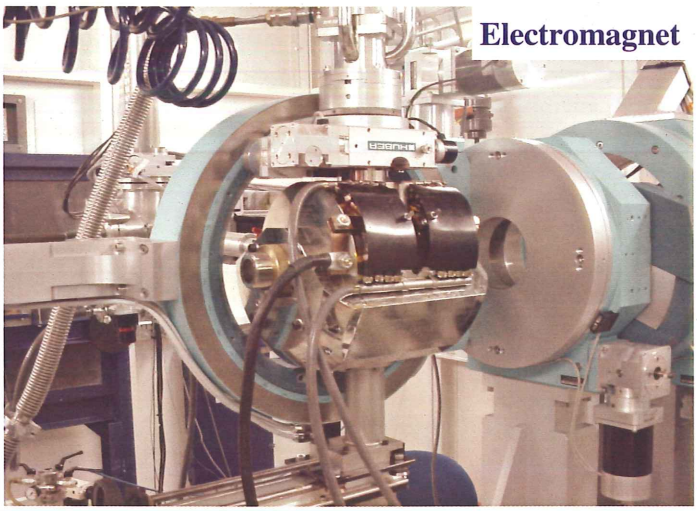
Introduction/Status

The XMaS beamline has been in operation now for two complete years. During this time 48 experiments have been performed by 15 visiting UK groups and a further 7 undertaken by UK and non-UK groups as part of the public allocation that CRG beamlines provide to the ESRF. The beamline has settled into routine operation with less than 5% "lost time" due to all causes. Moreover in the 12 month period up to October 1999 XMaS delivered more shifts than any other CRG. We continue to upgrade the beamline and various examples of beamline facilities installed within the past 12 months, or about to be installed, are featured below. Subsequent pages contain brief examples of just a few of the experiments carried out by users during the past 12 months.

Polarisation analyser

The polarisation analyser, shown in-situ on the 2-theta arm, has been operational for more than a year now and seen service with several users.

Electromagnet

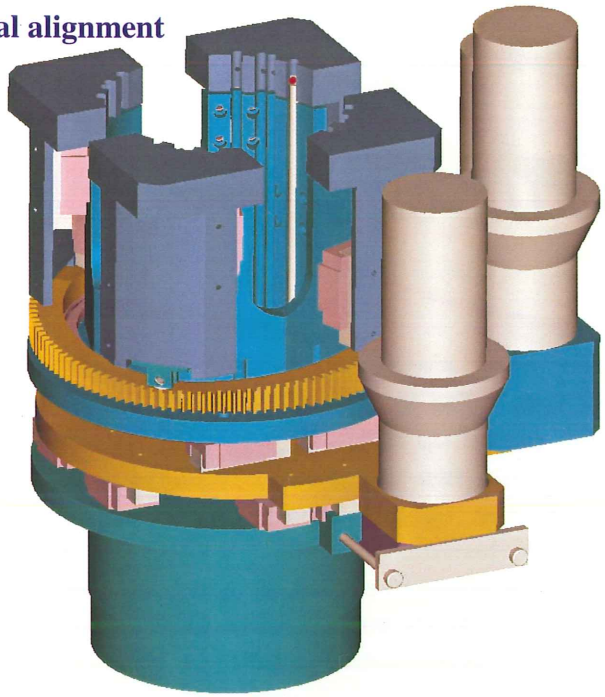


The new 1 Tesla electromagnet mounted on the diffractometer with the cryostat also installed. In its standard mounting it can be oriented in either of two orthogonal positions, providing transverse or longitudinal fields for 90° scattering. It is also possible to mount it in order to provide a vertical field.

Lower sample temperature

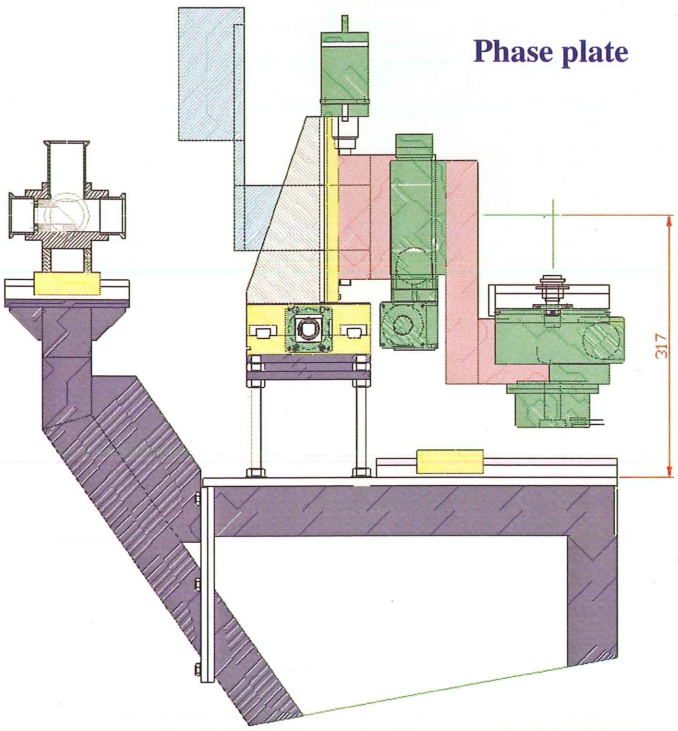
A new low temperature sample environment is presently under test. It is an adaptation of one of the beamline's existing APD displex cryostats and will provide a temperature down to 1.5° K - at present only > 10° K is possible. An "exchange gas" environment is an option with this cryostat. It will be available to users in the next allocation period.

Crystal alignment



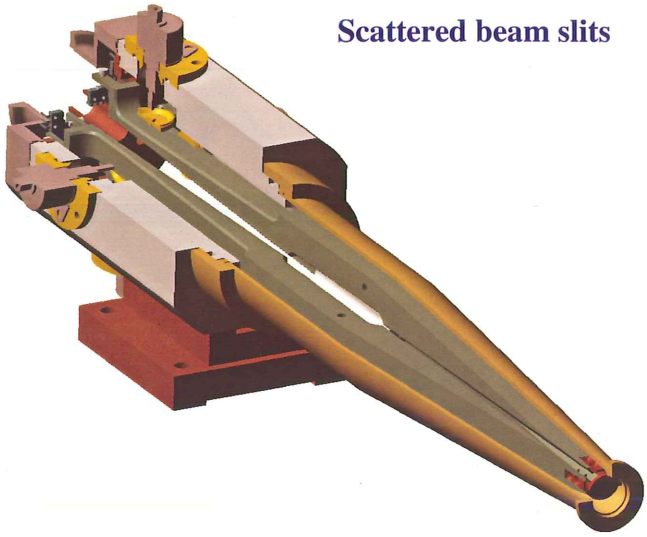
The new motorised cryostat mount, presently under construction, is a direct replacement for the current manually adjusted one. It will significantly enhance alignment for this sample environment and enable users to find more easily that "sweet spot."

Phase plate



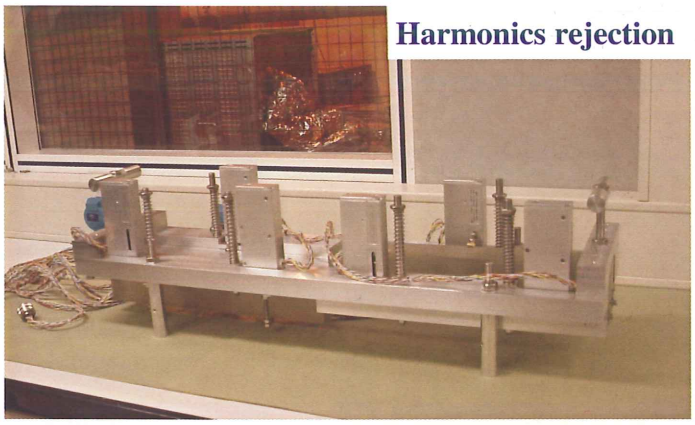
Schematic of the phase plate assembly - primarily for users interested in the delivery of circularly polarised X-rays to their sample. This will be located immediately upstream of the diffractometer and is removed from its frame when not in use. Commissioning is scheduled for early summer.

Scattered beam slits



Cut-away view of the tube-slit assembly, currently under construction, designed to provide slits very close to the sample position for beam definition and scatter elimination. The actuation hardware is remote from the jaws to enable maximum angular access without conflict. This addition complements the recently installed conventional motorised slits , also for the 2-theta arm of the diffractometer.

Harmonics rejection



The double mirror harmonics-rejection system is installed and commissioning, already begun, will be completed through the performance of a harmonics-sensitive experiment during April. This facility offers attenuation 10^3-10^6 of the high order harmonics in the otherwise monochromatic beam, with little sacrifice to the available flux at the selected energy.

Detectors

Two new detector systems have recently been purchased. One, a Cyberstar scintillation detector, provides a higher dynamical range than the existing Bicron scintillators. The Cyberstar ensures linearity up to 20,000 photons/s, and 100,000 photons/s with deadtime correction. These ranges are 2-3 times greater than the Bicron provide.

The other acquisition is a Eurisys Peltier-cooled solid state detector. This has an energy resolution similar to the existing LN_2 cooled Canberra system but, being significantly less massive, offers more flexibility for use on the diffractometer detector arm.

Some Recent Experiments

The phase transition of $SrTiO_3$ doped with calcium

R. A. Cowley, A. Yu. Babkevich - for further information contact R. A. Cowley at Oxford Physics, Clarendon Laboratory, Parks Road, Oxford OX1 3PU

The structural phase transition of $SrTiO_3$ has been exhaustively studied but is still not understood. In particular, the origin of the two divergent length scales close to the phase transition and the extent to which the long length scale is associated with defects are unresolved puzzles. We have studied a crystal of $SrTiO_3$ doped with 1% of Ca using X-ray scattering techniques and the XMaS diffractometer at the ESRF. The effect of only 1 % of Ca is to raise the structural phase transition by about 25K showing that in the neighbourhood of the Ca atoms the structure will be preferentially distorted. We have measured the x-ray scattering close to the satellite vector (3.5 0.5 0.5) * $2\pi/a$ between 250K and 8K. The width of the scattering is shown in Fig. 1 as a function of temperature. It decreases with decreasing temperature until about 130K, then it is resolution limited. The intensity of the scattering is shown in Fig. 2 and it decreases with increasing temperature, particularly abruptly near 130K. In more detail the widths are expected to have an exponent of about 0.7 whereas the 130K and 180K data suggest an exponent larger than unity. This suggests that the Ca has not only raised the transition temperature but has also given rise to a more rounded transition. A more detailed analysis of the line-shape of the scattering is needed to determine whether there are two length scale components. In Figs. 3 and 4 we show three-dimensional plots of the scattering near the (400) and (3.5 0.5 0.5) satellite reflection at 30K. Near (400) two types of tetragonal domains are visible corresponding to domains with their long axis parallel or perpendicular to the [100] direction. Only one peak is visible for the satellite because the domains with rotations of the oxygen about (100) have zero structure factors. Combining these results we can deduce the properties of both domains.

FIG. 1: FWHM vs T(K) for scans along [100] (H-scans) and [011] (K-scans)

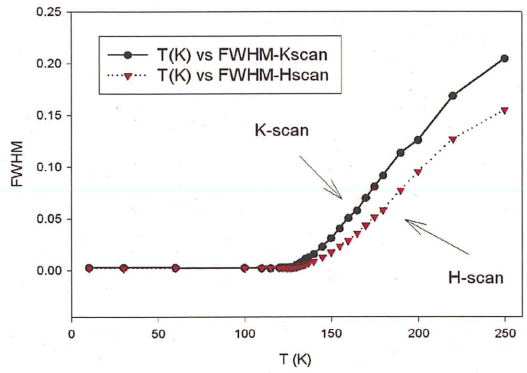


FIG. 2: Variation of the Intensity of a (3.5 0.5 0.5) satellite with a Temperature

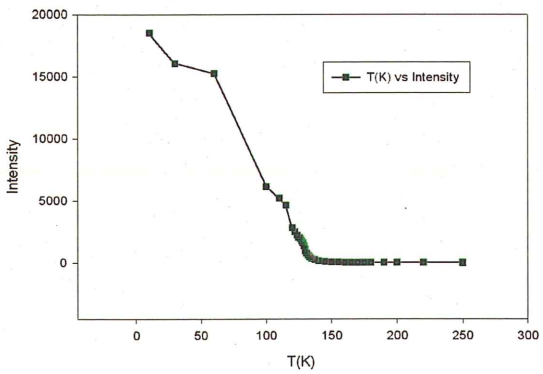


Fig. 3: Scattering around the (400) reflection measured at T=30 K

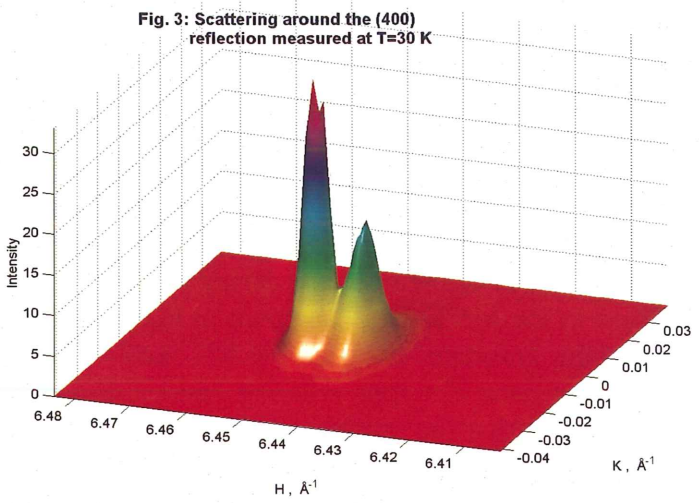
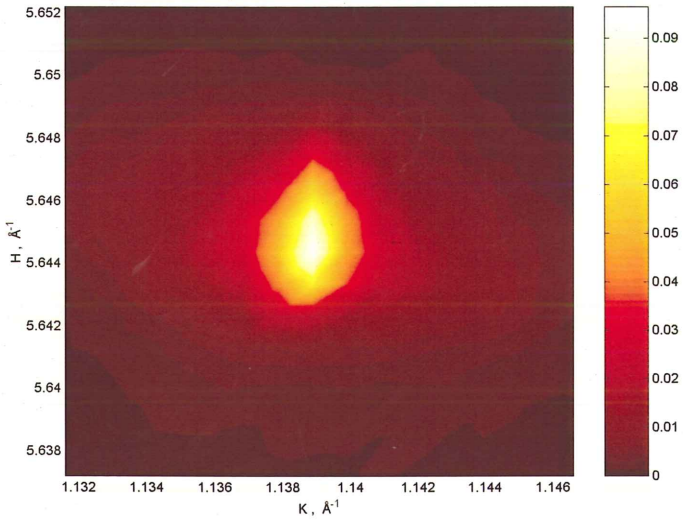


Fig. 4 Scattering from the satellite peak at T=30 K



Orbital ordering in the CMR manganite $Nd_{0.5}Sr_{0.5}MnO_3$

P. D. Hatton, C-H. Du, M. E. Ghazi, D-Y. Chen, S. Brown - for further information contact P. D. Hatton at Department of Physics, University of Durham, Durham. (p.d. hatton@durham.ac.uk)

Charge, spin and orbital degrees of freedom play important roles in the electrical and magnetic properties of the Colossal Magnetoresistance manganites. On the *XMaS* beamline studies have been made of the low temperature phase of $Nd_{0.5}Sr_{0.5}MnO_3$ using resonant x-ray scattering techniques at wavelengths close to the manganese K-edge. By utilising the different polarisation dependencies of charge, spin and orbital ordering (see Fig. 1) the individual wave-vectors were observed. Neutron and electron diffraction have demonstrated the presence of charge spin and orbital ordering in this phase. X-ray studies have demonstrated that the charge ordering displays a resonant enhancement close to the Manganese K-edge indicating that the charge ordering is due to spatial separation of Mn^{3+} and Mn^{4+} ions. Tuning to the Mn K-edge and scattering into the pi channel to suppress charge scattering, resonant enhancement was observed of the spin ordering wave-vector. The resonance occurs at an energy of 6.556 keV.

Orbital ordering can also be observed using resonant x-ray scattering. The dramatic enhancement of the orbital ordering peak (2 0 2.25) close to the Mn K-edge is shown in Figure 1. Note that the resonant energy is 6.552 keV, slightly different from the spin ordering energy. Finally it was noted that the temperature dependence of the orbital ordering peak intensity (Fig. 2) follows the expected behaviour (similar to the spin and charge). However the width is very different showing a marked increase close to the transition. This may be an indication of the orbital ordering displaying critical fluctuations close to the transition.

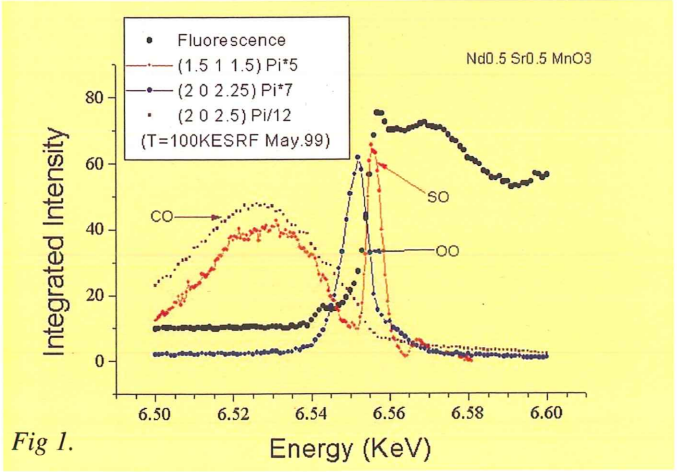


Fig 1.

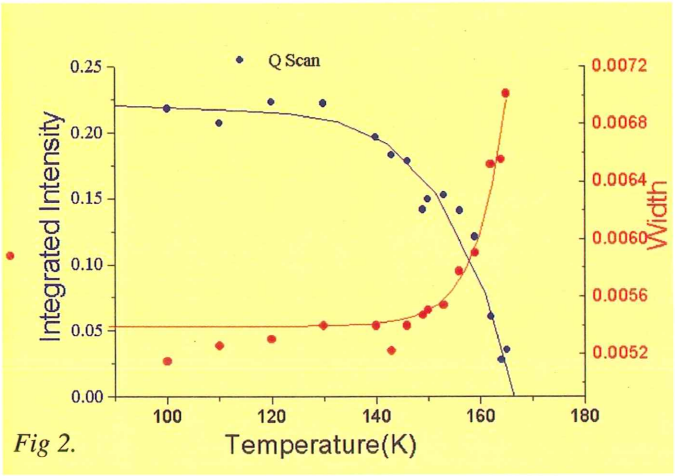


Fig 2.

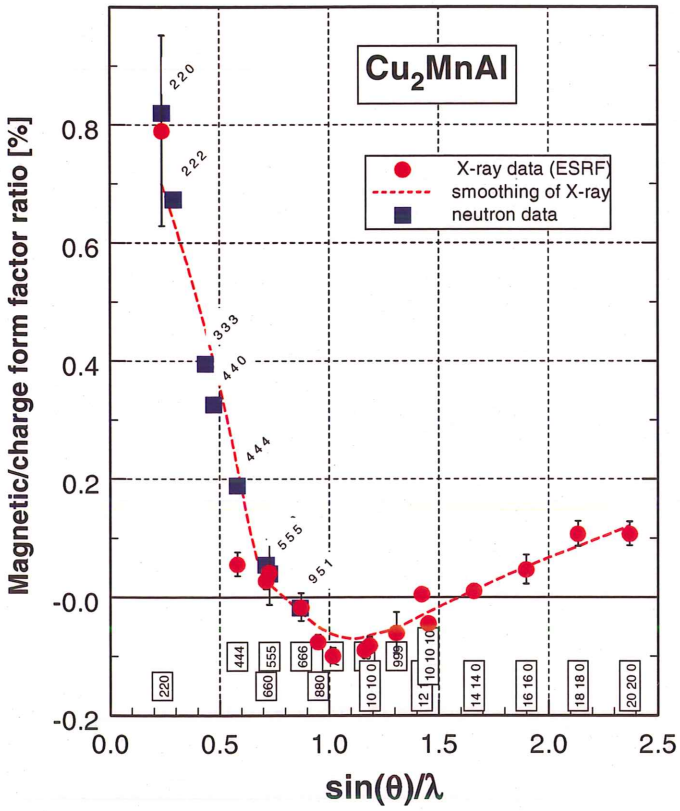
Magnetic form factor of Cu_2MnAl by white beam diffraction

M.J. Cooper, J. Duffy, M. Longfield, J. D. Wilmshurst, L. Dobrnzyski, E. Zukowski, S. Brown, A.Stunault - for further information contact M. J. Cooper at Dept. of Physics, Warwick University, Coventry CV4 7AL (m.j. cooper@warwick.ac.uk)

Data are presented here for the ratio of the magnetic and charge form factors for Cu_2MnAl, a ferromagnetic Heusler alloy with localised spin moments. Results were obtained from a white beam diffraction experiment at the XMaS beamline. This technique uses circularly polarised radiation obtained by going above/below the orbit so that an interference term is induced between the charge and magnetic scattering. The interference term can be isolated by periodically reversing the magnetic field and taking the difference. The ratio of the magnetic to charge form factors, $F_m(k)/F_c(k)$ deduced from the intensity change when the magnetic field is reversed is

$$\frac{I_+ - I_-}{I_+ + I_-} = \frac{E_x}{mc^2}\,\frac{P_c}{1-P_e}\,\frac{F_m(k)}{F_c(k)}$$

The results extend out to much higher values of $\sin(\theta/\lambda)$ than is possible using neutrons. Preliminary results are represented here with no corrections as yet applied. The errors for the measured X-ray data are weighted averages from several runs.



Experimental evidence for electron channelling in Fe/Au (100) multilayers

B. D. Fulthorpe, T. P. A. Hase, B. K. Tanner. P. A. Ryan, D. T. Dekadjevi, B. J. Hickey, S. Brown - for further information contact B. K. Tanner at Department of Physics, University of Durham, Durham. (b.k.tanner@durham.ac.uk)

Extensive structural studies have been undertaken at XMaS on epitaxial Au/Fe multilayers grown by molecular beam epitaxy on MgO and sapphire. These data have been used to interpret magneto-transport data on this system. From their analysis, we conclude that an electron channelling mechanism, due to strong specular reflection of the minority spin carrier at the Au/Fe interfaces, is responsible for the high conductivity in the (001) multilayers on MgO.

High resolution diffraction measurements showed good epitaxy in the MgO system, with sharp interfaces evident from the large number of satellite peaks visible in the reciprocal space maps.

A parabolic increase of the transverse satellite width with diffraction order was observed, as predicted theoretically for sharp, but rough interfaces.

Grazing incidence surface diffraction has been used to establish that the lattice perfection does not vary significantly with Au layer thickness in the (001) layers on MgO. Rocking curve widths (specimen scans about the surface normal) remained almost constant. Similar measurements on sapphire showed somewhat broader rocking curves (and six-fold symmetry due to the (111) orientation of the multilayer on this substrate).

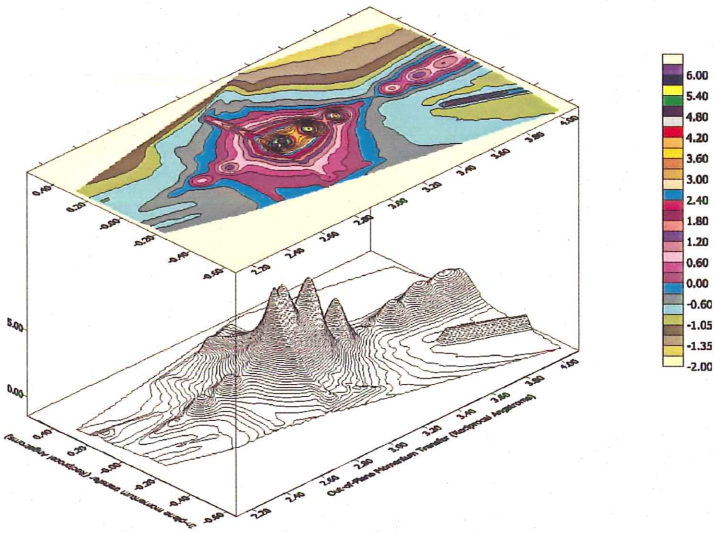


Fig. 1. Full reciprocal space map around the 002 reciprocal lattice point in Fe/Au

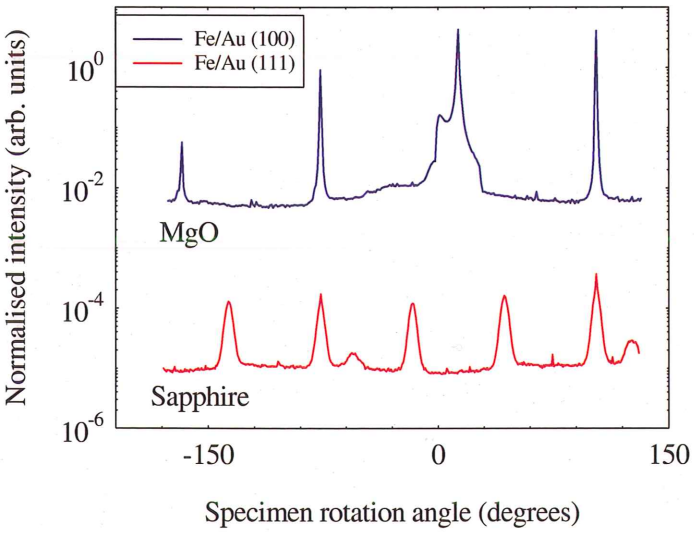


Fig 2. GIXD specimen rotation scan performed at the Au 220 diffraction condition, l=1.033Å.

Grazing-incidence X-ray diffraction from semiconducting polymer films

J. E. Macdonald, M. Durell, P. C. Jukes, M. Grell, R. A. L. Jones, S. Kawana, S. Brown, A. Stunault - for further information contact J. E. Macdonald at the Department of Physics and Astronomy, Cardiff University, Cardiff (macdonald@cf.ac.uk)

Electroluminescence from organic ultrathin layers has been explored for several decades but interest in conjugated polymers, attractive in view of their robustness and processability, has mushroomed since 1989 when Richard Friend et al generated yellow-green light from a poly (phenylene vinylene) (PPV) light emitting diode. The process relies on charge conduction via delocalised p-electrons along a molecular framework of alternating single and multiple bonds. PPV, formed by thermal conversion from precursors, offers little scope for control of the chain microstructure and alignment. By contrast, a recently developed alternative, poly (9,9-dioctylfluorene) (PFO) - a conjugated liquid crystal made up of an aromatic backbone of repeated linked benzene rings with long aliphatic side-chains - has a structure offering some ability for control through thermal processing. It emits light in the blue region of the spectrum.

Grazing angle diffraction techniques were employed to probe the surface and bulk regions of PFO samples. Two scattering geometries were employed which are sensitive to (A), surface normal and (B), in-plane correlations. PFO samples annealed at 130°C and 180-200°C, known from calorimetric measurements to have different liquid crystalline states, were investigated, (fig 1). The data indicate that the surface is more ordered in the nematic phase than in the crystalline phase, though, the bulk structure is more ordered in the crystalline phase. This apparent effect needs further investigation, particularly given the important role of the interface in potential electronic applications. Scans of the angle of incidence through the diffraction peaks appear to contain much information, (fig. 2) and further analysis is under way to elicit information about the degree of order as a function of depth.

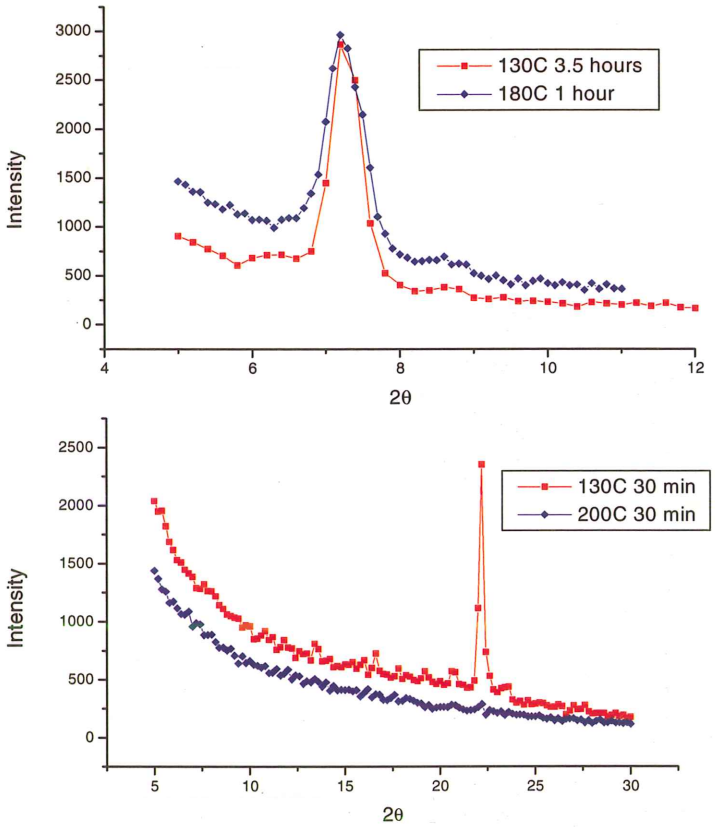


Fig. 1 Grazing incidence scans in geometries A and B for samples in the two liquid crystalline phases of PFO. The higher temperature phase shows nematic ordering whereas the lower temperature phase is smectic.

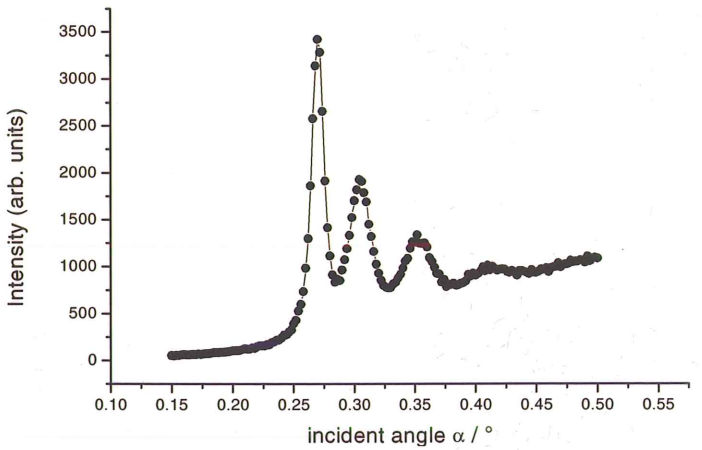
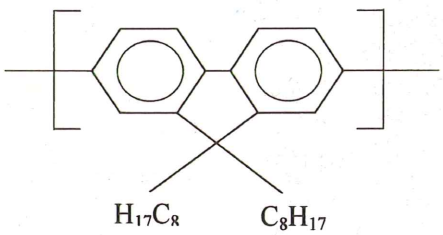


Fig. 2 Variation of peak intensity with angle of incidence for the peak at 7.2° (geometry A) in fig. 1. The oscillations arise from the film thickness and the overall intensity distribution depends on the depth of the ordered region relative to the surface.



The chain structure for poly(9,9-dioctylfluorene)

X-Ray voltammetry studies of the Au-Ni interface

C. A. Lucas, T. Nouar, J. Thornton, M. Ball, S. Brown - for further information contact C. A. Lucas at Department of Physics, University of Liverpool, Liverpool (clucas@liv.ac.uk)

Fig. 1. shows a 'cartoon' representation of the suggested interface between a Au(111) substrate and an electro-deposited Ni film. Earlier STM studies show that the Au(111) surface in a Ni-Watts bath (H_3BO_3, HCl, and NiSO), reconstructs with a (23 x $\sqrt{3}$) structure, with three equivalent rotational domains. The three domains form into a 'herringbone' pattern, with faults occurring at the elbows between domains. Other studies have revealed that Ni growth by electro-deposition, seeds at the elbow faults and extends across the surface as 'needles' until a Ni thin film is formed. With further deposition the Ni overlay regains its bulk structure. Very little is known, however, about the buried interface.

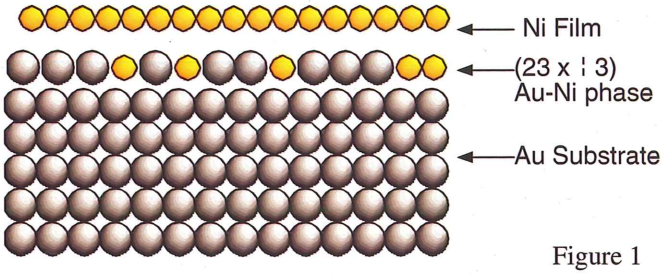


Figure 1

Fig. 2. shows the electrochemical cell used in the diffraction experiments. The reservoir can be filled with electrolyte during an experiment, via the two feed-throughs in both sides of the cell. The central column accepts a threaded collar that holds the crystal sample in place. A polypropylene film, secured over the top of the cell with a rubber O-ring, can be inflated/deflated by regulating the flow of electrolyte through the reservoir. The cell acts as a three-electrode system, allowing control of the electrochemical environment. In this experiment the cell contained a working electrode (the Au crystal surface), an Au ring counter-electrode and a Pd reference electrode.

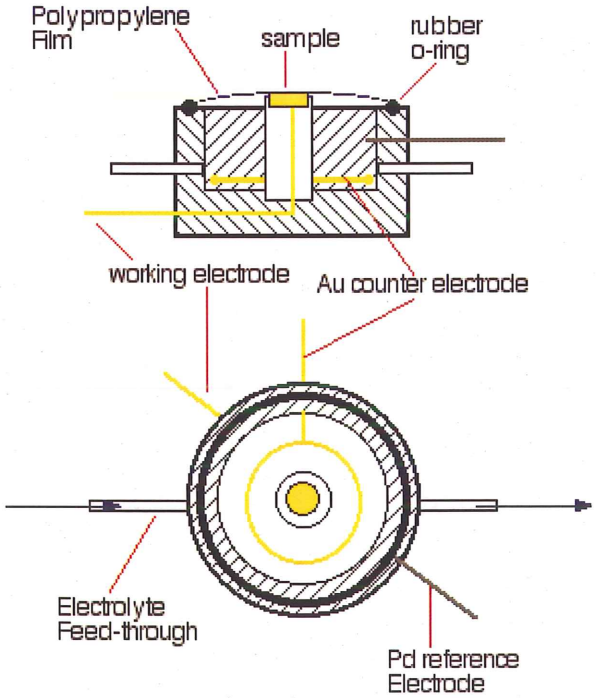


Figure 2

Fig. 3. shows a Cartesian projection of a mesh scan performed on a Au(111) crystal with an electrodeposited Ni over-layer. The peak at the centre of the plot is due to the crystal truncation rod and is surrounded by three satellites. In a hexagonal co-ordinate system they are equidistant from the central peak, and are located at the positions expected for the three rotational domains of the reconstructed (23 x $\sqrt{3}$) Au surface. This suggests a mixed Au-Ni structure is retained beneath the Ni over-layer.

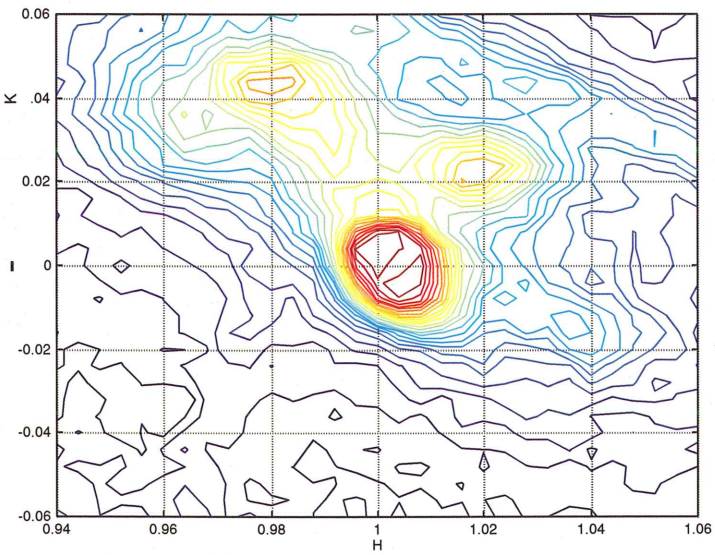
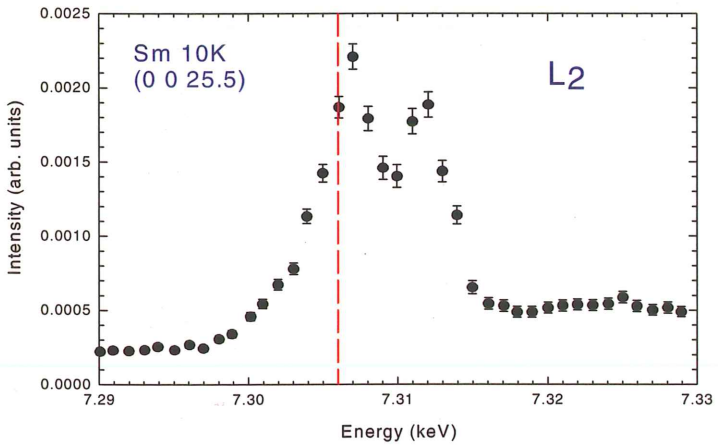


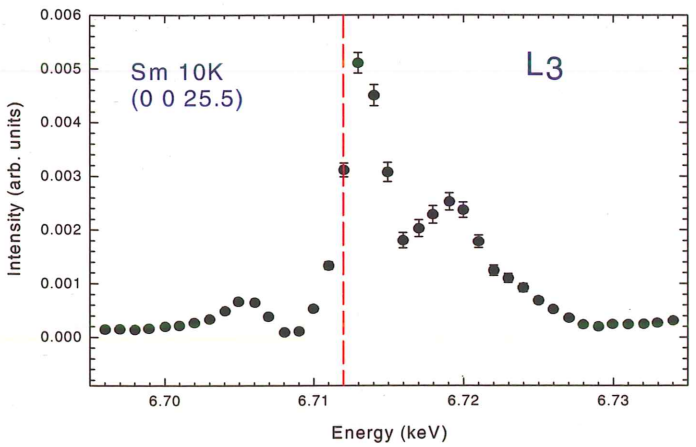
Figure 3

Resonant X-ray magnetic scattering from single-crystal Samarium

A. D. F.Herring, W. G. Stirling, N. Burton, M. M. R. Costa, A. Stunault - for further information contact W. G. Stirling at Department of Physics, University of Liverpool, Liverpool (stirling@liv.ac.uk)

The magnetic structures of single-crystal (bulk) samarium have been studied by resonant magnetic x-ray scattering. The energy dependence of the magnetic intensities is different for the L_2 and L_3 absorption edges (marked by vertical dashed lines). The L_3 edge has three magnetic resonances, a quadrupolar transition below the edge and two dipolar transitions above the edge. The L_2 edge has two peaks both due to dipolar transitions above the edge. Similar results have been observed from Sm thin film studies undertaken by A. Stunault et al. At present these results cannot be understood on the basis of atomic (resonance) theory.





News round-up

The experimental reports in the previous pages are all as yet unpublished. Please email the contact person if you are interested in any of them or wish to quote these results elsewhere.

New Web Site

At: http://www.esrf.fr/exp_facilities/BM28/xmas.html This contains the definitive information about the beamline including proposal/experimental report forms and an on-line beamline manual.

Living allowances

These remain at 350 Francs (53.3 Euros!) per day per beamline user, though actually paid in pounds sterling. XMaS will now support up to 4 users per experiment if you can make a case for the presence of the fourth experimentalist. The ESRF hostel, now comprising three blocks, appears adequate to accommodate all our users, though CRG users have a lower priority than the ESRF's own users. Do remember to complete the "A form," now web based, requested of you when you receive the ESRF invitation - all attendees must be listed since this is used to organise all site passes, meal cards and accommodation.

Mid-Term Review

EPSRC's review of XMaS takes place this summer. Thank you to everyone who helped by providing useful comments on our current operation. We hope that the release of the funds withheld pending review will allow us to continue to develop the facility. Looking ahead please note that we shall be organising a Users's Workshop in the Autumn to solicit your views on the beamline and the up-grade programme for the next two years. In 2001 we shall hold another meeting or meetings to get your help in formulating the proposal for a further 5 years (2002-2007) of XMaS activity.

Congratulations to Bill!

As many of you know Bill becomes the Director General of the ESRF from 1st January 2001. Clearly we shall see less of him at XMaS but he will remain involved as a co-investigator when there is no conflict of interest. Chris Lucas will maintain Liverpool's day-to-day involvement in the XMaS project.

Beamline people

There have been no changes in the beamline staff since the last Newsletter. Here is a brief update:

Project Co-ordinator - David Paul, (dpaul@esrf.fr), is the person who can provide you with general information about the beamline, application procedures etc. **NB the next deadlines for applications are Friday, April 14th, and Friday, October 13th.** The procedure is outlined on the last page of this newsletter. David should normally be your first point of contact.

Beamline Scientists - Simon Brown (sbrown@esrf.fr) and Anne Stunault (stunault@esrf.fr) share responsibility for helping the users as well as maintaining/developing the scientific capability of the facility. When you come to XMaS to perform an experiment one of them will be selected as your "local contact" - and would be the first person to turn to if things go wrong.

Technicians - Paul Thompson (thompso@esrf.fr) continues to provide the day to day technical support for the beamline. John Kervin (jkervin@liv.ac.uk) who is based at Liverpool University provides further technical back-up and spends part of his time on-site at XMaS.

Matt Longfield (longfiel@esrf.fr) and Jon Wilmshurst (wilmshurs@esrf.fr) are continuing their work on ferromagnetic diffraction and the polarisation conditioning of the beam (see the article on page 2) and both will leave XMaS this autumn.

Malcolm Cooper (csmc@spec.warwick.ac.uk) and Bill Stirling (stirling@liv.ac.uk) continue to travel between the UK and France and oversee the operation of the beamline. The administration for XMaS is handled by Sandra Beaufoy at Warwick University (sandra@spec.warwick.ac.uk); she receives the applications for beamtime and experimental reports during our proposal rounds. She also processes the travel/subsistence claims of our users - she is therefore a very important person!

The Peer Review Panel

The group that continues the splendid work of reviewing your applications for beamtime has been enlarged by the appointment of Sean Langridge (ISIS). It is chaired by Bob Cernik (Daresbury Laboratory) and its other members are Bob Cywinski (St Andrews) Jose Baruchel (ESRF) and Julie Staunton (Warwick Univ.). In addition either Malcolm Cooper or Bill Stirling attends their meetings. The group has a real job to do given the continued popularity of the facility.

Publish please, and keep us informed

Papers reporting work on XMaS are beginning to appear. We ask you to provide Sandra Beaufoy not only with the reference but also a preprint/reprint for our collection. Congratulations to Peter Hatton's Durham group for giving XMaS its first Physical Review Letter describing charge stripes in nickelates: it is currently in print. In addition studies of magnetic ordering in Sm thin films by the Nancy group together with Anne Stunault has been selected for the ESRF annual highlights publication.

When beamline staff have made a significant contribution to your scientific investigation you may naturally want to include them as authors. Generally, though, we do ask that you add an acknowledgement, of the form:

"This work was performed on the EPSRC-funded XMaS beam line at the ESRF, directed by W.G. Stirling and M.J. Cooper. We are grateful to the beam line team of S.D. Brown, D.F. Paul, A. Stunault and P. Thompson for their invaluable assistance, and to S. Beaufoy and J. Kervin for additional support."

Guidelines for Applying for Beam-time at the XMaS beamline

XMaS Pluo B3, ESRF, BP 220, 38043 Grenoble Cedex, France
Tel: +33 (0)4 76 88 24 36 Fax: +33 (0)4 76 88 24 55
web page : http://www.esrf.fr/exp_facilities/BM28/xmas.html email: dpaul@esrf.fr

Beamline Operation

The XMaS beamline at the ESRF, which came into operation in April 1998, has some 133 days of beam time available each year for UK user experiments, after deducting time allocated for ESRF users, machine dedicated runs and maintenance days. During the year, two long shut-downs of the ESRF are planned: 4 weeks in winter and 4 weeks in summer. At the ESRF beam is available for user experiments 24 hours a day.

Applications for Beam Time

Two proposal review rounds are held each year, with deadlines for submission of applications, this year, on **14 April** and **13 October** for the scheduling periods 1 August to 31 January, and 1 February to 31 July, respectively. An application form for beam time on the XMaS beamline is available from the XMaS web page (see above - click "Applying for Beamtime") and should be completed and sent to XMaS at :

Mrs S. Beaufoy
Dept. of Physics
Warwick University
COVENTRY CV4 7AL
UK

Tel. 02476 523965
Fax 02476 692016
e-mail: sandra@spec.warwick.ac.uk

It should be sent together with **12 copies** (reduced, 2-sided), i.e. a reduction by 70% to a double-sided single sheet A4 format. This is because we would like to have just one sheet per proposal to send to the Review Committee.

Technical specifications of the Beamline and instrumentation available are described in the *XMaS* web page.

When preparing your application, please consider the following:

- Proposals, which are submitted to a Peer Review Panel for appraisal, must be typewritten.
- All sections of the form must be filled in. Particular attention should be given to the safety aspects, and the name and characteristics of the substance completed carefully. Experimental conditions requiring special safety precautions such as the use of lasers, high pressure cells, dangerous substances, toxic substances and radioactive materials, must be clearly stated in the proposal. Moreover, any ancillary equipment supplied by the user must conform with the appropriate French regulations. Further information may be obtained from the ESRF Experimental Safety Officer, tel: +33 (0)4 76 88 23 69; fax: +33 (0)4 76 88 24 18.
- Please respect the space available on the form. Incomplete application forms may be rejected.
- Please indicate the dates that you would be unable to attend if invited for an experiment. This will help us to produce a schedule that is satisfactory for all.
- Please remember that the form will be reduced by 70% to an A4 format; additional sheets may not be forwarded for review. An experimental report on previous measurements must be attached to all subsequent requests for beam time. New applications will not be considered unless a report on previous work is submitted. Reports must be submitted within 6 months of the experiment - these should be sent to Mrs S Beaufoy at the address above. Forms for experimental reports are available from the XMaS web page – see above.
- The XMaS beamline is available for one third of its operational time to the ESRF's user community. Applications for beamtime within that quota should be made by direct submission of the normal ESRF application form to the ESRF - **Be aware, deadlines are earlier than for XMaS!** Applications for the same experiment may be made both to XMaS directly and to the ESRF. Proposals successfully awarded beamtime by the ESRF will not then be given beamtime additionally in the XMaS allocation.

Assessment of Applications

The Peer Review Panel for the UK-CRG considers the proposals, grades them according to scientific excellence, adjusts the requested beam time if required, and recommends proposals to be allocated beam time on the beamline.

Proposals which are allocated beam time must in addition meet ESRF safety and XMaS technical feasibility requirements.

Following each meeting of the Peer Review Panel, proposers will be informed of the decisions taken. If beam time has not been allocated, brief, general reasons only are given.