

Newsletter February 1999

Introduction/Status

The *XMaS* beamline has been in operation since April 1998, receiving visiting scientists from more than a dozen UK groups: in total 20 experiments had been completed on XMaS



The XMaS 11-axis diffractometer which has recently been augmented by the addition of a polarisation analyser.



THE UNIVERSITY of LIVERPOOL

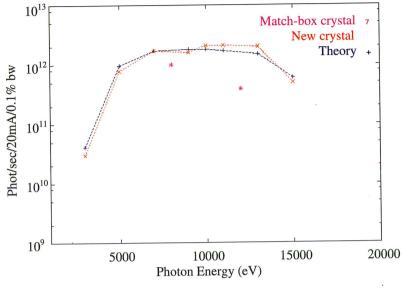
by Xmas(!). The beamline has a very full schedule for the half-year period just begun. In the pages that follow are some brief examples of the experimental work carried out to date. The development of the beamline continues and users can now benefit from the recently commissioned polarisation analyser. This follows an earlier, more general, improvement in the beamline's performance resulting from the replacement of the first crystal in the monochromator with one of a different design that resists better the thermal and pressure induced stresses tending to distort the diffracting surface. Its installation resulted in a gain in flux of approximately a factor of five at 10 keV and a halving of the rocking curve width to close to its theoretical value, reflecting the fact that the new crystal remains much flatter under normal operating conditions. A fuller description of its performance is given on the next page.

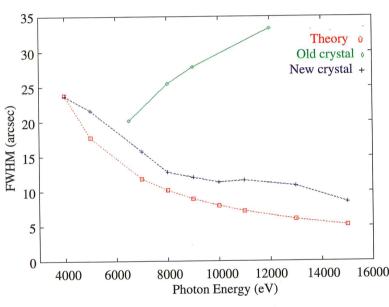


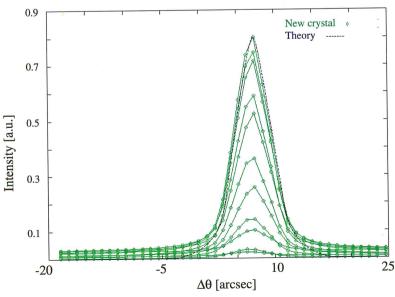
The new water cooled first crystal for the XMaS monochromator prior to installation. The cooling water is channelled through 22 cylindrical galleries running parallel to the beam.



Performance of the new *XMaS*Monochromator







The original Monochromator showed evidence of distortion due to both pressure and thermal effects. Its replacement, pictured on the front page, is an improvement in all respects.

Flux

Figure 1: The diagram to the left, illustrates one aspect of the marked improvement in performance provided by the replacement of the "match-box" design first crystal with the crystal. The blue "Theory" trace represents the calculated flux from 3.1 mrads width, 0.22 mrads height of incident beam passing through the beamline. It takes account of: - 0.85 mm thickness of Be; the reflectivity of the rhodium coating on the mirror; geometrical factors that limit the mirror to intercepting just 70% of the beam in the incident aperture and the reflectivity of the two mono crystals. The red data points represent measurements made with the new crystal installed. The purple points (2) are measurements for the original matchbox design.

Rocking curve width

Figure 2: This diagram plots the (convoluted) rocking curve widths at different energies for the two crystal designs for the full heat load and includes a comparison with the theoretical plot for a perfect crystal. It is clear that the bending of the diffracting surface of the matchbox design has severe consequences, particularly at higher energies. By contrast, the new design exhibits a performance that is close to that for a perfect crystal with the small amount of broadening at higher energies caused by the "thermal bump."

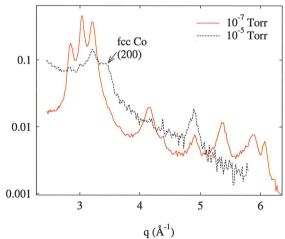
No pressure bump!

Figure 3: This diagram illustrates further the improvement over the old design. Data for the rocking curves was collected under very low heat load conditions. The procedure involved setting a small aperture in front of the first crystal and measuring the rocking curves for different vertical positions of this aperture. The curves stay centred, though they vary in amplitude in accord with the vertical intensity profile of the beam. This demonstrates that the diffracting surface is not bent by the excess pressure. In contrast the matchbox crystal produced rocking curves which were displaced by ±10 arcsecs between the centre and ends of the crystal due to the "pressure bump."

Measurement of Stacking Faults in CoPtCrTa Thin Film Recording Media

At present there is much interest in the effects of crystallographic defects in thin film magnetic recording media and their role in determining media noise and thermal loss of recorded signal. Stacking faults are common in thin film media, which can contain as much as 10% of Co in the non-ideal hcp phase. Accurate quantification of the percentage of stacking faults in thin film media is not trivial. Synchrotron radiation is employed with a grazing incidence geometry (GIXS) to eliminate the large background scatter from the underlayers and amorphous substrate. experiment at XMaS concentrated on characterising the stacking faults of a set of samples provided by Seagate Magnetics which had been produced at different sputtering pressures. Earlier qualitative HRTEM measurements had already provided evidence of increasing stacking fault density with rising sputtering pressure. This has been confirmed by the increased intensity of the scatter at the position of the fcc (200) reflection (see diagram). At present we are in the how? process of fitting the data to obtain a quantitative measure of the stacking fault density. The overall aim is to correlate the results of existing magnetic viscosity measurements with the obtained percentage of stacking faults.

Intensity (arb. units)



Horizontal 20 scans of thin film media samples produced at different sputtering pressures

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Surface Diffraction Studies of Ordering at PET Poly(ethylene terephthalate) Surfaces

The properties of polymeric surfaces and interfaces are central to many technologies: paints and coatings, adhesives, biocompatible materials, membranes etc. In contrast to the extensive data available semiconductors and other inorganic materials very little information is available for polymer surfaces.

This project is aimed at using grazing incidence x-ray diffraction (GIXRD) to probe chain conformation and ordering in the surface region to support or refute the ideas developed by theory and simulations. We used the XMaS beamline to perform GIXRD studies of 1000Å-thick film samples of polvethylene terephthalate (PET) deposited onto silicon substrates and annealed at a range of temperatures up to 180°C. The structure of the surface region (top 50Å) was compared with that of the bulk of the polymer by comparison of 20 scans with an angle of incidence of 0.15° (just below the critical angle for total reflection, 0.18°), with those performed using the steeper angle of incidence of 1.0°. We found that the topmost 50Å starts to crystallise at about 90-95°C whereas the bulk of the film starts to order at about 105°C. This opens the possibility of forming a crystalline surface on an amorphous bulk by simple thermal treatment. surprising feature of the results was that the scans in O₁ and O₂ showed different subsets of the crystalline PET Bragg peaks indicating a surface-induced molecular ordering throughout the film (fig. 1). This is consistent with the benzene rings in the PET chain aligning parallel to the surface. The implications of this result are currently being investigated further with AFM techniques at Cardiff.

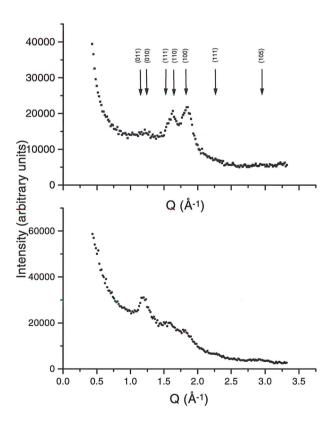
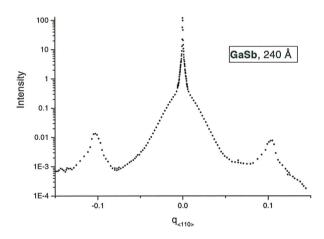


Fig. 1 GIXRD scans at an angle of incidence of 1° in (a) Q_{\perp} and (b) Q_{ℓ} showing preferred orientation of the PET polymer film due to interactions between the benzene ring and the surface.

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The Structure of GaSb Films

The growth of high quality heterostructures with large lattice mismatch (GaSb on GaAs) for use in optoelectronics has stimulated considerable interest in the dislocation structure and strain relaxation of epilayers. The only way to produce completely relaxed structures in such systems is to create a well-defined dislocation lattice confined to the interface. For a long time HREM has been a primary method of studying crystal growth and dislocations in epilayers. Nowadays x-ray synchrotron facilities allow the study of regular arrays of misfit dislocations by high-resolution X-ray diffraction. Measurements were recently made using the XMaS beam line on samples grown in Oxford by MOVPE techniques. Strained layers of GaSb were grown on GaAs (100) substrates. The scattering observed from one sample when the wave vector is scanned transversely to the (004) Bragg reflection is shown in the figure. The satellites surrounding the (004) reflection of GaSb are from a regular array of misfit dislocations. The sharp scattering at the (004) Bragg reflection shows that the atomic planes are flat on a length scale of 50,000Å while the origin of the broader component is unknown.



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Time Dependence of Spin Ordering in Holmium

At temperatures close to 17 K, the magnetic modulation wave-vector of holmium q(T) exhibits thermal hysteresis. We have carried out the first detailed study of the time dependence of q and developed techniques which allow observation of thermal activation of spin-slips under constant temperature conditions. Fig.1 shows the temporal decay of the q(t, T) = 1/6 peak on heating from 8.5 K. This plot clearly shows the strongly time dependent nature of the magnetic structure. There are important instrumentational implications associated with this time dependence as conventional experimental

techniques will show transitions occurring temperatures which are determined measurement time. A phenomenological model has been developed in which it is assumed that switching occurs through thermal activation of spin-slips over energy barriers formed by local interactions at the position of the slips. This model predicts that $ln(t)=E_b/k(T-T_c)$, where E_b is the local energy barrier, k is Boltzmann's constant and Tc is the critical temperature for the transition. The data presented in Fig 1 were analysed with use of this model. Fig 2 shows the results of fitting the data to the above function at various constant values of I / I₀. The fitted values for T_c of around 17.3 K are time independent and correspond to an anomaly observed in specific heat measurements. The fitted value for E_b of around 0.4 meV corresponds to the magnitude of the inter-planar exchange energy in holmium which is precisely the energy barrier associated with the product of a spinslip. In addition, the forms of the derivatives of each of the curves in Fig 1 clearly indicate the onedimensionality of the growth of spin-slip regions.

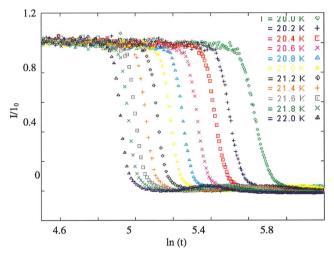


Fig.1 Temporal decay at the q=1/6 modulation wavevector

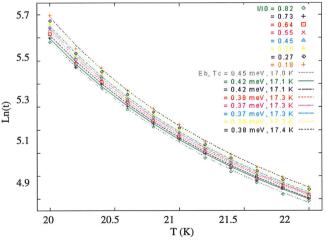
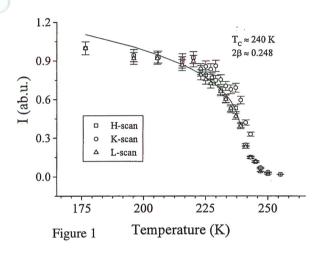


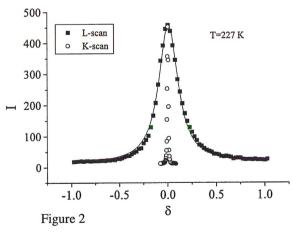
Fig.2 Fitted $ln(t)=Eb/k(T-T_c)+C$ curves

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Charge Stripes in La_{5/3}Sr_{1/3}NiO₄

Using x-rays of wavelength 1 Å the sample was oriented with the scattering vector parallel to [101] and scans were performed along H, K, and L axes in reciprocal space. The satellite reflections were located at $(h\pm 2\varepsilon, 0, l)$ where $\varepsilon = l$, h is even l is odd. The charge stripes are shown to be two dimensional in nature both by measurements of their correlation lengths ($\xi_a = 110 \text{ Å}$, $\xi_b = 130 \text{ Å}$ and ξ_c = 17 Å) and by the critical exponent of the charge stripe transition. No long range order was observed, even at low temperatures, indicating that they are disordered and quenched below the charge ordering temperature which is 240K. Figure 1 shows a plot of the integrated intensity of the (4.66, 0, 5) peak as a function of temperature. It can be fitted by a power law with exponent 0.248(3) showing the charge stripes to be 2-dimensional. Figure 2 shows K and L scans below the charge ordering temperature; the difference in width is easily apparent.

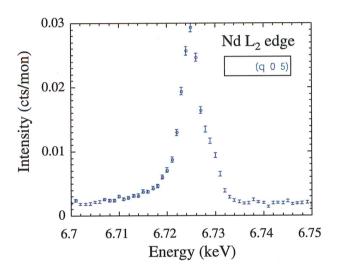


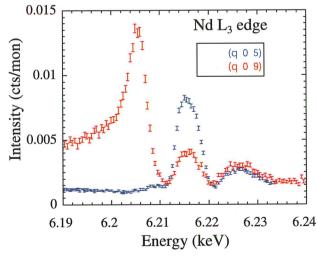


C-H Du, Y Su, J. P. Allen, P. D. Hatton, S Brown and S-W Cheong: for futher information contact P.D. Hatton in the Department of Physics at Durham University (p.d.hatton@durham.ac.uk)

X-Ray Resonant Exchange Scattering in Neodymium

The shape of the resonant magnetic response at the L_{II} edge in Nd metal appears comparatively simple - a single peak with a tendency to a shoulder on the high energy side (see Figure 1). It is clear from our earlier work [PRB 57, R8095, (1998)] that this is an almost 'dipole' response, arising from the induced magnetism of the d-electrons. However, the weaker L_{III} response shows a multi-peak structure (see Figure 2), which must arise from the interference of more than one process. The q-dependence of the lowest energy peak probably indicates that it is a 'quadrupole' response to the f-electron magnetism. The excellent degree of polarisation of the beam, and the availability of the polarisation analyser for the scattered photons will allow us to check these ideas, and give a much fuller picture of how the magnetic and atomic properties of neodymium are related to its X-ray magnetic response.





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Magnetic Structure of DyCu2 and GdCu2

Here we present the result of recent resonant magnetic x-ray scattering experiments on $GdCu_2$ and $DyCu_2$. The samples were cooled to 10K and the experiment, performed in vertical scattering geometry, used polarisation analysis (PA) of the scattered beam for the first time ever on XMaS. PA allows the magnetic $(\sigma - \pi)$ and charge $(\sigma - \sigma)$ scattering to be separated. It also reduces the background level, which results from the high fluorescence signal. The main results are:

(1) The magnetic phase diagram of DyCu₂, as deduced from the resonant magnetic X-ray scattering experiment, is consistent with the one suggested from old neutron scattering results, i.e. an incommensurate phase at high temperatures, followed by a commensurate magnetic phase with a propagation vector of $\mathbf{q} = (\frac{2}{3}, 1, 0)$ at low temperatures. The high q-resolution allowed the variation of the propagation vector with temperatures in the incommensurate magnetic phase to be investigated precisely.

(2) The magnetic structure of $GdCu_2$ (magnetic propagation vector $q=(\frac{2}{3}+\epsilon,\ 1,\ 0)$ as well as the temperature variation of the magnetic intensity were determined precisely for the first time. The propagation vector varies smoothly over a wide temperature range and no lock-in transition into a commensurate phase was observed down to T=10K. The good temperature stability of the diffractometer and its versatility facilitated the measurement of the propagation vector and the integrated intensities at various temperatures and at about 30 different Bragg positions. This will allow us to determine the moment direction.

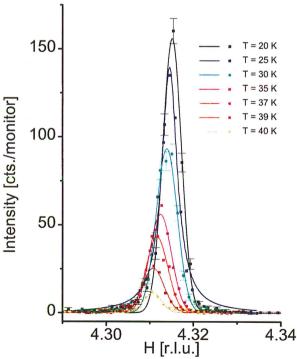


Fig. 1: Temperature dependence of the magnetic propagation vector $q = (5-h\ 1\ 0)$ and the magnetic intensity. An ordering temperature of $T_N = 40.5 K$ has been deduced

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Magnetic Structures and Phase Transistions of UAs-USe Solid Solutions

Scattering from the (0, 0, 6) charge reflection has revealed a distortion of the lattice of single crystal UAs_{0.82}Se_{0.18}. See Fig 1. At 134K this material is paramagnetic; scans about the (0, 0, 6) position show a single sharp Bragg reflection, and hence the crystal lattice has cubic symmetry. At lower temperatures the Bragg peak 'splits'. This is considered to arise from two different lattice reflections, which correspond to the a and c lattice parameters. A single magnetic domain of the 2k magnetic structure may exhibit a distortion along the caxis since the magnetic moments are orientated within the ab plane. A fit to the low temperature reflection gives a distortion of $\delta c = 0.0014c$. In comparison, the tetragonal lattice distortion for UAs is 0.0015c as measured by McWhan et al. The larger scattering vector (the higher positioned peak in reciprocal space) about the (0, 0, 6) position corresponds to scattering from the a and the b lattice parameters. The smaller scattering vector corresponds to the c lattice parameter. The larger scattering vector is more intense than the lower scattering vector, and thus c/a > 1. A contour plot of the lattice distortion at 12K taken around the (0, 0, 6) position which suggests tetragonal symmetry is shown in Fig 2.

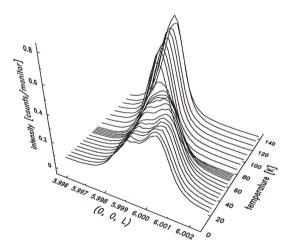


Figure 1: Scans of the (0, 0, 6) lattice reflection upon lowering the temperature of single crystal UAs_{0.82}Se_{0.18}.

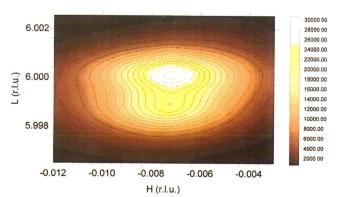


Figure 2: A contour plot of the (0, 0, 6) lattice reflection taken at a temperature of 16K

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First Surface Diffraction Experiments on the XMaS beamline.

An important application of the XMaS diffractometer was envisaged to be ex-situ surface X-ray diffraction experiments. Such measurements, usually undertaken in UHV, are particularly valuable in probing the inplane structure of thin multilayer structures only a few nanometres in thickness. We have found that the large number of highly stable axes on the XMaS instrument makes these experiments extremely straightforward to perform. Our first data were emerging within a few hours of starting to set up. A series of epitaxial Fe/Au multilayers grown by molecular beam epitaxy on either a MgO substrate with an Fe seed layer (showing high magnetoresistance), or a sapphire substrate with a Nb buffer (showing low magnetoresistance) have been studied. In grazing incidence diffraction, the scattering planes are perpendicular to the plane of the film and measurements are sensitive to in-plane strain and atomic disorder. It was necessary, therefore, to utilise the horizontal scan axes of the diffractometer with accurate phi rotation, the whole diffractometer having been tilted to give an incident beam always below the critical angle for total external reflection. Well defined 4- and 6- fold symmetries show the Au layer to be

deposited with good (100) and (111) epitaxy in the MgO and sapphire systems respectively. Sharp, high intensity, peaks indicate little in-plane crystalline disorder. In contrast, scans performed at the Fe 022 reflection for both systems show the crystalline quality of the deposited layers in both systems to be poor and of comparable perfection in each case. The sharp peaks in the Fe diffraction scan are due to the overlapping Au diffraction peaks. Analysis of allowed reflections shows a mixture of fcc and bcc Fe present in the multilayers grown on sapphire. Strong Nb 011 peaks were detected in the sapphire system, (inset to the figure), even though the Nb buffer layer is well beyond the depth penetration of the incident beam. The symmetry indicates (111) oriented epitaxial growth, similar to the Au layers, in contrast to RHEED measurements which show that the Nb buffer layer is deposited with a (110) epitaxy. Grazing incidence fluorescence measurements, performed at Durham, confirm the presence of Nb at the multilayer surface, suggesting a possible surfactant role for Nb.

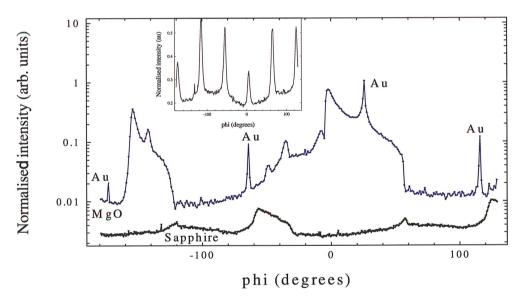


Figure 1. Grazing incidence surface diffraction measurements performed at the Fe 022 reflection for both systems. The sharp diffraction peaks are due to overlapping Au diffraction peaks. The strong Nb 011 diffraction peaks of the sapphire system are shown in the inset.

B.D.Fulthorpe, S.B. Wilkins, T.P.A.Hase, J.Clarke, B.K.Tanner, P.A.Ryan, D. T. Dekadjevi, B.J.Hickey – for further information contact B.K. Tanner at the Department of Physics, University of Durham (B.K.Tanner@durham.ac.uk)

News round-up

The experimental reports in the previous pages are all as yet unpublished. If you are particularly interested in any one of them please email the contact person. If you wish to quote these results elsewhere please obtain their approval first.

Living allowances

In line with ILL we have raised our per diem for beamline users to 350 Francs (53.3 Euros!) per day. It is still paid in good old pounds sterling. So far all our users have been accommodated in the ESRF hostel (CRG users have a lower priority than ESRF's own users) and, with the opening of the new C Block, we anticipate no problems with getting you on site, but David Paul needs to know your plans as far ahead as possible.

Beamline people

There have been some changes in the beamline staff since the construction phase. Here is a brief update:

Project Coordinator - David Paul (dpaul@esrf.fr). David is the person who can provide you with general information about the beamline, application procedures etc. **NB the next deadline for applications is April 16th** and the procedure is outlined on the last page of this newsletter. David should be your first point of contact; because he was responsible for the beamline's detailed design he knows everything about it!

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. If you come to XMaS to perform an experiment you will be "looked after" by one of them.

Technicians - Paul Thompson (thompso@esrf.fr) provides the technical support for the beamline and is therefore the first person to turn to if things go wrong - which, of course, they hardly ever do on XMaS! John Kervin (jkervin@liverpool.ac.uk) who is based at Liverpool University provides further technical back-up and spends part of his time on-site at XMaS.

Recently two new faces have appeared in PLUO B3 Matt Longfield (longfiel@esrf.fr) and Jon Wilmshurst (wilmshurs@esrf.fr) they are working on ferromagnetic diffraction and the polarisation conditioning of the beam which in plain English means they have until Autumn 2000 to find out how to install a phase plate and use it to help us study ferromagnets.

In addition, flitting between the UK and France, Malcolm Cooper (csmc@spec.warwick.ac.uk) and Bill Stirling

(stirling@liv.ac.uk) oversee the operation of the beamline. All the administration for XMaS is handled Sandra Beaufoy at Warwick University (sandra@spec.warwick.ac.uk); in particular she processes the travel/subsistence claims of our users. She is therefore a very, very important person. Finally David Laundy and Dave Bowyer have now returned to their old habitats in the UK, thereby reducing the excess of "Davids" on our beamline, but we are pleased to see them returning here from time to time. Finally Bruce Forsyth (aka Father Christmas) has retired but also returns to haunt us.

The Peer Review Panel

The group that reviews your applications for beamtime 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 popularity of the facility. The approach that they have taken to allocating time will be up for discussion at the forthcoming workshop - see below.

Stop Press!

XMAS Workshop: If you have not heard already there is a one-day workshop for current and intending XMaS users at Warwick on Tuesday 16th March beginning at coffee time (11:00). Its main purpose is to discuss the continuing development of the beamline in the light of its current performance. We want to know what you think the priorities should be. Bob Cernik, who chairs the Peer Review Panel that assesses your applications will also explain how they are dealing with the applications for beamtime and seek your comments. If you are not yet signed up for it please contact Sandra Beaufoy (there is no fee and we shall be able to reimburse "reasonable" travel costs for attendees.

Publish Please!

We look forward to seeing more and more of your XMaS research in print. When our facility is reviewed it will be refereed papers, not newsletters, that will ensure a rosy future for XMaS and the same is, of course, true for your prospects in the RAE.

When beamline staff have made a significant contribution to your scientific investigation you may naturally want to include them as authors. Generally we ask that you simply 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

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 adlines for submission of applications on 16 April and 16 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 (available from XMaS at the address above in paper form and also as a Microsoft Word file on the ESRF's ftp server: ftp.esrf.fr in the directory /tmp/xmas) should be completed and sent to XMaS at:

> Mrs S. Beaufoy Dept. of Physics Warwick University COVENTRY CV47AL UK

Tel. 01203 523965 Fax 01203 692016

e-mail: sandra@spec.warwick.ac.uk

It should be sent together with 12 copies (reduced, 2sided), 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.

specifications of the Beamline Technical instrumentation available are described in the ESRF Beamline Handbook, which can be consulted on the World Wide Web at www.esrf.fr.

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 Experimental conditions completed carefully. 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 will not be forwarded for review. An experimental report on previous measurements must be attached to all subsequent requests for beam time. 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 XMaS at the address above and also the ftp server at the ESRF - 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. 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 beamtime additionally in the 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.