# New Developments at the *XMaS* Beamline For Magnetic and High Resolution Diffraction

P.B.J. Thompson[1], L. Bouchenoire[1], S.D. Brown[1], D. Mannix[1], D.F. Paul[1], C. Lucas[2], J. Kervin[2], M.J. Cooper[3], P. Arakawa[4], G. Laughon[4]

*[1.]XMaS, The UK-CRG, ESRF,BP220, F-38043 Grenoble CEDEX, France.*
*[2] Dept of Physics, University of Liverpool, Liverpool, United Kingdom,*
*[3] Dept of Physics, University of Warwick, Gibbet Hill Road, Coventry, CV4 7AL, United Kingdom,*
*[4] American Magnetics Inc, P.O. Box2509, 112 Flint Road, Oak Ridge, TN 37831-2509, USA*

**Abstract.**. We report here on a number of developments that include enhancements of the sample environment on the *XMaS* beamline and the flux available at low energy. A 4 Tesla superconducting magnet has been designed to fit within the Euler cradle of a six circle Huber diffractometer, allowing scattering in both horizontal and vertical planes. The geometry of the magnet allows the application of longitudinal, transverse horizontal, and vertical fields. A further conventional magnet (~ 0.1 T) to minimize air absorption at low energies (~ 3KeV) has been designed for two circle applications, such as reflectivity. A novel in-vacuum slit screen has been developed, also minimizing absorption at low energies. New equipment for performing in-situ studies of surfaces in the electrochemical environment has been developed to allow control of the solution and sample temperature over the region of -5C to 80C. Preliminary experiments on the surface reconstructions of Au(111) in an electrolyte have been performed, whilst commissioning at the same time a MAR CCD detector for the beamline.

## INTRODUCTION

The *XMaS* beamline [1,2] at the European Synchrotron Radiation Facility (E.S.R.F.) has been designed to perform high resolution diffraction and magnetic scattering since April 1998. A comprehensive description of the beamline has been given by Brown *et al*. (2001)[2].

During recent years, much emphasis has been made to increase the range of sample environments on the beamline. A 4 Tesla superconducting magnet is being designed. Variable temperature electrochemical cells have been developed that can be operated within a magnetic field to explore the solid-liquid interface under these interesting conditions. Our detector capability has been augmented with the recent arrival of a MAR CCD two dimensional detector. Significant reduction in air path attenuation for certain low energy experiments has been achieved, e.g. an-in vacuum / cryostat has been built for reflectivity experiments..

## RECENT DEVELOPMENTS

### Sample Environment

#### *Superconducting Magnet*

American Magnetics, Inc. has designed a superconducing split coil magnet system to be used for synchrotron x-ray diffraction experiments. The magnet system is designed to operate without the use of liquid helium (i.e., cryogen free). The superconducting magnet is wound of twisted multifilamentary Niobium-Titanium (NbTi) superconductor embedded in a copper matrix. Twisted filaments maximize magnetic stability and minimize magnetic hysteresis. The former for the magnet coil is constructed of non-magnetic stainless steel alloy. Quench protection diodes are mounted with the magnet. The magnet cryostat is manufactured of non-magnetic stainless steel and aluminum and provides up to 180° radial access to the central field. High temperature superconducting (HTS) current leads are installed to energize the magnet. A commercial Sumitomo two stage, closed-cycle refrigerator is used to cool the magnet system. The cryostat internal support structure is designed to maintain the position of the magnet while the cryostat is oriented in varying positions. The support structure must be capable of providing lateral, longitudinal and torsional support while minimizing thermal loads to the magnet. The variable temperature insert is based around a standard two-stage displex, capable of reaching 10K. However, a third stage has been developed by the cryogenics group at the I.L.L., in Grenoble. This novel device is capable of operating down to 1.7 K using $^4$He and 1K if $^3$He is used. It may also be operated over a wide range of angles without a degradation of the base temperature. The rated field of the magnet at an operating temperature of 4.2 K is expected to be 4 Tesla, with a homogeneity of +/-1.0 % in a 1 cm diameter spherical volume at an approximate operating current of 75 Amperes.

The *XMaS*/AMI superconducting magnet has been designed to fit within the Euler cradle of the Huber diffractometer and allows versatile field orientations as shown in Figure 1. The geometry allows the magnet to be turned along the vertical axis through 90 degrees, facilitating application of magnetic fields both along and transverse to the incident beam direction. An efficient yoke configuration occupies the lower half of the vertical scattering plane, leaving the other half open for the cryostat and scattered beam. The design of the coil former has been optimized to allow for a maximum number of turns within the geometrical constraints of the diffractometer.

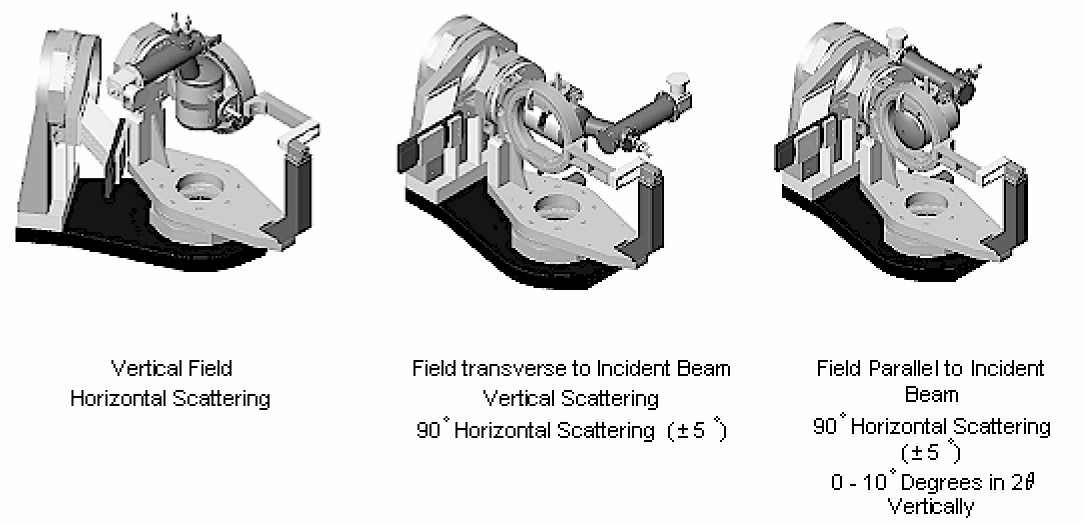


**FIGURE 1.** The *XMaS*/AMI superconducting magnet mounted within the Euler cradle on the Huber diffractometer, illustrating the three possible scattering geometries.

*Electrochemical Sample Environments*

A new trend in the study of surfaces is, away from the traditional ultra-high vacuum (UHV) environment, to explore surface phenomena in more complex environments that are directly related to technological applications and thereby gaining a fundamental understanding of ‘real’ surface processes. The *XMaS* beamline is ideal for pursuing these studies due to the flexibility of the diffractometer, both in terms of providing a range of possible scattering orientations and accommodating ‘custom-built’ sample chambers. The general experimental procedures used in x-ray diffraction measurements of electrochemical systems have been described in detail previously [3]. Figure 2 shows the modified cell that we have designed to incorporate temperature control of the sample environment into the experimental set-up. This incorporates a water-cooled Peltier device attached to the base of the electrochemical cell. A thermocouple, in close proximity to the liquid reservoir in the cell, allows control of the temperature over the range, -5°C - 80°C.

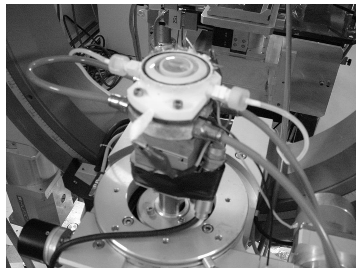

**FIGURE 2** Peltier cooled electrochemical cell mounted onto the *XMaS* diffractometer

Preliminary experiments using the cell have featured Au(*hkl*) single crystal electrodes, in particular, the lifting and forming of the surface reconstructions of Au(111)-(23x√3). In addition we have recently studied the growth of Cu thin films on the Au(111) surface as part of the commissioning of a Mar CCD detector on the beamline. The use of an area detector for this work enables time-dependent studies whereby the growth of the Cu film can be examined in real time by taking ‘snap-shots’ of the diffraction from the surface as the electrodeposition proceeds. Electrodeposition of Cu onto Au is a fully reversible process that is controlled by the applied electric field and so several growth/dissolution cycles can be studied in one experiment. Figure 3 shows two images of the scattering measured at the reciprocal lattice position (0, 0, 3.385) (in the conventional hexagonal reciprocal space notation used for the Au(111) surface).

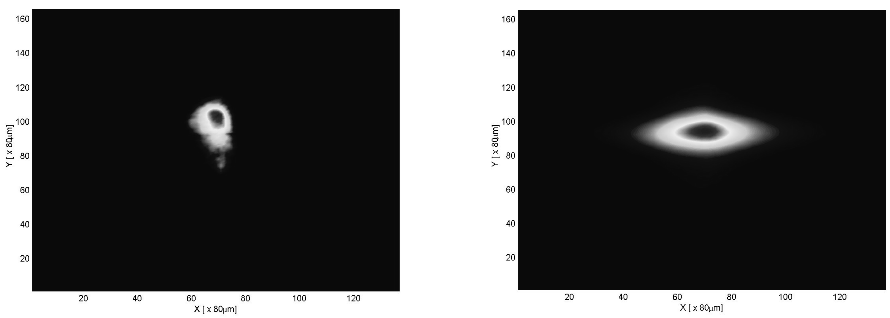


**FIGURE 3** Two images of the scattering measured at the reciprocal lattice position (0, 0, 3.385) for the Au(111) surface. The image to the left shows scattering from the Au (111) surface crystal truncation rod and the image to the right shows the effects of the copper deposition.

At this reciprocal lattice point there is scattering from the crystal truncation rod [8] of the Au surface and this is observed prior to Cu deposition (left panel). After deposition of Cu the scattering changes dramatically as this is the reciprocal space position where diffraction from Cu(111) planes also occurs. The spot profile becomes extended in the horizontal plane consistent with diffuse scattering from the Cu deposit. These two images represent the two end points (clean surface and Cu film deposited) of a complete data set which was taken with a 10 second time resolution both during the growth of the Cu film and during Cu dissolution.

## Low Energy Optimization

### *In-Vacuum Magnet-Cryostat*

In order to allow the application of a magnetic field at low temperatures and remove entry and exit window absorption from the beam path an in-vacuum magnet-cryostat has been designed and fabricated. The main body of the vacuum vessel has been constructed from aluminum and is supported on the *XMaS* XYZ mount designed for a 410 Huber circle [4] to allow for precise sample alignment. To ensure efficient cooling, the coil of the electro-magnet is mounted ex-vacuum, below the vacuum vessel. The magnet yoke, which is made from ARMCO grade iron passes through the base of the vacuum vessel, sealed by double O-rings. Inside the vacuum vessel, the tips of the adjustable magnet pole pieces support a copper sample stub via three ceramic balls to minimize thermal losses. An ARS DE202 displex is mounted on the Huber 512 chi cradle with rubber dampers to minimize vibration from the cold head. Sample cooling is facilitated by a very short copper braid from the tip of the second stage of the cryostat to the sample stub. A radiation shield, which extends to a few mm above the sample is also mounted to the first stage of the displex. The incident and outgoing beam paths consist of two large bellows, terminating in ISO-KF 40 flanges. These allow scattering angles of 30 degrees in theta and 60 degrees in two-theta.

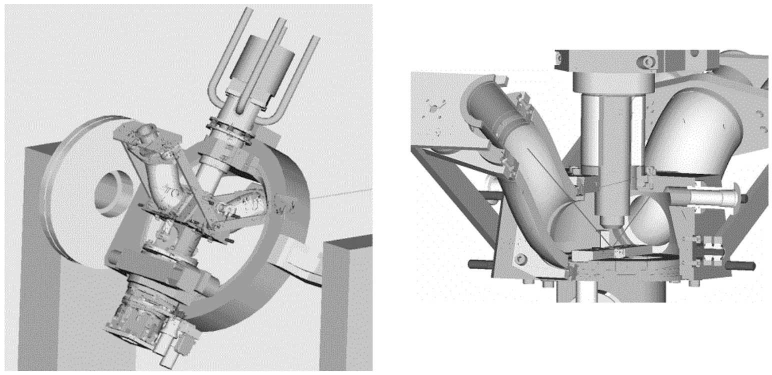

**FIGURE 5** Schematic diagrams of the in-vacuum magnet-cryostat. The beam path and magnet pole pieces can be clearly seen in the figure to the right. .

*XMaS In-Vacuum Slit Screen*

The principal design criterion for this slit screen was to produce a small compact assembly that does not exceed the physical dimensions of the vacuum flight tubes employed on the *XMaS* beamline. The assembly also had to be compatible with the *XMaS* "tube slits" [4]. The independent actuation of the four tungsten blades is achieved through the use of miniature in-house designed vacuum feedthroughs, allowing the motors to be mounted externally. The maximum opening aperture of this screen is 12mm × 12 mm and each jaw can be independently positioned to within 2μm. All of the axes have limit switches to prevent mechanical damage.

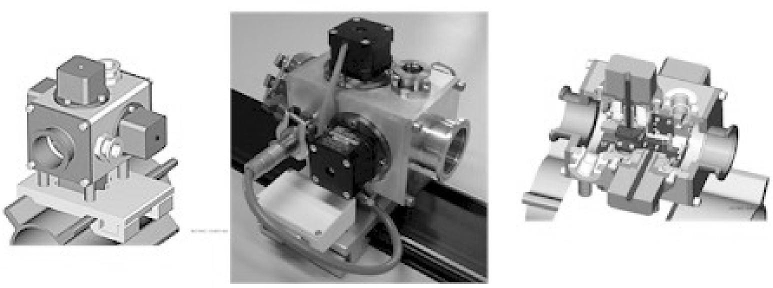

**FIGURE.6** *XMaS* in-vacuum slit system

# CONCLUSIONS

The new *XMaS*/AMI superconducting magnet will provide a 4 Tesla magnetic field that can be used in a variety of scattering geometries and can be used with a sub 2 K cryostat. Due to the novel design of this magnet, a very large area of reciprocal space will become available in this very interesting sample environment. A MAR CCD detector has been installed onto the beamline and has commissioned by performing in-situ real time electrodeposision experiments. The beamline has been optimized for low energy experiments, this has been achieved by commissioning a miniature in-vacuum slit screen and vacuum cryostat-magnet.

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