Institution: University of Liverpool

Unit of Assessment: 9 - Physics

Title of case study: XMaS: Commercialising innovative X-ray instrumentation and providing research and development opportunities in emerging technologies

1. Summary of the impact (indicative maximum 100 words)

This case is primarily based on the economic benefit derived from commercialisation of intellectual property arising from our research programme in materials at the XMaS beamline at the European Synchrotron Radiation Facility at Grenoble. The company *Huber Diffraktiontechnik GmbH and Co. KG* have had direct commercial benefit from exploitation of instrumentation we have developed, in collaboration with Warwick University, to address the specific research challenges described below. A second, indirect, impact of XMaS is knowledge transfer through the career progression of trained specialists in positions at other large scale science facilities and in the private sector.

2. Underpinning research (indicative maximum 500 words)

In the late 1980s the community interested in actinide magnetism and the recently discovered phenomenon of x-ray resonant magnetic scattering (XRMS), highlighted a need for an x-ray synchrotron beamline facility for the investigation of magnetic structure and associated phenomena in single crystals. To maximise impact in actinide materials it was identified that the higher flux and overall beam characteristics of a third generation source were required and so the facility should be based at the European Synchrotron Radiation Facility (ESRF).

An EPSRC award was made to Professor Stirling (University of Liverpool) and Professor Cooper (University of Warwick) to design, build and commission a flexible beamline to exploit an x-ray energy window between 3 and 20 keV. The design consisted of a non-magnetic six circle diffractometer with a standard suite of sample environments and detectors suitable for exploiting the high flux and beam polarisation to study magnetic materials. The design of the optical components (mirrors and monochromator) as well as any air-paths was optimised for low energy XRMS operations to facilitate actinide studies. As the polarisation of the incident beam is a critical parameter for the study of ferromagnetic materials, it was quantified during the early build phase by measuring the ratio of magnetic to charge form factors of nickel. These measurements showed that the design and beamline characteristics were suitable for studies of a wide range of magnetic materials [1]. This beamline facility is known by the acronym XMaS (X-ray Magnetic Scattering).

Due to the robust and flexible design of the beamline described above, the user community (including Liverpool academics Lucas and Stirling) expanded rapidly to include new scientific areas and challenges beyond actinide magnetism. These experiments used the beamline in different ways and highlighted areas that could be optimised and made more efficient through better control of the sample position and slit defined beam paths.

This led to a research and development programme involving University of Liverpool research staff (Brown, Thompson, Bouchenoire, Kervin) and Liverpool academics (Lucas, Stirling) to design, develop, test and commission new components for performing diffraction experiments. These developments have been published in a series of articles in peer-reviewed journals [1-5].

These include the development of “tube slits” for reducing background and the XYZ cryostat mount for highly accurate and efficient sample positioning during “beam-on” conditions [2]. New sample environments such as magnetic fields (1 T electromagnet and 4 T superconducting magnet), wet chemical cells [3] and the ability to apply electric fields at low temperatures (1 K) were also designed, completing phase II (2002-7) of the XMaS project.

In XMaS Phase III (2007-12), it was investigated how the operational energy could be lowered to 2.3 keV. This involved developing and researching new evacuated sample environments, thinner Be windows and new detectors. For the first time, attenuators compatible with UHV operation had to be developed to overcome absorption problems [4]. New methods for controlling the incoming polarisation of the beam, back compatible with previous developments, were also required. Through the development of the phase plate and subsequent development of the PE loop system on XMaS, a strong collaboration with the National Physical Laboratory (NPL) was formed [5]. As a result of this collaboration, Liverpool is now a project partner with NPL on a European Metrology Research Programme to study novel electronic devices based on control of strain at the nanoscale in multiferroic and ferroelectric materials.

The continual developments throughout the XMaS project now mean that it is a facility for the study of a broad range of material science. The collaborative project is on-going having been refunded recently as an EPSRC mid-range facility allowing continuation of research on new capabilities and projects [6].

The XMaS beamline is jointly managed and operated by the Universities of Liverpool and Warwick. Staff on the project from the Department of Physics at the University of Liverpool are: Project Directors; W. G. Stirling (1991-2000), C. A. Lucas (2001-), Research Staff; A. Stunault (1997-2000), S. Brown (1997-), P. Thompson (1997-), L. Bouchenoire (2002-), D. Mannix (2001-2007), P.S. Normile (2008-2010), D. Wermeille (2011-), J. Kervin (1997-).

3. References to the research (indicative maximum of six references)

[1]. *Magnetism in nickel and Synchrotron Beam Polarization studied by X-ray diffraction*,
D. Laundy ,**S. Brown**, M.J. Cooper**,** D.F. Paul, D. Bowyer, **P. Thompson**, **W.G. Stirling** and J.B. Forsyth,
J. Synchrotron Rad. 5, 1235-9, (1998).DOI:10.1107/S0909049597019420

[2]. *The XMaS beamline at ESRF: instrumental developments and high resolution diffraction studies,*
S.D. Brown, **L. Bouchenoire**, D. Bowyer, **J. Kervin**, D. Laundy, M.J. Longfield, **D. Mannix**, D.F. Paul, **A. Stunault**, **P. Thompson**, M.J. Cooper, **C. A. Lucas** and **W.G. Stirling**
J. Synchrotron. Rad. 8, 1172 (2001).

[3]. *Temperature-induced ordering and phase transitions in metal/adsorbate structures at electrochemical interfaces,*
C. A. Lucas, **P. Thompson**, **M. Cormack**, **A. Brownrigg**, **B. Fowler**, D. Strmcnik, V.R. Stamenkovic, J. Greeley, A. Menzel, H. You, and N. M. Markovic,
Journal of the American Chemical Society, 131, 7654 (2009).

[4]. *Optimizing the XMaS beamline for Low Energy Operations to maximize benefits from the ESRF Upgrade Program*,
P.B.J. Thompson, S.D. Brown, L. Bouchenoire, D.F. Paul, **D. Mannix, P.S. Normile**, O. Bikondoa, T.P.A. Hase, **J. Kervin , C.A. Lucas** and M.J. Cooper,
AIP Conf. Proc. 1234, 407 (2010). DOI:10.1063/1.3463226

[5]. *Simultaneous measurement of X-ray diffraction and ferroelectric polarization data as a function of applied electric field and frequency,*
J. Wooldridge, S. Ryding, **S.D. Brown**, T.L. Burnett ,M.G. Cain, R.J. Cernik, R. Hino, M. Stewart and **P.B.J. Thompson**,
J. Synchrotron. Rad. 19, 710 (2012).

Grants/awards:

[6]. Total EPSRC commitment - £18M

1992: Phase I (build) GR/G02338/01 GR/J79263/01,
1997: Phase I (operation) **GR/R14989/01, GR/L39698/01**, GR/M21706/1
2002: Phase II Operation **GR/R87420/01**,
2007: Phase III operation EP/F000375/1 and **EP/F000766/1**,
2012: Phase IV – **Mid-Range Facility Grant** (£6.3M) which runs until Sept. 2017.

*Bold items relate to Liverpool authors/PIs.

4. Details of the impact (indicative maximum 750 words)

The direct **economic impact** of the XMaS project has been through the commercialisation of intellectual property generated as part of the research infrastructure developments that have occurred as new advances in synchrotron based experiments have been implemented. Commercial licence agreements (currently 9 agreements for individual instruments, shared between Liverpool and Warwick) are held in partnership with *Huber Diffraktiontechnik GmbH & Co,* which is a world leader in specialist x-ray equipment. By including the commercialised equipment in its catalogue, our research is disseminated to industry, central facilities and academic users. Since 2008, 22 instruments have been sold (mainly to synchrotron facilities worldwide) which equates to over £700k sales turnover. The instrumentation which has been licenced to Huber includes:

- An X, Y, Z cryostat carrier for Huber 512/410 Eulerian cradle, maintaining 40 micron diffractometer sphere error with sub-micron precision in X, Y & Z (model no. 512.12M);
- An in-vacuum “Tube Slit” assembly enabling slit screen to be mounted within a few mm of sample (model nos. 3016 and 300260);
- An in-vacuum slit screen (model no. 3017);
- An in-vacuum x-ray polarization analyzer (model no. Polanvac).

According the Managing Director of Huber, “The XMaS impact is significant in its overall contribution to our business as the systems we sell extend our exposure and hence attract a wider international market for our products. XMaS instrumentation with its insight into the international community’s needs has widened our sales market and added value to our systems. The inclusion of the licensed products in our portfolio has enabled to the company to grow year on year despite the current economic environment.”[1].

An article in the EPSRC newsletter Connect describes the impact of the XMaS instrumentation as “big business” highlighting that “research can have impact through innovative instrumentation as well as science”. [2] The take-up of the XMaS-developed research infrastructure enables enhanced capability and capacity at synchrotron radiation facilities worldwide through increased efficiency of experimental procedure. This can have significant cost saving benefits as experiments cost >£10k per day. For example, one of the biggest sellers, the XYZ cryostat mount, has enabled large efficiency gains to be realised (up to 20% of measurement time saved) by allowing motorised repositioning of the sample). [3].

The second type of impact generated by XMaS arises from **knowledge transfer through the provision of trained specialists and know-how**. The demand for synchrotron radiation extends well beyond the academic community to industry and government organisations. This means that the latest generation of machines are currently being built while existing machines are upgraded to provide more flexible experimental stations. This leads to a requirement for sophisticated instrumentation to improve the efficiency of SR experiments in a wide range of scientific fields, and for trained specialists for this industry.

Since the XMaS project has been running, several new synchrotron facilities have been built or upgraded. Through knowledge transfer and direct interaction with these facilities, which include the LNLS in Brazil and the UK Diamond Light Source (DLS) facility, we have enabled commercial projects to be completed more efficiently and cost effectively. This was typified by the joint appointment of a post-doctoral researcher who worked on XMaS for two years before transferring to DLS [4]. Examples of exchange of processes include installation of an electric-field capability developed with NPL at DLS and, more recently, the Swiss Light Source have implemented our methods for controlling the incident polarisation. In conjunction with commercial partners, such as NPL and DLS, we co-host, sponsor and run specialist meetings and workshops. A series of meetings co-hosted by the NPL led to the development of a new web resource detailing metrology standards for electrical measurement. [5] The success of this endeavour resulted in XMaS and

NPL collaborating as partners in the EURAMET-EMRP project which brings the premier European metrology labs together with commercial partners such as IBM, New York. [6]

The XMaS project has a commitment to provide the next generation of material scientists that have specialist expertise in advanced synchrotron radiation techniques. As well as contributing directly to the running of synchrotron facilities, the diverse range of people trained on XMaS have transferred their knowledge and experience to their current roles that span industry, other large scale scientific facilities and education in schools. Since 2008, more than 50 post-doctoral and 150 postgraduate researchers have been trained on the beamline. In addition to those now in academic posts, many of these researchers currently work at central facilities worldwide with 15 working at the UK national x-ray (DLS) and neutron (ISIS) centres. Many other trained researchers have progressed into careers in directly related industries.

Dr. Mark Gallagher is a project scientist at Instrument Design Technology (IDT), which is an established leading supplier of complete beamline, systems & components to the global synchrotron community [7]. He states, "My introduction to the speciality of synchrotron radiation began as a beam line user at XMaS. Many of the skills required on a daily basis in my current profession were conceived during this period. This was my first personal experience of how instrumentation development in the field is critical to scientific advancement. It is difficult to envisage a more appropriate experience suitable for my present position and has added great value to my own role within the company."

5. Sources to corroborate the impact (indicative maximum of 10 references)

[1] A letter of support from the Managing Director of Huber corroborates XMaS's contribution to Huber's business, enabling them to attract a wider international market for their products.

[2] The impact of XMaS through its innovative instrumentation is described in an article in the EPSRC newsletter Connect.

[3] A letter of support from the Associate Division Director at the Argonne National Laboratory corroborates that the XMaS-developed research infrastructure has enhanced capability and capacity at synchrotron radiation facilities worldwide which has created cost saving benefits enabled large efficiency gains.

[4] A letter of support from the Principal Beamline Scientist at Diamond Light Source can corroborate that through knowledge transfer and direct interaction with new synchrotron facilities, projects have been completed more efficiently and cost effectively.

[5] A new web resource for commercial partners was set up detailing the metrology standards for electrical measurement

[6] A letter of support from the Principal Research Scientist at the National Physical Laboratory corroborates the scientific and commercial impact that the collaboration with the XMaS beamline has had for NPL.

[7] A letter of support from a project scientist at Instrument Design Technology corroborates the value of XMaS's instrumentation development on professional careers within a commercial organisation that supplies complete beamline, systems & components to the global synchrotron community.