Diamond-II
Proposal for flagship project
BERRIES
(Bright Environment for x-ray Raman, Resonance Inelastic and Emission Spectroscopies)

Science Group: **Spectroscopy**

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Outline proposal for a flagship beamline project Diamond-II

1. Summary/Impact statement

Taking advantage of the added capacity and increased brightness offered by Diamond-II, and the availability of the long straight (straight 17), we propose to build a new beamline to perform photon-in/photon-out spectroscopies. The new beamline, called BERRIES (Bright Environment for x-ray Raman, Resonant Inelastic and Emission Spectroscopies), will be a high flux beamline offering two new techniques to the Diamond user community: pink-beam X-ray Emission Spectroscopy and X-ray Raman Scattering. These emerging techniques will support discoveries in several scientific areas important to the UK's research priorities, such as chemistry and catalysis, energy materials and nuclear waste management among others. BERRIES will also support state of the art High Energy Resolution X-ray Absorption Spectroscopy, X-ray Emission Spectroscopy and Resonant X-ray Emission Spectroscopy, complementing the existing capabilities offered by the spectroscopy group. Its small focal spot and high flux will enable to perform these techniques in time and spatially resolved studies, as well as perform experiments on small samples, such as those contained in pressure vessels.

2. Scientific Case

Photon-in/photon-out spectroscopies have become powerful tools for the study of chemically specific electronic and geometrical structures. Several techniques are encompassed under this generic term: High Energy Resolution Fluorescence Detected X-ray Absorption Spectroscopy (HERFD-XAS), Non-Resonant X-ray Emission Spectroscopy (NXES or just XES), Resonant X-ray Emission Spectroscopy (RXES) and X-ray Raman Scattering (XRS). These techniques extends the range of applications of conventional X-ray Absorption Spectroscopy (XAS), and address some of its shortcomings[1,2]. For example, XRS uses hard X-rays for performing spectroscopy of light elements, such as O, C, Li, Be, etc... making this technique a bulk sensitive probe that can be used for performing experiments *in situ* and with a broad range of sample environments. Recently, XRS has been also used to perform X-ray imaging on samples, even when complex sample environments are used. This field of research is in the early stages of development but has shown great promise for the study of heterogeneous sample systems such as catalysts, batteries and fuel cells[3]. Furthermore, making use of the dependency of XRS with momentum transfer, q, information about higher order transitions can be obtained. At low q, XRS is dominated by dipole transitions and the spectrum becomes equivalent to XAS, but at high q values, XRS becomes sensitive to dipole forbidden transitions. Owing to this, the entire unoccupied density of states is accessible with this technique[4]. XES on the Valence-to-Core emission line (VtC-XES), on the other hand, has all the benefits of standard XAS but overcomes its main limitation: the inability to distinguish ligands that are neighbouring in the periodic table, such as carbon, nitrogen and oxygen.

The capabilities that the proposed beamline will bring to Diamond will have an impact in many areas of science, as was described in the Diamond-II science case. In this outline proposal we will focus on the impact that BERRIES will have on Chemistry and Catalysis, Energy Materials and Environmental Sciences.

Chemistry and Catalysis.

XRS has proven to be a very useful spectroscopic tool for the investigation of low-Z elements in materials under challenging conditions[5], in gas and liquid phases[6], for studying chemical bonding[7] or carbon-carbon 3D mapping[8]. Its bulk sensitivity and the possibility of measuring samples that are incompatible with vacuum conditions, combined with an unparaleled spectral-feature content given by the non dipole-limited nature of observable electronic transitions[9], opens new opportunities for catalysis experiments. The technique can unveil the coordination of light elements (carbon, nitrogen) in oxide supports and probe the interaction of the adsorbed molecules with the catalysts, providing structural, electronic and spin insight via access to the crystal field at the L-edges of transition metals[10].

As highlighted in the Diamond-II science case, XRS will also play an important role in broadening our understanding of the nucleation of organic solutes during the first stages of crystallisation. This is critical information if we are to develop methods to control the physical and chemical properties of crystalline products. *In situ* studies of these mechanisms are hard to perform in the soft X-ray regime, but a recent study[11] of the crystallisation process of imidazole has shown how XRS can be used to study relevant systems *in situ*, characterising the solute speciation during the nucleation process.

VtC-XES and RIXS are routinely applied to compounds relevant to both homogeneous catalysts and intermediates in heterogeneous reactions[12]. In recent years, this versatile technique has been used in some of the major fields of catalysis, such as adsorption processes, photocatalysis and bond-activation, mesoporous media for gas conversion and electro-catalysis. With the proposed combination of a high-flux beamline, small focal spots, pink beam and state of the art emission spectrometers, BERRIES will be able to provide access to a suite of complex and completely new experiments. Beyond the capability to perform site selective HERFD-XANES with high energy resolution for the separation of elements with different ligands[13], the micron size beam will enable the recording of XES in a spatially resolved manner, allowing the mapping of catalyst beds in similar fashion to conventional XAS studies[14]. In addition, the availability of an emission spectrometer working in a dispersive configuration[15] will enable time-resolved collection of RIXS maps[16]. The combination of such an instrument with pink-beam will also enable VtC-XES studies in a time-resolved manner, or in more dilute samples, as described in the Diamond-II science case.

Energy Materials.

The capability of XRS to probe the electronic and structural changes in light elements such as Li, B, N, O, with bulk sensitivity and under *in situ* and *operando* conditions, make the technique very relevant to the study of energy materials, as identified in the Diamond-II science case.

XRS has been used to study the electronic structure of lithium battery interphase compounds[17] and to monitor the temperature evolution of the O K-edge in $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8\text{-}0.03}Ni_{0.03}O_{3\text{-}\delta}$ cathodes, used for intermediate-temperature solid oxide fuel cells (IT-SOFCs)[18]. XRS has also been used to study the electrolyte solution in a flow cell by following the C and O K-edges[19].

XRS also enables bulk studies of L-edges of the transition metals in battery materials, which are often sensitive to oxidation state change, as shown in a recent experiment performed by Tian et al.[20] on $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$ cathode materials as a function of the state of charge (Figure 1).

XRS has also been used successfully for the study of lithium borohydrides[21]. These are promising materials for use in hydrogen storage technology, and the experiments identified changes in the speciation of the lithium and boron as hydrogen is sorbed.

Operando studies by XES and HERFD-XANES of the first-row transition metals in batteries provides clearer and more detailed information on the small changes that can happen during cycling and on the effect of repeated cycling[22]. The techniques can also provide information about the spin state of the metal centre. The high flux available at BERRIES, together with the small beam size will facilitate mapping of batteries during electrochemical cycling, bringing important insight into the rate limiting steps of charging and discharging. Moreover, fast measurement of the $K\beta_{1,3}$ emission using pink beam together with a dispersive spectrometer will provide the opportunity to study changes in the electronic structure of metal ions during fast charging processes in batteries[23].

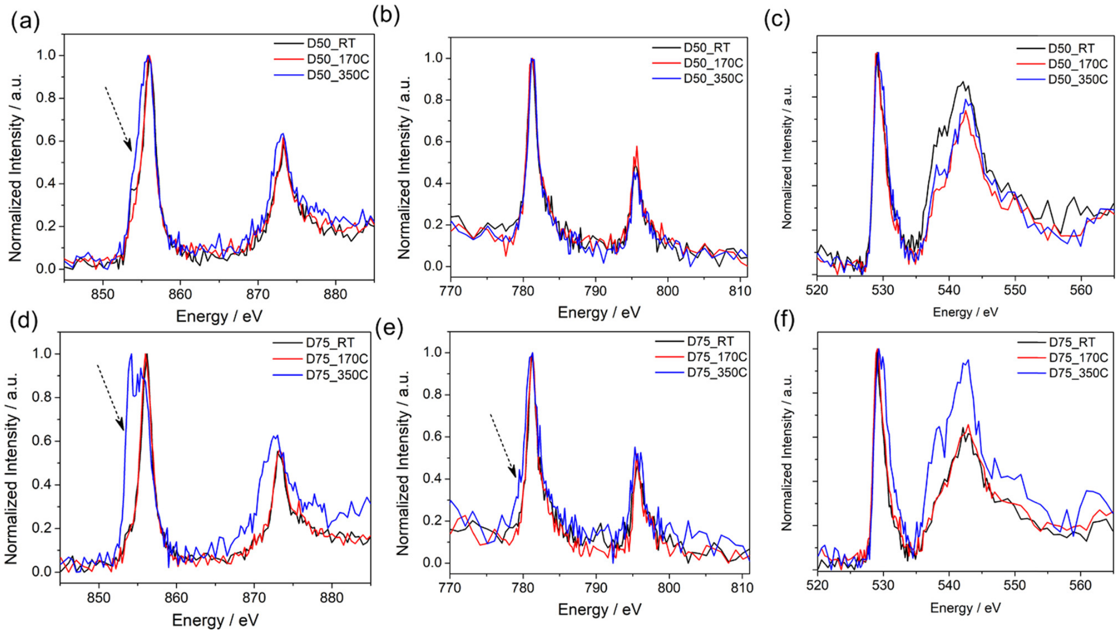

Figure 1. X-ray Raman Scattering data showing (a, d) Ni L-edge, (b, e) Co L-edge, and (c, f) O K-edge for (a, b, c) 50% de-lithiated and (d, e, f) 75% de-lithiated $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2$ powders at room temperature and after heating to 170 or 350 °C (taken from ref 19).

Earth, Environment and Planetary Science

Understanding the electronic structure and speciation of actinide materials is extremely important in characterising nuclear waste forms and predicting their long-term behaviour; this is essential for building the safety case for a geological disposal facility. Both L- and M-edge X-ray spectroscopy are commonly used to characterise such materials and the dispersive spectrometer on the XES branch will enable much faster RIXS studies of L-edges providing information on the density of states in the valence and conduction bands, and also deliver sensitivity to doping of uranium compounds by other actinides[24].

XRS, on the other hand, provides access to the $O_{4,5}$-edges of uranium (5d-5f transition), which have been shown to probe directly the configuration of the *f*-state, relevant in the study of highly correlated materials such as URu_2Si_2[25] .

The high flux and small beam sizes offered by BERRIES will also be essential for studies in Earth and planetary science, when high pressure vessels such as diamond anvil cells (DAC) are used to mimic the conditions of matter in the interior of the Earth and on other planets. The use of XRS will allow the study of earth abundant light elements such as Si and C, at extreme conditions, as has been demonstrated by Lee et al.[26] in their study of the atomic configuration of silicate glasses at the bottom of Earth's mantle at pressures higher than 100 GPa.

3. Benefit to the Diamond research community

The addition of BERRIES to the beamline portfolio will offer completely new capabilities at Diamond for hard x-ray photon-in/photon out spectroscopy, as well as complementing the capabilities of existing instruments.

Firstly, this new instrument will offer two techniques that are currently not accessible at Diamond, XRS and pink-beam XES. These techniques, that have only recently become available at synchrotron sources, are showing great potential in several areas of science. This makes the BERRIES proposal very timely, as many synchrotrons are in the process of upgrading their existing spectrometers so higher quality XRS can be performed by capturing a large solid angle, or they are planning new instruments to perform pink-XES as part of their upgrades, as is the case of the APS. As described in the Diamond-II Science Case, XRS is already having a significant impact on the study of energy materials, allowing the study of the K and L absorption edges in the low energy regime with bulk sensitivity and in real devices. In chemistry, some interesting studies are starting to emerge on the study of crystallization. XES on the other hand, has already been proven as an important technique for the study of the evolution of the electronic structure of *in situ* catalytic reactions. The possibility of performing XES with a broad energy band-pass (pink-XES) that considerably decreases the time needed for data collection, opens the door to perform *operando* studies. This will also be of great utility in the study of energy materials, as XES experiments on fast battery cycling will then be possible.

Secondly, BERRIES will also deliver XES, RXES and HERFD-XAS. Although these techniques are already offered at I20-Scanning, the higher flux and smaller focal spot available at BERRIES will enable studies with higher energy resolution and in a spatially resolved manner. This will open new opportunities in many scientific areas, both for academia and industry. The study of catalytic reactions with a probe that provides detailed information about the electronic and geometrical structure in a spatially- and time-resolved manner, will increase our understanding of the mechanisms of vitally important reactions. The small beam size will enable XES studies on samples under high pressure conditions, not currently possible at Diamond. The increased flux available at BERRIES will support the study of radioactive waste, as the small amount of radioactive material, required for safe handling is often a handicap for these types of experiment.

With the development of this beamline Diamond can retain its international competitiveness in the evolving field of X-ray spectroscopy, whereas the facility risks falling behind if it cannot offer to the user community the new and promising techniques, XRS and pink-beam XES. These rapidly developing methods have already started to demonstrate significant impact in several scientific areas that are important for the UK's research priorities.

4. Outline Specification

BERRIES will take advantage of the added capacity and higher brightness offered by Diamond-II. We are proposing to build the beamline on the available long straight section (Straight 17) as the use of multiple undulators is needed to deliver the required flux for the instrument. The beamline consists of two experimental hutches, the first will operate in the energy range from 4keV to 20keV and will be optimized for performing HERFD-XAS, RXES and pink-beam XES, while the second will house an X-ray Raman Spectrometer operating at two energies, 6.46 keV and 9.69 keV.

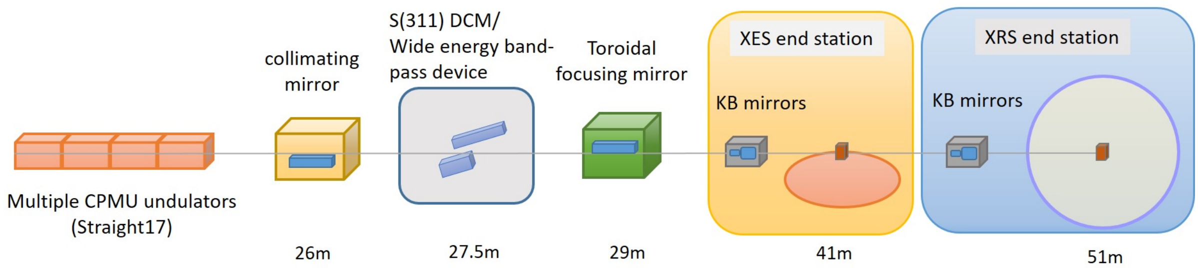

Figure 2. Proposed BERRIES layout, showing the position of the different optical elements and the two separated experimental hutches.

The proposed optical layout for the beamline is shown in Figure 2. A pair of 2m CPMU undulators will deliver the flux to the first optical element housed in the optics hutch via a vertical collimating mirror. Downstream of the mirror, a high energy resolution monochromator will be located. For those experiments that require the use of pink-XES, the beamline will also be equipped with a broad energy band pass device, either a multilayer monochromator or a transmission prism use to separate the undulator harmonics, such as the one developed by Inoue et al.[27].

The beam will be further focussed by a pair of KB mirrors placed in each of the experimental hutches. The first experimental hutch will house a state-of-the-art XES spectrometer capable of operating in a point-to-point configuration to perform HERFD-XAS and RXES. A dispersive spectrometer will also be available to perform time-resolved XES experiments using pink beam.

The second hutch will house a highly efficient XRS spectrometer with several groups of analyzers aligned in a fixed near-backscattering geometry. Each group of analyzers will be placed at different locations so that the required momentum transfer range, q, can be covered. Each group will have a dedicated pixel detector to allow imaging of the beam collected by the analyzers to allow X-ray imaging with XRS[28]. This is a novel application of the technique that is under current development. Currently the spatial resolution is limited by the detector pixel size and the quality of the crystal analyzers.

The outline specifications for both end-stations using the described optical layout is given in Table 1.

End Station	**XES**	**XRS**
Energy range (keV)	4-20	6.46 and 9.69
Flux using Si(311) DCM	> 5×10^{13} ph/s at 7112eV	> 5×10^{13} ph/s at 6460eV and 9690eV
Beam size (FWHM) (μm)	17 × 25 (V×H)	21 × 20 (V×H)
Geometry of the Spectrometer	Rowland circle geometry. Point-to-point & Dispersive configurations	Backscattering Rowland configuration with Si(nn0) analysers
Total energy resolution	< 1eV	0.2 to 0.8 eV
Detectors	Pixel detectors	Pixel detectors

Table 1. Outline specifications for the end-stations of the proposed beamline.

5. State of the art benchmark

High energy resolution XES spectrometers are widely available at third-generation synchrotron sources around the world. A key performance differentiator between the XES spectrometers is the number of crystal analysers, which increases the detection efficiency of the instrument as you add more analysers. Current point-to-point spectrometers are most commonly equipped with five analysers,[29,30] but spectrometers with seven, ten and fourteen analysers have recently been built at SSRL 6-2b beamline[31], CHESS C-line[32], and ESRF FAME[33], respectively. Alternatively, dispersive spectrometers designed to perform XES in single shot measurements are becoming more widely available[15]. The high flux, small focal spot and optimum spectrometers design planned for BERRIES will make it very competitive for XES studies at an international level. In particular, the capability of performing pink-beam XES offered by the proposed beamline is currently only available at the ROBL beamline at the ESRF by using a multilayer monochromator[34], but with a lower X-ray beam flux compared with what will be available at BERRIES. It is important to notice that the future upgrade of the spectroscopy beamline at the APS, expected to start operations in 2021, includes the capability of performing XES with a broad energy bandpass using a multilayer monochromator.

In contrast, there are not many XRS instruments available in the world, as the expansion of this technique has only happened recently thanks to the development of efficient spectrometers that cover a large q range, together with the advent of high-brilliance beamlines and the development of advanced theoretical tools. One of the most sophisticated XRS instruments currently available is at beamline ID20[35] at the ESRF, where 72 crystals are used to collect the scattered Raman photons at different q values using three 1m undulators as the X-ray source to provide an incident flux of 7×10^{13} ph/s at 9.7 keV in a 16×8 μm^2 (H × V) beam size (according to the ESRF upgrade Technical Design Study[36], the flux at ID20 after the upgrade is expected to increase by a factor of 2, while the beam size is expected to be reduce by factors of 1/7 and 1/3 in the horizontal and vertical directions, respectively). Beamline 6-2b at SSRL, equipped with a 0.9-Tesla wiggler source, has also recently developed a new XRS instrument with 40 diced-bent crystals at low-q and 14 diced-bent crystals at high-q[37]. Other spectrometers are currently being developed and/or optimized for XRS studies in other sources. For example, the Spectroscopy Group at the APS is building a new XRS spectrometer equipped with over 100 crystals covering a large q range on a 5m undulator source based on their existing LERIX spectrometer[38], while the GALAXIES[39] beamline at Soleil, delivering 1.5×10^{12} ph/s at the sample with a beam size of 20 x 80 (V × H) µm2, is currently upgrading their instrument to 40 analyser crystals. The use of 2x2m CPMU undulators, combined by a large number of crystal analyzers able to cover a large solid angle around the sample, will make BERRIES competitive at the international level for performing XRS experiments.

6. Community engagement

As part of the preparations for a full scientific case for the proposed beamline to be presented to the Diamond Science Advisory committee in November, we are planning to engage with the user community through the creation of a working group with a representative cross section of potential users drawn from both academic and industrial institutions. We will also ensure that large government initiatives such as the UK Catalysis Hub and the Faraday Institution are represented. We expect this group to collect the requirements of their colleagues and collaborators and communicate them to the working group.

In addition, a champion for the project will be chosen. The champion will need to lead the full case that will presented to the Diamond management, SAC and DISCo.

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