# Diamond Light Source

# Outline Proposal for a Phase III beamline

## DIAD: Dual Imaging And Diffraction

**Case prepared by:**

Dr Alison Davenport (U. Birmingham)
Prof Peter Lee (Imperial College London)
Prof Ian Sinclair (U. Southampton)
Prof Philip Withers (U. Manchester)
Dr Felix Hofmann (U. Oxford)
Dr Howard Stone (U. Cambridge)
Prof Michael Preuss (U. Manchester)
Dr Philipp Thurner (U. Southampton)
Dr Alex Porter (Imperial College London)

Dr Michael Drakopoulos (Diamond)
Dr Christoph Rau (Diamond)
Dr Igor Dolbnya (Diamond)
Dr Thomas Connolley (Diamond)
Dr Robert Atwood (Diamond)

# DIAD: Dual Imaging And Diffraction

## 1. Summary

DIAD is a dual beam instrument to be built on a 3 T superbend magnet with a unique design delivering almost concurrent (chopped) imaging (1 μm resolution) and diffraction (min. beam size 1 μm) with the primary aim of relating monochromatic or Laue micro-diffraction information (either strain mapping or phase identification) to 2- and 3D dynamic imaging of complex structures. Other beamlines around the world can measure diffraction and imaging on the same sample via complicated mode changes or major sample movements: DIAD will measure both concurrently. Its unique dual beam design represents a step change in the possibilities of, for example, observing changes in shape AND stress state of structures during deformation and cracking, or determining morphology AND chemistry of phases during chemical reactions. Its medium-energy range (~4-40 keV) is ideally suited to materials and biological applications where strain-induced transformations control microstructure, and microstructure controls mechanical, and many other types, of performance. Applications are wide-ranging, with examples from osteoporosis and tissue scaffolds to batteries, and stress corrosion cracking to carbon sequestration. Materials include light alloys (Ti, Al, Mg), bioactive glasses, biological structures, polymer matrix composites, and devices such as fuel cells. DIAD is designed to be complementary to I12 (imaging and diffraction of engineering structures at 50-150 keV), I13I (50 nm to 1 μm resolution, but no diffraction), I08 (STXM, 20 nm resolution, 250-3000 eV) and B24 (cryo-TXM, up to 2.5 keV), providing a suite of imaging beamlines that will give a strong focus for the developing UK imaging community and be complementary to UK University Centres and developing facilities at RCaH.

## 2. Scientific Case

The properties of many materials depend on the interaction between microstructure, stress state and/or phase. DIAD's near-simultaneous wide field imaging and local diffraction will open up a multitude of applications where the microstructure is greater than one micron, and the crystallographic/strain state is either nanoscale (powder diffraction mode) or >1 μm (Laue). We choose to highlight a few of the many possible applications below.

### Biomedical research

This area will be a major beneficiary: DIAD will address the key question in regenerative medicine: *why, despite encouraging results in small animal models, has bone or cartilage regeneration using tissue engineering scaffolds failed to achieve clinical success in humans*? A crucial aspect is determination of the optimum stress level to promote bone growth. DIAD is the ideal and unique tool to explore how stress is applied within a scaffold-bone construct. 3D structure can be quantified with the imaging beam, and the stress-state within developing bone measured with local powder diffraction patterns of carbonated apatite nano-crystals. Effects of changing chemical environments and biodegradation on bone growth and mineral content can be mapped with K-edge subtraction tomography. Phase contrast imaging will detect early stages of osteoblast growth and tissue formation, prior to mineralisation.

Similar studies are important for bone augmentation, orthopaedic and dental applications, where the stress state at surfaces is fundamental to understanding load transfer and hence stress shielding leading to implant loosening. Measurement of stress state during dynamic loading around bone implants will improve design of long-lasting joint-replacements for our aging population. Furthermore, in situ measurements will provide new insight into bone failure mechanisms, for example, by studying the strain state behind, and ahead of, the crack tip during crack propagation and using *in situ* rigs to load cycle bone while simultaneously imaging and measuring strain. Studying damage accumulation during *in situ* testing will show the efficacy of both normal bone toughening mechanisms and the influence of pathologies such as osteoporosis or osteogenesis imperfect on failure. The imaging mode will allow direct meshing and finite element simulation of stresses and joint motion leading to development of new therapies and improved design of synthetic materials with bio-inspired architectures.

### Engineering failure in light alloys and composites

Life prediction of engineering composites and alloys requires understanding of crack behaviour at the microstructural scale in response to local stresses within individual crystallites at the crack tip. This is also a key challenge for design of novel damage-tolerant micro/nano-structured materials. Whereas I12 is specialised for larger engineering components (cm scale) and samples, DIAD will be optimised for smaller (mm) samples/test-pieces and, as the medium energy complement of I12, it is ideally suited to achieve a 10-fold increase in spatial resolution for stress mapping and structural imaging to tackle such issues. It is a unique and versatile tool to tackle these problems through concurrent measurement of microstructure, strain/phase evolution from macroscopic behaviour to micron-scale individual crystals (high resolution polychromatic micro-diffraction), and the integrated behaviour of nanocrystallites (powder diffraction). Combining imaging and diffraction will lead to step changes in understanding existing materials as well as the design of new crack-resistant ones. Important issues include cracking (imaging) and crack-tip shielding (diffraction) in tough hierarchical biomimetic biomaterials, damage propagation in carbon fibre composites (key to new aerospace designs),  novel light alloy metal-matrix composites e.g. Al-Ti (for significant weight and hence energy savings) Diffraction contrast microscopy can provide insights into deformation and fatigue in 3D at the grain scale in a wide range of materials; it could provide both average and local grain reorientations during straining and the interaction of cracks with different grain orientations; helping us understand the basic physics of slip at the grain by grain level and its role during crack initiation and propagation.

### Corrosion of metals

Corrosion of metals is an area of great economic impact where in situ methods are essential for fundamental mechanistic understanding. Recent work has detected corrosion products on rapidly dissolving surfaces with local diffraction, but this was achieved “flying blind”: with DIAD, phase development can be related to corrosion attack morphology, providing data for developing corrosion prediction models. It will also be ideal for studying stress corrosion cracking in light alloys (Al, Mg, Ti), relating local strains to crack propagation.

### Batteries and fuel cells

Solid oxide fuel cell (SOFC) electrodes are commonly porous composites providing intimate contact of electronic, ionic and pore phases: the electrode microstructure dictates the cell’s catalytic performance. The cermet NI-YSZ is a common anode, and cell operation must be carefully managed to accommodate stresses from thermal and redox cycling to prevent microstructurally driven degradation and failure. A combined understanding of stress distribution and high resolution imaging will unravel stress-related failure processes, providing design and operating criteria for enhanced SOFC peformance.

During charge cycling in conventional Li-ion batteries, Li intercalation into graphite electrodes results in ~13 vol% expansion. In high capacity "alloying-type" systems such as Si or Sn based anodes, volume changes >300 vol% are seen, providing a major challenge to battery durability. Combined stress and tomography measurements will improve understanding of microstructural degradation and fracture during cycling, facilitating better battery design. Scaling up Li-ion batteries from small devicesfor portable electronics to much larger batteries for transport and grid storage will require a step change in our understanding of how electrode materials transform and degrade over a wide variety of time and length scales. The ability to extract maps of structure and local strains in particles during cycling of intact batteries will provide vital insight into failure mechanisms in these systems.

### Methane hydrates

Methane hydrates represent one of the largest carbon inventories on the planet. It is claimed that 1% recovery would more than double current natural gas reserves. $CH_4$ is also a powerful greenhouse gas with a major potential role in climate change, particularly via oceanic sediment breakdown. The physical characteristics of natural gas hydrate-bearing sediments remain poorly quantified. Simultaneous tomography and diffraction data from natural, model and bulk hydrate samples under controlled pressure, temperature and fluid saturation/flow can support understanding and multi-physics simulation of of hydrates and host sediment behaviour, as well as hydrate structures, formation kinetics and sediment micro-morphology.

## 3. Outline specification

A 3T “super” bending magnet will provide x-rays in an energy range of 4-40 keV, which is ideal for imaging biological and polymeric materials and light alloys Mg, Al and Ti (and small Fe structures). The relatively wide fan beam of X-rays will be split horizontally into two almost parallel beams by a pair of identical plane mirrors, with a large beam for imaging, and a small beam for diffraction. The imaging beam will be 2 mm wide and 2-15 mm high.

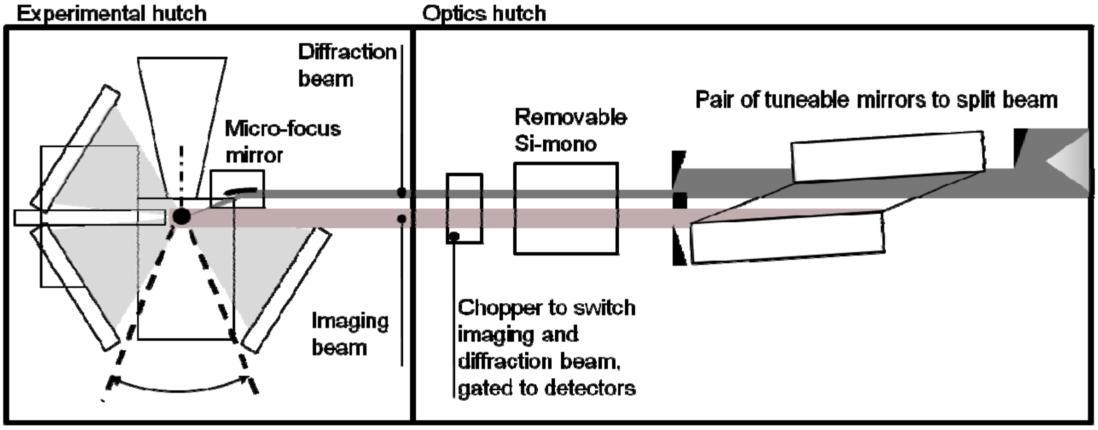

Fig.1: beamline layout from above: the beam and focusing mirror for diffraction will be in vacuum as close as possible to the sample.

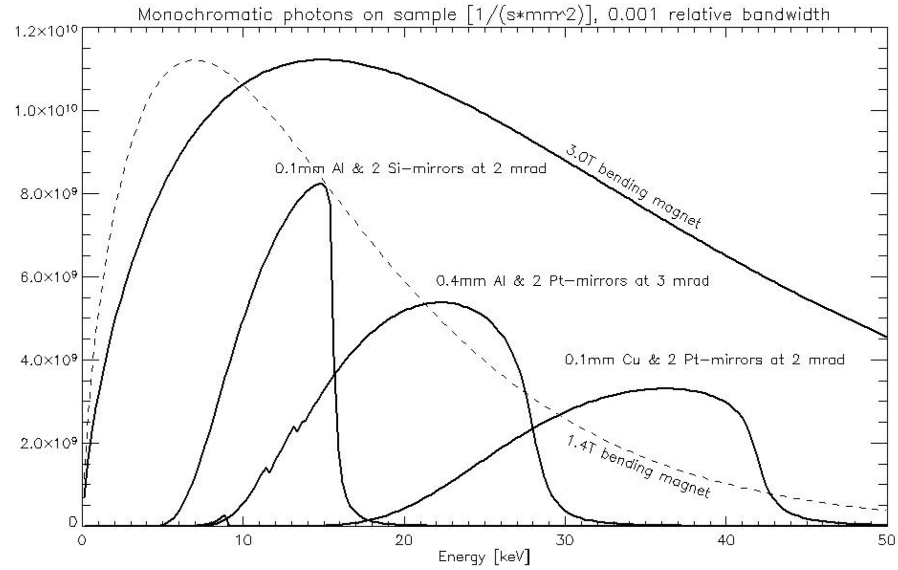

Fig. 2: Imaging beam intensity as a function of energy using different mirrors and filters.

The mirror pair not only seperates the imaging beam from the diffraction beam, but also narrows its spectrum. In combination with transmission filters, it produces an intense pink beam of more than $5\times10^{13}$ photons/(s×mm$^2$) at the sample position. Combining different filters with different mirror angles (in fixed exit geometry) or mirror coatings, the spectrum can be tuned through the entire energy range (Fig.2).

After the mirrors, a Si monochromator can be inserted. When inserted, we have a horizontal pair of monochromatic beams (at identical energy). A slow chopper behind the monochromator switches between the imaging and diffraction beams, electronically gating the detectors to recorded images and diffraction patterns without interference.

Finally, at a distance of ~300 mm from the sample, a static ellipsoidal focusing mirror (Pt-coated, with ~1.7 mrad central ray incidence angle) produces a focused diffraction beam (for µ-Laue (white beam) or monochromatic diffraction) to merge with the imaging beam at the sample position. By using either different mirrors or a single movable mirror, the focused spot size can be varied from ~1 µm to larger diameters (e.g. 10 µm, 50 µm, to be determined). A maximum size of 300 µm can be obtained using a flat mirror. All optics up to the sample are under vacuum, including the focusing mirror.

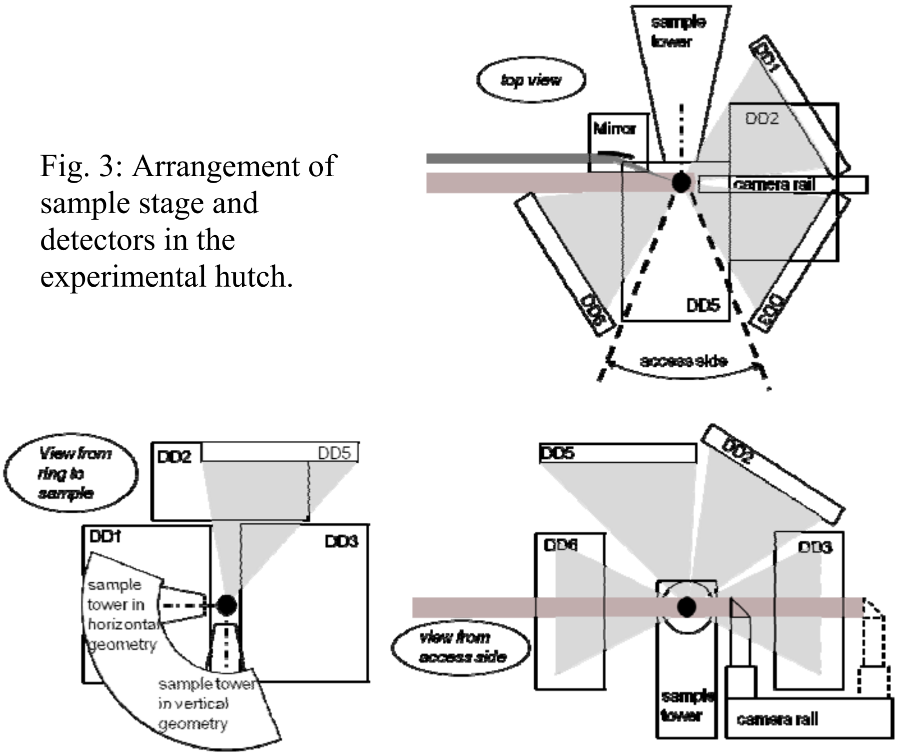

Fig. 3: Arrangement of sample stage and detectors in the experimental hutch.

The experimental hutch houses imaging and diffraction detectors and sample manipulation (Fig.3). Six large area flat panel diffraction detectors (DD1-DD6) are arranged to give a large reciprocal space coverage, including back-reflection (DD6) for high resolution strain mapping. Two interchangeable imaging cameras mount on a camera rail, one with ~2 mm field of view and 1 µm resolution and the other with a 15 mm field of view and 10 µm resolution. The variable sample-camera distance accommodates phase contrast imaging. The “sample tower” aligns the tomography rotation axis to any angle between vertical and horizontal and thus orients the sample into the 2 × 15 mm field of view, as required. Manual or automated sample manipulation as well as insertion of a sample environment chamber is through the “access side”. For larger sample chambers, individual detector panels can be temporarily removed.

Energy resolving pixel-detectors (pioneered by Cernik using CdZnTe-based detectors for “TEDDI”) could be an important future upgrade to complement our white beam micro-Laue diffraction technique and enable time-efficient white beam diffraction tomography.

It is recognised that a crucial part of making this line successful will lie in highly effective use of software both for very rapid tomographic reconstruction and extraction of diffraction data “on the fly”, so that the data quality can be monitored as it is being collected. It will be important to learn from other beamlines where this is successfully implemented.

## 4. Community

There has been an enthusiastic response from diverse research groups, with >90 responses, of whom >80 expressed interest in imaging combined with diffraction. In addition to combined diffraction+ imaging work, there is an extremely strong and unfilled demand for imaging in applications that need the 4-40 keV photon energy range. These include soils and plant roots, organisms such as bees, archaeological artefacts and fossils, tissue structure including, lung, heart, brain and vascularisation, multi-phase flow and hydrocarbons in porous rock, microbial corrosion, bioactive glasses, solidification structures, highly porous materials and foams. This community is on the whole inexperienced in diffraction methods and represent an outreach opportunity, leading to a vibrant user community for DIAD, and extending that for I13I, which aims for 50 nm resolution imaging following commissioning at 1 µm resolution

The user community responses cover many areas encompassing RCUK themes such as ageing (>40), energy (>40), nanotechnology (>40), environmental change (~30), food security (~20), digital technology (~20) and security (>10) as well as >30 users either based in or collaborating with industry. In addition to highly experienced groups (featured above), there are many groups who have not used synchrotrons before, indicating that DIAD reaches out to a new user community, particularly through high profile University Centres including those at Manchester and Southampton. Together with these, the complementary imaging capabilities at Diamond and Imat at ISIS, and facilities at RCaH, DIAD can play an integral part of a national strategy for imaging.