Soft-Condensed Matter Science has a bright future via the Diamond-II upgrade

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In 2020, the most significant application of Soft-condensed matter (SCM) science was, undoubtedly, the development of the COVID vaccines by Pfizer-BioNTech and Moderna which are composed of a synthetic messenger RNA encapsulated within a lipid nanoparticle [1]. SCM science drives continued improvements to quality of life as SCM science is integral to a broad range of industrial sectors. These sectors include food safety and waste, agriculture, polymers, coatings, additive manufacturing, lubricants and additives, personal care, electronics, energy storage, transportation, health and medicine.

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SCM materials occur over a range of physical states that include liquid, semi-solid, waxes, glasses and aerosols. This necessitates a complementary and diverse suite of instrumentation and methodologies for understanding how molecular details give rise to the desired macroscopic, bulk properties of the material. Diamond Light Source, operating since 2007, is the United Kingdom's synchrotron light source and provides four instruments (beamlines) for investigating SCM materials: B21 (high-throughput small-angle X-ray scattering for liquids), B22 (infrared nanospectroscopy and imaging), B23 (ultra-violet circular dichroism spectroscopy) and I22 (general purpose small angle X-ray scattering and diffraction for non-crystalline materials).

Diamond is a circular, electron accelerator that maintains electrons in a large orbit (561.6 meter circumference) near the speed of light *(Figure 1)*. This creates synchrotron radiation that emanates tangentially as fine beams from the synchrotron ring providing infrared (IR), visible and ultra-violet light and X-ray radiation for experiments. IR-spectroscopy informs on the chemical environment of a material and can be applied to liquids, soft materials, paintings and even geological samples. B22's IR-imaging can provide real-time IR-spectroscopy of living cells at sub-cellular resolution *(Figure 2A)* allowing metabolism to be followed in real-time. B23 exploits polarised UV light from the synchrotron to study materials with chiral properties (chirality refers to molecular arrangements that have complementary mirror images). B23 recently discovered the emergence of magneto-optical properties in polymer thin-films at a spatial resolution of 50 microns *(Figure 2B and C)* [2]. B21 and I22 use X-rays for scattering and diffraction from non-crystalline materials. X-rays allow for increased penetration depths into the material under investigation informing on internal molecular, hierarchical structures. Combined, B21 and I22 can probe samples at molecular- to nano-length scales using the so-called wide (WAXS) to small (SAXS) angle X-ray scattering range *(Figure 1)*.

Exploiting the missing information link between nano- to micron-length scales

Non-crystalline materials represent the vast majority of everyday materials we encounter. These include plastics and polymers, chocolates, wood and wood products, lubricants, lotions, cleaning products, ice cream, yogurt, liquid crystal displays, and medicines to name a few diverse examples. The bulk properties and functional aspects of many SCM materials derives from the hierarchical structures that are partially ordered on molecular to micron length-scales *(Figure 2)* [3]. SAXS has been the primary technique for structural investigations of such partially ordered systems with B21 and I22 reliably providing UK scientists with world class instruments for studying soft-matter problems since 2009. Diamond's complementary suite of beamlines can cover a wide range of length- and time-scales.

However, there is a missing window of mesoscopic information between the nano- to micron-length scales. For this reason Diamond is planning a competitive, world-class upgrade, called Diamond-II, that will generate finer and brighter beams of synchrotron light. The orbiting electrons travel in packets with distinct shapes that effect the quality of the synchrotron radiation. Diamond-II will exploit recent advances in miniaturisation and magnets to shape the electrons into smaller, rounder shapes producing optimal X-rays beams for ultra-small angle X-ray scattering (USAXS) studies.

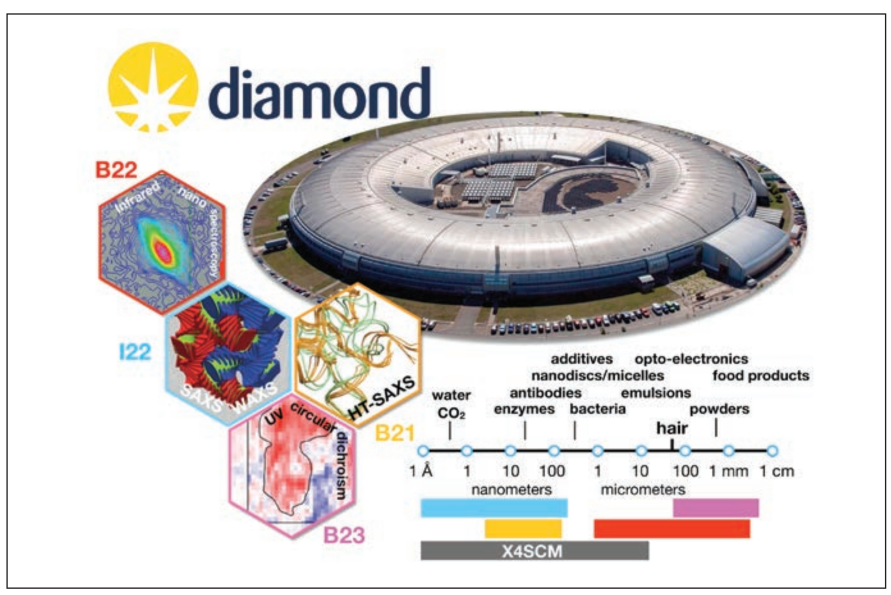


Figure 1. Diamond Light Source at the Harwell Science and Innovation Campus. Synchrotron ring operates 33 beamline instruments. Soft-Condensed Matter group consists of the four beamlines, B21 (high-throughput SAXS), B22 (infrared nanospectroscopy), B23 (UV circular dichroism) and I22 (general purpose SAXS & WAXS). Currently, there is a gap in the accessible length-scales that would be filled by Diamond-II X4SCM beamline.

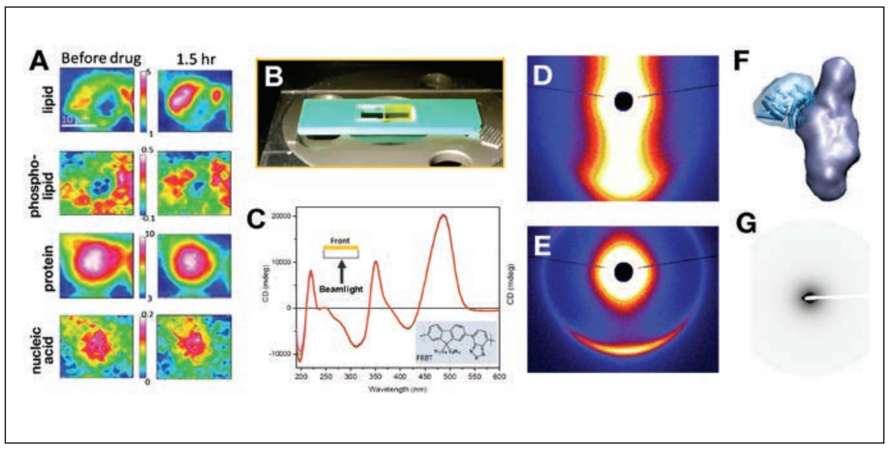
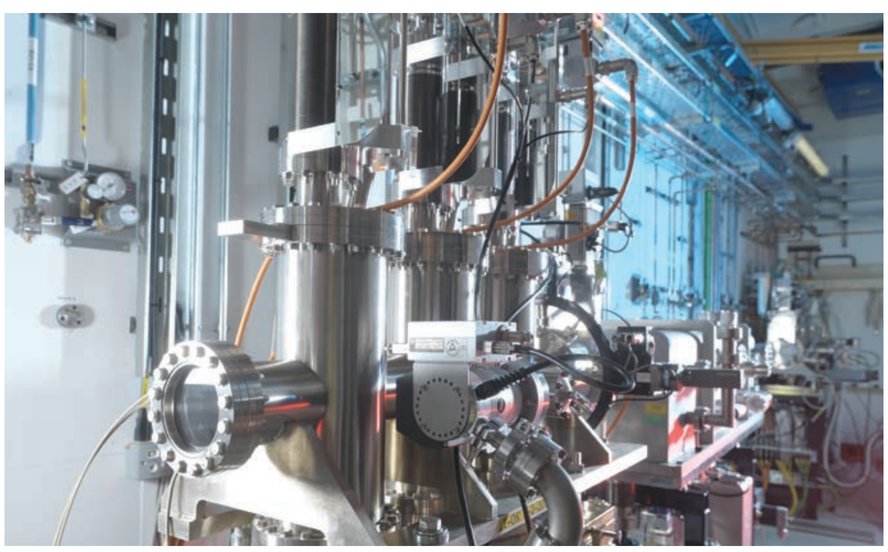


Figure 2. Capabilities of the SCM group at Diamond Light Source. A. B22 Infrared imaging of a single living cell. Lipid, phospho-lipid, protein and nucleic acid refer to infrared wavelengths unique to each molecular class. Panel shows changes in the cell following drug treatment. B. Thin-film polymeric sample on microscope slide in CD-imaging. C. Single, 50-micron CD spectrum from sample in B. D. I22 SAXS image of sample under rheologic stress. E. Same sample in panel D reaching equilibrium. F. Biphasic volumetric reconstruction of maltose-binding protein tagged to a recombinantly expressed 65 kiloDalton protein from solution-state, high-throughput SAXS. G. Representative bioSAXS image.

To exploit the new beam properties in Diamond-II, we are proposing the X-rays for the Soft-Condensed Matter (X4SCM) beamline, a world-leading instrument for SCM Science. It will offer the first camera to simultaneously observe the micron- to molecular-scale ordering, whilst offering access to a high brightness, polychromatic X-ray source for time-resolved studies. X4SCM will provide two instrumentation modalities: HIERARCHY, for structural investigations capturing molecular to micron length-scales (USAXS-to-WAXS) using monochromatic X-rays and SNAPSHOT, for high-speed, time-resolved studies enabling cutting-edge dynamics investigations using high-flux, highly-focused 'pink' X-rays.

The USAXS region observes the current missing information by probing length scales that are in the hundreds to thousands of nanometers. SCM materials are formed from molecular- to nano-structured building blocks that deform under moderate thermal fluctuations or mechanical stresses [3]. The size, shape and spatial distribution of these structures determines many intensive material properties such as viscosity, ductile and tensile strength, and compressibility. As composition of materials can be varied, there are several questions that are routinely investigated to understand an emergent property such as what are the size and shape of structural features present in the material? How are these features spatially arranged or correlated? And do these features relate to a desired mechanical, electronic, interfacial or biomedical property?



Optics hutch of beamline B21. Copyright: Diamond Light Source Ltd 2021

Understanding these questions have been particularly important in polymer composites with dispersed nanoparticles (polymer nanocomposites). Akin to concrete, polymer nanocomposites contain nanoparticles acting as an aggregate bonded together by the suspending polymer. The type and concentration of the suspended nanoparticles (dispersed state) fine-tunes the mesoscale structural features within the nanocomposites, however, the necessary molecular to micron-scale descriptions of these dispersed states remains unknown. Routine USAXS-to-WAXS measurements of varying polymer nanocomposite blends hold the key to unlocking the great promise of polymer nanocomposites as a future, designer material.

Currently, USAXS is an uncommon, specialized measurement using either a Bonse-Hart camera or an extremely long camera length where the investigator must choose either USAXS or SAXS during a measurement, never both simultaneously [4]. Bonse-Hart cameras provide exceptional resolution at the cost of extended exposure times. France and Japan both maintain USAXS instruments with camera lengths of 31 and 160.5 meters, respectively [5]. Here, a complete USAXS-to-WAXS picture requires several measurements in different camera configurations and for samples that exhibit X-ray radiation sensitivity, the measurements would be repeated on many samples.

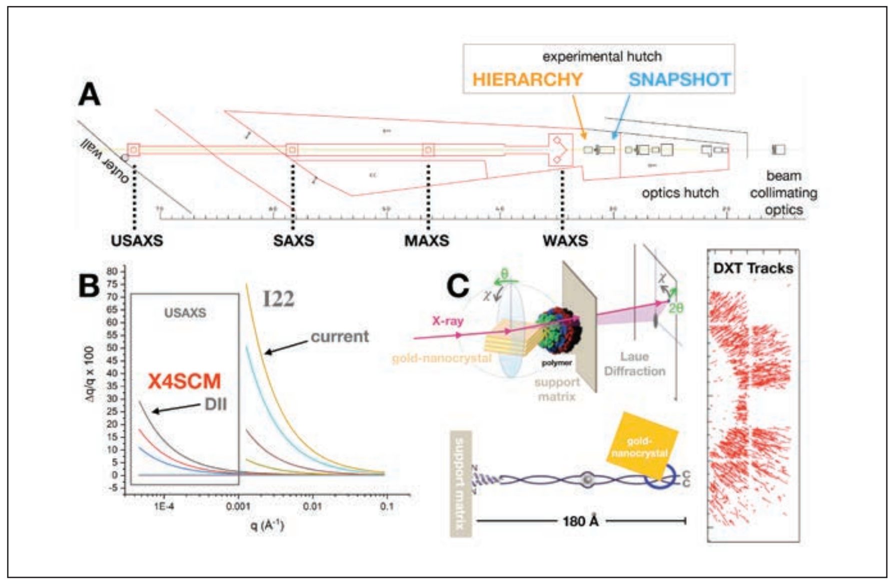


Figure 3. A. Proposed layout for X4SCM. Dual end-station design supporting HIERARCHY, for USAXS-to-WAXS measurements along a 38-meter long camera with four strategically placed detectors and SNAPSHOT, for dynamics studies of polymers using high-flux, polychromatic X-rays. B. Gain in USAXS region in comparison to current capabilities on I22. C. Diffracted X-ray tracking (DXT). Polymers are individually labelled with single gold nanocrystal. Polychromatic X-rays support Laue diffraction of nanocrystal. Rotation or wobbling of gold produces motions of the diffracted spot that are recorded on a high frame rate detector. Recent experiments at Diamond used gold-nanocrystal attached to a 180 Å long coiled-coil protein, 1000s of observed tracks (right panel) were used to measure available dynamics of the protein.

HIERARCHY will have 4 strategically placed detectors arranged along a 38-meter camera allowing for simultaneous measurements from the USAXS to WAXS regions *(Figure 3)* thereby reducing exposure and measurement times whilst providing a complete picture of the micron to molecular length scales during a single exposure. The HIERARCHY modality of X4SCM will provide a complete picture of test materials in operando enabling valuable insights for material engineering.

DXT and XFMS will offer UK and EU science communities unique experimental capabilities.

Many SCM materials exist in meta-stable states that deform under moderate forces with slow (greater than seconds) relaxation times. These time scales are accessible at existing synchrotron SAXS instruments, however, observations at time-scales relevant to molecular thermodynamic relaxations (sub-milliseconds) are not readily available. The SNAPSHOT modality of X4SCM will introduce two new techniques to the Diamond science community, namely Diffracted X-ray Tracking (DXT) and X-ray Foot-printing Mass Spectrometry (XFMS) [6-7].

DXT *(Figure 3C)* is a single molecule technique that tracks the motions of gold nanocrystal-labelled materials using the polychromatic X-ray mode of X4SCM. Polychromatic X-rays maintain the so-called Laue diffraction condition, allowing the wobble and rotation motions of an individual gold nanocrystal, tethered to a polymer, to be tracked in real-time (200 nano-second resolution). DXT informs on the polymers intrinsic degrees-of-freedom and has been applied to synthetic rubber formulations and several protein systems including membrane proteins, a class of proteins implicated in many diseases and conditions.

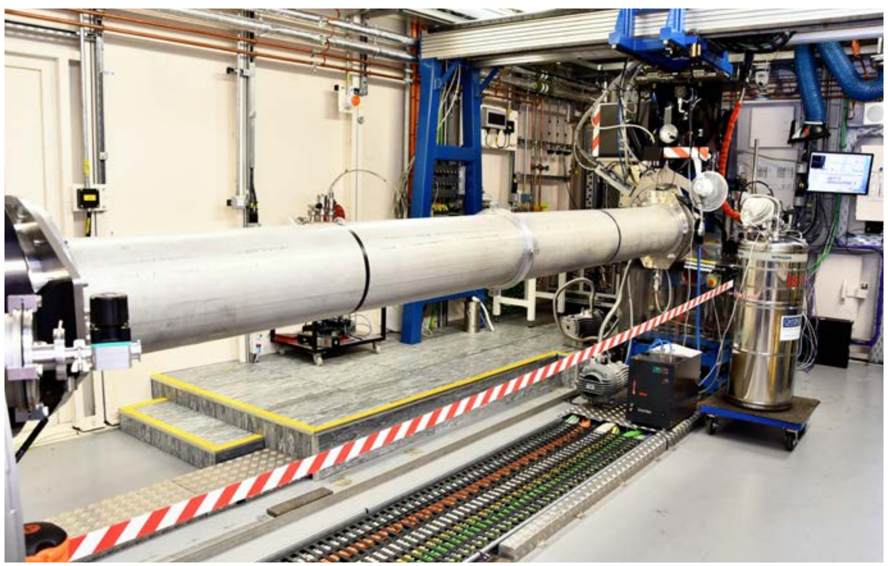
In contrast, XFMS uses the polychromatic X-ray source to induce localised, hydroxy-radical damage of the polymers. In an aqueous environment, X-rays produce short lived (less than nano-second) hydroxy radicals that react specifically with aliphatic hydrogens in the polymers to form distinct chemical adducts that are subsequently identified and mapped with high-mass accuracy mass spectrometers. For self-assembling polymers and biopolymers, XFMS maps in time the accessibility of residues to water molecules, within cavities or at interfaces, that are transiently exposed. The X4SCM proposal adds DXT and XFMS to the Diamond user program offering unique experimental capabilities to the UK and EU science communities.

Beamline I22. Copyright: Diamond Light Source Ltd 2021

New Rules will fundamentally determine the design principles of material engineering

SCM materials self-assemble from molecular constituents forming dynamic hierarchical structures that are partially ordered on molecular to micron length-scales. The desired mechanical, electrical or optical responses to external stimuli are macroscopic properties. These are emergent properties resulting from the chemical composition, network of non-covalent and covalent interactions and hierarchical level of organisation. X4SCM optimally exploits the Diamond-II upgrade, providing a seamless picture from the molecular to micron length scales allowing the UK science community to uncover, understand, and describe, in detail, the molecular rules for hierarchical assembly. These rules fundamentally determine the design principles of material engineering.

For example, X4SCM will be critical to addressing challenges in renewable resources by promoting the development of biomaterials into widespread commercial and medical applications. Cellulosic biomass production far exceeds demand of petroleum based polymers, yet, the widespread conversion of cellulosic feedstocks into useable materials remains an unrealised goal. The Diamond-II upgrade and X4SCM will usher in a new era of competitive and economic advantages for the UK by strengthening core SCM research and development groups across the UK. This investment in X4SCM ensures the world-class competitiveness of the UK's national synchrotron and enables SCM to continue to improve every aspect of quality of life.



Beamline I22. Copyright: Diamond Light Source Ltd 2021

Diamond Light Source Aerial View. Copyright: Diamond Light Source Ltd 2021

For more information about the Diamond-II X4SCM beamline please contact:
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