

UK-ESS Instrument Project

Instrument Science case

Introduction

The Instrument sub-project is tasked with delivering neutron instruments into the ESS operational beamline portfolio. At present the scope of the project is to deliver the Loki small angle scattering instrument and to complete phase 1 for the Freia reflectometer. Alongside this activity the project team are working with the CNR to prepare for the Vespa, spectrometer, scope setting meeting.

The wider scientific and impact benefits are detailed in the UK-ESS business case: UK-ESS-17070.

This document sets out the science case for these three instruments.

Science Case

Loki

Small angle neutron scattering is a technique that is applied across a spectrum of scientific disciplines, with users from chemistry, physics, biology, materials science, engineering and geoscience. LoKI is designed primarily with the needs of the soft matter, biophysics and materials science communities in mind and the trend in all of these fields is towards complexity and heterogeneity.

Complexity manifests itself in the study of multi-component systems studied as a function of multiple environmental conditions (e.g. pressure, temperature, shear, magnetic field) simultaneously. In order to be able to examine the possible parameter space, a combination of faster measurements, measurements on smaller sample volumes, and measurements with good signal-to-noise is required.

Heterogeneity is seen both spatially and temporally. Spatial heterogeneity is manifested as different structure at different length scales, from the nanometre scale to the millimetre scale. This can be driven by applied stimuli such as shear, flow, electrical or magnetic fields, or by intrinsic structural features of the material. Examples of the former are shear banding in surfactant systems and the flow re-orientation of polymers. Examples of the latter are nano-composite materials, multi-component gels, and porosity in rocks. To address this spatial heterogeneity requires a wide Q range to examine the sub-micrometre length scales and small beams to examine the heterogeneity on the millimetre scale. Furthermore, since these heterogeneities are often driven by non-equilibrium conditions, the accessible Q range must be measured simultaneously.

Temporal heterogeneity is seen in the form of stimulus-response experiments (e.g. shear relaxation), in the kinetics of formation of materials when the components of the material are mixed (e.g. mixed micelle formation) and in the growth of biomolecule aggregates such as fibrils.

In order to examine these systems with sufficient time resolution a high neutron flux is required and a wide simultaneous Q range is needed.

Not only are the systems of interest becoming more complex and heterogeneous, but they are also becoming smaller in volume. Examples of this are the small amounts of protein complexes that can be purified and deuterated, thin film systems such as organic photovoltaics, and bio-mimetic or polymer membranes. In these cases it is vital that the instrumental background be as low as possible in order to discern the small scattering signal obtained.

Through the use of neutron event recording, which identifies the time of arrival of each neutron at the detector, LoKI will allow the scientist to make this decision on the fly and variably as a function of Q. This is valuable for materials such as block-copolymer blends where there are strong peaks in the data that require high resolution but the inter-peak scattering is of lower intensity and so requires more neutron counts but can make use of lower resolution. In particular there is a need to have good Q resolution at high Q, for example in studies of fibres, liquid crystals or multi-lamellar systems such as lipid or surfactant vesicles. The ability to make use of longer wavelength, higher time resolution, neutrons at high angles will be key to being able to make use of post-measurement resolution tuning. This requirement demands a large solid angle of detectors and thus potentially a large area detector close to the sample.

The ability to reach Q values above the typical 0.8 to 1.0 \AA^{-1} opens up significant areas of new science when combined with wide-angle scattering studies and the rapidly advancing fields of materials simulation. The study of, for example, ionic liquids and their ordering in the presence of solutes calls for the combination of SANS, wide-angle scattering and atomistic or coarse-grained molecular dynamics simulations. The analysis techniques used in biological solution scattering, such as ab-initio shape reconstruction, require data out to high Q as do studies of nano-composite materials where the size of the particles or domains may be only a few nanometres.

Non-equilibrium studies often use complex sample environment and require strong integration of the sample environment with the neutron measurement in order to tie sample conditions tightly to the measured scattering. Space is also required for the use of in-situ complementary measurement techniques, for example light or x-ray scattering, or UV spectroscopy simultaneously with the neutron scattering measurement. LoKI has a flexible sample area that can be easily re-tooled for different experiments through the use of interchangeable sample environment platforms.

Whilst some of the types of experiment mentioned above are done at present, they are often tour-de-force studies pushing the capabilities of today's instruments. LoKI provides a world leading combination of high neutron flux and wide simultaneous Q range with the ability to study small samples.

With these characteristics, LoKI will enable scientists to answer the challenging materials science questions of tomorrow in fields from health and aging, to sustainability and energy security.

Freia

Neutron reflectometry covers a very broad spectrum of science involving the growth, self-assembly, structure and interactions of a wide variety of thin films and has an impact on all the core areas of the ESS materials science case. This leads to a broad range of requirements on sample size, resolution and bandwidth in all types of reflectometry experiments.

The range of scientific challenges to be met in soft condensed matter and the life sciences is broad, and requires a number of different collimation options and specialist sample environments to carry out measurements at different types of interfaces. However, a common feature is that in order to be able to examine the relevant parameter space in increasingly complex materials, faster measurements, measurements on smaller sample volumes, and measurements with good signal-to-noise are required. Similar issues are equally relevant in a wide range of materials chemistry and hard condensed matter science. Consequently, the ability to match the experimental throughput to the ESS source performance in terms of the time needed for sample changes, data processing and analysis constitutes one of the key challenges in maximizing the scientific output of the instruments. While the sensitivity of neutrons to structural features offers a significant advantage in all types of multicomponent systems, there is a clear trend to follow time-dependent processes due to the development of time-of-flight reflectometers in the past three decades at facilities world-wide. These processes include, but are not limited to:

- Self-assembly of surfactants, polymers and proteins at solid and liquid interfaces
- Rearrangement processes in thin films: e.g. interdiffusion, inter-layer movement
- Encapsulation and release of components in e.g. plastics, polymer blends, drug delivery
- Switchable materials that respond to external stimuli (chemical, electrical, magnetic)
- Surface reactions e.g. enzyme catalysis, oxidation, surface functionalisation etc.
- High-throughput screening of e.g. biological/medical samples or industrial conditions
- Liquid-liquid interfaces: e.g. heavy metal extraction and oil-recovery processes

Time-of-flight (tof) neutron reflectometry offers the possibility to record a range of Q-values simultaneously, and determination of both structure and chemical composition as a function of time during such processes. The usefulness of tof-reflectometry critically depends on the ability to match both the time-resolution and the dynamic Q-range of the measurements to the structural changes investigated. The main challenge for kinetic measurements is to record the full range of interest simultaneously without need to move the sample or reconfigure the instrument. The length scales of interest for neutron reflectometry span 1\AA - 1000\AA , so variable wavelength resolution options are required. For the majority of purposes in specular reflectometry a Q-range of $0.005 - 0.5 \text{\AA}^{-1}$ is sufficient, however, for surface diffraction experiments from multilayer samples, access to Q up to 1\AA^{-1} is often required. Liquid-liquid interfaces and many sample environments such as rheometers further require an inverted beam geometry in which the beam impinges on the interface from below.

Off-specular reflectometry and grazing incidence SANS are expected to become mainstream techniques at ESS. Polarisation analysis is typically not required in soft matter experiments, but polarised reflection from magnetic reference layers for magnetic contrast variation is a popular method.

Vespa

The fundamental idea behind the *Neutron Vibrational Spectroscopy* (NVS) technique is analogous to that exploited in optical spectroscopy: a sampling probe, carrying energy larger than that of the internal excitations, is directed at the sample. The resulting neutron energy loss upon excitation of a vibrational mode gives direct information on the vibrational energy level structure of the sample. Vibrational spectroscopy is a fundamental technique used constantly in educational, research, and industrial laboratories all over the world. Its applications in the investigation of solids and liquids, soft matter, complex fluids, and biomaterials are well-known. Indeed, it has become an essential tool in medical applications, forensics, environmental compliance, and quality control to cite but a few common uses. Fundamentally, vibrational spectroscopy probes potential energy surfaces and interatomic interactions. Alone or in combination with other techniques, vibrational spectroscopy permits the identification of bonds and functional groups, as well as the transformations that occur when bonds are broken and made in chemical reactions (e.g. in catalysis or thermal decomposition). The vibrational density of states is of great interest in itself, being related to various thermodynamic properties such as specific heat or entropy. The vibrational spectrum is affected by configurational changes in molecules thereby also providing structural information. NVS exploits the large incoherent scattering cross section of the hydrogen nucleus. Proton dynamics or vibrations connected to the movement of 'H' atoms can be easily detected spectroscopically, even if 'H' is dissolved at very low concentrations in materials composed mostly of heavier atoms. For this reason, the technique attracts a high interest in the scientific community operating in the fields of chemistry, materials science, physics and biology, with a particular emphasis on applications.

One of the big advantages of an NVS instrument resides in the possibility to get a picture of the complete spectrum "in one shot". The science case pushes us to optimize the instrument for the fingerprint (60-220 meV) region, however extending the energy range to 500 meV comes at no extra cost. In other words, the high-speed neutrons can be used (or not used) without essentially changing the instrumental design. The instrument length has been optimized to provide a broad spectral range while maintaining the desired high energy resolution ($\approx 1-2\%$ of the incoming neutron energy). The possibility of optimizing the resolution at the expenses of the count rate or, on the contrary, sacrificing the resolving power to increase the instrument flux at the sample position, is a bonus that makes VESPA unique among many other indirect geometry instruments worldwide. Nonetheless, once a configuration is selected, the full spectra are collected in a single ESS period, thus making kinetic or parametric experiments feasible and easy to interpret.