12th International Workshop on Sample Environments at Scattering Facilities

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Book of Abstracts

Abstract # 17

Brand New Helium Recovery and Liquefaction System for the NIST Center for Neutron Research

In 2024, NCNR unveiled its long-anticipated Helium Recovery and Liquefaction System, designed for optimal functionality. This system efficiently captures Helium gas from cryostats, magnets, transport Dewars, and pump exhausts. Boasting a high-pressure storage capacity of 2,000 liquid liters and a liquefaction rate of 300 liquid liters per day, it fulfills NCNR's current neutron reactor operation needs, supporting experiments and equipment tests.

A notable feature is its modular design, incorporating a stainless-steel Helium recovery manifold, 180-cylinder high-pressure storage, and three GM cold heads-powered liquefaction units. This modularity adapts to evolving facility needs, potential liquid Helium demand increases, maintenance requirements, and certification standards for high-pressure cylinders.

Operating seamlessly with full automation, the system requires minimal operator intervention, focusing on liquid Helium transfer and routine maintenance. Throughout the facility, thirty customdesigned End-User Stations efficiently capture and reheat Helium gas to prevent pipe condensation.

Strategic Helium Monitor Stations along the recovery manifold's main branches monitor gas flow rate and purity, automatically venting contaminated gas to prevent system-wide Helium inventory contamination and extensive downtime for cleanup.

Further details and specifications of the system as well as lessons learned will be presented.

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Presenter: Dr VEKHOV, Yegor (University of Maryland / NIST Center for Neutron Research)

Contribution Type: Oral

Abstract # 18

New laser furnace for the STRESS-SPEC instrument

Components and materials from new production processes, i.e. additive manufacturing (AM), require highly flexible sample positioning systems for residual stress diffraction experiments. Therefore, the STRESS-SPEC group has pioneered the use of industrial robots for sample handling and positioning at neutron diffractometers [1, 2]. To fully exploit the capabilities of the robotic positioning system, a dedicated sample environment is essential. In this talk, we will present a recently developed lightweight laser furnace with a large neutron acceptance angle, which allows the investigation of samples at elevated temperatures up to 1200 °C, while benefiting from the positioning flexibility of a 6-axis industrial robot. Some features and example use cases of the laser furnace are presented. The furnace control rack was built with interoperability in mind, allowing control of various other sample environment devices as well.

Furthermore, we will also give an outlook of an ongoing development of a lightweight mechanical tensile testing machine that can also be mounted on a 6-axis industrial robot.

References

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Contribution Type: Oral

Abstract # 19

CRYOGENIC GONIOMETERS AT NCNR, NIST

We would like to introduce low-temperature goniometers for neutron research that can operate at ultra-low temperatures to 10 mK and under external magnetic fields to 20 T. These devices allow the realigning and rotation of a single crystal inside a cryostat during a neutron experiment without significant disturbing the experimental conditions. The application of this device for neutron experiments on single crystal both will reduce the amount of time preparing or modifying experiments and, for some cases, will make some experiments possible.

Figure 1 represents set of devices which can be used with different types of cryostats and cryo-magnets. The different types of attocube Low Temperature Nanopositioners have been used to provide rotation and alignment of the samples. Characteristics of motors allow to use these devices at extreme conditions and inside the limited space. We are demonstrating several Goniometer designs which allow sample alignment and rotation around variable axes. These devices have maximal transparency of the sample surrounding for neutron beam as well as providing shielding from possible disruption of the signal from sample environment.

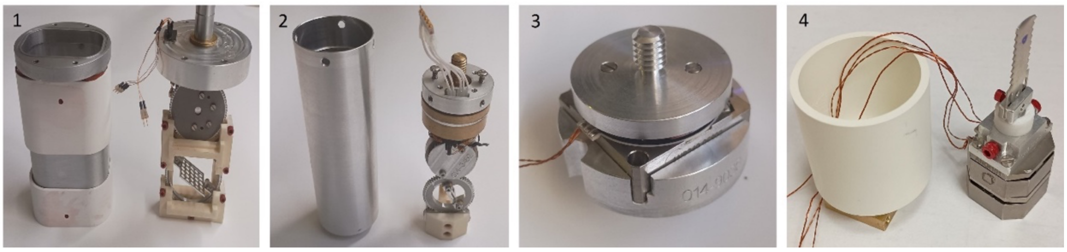

Figure 1. Low temperature Goniometers: 1.70 mm Goniometer (360 degrees of rotation around horizontal axis); 2. 50 mm Goniometer (360 degrees of rotation around horizontal axis); 3.50 mm Rotator (360 degrees of rotation around vertical axis); 4. Small angle Goniometer (alignment around horizontal axes).

Our special attention has been given to the problem of sample thermalization at ultra-low temperatures. We implemented hardware and software solutions to efficiently thermalize sample at wide temperature ranges as well as reduce overheating occurs for devices during motor operation. Multiple tests demonstrated successful solution of sample thermalization problems for different cryostats. Also, we demonstrated non-interrupted well controlled rotation and alignment of the sample in wide range of temperatures.

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Contribution Type: Oral

Abstract # 20

A versatile humidity generator

Following the very successful development of the ultimate humidity chamber with colleagues (and friends) of the HZB [1] which is often used on D16 [2], we have been asked by several teams of users to develop a fast humidity generator combining H2O, D2O and/or an optional liquid/gas for carrying out diffraction, reflectometry and inelastic neutron scattering experiments.

This humidity generator uses the well-known technique which consists in mixing a dry fluid with a fluid saturated at 100% humidity with controlled flow rates [3,4]. The generator comes with three bottles filled with H2O, D2O and an optional liquid chosen by the user. From these bottles, the system fills pairs of bubbling saturators which are thermally controlled by Peltier elements mounted on a common plate whose temperature is regulated with a fluid circulating through a thermostatic bath. As for the dry line, it is fed with nitrogen or an optional gas circulating in a heat exchanger thermalised in the same way.

From the touchscreen, the user selects the wanted temperature, relative humidity, total flow rate and the flow ratios of D2O and optional liquid/gas. The system calculates the individual flows to reach the wanted conditions and starts circulating the gases. When working with D2O, H2O and dry N2 the system calculates the exact flows from known equations. With other liquids and gases, the user retrieves the flows and all other parameters allowing to describe the mixture. The system automatically refills the chambers during operation when necessary to allow the operation of long experiments. Of course, it is also possible to flush the system with different dry gases and control individually all components (Peltier elements, mass-flow controllers) with an administration mode.

[1] J. Gonthier, M.A. Barett, O. Aguettaz et al., BerILL: The ultimate humidity chamber for neutron scattering, J. Neutron Research 21 (2019) 65

[2] V. Cristiglio, B. Giroud, L. Didier and B. Demé, D16 is back to business: More neutrons, more space, more fun, Neutron News 26(3) (2015) 22

[3] H. Arima-Osonoi, N. Miyata, T. Yoshida et al., Gas-flow humidity control system for neutron reflectivity measurements, Rev. Sci. Instrum. 91 (2020) 104103

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Contribution Type: Oral

Abstract # 21

The Latest Status of Sample Environment at CSNS

The sample environment at the Chinese Spallation Neutron Source (CSNS) has been providing in-situ experimental services since the commissioning of the instrument in 2018. As of February 2024, CSNS has completed 11 rounds of open operation, and the demand for beam time exceeds the supply. The neutron beamlines have completed over 1300 research projects. It has evolved from initially serving as a cryostat to now supporting a wide range of sample environments, including low temperature, high temperature, high pressure, and magnetic field, catering to different user needs. The development has enabled the transition from single-sample environment experiments to multiple-sample environment experiments, showcasing the progress from 0 to 1 and from 1 to many in terms of experimental applications. This article primarily introduces the operation and usage of the sample environment at CSNS. It also highlights recent developments and optimizations in sample environment. Additionally, some experimental applications conducted in collaboration with users are also presented.

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Presenter: HU, Haitao (IHEP)

Contribution Type: Oral

Abstract # 22

Uniaxial Pressure for Neutrons and X-rays

Material Physics experiments often explore competing and intertwined orders. Here I will present our recent advances in using uniaxial pressure as a clean "surgical" tool to tune these orders while simultaneously obtaining microscopic insights via scattering experiments. The realizations of experiments are achieved through technical developments by minimizing the background and enabling the tuning in-situ [1].

First I will discuss our efforts to observe weak signals by designing a low-background uniaxial strain cell, optimizing the experiment based on neutron-tracing simulations and using aggressive focusing and energy analysis. I will illustrate the process with two experiments where we studied incommensurate magnetism [2] and superconductivity [3] in cuprates.

To achieve the fine-tuning in-situ, we have designed a new in-situ uniaxial device for large-scale facility research based on an actuator-motor mechanism, efficient feedback loops and the sampleholder design enabling rapid exchange of the samples [4]. I will demonstrate the advanced capabilities of this device by reporting the control of charge and structural degrees of freedom as studied by X-rays in an archetypical cuprate [5,6].

[1] Simutis et al., Swiss Neutron News 62, 14 (2023)
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[3] Küspert et al., arXiv:2312.03650 (2023)
[4] Simutis et al., Review of Scientific Instruments 94, 013906 (2023)
[5] Guguchia et al., PNAS 121 (1) e2303423120 (2023)
[6] Thomarat et al., arXiv:2401.13526 (2024)

Primary author: SIMUTIS, Gediminas (Paul Scherrer Institute)

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Contribution Type: Oral

Abstract # 23

High Pressure Research at SINQ

The Swiss neutron source SINQ at Paul Scherrer Institute has recently undergone an extensive upgrade improving the versatility of several instruments as well as increased neutron flux.

The increased performance opens up more opportunities for advanced-sample-environment research, such as experiments involving high pressure. In this contribution, we will present the current status of high-pressure sample environment at SINQ and will discuss our future plans.

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Contribution Type: Poster

Abstract # 24

High Temperature Capabilities and the Development of the Levitation Program at ORNL's Neutron Sources

The High Temperature Sample Environment (HTSE) group at Oak Ridge National Laboratory (ORNL) supports experiments at both the High Flux Isotope Reactor (HFIR) and the Spallation Neutron Source (SNS). The HTSE group deploys and maintains over 30 pieces of high temperature equipment, such as furnaces and high temperature closed-cycle refrigerators, and develops custom sample sticks for multi-condition experiments including hazardous gas flow and applied voltage. The group is also developing a suite of levitators, including three actively in the user program (one electrostatic and two aerodynamic) and four that are either in active development or currently being commissioned.

This talk will cover general capabilities of the high temperature program, as well as showcase some of the most recent developments including the integration of rapid cooling techniques, improvements to thermometry, and imaging characterization of thermal deflection and sample alignment. The newest levitation systems will also be discussed, including results of the commissioning of a solution electrostatic levitator on a neutron diffraction beamline, the development of a pressurized levitator for volatile materials at extremes, and progress on the creep electrostatic levitation system.

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Presenter: QUIRINALE, Dante (Oak Ridge National Laboratory)

Contribution Type: Oral

Abstract # 25

IN SITU X-RAY DIFFRACTION STUDIES OF STRESS-INDUCED TRANSITIONS IN SINGLE CRYSTAL PIEZOELECTRICS – DEVELOPMENT OF NEW STRESS FACILITY

To exploit the unique properties of piezoelectric single crystals – which includes their extremely large actuation stroke, their high strain sensitivities and their very high electromechanical conversion efficiencies - several key challenges remain. We have explored the crystallographic stability and dynamic characteristics of the materials, as functions of driving parameters such as electrical field, temperature, and mechanical stress, using a novel purpose-built instrument retrofitted to a modern Xray synchrotron beamline.

A dedicated stress rig ('ESPYRig') has been designed and manufactured by Electrosciences Ltd and the University of Liverpool, to explore the static and dynamic structural, electrical and optical response of a range of materials whilst under controlled applied uniaxial loads, temperatures, electric fields, and magnetic excitation. Loads to 2kN, stroke up to 100microm, electrical field isolation to 10kV, and full line of sight access to the surface of the sample for in situ diffraction have been realised within a fully automated computer-controlled system and designed to fit to a wide range of goniometers and laboratory workbenches.

In this presentation, we describe the operation of this new instrument and how it has been used successfully on several X-ray facilities and also, offline as a lab-bench operando materials test lab to characterize time-resolved in situ properties of advanced piezo crystals and traceable standards. Stress, strain, X-ray intensity, and field are all measured simultaneously and recorded using a purpose-developed data acquisition system. The instrument is now commercially available, through Electrosciences Ltd.

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Contribution Type: Poster.

Abstract # 26

A 1 GPa kit for all neutron users

Over the past decade, the demand for high-pressure systems has doubled. To meet this growing need while ensuring safe and successful experiments, we have upgraded our pressure generators and expanded our range of continuously loaded pressure cells.

We have significantly improved the 1 GPa liquid and gas pressure generators with:

- a comprehensive revamp of the automation program improving the reliability,
- a modern user interface easing control and maintenance,
- additional sensors and controls enhancing safety,
- programmable pressure ramps controlled with greater precision,
- remote control and data archiving capabilities.

We have also designed and built five 'p•T' controllers taking care of pressure equipment at cryogenic temperatures. A p•T controller takes the lead and becomes the companion of an instrument control workstation. It manages the pressure/temperature requests by adding/removing exchange gas around the high-pressure capillary of the sample stick [1], and dispatching commands to the temperature and pressure controllers at appropriate times.

With or without p•T controller, the experiments have become extremely user-friendly. This ensures biologists, chemists and physicists, including those new to high-pressure techniques, to fully leverage our equipment's capabilities safely and effectively at room and cryogenic temperatures.

Our series of pressure cells is optimised for experiments up to 1 GPa on different types of instruments. Each pressure cell offers an optimum balance between maximum operating pressure, sample volume, access to incident and scattered beams, and the adoption of materials appropriate to the scattering technique.

This includes cells specially designed for powder diffraction not producing Bragg peaks, or for neutron spin-echo and small angle neutron scattering offering outstanding performances with a very low neutron background, a high beam transmission and a reliability proven for several years.

For inelastic scattering, we have developed together a compact non-magnetic Ø6 mm sample bore double-wall cell. Accommodating up to 1 GPa pressure with either liquid or gas mediums, this cell outperforms others by achieving higher pressures and providing improved neutron transmission and signal-to-noise ratios.

[1] E. Lelièvre-Berna, B. Démé, J. Gonthier et al., 700 MPa sample stick for studying liquid samples or solid-gas reactions down to 1.8 K and up to 550 K, J. Neutron Research 19 (2017) 77

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Contribution Type: Poster

Abstract # 27

Neutron diffraction above 20 GPa and below 100 mK

The Paris-Edinburgh press is routinely used on diffractometers and starts to be used on inelastic spectrometers. A vast majority of the experiments are performed with one of our cryostats hosting a magnetic or non-magnetic VX-5 type press down to 4 K, and even sometimes down to 1.8 K [1].

In order to search for or investigate transitions occurring at lower temperatures, we have installed a VX-5 type Paris-Edinburgh press inside a Triton DR-200 dilution refrigerator delivered by Oxford Instruments Nanoscience [2].

While cooling the press to temperatures below 100 mK is relatively easy with a Triton, the cooldown time is long, reaching almost 3 days. For this reason, experiments are scheduled in a nonconventional way. For example, after loading the sample and closing the cryostat, we start collecting data at room temperature and high pressure. We then remove the cryostat from the sample table, cool down the press while another experiment is carried out, and put back the cryostat for collecting data at ultra-low temperature.

We shall present how the Paris-Edinburgh press is attached to the dilution refrigerator, how the high-pressure capillary is cooled down, why the addition of a liquid nitrogen precool loop is not a good idea, and the results of the commissioning.

[1] S. Klotz, Th. Strässle, B. Lebert et al., High pressure neutron diffraction to beyond 20 GPa and below 1.8 K using Paris-Edinburgh load frames, High Pressure Research 36 (2016) 73
[2] https://nanoscience.oxinst.com/dry-systems

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Contribution Type: Poster

Status: ACCEPTED

A new T-gradient monochromator for IN13

The thermal high-energy resolution backscattering spectrometer IN13 [1] operated at the ILL by a French–Italian CRG has recently undergone a major technical upgrade as part of ILL's Endurance program.

Among the numerous components replaced or upgraded, the monochromator used to control the incident energy has been fully redesigned to exploit the new incident guide and cover a wide range of energies.

This monochromator is built from three 10-mm thick CaF_2 crystals which underwent controlled plastic deformation in order to adapt their mosaicity to the increased beam divergence of the new guide, and by then also increase the flux at the sample by a factor 4.

The wanted incident energy is obtained by changing the temperature of the crystals, i.e. by changing the d-spacing of the (422) planes. The cryofurnace hosting the crystals allows to regulate ramps of temperature between -200°C and +250°C, i.e. to scan energy transfers from -125 to 150 μeV. As an option, it is possible to further increase the flux by applying a temperature gradient across the thickness of the crystals.

We will present how this gradient is applied with thermally decoupled aluminium plates while keeping the crystals alignment. We will also describe how the PLC controls temperature changes automatically and reliably.

[1] https://www.ill.eu/in13

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Contribution Type: Poster

Abstract # 29

The ultracold neutron source SuperSUN

SuperSUN is designed to produce ultracold neutrons (UCN) for long-duration storage experiments, and is the first user instrument in the category of “high-density” UCN sources (in contrast to the “high-flux” sources that deliver high-rate UCN beams).

The source is fed with cold neutrons by an octagonal guide [1] to best match a 3-m long circular cold-neutron replica guide with a m=3 supermirror coating, which is installed inside the cryostat. In this “convertor volume”, cold neutrons become UCN by scattering inelastically in isotopically pure superfluid ^{4}He at 0.6 K. After several minutes of accumulation, UCN are extracted by opening a valve [2]. The cold neutron beam can be independently shut off to stop UCN production.

The cryostat includes a 100-litre liquid helium reservoir, featuring a recondensing system to compensate the loss due to boil-off. A 1-K pot is filled from this reservoir via a cold valve, and is used to provide isotopically purified superfluid ^{4}He for the convertor through a superleak filter. It also serves to precool and liquefy circulated ^{3}He gas for the final cooling stage. The Joule–Thomson thermal expansion of the ^{3}He is performed after a fixed impedance, placed at the entrance to the ^{3}He/^{4}He heat-exchanger that is used to cool the convertor.

All pumps, valves, and superfluid levels are controlled by a PLC and monitored via a touch-screen HMI, which can also be accessed remotely. The cryostat is cooled using 5 commercial cold-heads, and refilled with 80 litres of liquid helium once a week. The base temperature at 0.6 K is stable to better than 5 mK over several weeks.

During initial operation in 2023, SuperSUN detected 3.8 10^6 UCN from a single filling cycle of the 14-litre source volume, at a reactor power of 48.6 MW. This confirms the expected performance in terms of integral UCN output [3]. SuperSUN will be upgraded to provide polarised UCN, using a 3-m long superconducting octupole magnet that will act as a magnetic trap around the replica guide in the convertor volume.

[1] S. Degenkolb, M. Kreuz and O. Zimmer, A tapered transition guide with irregular octagonal cross-section, J. Neutron Research 20 (2018) 117

[2] E. Chanel, S. Baudoin, M.-H. Baurand et al., Concept and strategy of SuperSUN: A new ultracold neutron converter, J. Neutron Research 24 (2022) 111

[3] D. Wurm et al., The PanEDM neutron electric dipole moment experiment at the ILL, EPJ Web of Conferences 219 (2019) 02006

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Contribution Type: Poster

Abstract # 30

A fully modernised instrument suite with new sample environment capabilities

Time to celebrate! The Endurance Programme [1] is now complete and the results of recent experiments clearly show that ILL has made a massive step forward by replacing or upgrading the cold and thermal neutron guides and half of the instrument suite.

The "neutron environment" team has contributed to this investment programme by developing and commissioning an ultra-compact Be filter for D22, a cryofurnace regulating the T-gradient applied on the CaF_2 monochormator of the backscattering instrument IN13, and a very reliable ultra-cold neutron source reaching unprecedented performances [2].

In addition, to boost sample environment capacities and capabilities, we have upgraded all Ø49 cryostats with double heat-exchangers, built a second Cryocradle for orienting single crystals quickly in the zero-field polarimeters Cryopad, built ten dilution inserts and additional gas handling systems for cryostats and cryomagnets, acquired ten 6-position sample changers for SANS and ordered a 12 T high-Tc superconducting magnet which will host an ILL variable temperature insert.

WehavealsodesignedaversatilehumiditygeneratorcomplementingtheultimateBerILLhumi dity cells [3], commissioned a Langmuir setup for the horizontal reflectometer FIGARO and equipped a dilution cryostat with a Paris-Edinburgh press. Substantial efforts have also been injected in the high-pressure field with the PSI team to push the limit of continuously loaded cells to 1 GPa.

A brief overview of this work will be presented, supplemented by posters.

[1] https://www.ill.eu/users/instruments/modernisation-programmes/endurance
[2] https://www.ill.eu/news-and-events/news/scientific-news/supersun-instrument-ready-for-use [3] J. Gonthier, M.A. Barett, O. Aguettaz et al., BerILL: The ultimate humidity chamber for neutron scattering, J. Neutron Research 21 (2019) 65

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Contribution Type: Oral

Abstract # 31

SAMPLE ENVIRONMENT AT HZB

The Sample Environment Group at the Helmholtz-Zentrum Berlin is dedicated to the development and implementation of advanced sample environment equipment and methodologies at the BESSY II beamlines across various focus areas. In recent years, significant emphasis has been placed on in-situ and operando measurements, particularly in the fields of electrochemistry and catalysis experiments. Through several key projects [1,2,3], we have been developing customized cells and the necessary support infrastructure to enhance these experimental capabilities.

In our projects, we explore the benefits standardizing hardware [3] and software communication. The latter relies on the implementation of the Sample Environment Communication Protocol (SECoP). For SECoP, we have further worked on the completeness of metadata through the SECoP@HMC project, in collaboration with several partners and the Helmholtz Metadata Collaboration [4].

The requirement for fully automated experiments with high throughput has driven the development of modular robotic sample changers for different applications, e.g. for catalysis in the ROCKIT project [3]. Here, again, we benefit from shared development efforts aimed at standardizing procedures and hardware across multiple facilities.

Within the League of European Accelerator-based Photon Sources (LEAPS) the project LEAPSINNOV [5] has entered its final year. Here we were participating in the developments for “New positioning and scanning systems for speed and accuracy” specially focusing on the development of interferometry systems for nano-positioning applications. Collaboration in these projects has been crucial in achieving our goals.

The Helium-Management project, our collaboration for the holistic monitoring of Helium, is progressing to the next stage, where we are preparing for commercialization. This will make our developments accessible to a broader community.

In this presentation, we will showcase the latest developments in the aforementioned topics.

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[4] https://helmholtz-metadaten.de
[5] https://www.leaps-innov.eu

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Presenter: KIEFER, Klaus (Helmholtz-Zentrum Berlin)

Contribution Type: Oral

Submitted by KIEFER, Klaus on Wednesday 10 July 2024

Abstract # 32

Facility News from PSI

The operation and improvement of its large scale facilities is one of PSI's core missions. The ongoing upgrade of the synchrotron and the planed upgrade of the muon extraction are very prominent showcases. At SINQ, new instrumental capabilities and a diversifying user demand affect the work of the sample environment. I will provide an overview of the ongoing projects and elucidate the opportunities and challenges for the sample environment group.

Primary author: Dr BARTKOWIAK, Marek (Paul Scherrer Institut)

Presenter: Dr BARTKOWIAK, Marek (Paul Scherrer Institut)

Contribution Type: Oral

Status: ACCEPTED

Submitted by Dr BARTKOWIAK, Marek on Monday 15 July 2024

Abstract # 33

Nifty Tricks with Dilution Sticks

The support of neutron scattering experiments at ultra-low temperatures and high magnetic fields has been an earmark of the sample environment support at SINQ. We would like to present a selection of tricks that we developed to make some experiments possible and our life easier.

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Contribution Type: Poster

Status: ACCEPTED

Submitted by Dr BARTKOWIAK, Marek on Monday 15 July 2024

Abstract # 34

The MAX IV Facility Report

The MAX IV Laboratory is a synchrotron-radiation research facility built on a linear electron accelerator feeding electrons to two storage rings operated at 1.5 and 3 GeV, respectively. The MAX IV facility provides an ultra-low electron-beam emittance with a very high X-ray brilliance. The facility covers research using short X-ray pulses on the linear accelerator, UV-, and soft X-rays (< 2 keV) at the 1.5 GeV storage ring, as well as hard X-rays (2 - 50 keV) at the 3 GeV ring.

The MAX IV Laboratory has a team dedicated to Sample Environments and Detector Support (SEDS), belonging to the MAX IV Beamline Office (BO). Currently, it consists of three staff members: Researcher Stefan Carlson (team leader), research engineers Artur Domingues, and Christopher Ward. The SEDS team supports end stations at beamlines with instrumentation development, spare-equipment pool, and fast access workshops for mechanical and electronics work. In addition, labs for assembly and testing of sample environments and detectors are operated by the SEDS team.

A brief report on the latest development by our sample-environment team and MAX IV beamlines will be given.

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Presenter: CARLSON, Stefan (MAX IV Laboratory)

Contribution Type: Oral

Abstract # 35

DESIGN CONSIDERATIONS OF SPLIT PAIR MAGNETS FOR NEUTRON SCATTERING APPLICATION

High B field magnets with wide vertical and horizontal angles access are an essential and integral part of the modern neutron beam line facility. The demanding specifications result in high peak B field densities in the superconducting wires, large electromagnetic forces on the coils, high mechanical loads on the magnet formers and high stored energy density. Such magnets require complex quench management schemes for magnet protection.

Moreover, the presence of high magnetic field imposes design challenges on the beamline construction itself. The use of magnetic and high electrically conductive materials must be controlled to limit magnetic forces on the beamline components and sensitive equipment. It is well known that ferromagnetic materials in the vicinity of superconducting magnets can cause large static forces and should be avoided in beamline construction. Dynamic forces that can occur due to eddy currents during a quench have been much less well studied. These can be made worse by the use of non-magnetic, but highly conducting, aluminium as a construction material. This is a particular problem for vacuum tank instruments where slits cannot be easily introduced.

An example of a 14 T split pair magnet design for two ESS beamlines is described with detailed magnetic force calculations for TREX and CSPEC sample environments. Surprisingly, high forces are seen, comparable to or greater than typical magnet design parameters. The design limitations and the solutions deployed for specific applications to meet these challenging demands at ever higher magnetic fields are described.

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Co-authors: DrTWIN,Andrew(OxfordInstruments); DrBURGOYNE,John(OxfordInstruments); Dr HOLMES,Alexander(EuropeanSpallationSource); DrDEEN,Pascale(EuropeanSpallationSource); Dr FRANZ, Christian (Forschungszentrum Jülich GmbH)

Presenter: Dr VIZNICHENKO, Roman (Oxford Instruments)

Contribution Type: Oral

Abstract # 36

CAN A TINY DROPLET OF QUANTUM FLUID STOP UP-HILL FLOWING LIQUID AND HOW IS IT RELATED TO SAMPLE ENVIRONMENT?

Since medieval times, water flowing uphill was considered to be a miracle (for example, this effect was one of the Seven Wonders of Fore Abbey). However, in the quantum world superfluid helium 4He can defy gravity and is able to climb out of any container in a thin film that moves with such a pace that the container is drained in minutes. Here we present a study of pure 4He and 3He/4He mixture films using specular neutron reflection, in the temperature range of 150 mK to 1.5 K [1]. Thanks to the exceptional sensitivity and precision of this technique, we have observed a phaseseparated 3He/4He mixture film at 170 mK, and have been able to watch the gradual dissolution of its 3He top layer into 4He with increasing temperature, in agreement with current theories. Furthermore, the surprising behavior of the helium mixture at 300 mK hints at the possibility of an as-yet unstudied geometrically restricted phase transition. The subsequent restoration of the layered structure at 1.5 K was equally unexpected. We also developed a sample environment application based on the surprising behavior of the helium mixture at 300 mK.

References

[1] Kirichek, O., Lawson, C.R., Kinane, C.J. et al. Density profile of 3He in a nanoscale 3He-4He superfluid film determined by neutron scattering. Commun Phys 7, 181 (2024).

Primary authors: KIRICHEK, Oleg (ISIS neutron and muon source); Dr JONES, Alex (ISIS); KINANE, Christy (ISIS Neutron Source); Dr LAWSON, Chris (ISIS)

Presenter: KIRICHEK, Oleg (ISIS neutron and muon source)

Contribution Type: Oral

Abstract # 37

AUTOMATED GAS SUPPLY AND ANALYSIS SETUP FOR OPERANDO X-RAY INVESTIGATIONS OF CATALYTIC PROCESSES

ROCK-IT(Remote, OperandoControlled, Knowledge-driven, andIT-based)isacollaborationproject of the four German Helmholtz institutes DESY, HZB, HZDR and KIT. The project aims to establish a fully automated experimental setup that enables remote-controlled operando catalytic experiments at synchrotron light sources.

As a demonstration reaction, the hydrogenation of CO2 to methane has been chosen, an important Power-to-X process:

$CO_2 + 4 H_2 \quad CH_4 + 2 H_2O$; $\Delta HR = -164$ kJ/mol

The gas supply and analysis presented are defined according to the selected demonstration reaction, which is conducted at ambient pressure and reaction temperatures up to 600 °C. During methanation, H2, CO2 and an inert gas for dilution are supplied via mass flow controllers. Various gas analysis methods, including mass spectrometry, gas chromatography and FTIR spectroscopy, can be used to analyze the reaction products. These methods allow both quantitative and qualitative analysis of the resulting products and can be applied to other gas catalytic reactions as well. To analyze liquid by-products of catalytic reactions, the gas stream coming from the reactor must be dilutable and heatable to at least 100 °C.

In addition to the analytical data, process parameters such as temperature, gas flow and pressure are continuously recorded during the experiment. All relevant devices in the system must have interfaces for digital data acquisition and device control, as well as options for in-situ calibration measurements.

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Presenter: RULAND, Gina (HZB)

Contribution Type: Poster

Abstract # 38

SIZE-EXCLUSION CHROMATOGRAPHY AND SMALL ANGLE NEUTRON SCATTERING AT ORNL

Size exclusion chromatography (SEC) is a routine method to purify bio-macromolecular complexes. SEC has regularly been combined with small angle scattering at X-ray sources (SEC-SAXS). This enables the removal of aggregated material and allows the study of complex materials. However, there are a limited number of examples of SEC being combined with small angle neutron scattering (SANS) experiments due to flux limitations as well as higher sample volume and concentration requirements.

While there are limitations with flux and sample volume and concentration, SEC-SANS has now been developed and commissioned for user-friendly operations at the Bio-SANS instrument (Figure 1). The system is composed of an auto-sampler for sample injection, a modular HPLC for sample purification, and multiple flow cells designed for SANS measurements. In addition to the ability to measure complex materials, the autosampler and flow cells allow automation of contrast variation series measurements. EPICS integration of the HPLC system enables control through the Bio-SANS data acquisition system to synchronize the neutron scattering measurements with the chromatographic purification.. Use with time-sliced data enables identification of varying protein species present in the SEC elution profile. Development and operations of the SEC-SANS will be presented.

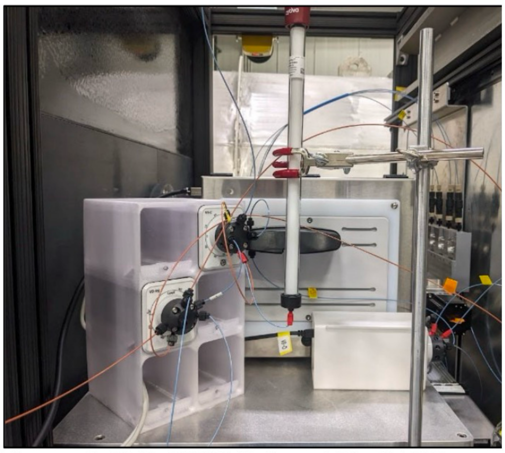

Figure 1. SEC-SANS Setup at Bio-SANS

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Presenter: Dr HEROUX, Luke (Oak Ridge National Laboratory)

Contribution Type: Oral

Abstract # 39

Present status of the cryogenic and magnet sample environment at the J-PARC MLF

At the Materials and Life Science Experimental Facilities (MLF) of the Japan Proton Accelerator Research Complex (J-PARC), some pieces of the cryogenic sample environment equipment are shared by several instruments, while the others are managed by each instrument. The Cryogenic and Magnet group at the MLF supplies a GM cryofurnace, a top-loading 4He cryostat, a 3He refrigerator, a dilution refrigerator insert, and a 7 T vertical field superconducting magnet since JFY2016 and the frequency of the use is increasing [1].

In recent two years after the last ISSE Workshop, we improved the 7 T superconducting magnet and dilution refrigerator systems in the following aspects:

(1) the expansion of the available instrument (BL23),

(2) detailed studies of the attainable lowest temperature according to the magnetic environment of the sample and the sample holder,

(3) optimization of the amount of the exchange gas, regarding the attainable lowest temperature and the background scattering,

(4) installation of the remote and automatic liquid He transfer system.
In the poster presentation, these research and development outcomes will be reported.

Reference

[1] M. Ishikado *et al*, JPS Conf. Proc. 011010 (2024).

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Presenter: ISHIKADO, Motoyuki (CROSS (Comprehensie Research Organization for Science and Society))

Contribution Type: Poster

Abstract # 40

LOW TEMPERATURE SAMPLE ENVIRONMENT CAPABILITIES AT HFIR: DEVELOPMENTS AND UPGRADES

There is a growing demand for low temperature (LT) (< 100 K) and ultra-low temperature (ULT) (<1 K) experiments at neutron scattering facilities with more than half experiments at High Flux Isotope Reactor (HFIR) requiring cryogenic sample environment equipment. This demand drives efforts to enhance the efficiency of these experiments through faster cooling, more reliable operation, and improved automatization and remote control. Here, we present the overview of ongoing projects to expand and develop new low temperature capabilities at HFIR. These projects include developing new experimental protocols for cooling and temperature regulation of ULT inserts, incorporating neon as the exchange gas within the inner vacuum can; designing an in-house dilution refrigerator gas handling system; upgrading to a new generation of cryostat control and cryogenic autofill systems; implementing an ULT sample changer platform that enables simultaneous cooling and rapid switching of multiple samples at ultra-low temperatures. These developments pave the way for enhancing efficiency and reliability of low-temperature experiments, ensuring the continued success of the neutron scattering user program at HFIR.

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Presenter: NASYEDKIN, Kostyantyn (Oak Ridge National Laboratory)

Contribution Type: Oral

Abstract # 41

PULSED MAGNET SYSTEM AT MLF IN J-PARC

Neutron scattering experiments have been performed in high magnetic fields. Superconducting magnets are used to generate fields up to a maximum of 17 T [1]. To generate a steady magnetic field of exceeding 20 T, large equipment is required. For example, a resistive and superconducting hybrid magnet was equipped with massive equipment with 4 MW generating up to 26 T in the Helmholtz Zentrum Berlin (HZB) [2], however, the research reactor was already shut down in 2019. Recently, pulsed magnetic equipment has been developed in some scattering facilities such as ILL [3]. It was difficult to build a large equipment in the MLF with the beamlines already in place. On the other hand, pulse magnetic field devices do not require large amounts of power, and it is possible to make compact setups. In this talk, the developments of pulsed magnetic field equipment in MLF will be presented in the following two directions. (1)A compact high magnetic field system of up to 40 Tesla

A high magnetic field system collaborating with Dr. Nojiri has been developed [4]. The equipment was designed to be compact and movable to allow it to be transported to multiple beamlines. The pulsed magnet system comprises a vacuum chamber, a closed-cycle refrigerator for sample cooling down to 4 K, and a nitrogen bath made of stainless-steel tube. The coil is made of a silver–copper alloy wire with low resistance and high rigidity, and it is immersed in the nitrogen bath to reduce the resistance and quickly remove the Joule heat generated by the pulsed current. The magnetic field is generated upto 40 T.Thepulse widthisseveralmilliseconds. Severalscatteringexperiments using the pulsed magnet have been already performed [5].

(2) 100 milli-seconds long pulse magnet system

Recently, a longer pulsed magnet system collaborating with Dr. Kohama and Dr. Nakajima has been developed utilizing electric double-layer capacitors [6]. The coil is made of copper. The cryostat to insert the coil and cool the sample was made by modifying the 40 Tesla magnet. For the extremely long pulse width, an entire time of flight spectrum can be measured in magnetic fields up to 20 T. Moreover, the control of the pulse shape is possible for effective data acquisition.

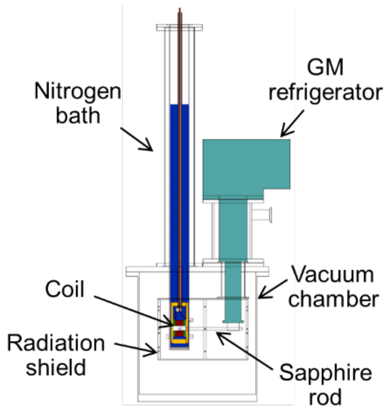

Figure 1: Cross section of the pulsed magnet.

References
[1] M. Meissner and P. Smeibidl, Neutron News 12:3, 12-18 (2001).
[2] M. Steiner et al., J. Phys. Conf. Ser. 2006, 51, 470-474.
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[4] M. Watanabe, T. Kihara and H. Nojiri, Quantum Beam Sci. 2023, 7(1), 1.
[5] S. Holm-Janas, et al., Phys. Rev. B 109, 174413 (2024).
[6] T. Nakajima et al., Phys. Rev. Research. 6, 023109 (2024).

Primary author: WATANABE, MASAO (J-PARC center)

Presenter: WATANABE, MASAO (J-PARC center)

Contribution Type: Poster

Abstract # 42

TIME RESOLVED ON DEMAND SERIAL CRYSTALOGRAPHY FOR USE AT SYNCHROTRON AND XFEL FACILITIES

The XFEL Hub at Diamond Light Source (DLS) identified a need to develop a "Drop on Demand" sample environment to enable a large user community interested in enzyme catalysis and drug discovery [1]. The instrumentation will drive a strong collaboration between initially, DLS and SwissFEL free electron laser.

Typical methods utilized for serial crystallography - fixed target or slurry injection have inherent drawbacks. They do not allow for sample reaction such as time resolved gas reaction, laser reaction or fluid reaction and suffer increased background.

An existing system created at Stanford [2] was used as the inspiration. The new DLS system is more compact, uses smaller drops at a higher frequency and is engineered to be easily portable. The continuous tape drive concept enables flexibility of the experiment conditions. A Polypico micro-dispenser set at a fixed distance from the X-ray beam ejects sample droplets (containing crystals), of diameter ~80 µm (270 pl), at a rate up to 5000 Hz onto a seamless Kapton® tape. The sample droplet is then exposed to a secondary condition to create a reaction of the sample, this varies between a second injector adding additional liquid drops, passing the sample through a gas reaction region or subjecting the sample to laser or LED light. The timing of the reaction is set by accurately controlling the drive speed of the Kapton® tape and adjusting the position of the secondary injector. The tape drive is a maxon servo motor in close loop with a Renishaw rotary encoder, controlled by Delta Tau Power PMAC. The system demonstrates a velocity jitter low enough to hit 95% of the drops with the 10 µm X-ray beam on beamline VMXi at DLS.

For the system to be a continuous delivery at 5000 Hz and 200 mm/s the Kapton® tape is constantly cleaned and dried post sample/X-ray interaction by passing it through a 3D printed series of chambers consisting of jetted water and compressed gas.

To enable different experiment conditions the sample delivery system sits in an "Environmental Chamber" which allows the control of humidity up to 95 %, low oxygen to < 50 ppm, temperature to 22 ± 0.2°C. This is achieved using nitrogen purging, in house designed humidity generator and proprietary monitoring sensors.

The initial design has been demonstrated on beamline VMXi at DLS. It will enable the collection of X-ray Diffraction (XRD) using proprietary Dectris 4M, alongside X-ray Emission Spectroscopy (XES), using in house designed Von Hamos Crystal Array and Tristan 2M detector.

References

[1] Business Case – Serial MX-XES Diamond-SwissFEL, A.M. Orville 07/12/2020

[2] Fuller, F. D., 2017. Drop on demand sample delivery for for studying biocatalysts in action at X-ray free-electron lasers. *Nature Methods, Nature America,Inc., part of Springer Nature..*

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Presenter: MALE, Andrew (Diamond Light Source LTD)

Contribution Type: Oral

Abstract # 43

Approaches in Relative Humidity for Neutron/X-ray Reflectometry and Liquid Pressures for SANS

Humidity control on thin films for neutron and x-ray reflectometry is highly dependent on various factors that make this technique challenging. Accurate temperature control is paramount in achieving satisfactory results. Sample, chamber, atmospheric temperatures, and humidity are tightly correlated and dependent on the measurement and controls conditions. Here we discuss different approaches in achieving on-situ humidity control across a wide range of sample temperatures and relative humidities.

Moreover, we present the latest update on the NCNR's HP-BioSANS Liquid Insertion Pressure System for SANS (LIPSS) project that aims to provide liquid pressures of up to 350 MPa at temperatures between -20°C and +65°C utilizing small sample volumes.

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Presenter: Mr LEAO, Juscelino B. (NIST Center for Neutron Research) Contribution

Type: Oral

Abstract # 45

Pulsed Magnetic Fields at Bessy II

High magnetic fields allow the stabilization of otherwise inaccessible new quantum states of matter. A scattering technique is imperative for a deep and thorough microscopic understanding of these exotic high field phases in correlated electron systems. Techniques employed at UE46_PGM-1 include polarization-dependent x-ray absorption and resonant soft x-ray scattering experiments, covering a wide range of materials and scientific problems.

Recently, high -field (30 T) x-ray magnetic circular dichroism (XMCD) measurements in an ultrahigh-vacuum chamber have been successfully performed, as shown in the schematic

drawing in Fig. 1.[1?]

For pre sample characterization measurements an improved copy of this working horse instrumentation is actually in development at the UE46_PGM-1 beam line in close cooperation with Prof. Nojiri (Institute for Materials Research of the University Tohoku, Japan). A second capacitor bank has recently been commissioned at HZB. A new coil for an even slightly higher magnetic field of 33 T has successfully been tested at HZB together with Prof. Nojiri. A further cryostat for the beam line setup has been ordered from Janis/LakerShore and will be delivered end of 2024.

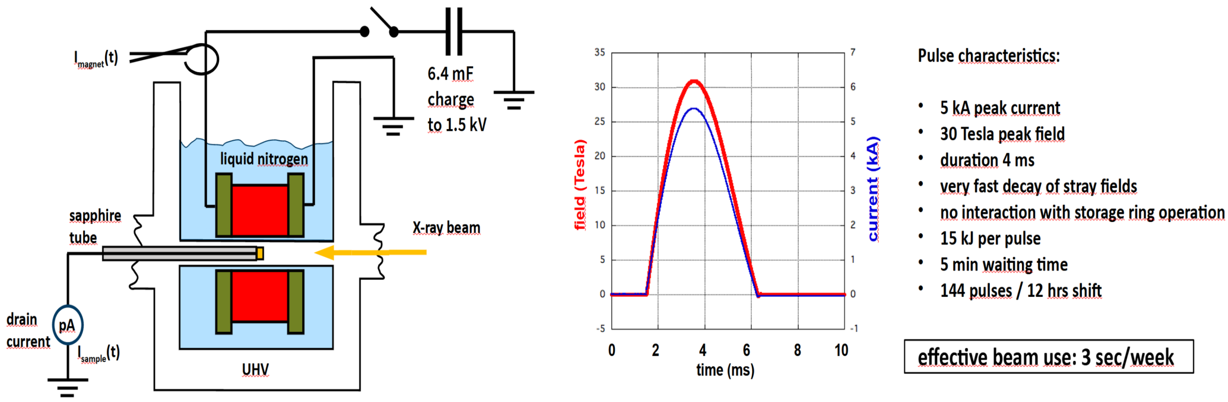


Fig. 1: Principle of x-ray magnetic circular dichroism (XMCD) measurements

References

[1] Eugen Weschke et al, Workshop: Frontiers of Synchrotron and XFEL Research at HMF, Dresden, Nov 2023

Primary author: WAHLE, Robert (Helmholtz-Zentrum Berlin)

Co-authors: Dr WESCHKE, Eugen (Helmholtz-Zentrum Berlin); Dr PROKHNENKO, Oleksandr (Helmholtz-Zemtrum Berlin); Prof. NOJIRI , Hiroyuki (Institute for Materials Research, Tohoku University)

Presenter: WAHLE, Robert (Helmholtz-Zentrum Berlin)

Contribution Type: Poster

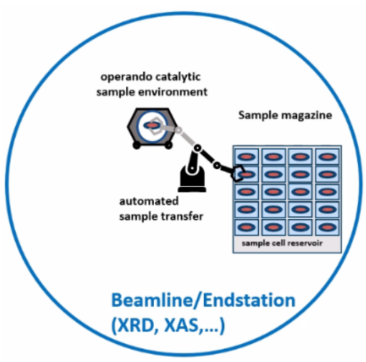
Abstract # 46

Automated Sample Handling for Enhanced Remote Access in Operando Catalysis Experiments at Synchrotron Beamlines

In the context of the ROCK-IT project [1], the development of sophisticated automated sample environments is crucial for enhancing the efficiency and accessibility of complex in-situ and operando catalysis experiments at large-scale research facilities. At X-ray absorption spectroscopy beamline P65, a fully integrated robotic system has been designed to manage samples with precision, thereby ensuring consistent and reliable experimental conditions.

The system employs a Universal Robots UR10e robotic arm equipped with a RobotIQ 2F-85 gripper to automate the handling of catalyst samples. Each sample is secured within a glass capillary tube attached to a standardized holder, uniquely identified by a QR code. The QR code stores the sample holder ID which is linked to the sample ID, enabling seamless tracking throughout the experimental process.

Figure 1: UR10e Robotic arm with RobotIQ 2F-85.



Upon initiation from the Tango control system, the robotic arm controlled via a Tango server, begins by scanning the sample magazine, which holds up to 12 samples, to locate the first sample in the measurements queue. Using a camera system in conjunction with AruCo markers, the robot precisely identifies and positions the sample holder at the measurement station. The sample holder is then clamped securely into place using pneumatic cylinders. Next, the system automatically attaches side gas inlets and conducts a leak test, ensuring safe operation before introducing any hazardous gasses.

Figure 2: Automated sample changer transferring the samples from the magazine to the sample environment and vice versa.

The sample is subsequently heated using a hot air blower, readying it for high-resolution operando measurements. This comprehensive automation of the sample environment not only reduces manual intervention but also enhances the reproducibility and safety of high-temperature experiments. By integrating standardized components and robotics into the sample environment, the ROCK-IT project aims to establish a more resilient and efficient experimental workflow. This approach facilitates remote access, lowers barriers for industrial users, and aligns with broader efforts in the automation and standardization of sample environments at synchrotron facilities.

References

[1] https://www.rock-it-project.de/

Primary author: EHAB, Shrouk (DESY)

Co-authors: SCHOEKEL, Alexander (DESY); Dr WELTER, Edmund (DESY)

Presenter: EHAB, Shrouk (DESY)

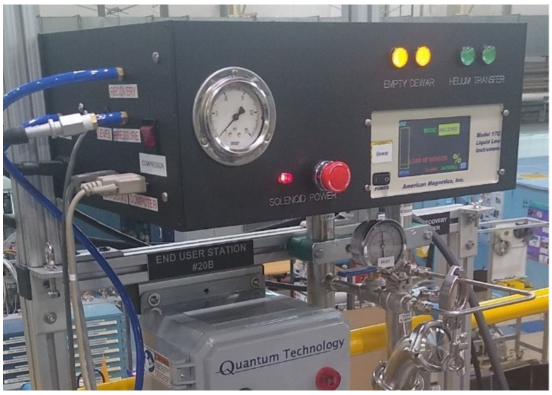
Contribution Type: Oral

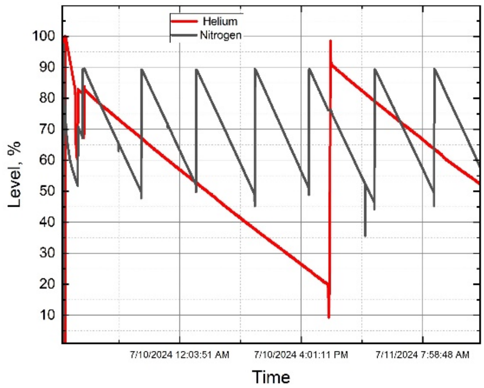
Abstract # 47

AUTOMATIC LIQUID HELIUM AND NITROGEN REFILL SYSTEM

Implementation of Automatic liquid helium and nitrogen refill system provide us opportunity making more efficient both helium consumption and convenience of operation of “wet” cryostats and magnets at NCNR. This system allows both automatic and user initiated liquid helium and nitrogen refill. Computer program provide control and monitoring of auto-refill process by instrument scientists and members of sample environment group.

Key element of this system is control box represented on Figure 1(a). Control box contains the combination of solenoid valves and compressor providing pressurization and de-pressurization of transport dewars to perform transfer of cryogenic liquids. Indicators of different regimes as well as emergency button placed on the front panel. Electrical, data and pressure lines connectors are on the side panel. Pressure lines connect the control box to user station of Helium Recovery system creating complete structure for efficient and convenient operation of helium cryostats at neutron experiments.





(a) (b)

Figure 1. (a) Control box of automatic refill system. (b) Chart of automatic refill of Helium cryostat with liquid helium and nitrogen.

Figure 1(b) represent process of automatic refil of helium cryostat with cryogenic liquids. Autorefil initiated automaticaly and starts when level of liquid helium in the cryostat drops to 20% and liquid nitrogen to 50%. Autorefil stops when level of cryogen liquids reach the 90%. These parameters preset before neutron experiment, however autorefil can be initiated and stoped within the experiment using software designed to control parameters of Helium cryostat.

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Presenter: Dr VEKHOV, Yegor (NIST)

Contribution Type: Poster

Abstract # 48

EXPANDING TEMPERATURE RANGE OF NEUTRON POWDER DIFFRACTION EXPERIMENTS USING WEAK THERMAL LINK

We present the outcomes of development project at ISIS Neutron & Muon source, which enhance our ultra-low temperature sample environment offering. We used a weak thermal link, to give an expanded temperature range for neutron diffraction of powder samples.

For the cooling of powder samples, ISIS offers a vacuum can in which the sample is sealed against a flange on a dilution refrigerator insert using indium wire. There is a pair of capillary tubes between the flange and room temperature, allowing the dosing of helium exchange gas into the can once the dilution refrigerator is at its base temperature. In order to terminate a parasitic heat load caused by superfluid helium film we have used 0.1% ^{3}He in ^{4}He mixture [1]. We have expanded this facility by offering an optional weak thermal link between the dilution refrigerator and sample can. This increases the maximum sample temperature from 1.0 K to 3.5 K before the dilution refrigerator becomes unstable, significantly increasing the speed and convenience of measuring samples with a phase transition in this temperature range, particularly in cases where a phase transition exists between 1.0 K and 1.6 K, which was otherwise inaccessible with this setup.

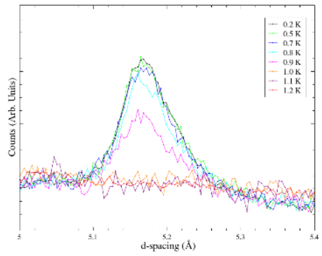

Figure 1: Phase transition observed around 1 K on a sample cooled using the weak link assembly.

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[1] Kirichek, O., Lawson, C.R., Kinane, C.J. et al. Density profile of ^{3}He in a nanoscale ^{3}He-^{4}He superfluid film determined by neutron scattering. *Commun Phys* **7**, 181 (2024).

Primary authors: Dr JONES, Alex (ISIS Facility); Dr LAWSON, Chris (ISIS); DOWN, Richard (ISIS); KIRICHEK, Oleg (ISIS neutron and muon source)

Presenter: KIRICHEK, Oleg (ISIS neutron and muon source)

Contribution Type: Poster

Abstract # 49

A custom designed cryogenic free Adiabatic Demagnetization Refrigerator (ADR) for ultra low temperature experiments at MLZ

The MLZ has two new custom designed liquid helium-free ADR (Adiabatic Demagnetization Refrigerator) systems, which will be used in neutron-scattering experiments at ultra low temperatures, without wasting cost-intensive and rare liquid helium.

The new systems are equipped with two ADR cooling units and allow making measurements in a continuous mode from the room temperature down to 300 mK. In a single shot mode, the temperature range can be extended even further. The ADRs are currently in a commissioning phase and during preliminary tests, we reached 90 mK under laboratory conditions. Typical curves for cooling will be presented on the poster as well as dimensions of the ADRs, sample space, the sample loading process and further characteristics.

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Presenters: BILLER,Regina(HeinzMaier-LeibnitzZentrum(MLZ),TechnischeUniversitätMünchen, Garching, Germany); RUBANSKYI,Valentyn(JülichCentreforNeutronScienceJCNSatHeinzMaier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH, Garching, Germany)

Contribution Type: Poster

Abstract # 50

ULTRA LOW TEMPERATURE NOISE THERMOMETRY AND ITS APPLICATIONS AT ISIS NEUTRON AND MUON SOURCE

The ISIS Neutron and Muon Source undertakes approximately 100 experiments per year at temperatures below 1K. At these temperatures, there is a clear need for accurate, precise, and wellcalibrated thermometry, which is resilient to the challenging sample environments found in large science facilities. Commonly used resistive thermometers, whilst offering convenience and operational simplicity, struggle under conditions involving radiation flux and magnetic fields. Additionally, they can be prone to self-heating and have a relatively high cost when bought fully calibrated. The magnetic field fluctuation noise thermometer (MFFT-1) from Magnicon can be utilized to overcome these difficulties. It operates through the Johnson-Nyquist noise theorem to link thermodynamic temperature to thermal fluctuations of voltage in a metal, allowing it to function as a primary thermometer. Using a superconducting quantum interference device (SQUID), it can read precise temperatures in the millikelvin range.

Inthiswork, welooktoimprovetheuseoflowtemperatureresistivesensorsinthehigh-throughput environment of an operational large-scale facility. Firstly, by installing the MFFT-1 into a dilution refrigerator we were able to create a methodology and mounting solution for the rapid calibration of cheap commercial resistors, thus enabling the creation of our own low-cost resistive thermometers able to measure down to millikelvin temperatures. Secondly, this provision enables us to characterize the behavior of both SQUID and resistance-based thermometers in the extreme environments found at neutron and muon sources. This incorporates the behavior of our sensors under radiation, whilst also including a scheme to operate the MFFT-1 in moderate magnetic fields, allowing calibration of the magnetoresistance effect in our resistive sensors. Lastly, we use these tools to understand the effect of 'beam heating' upon samples placed in our neutron instruments. This has been historically enigmatic due to the poor understanding of radiation on resistive sensors, coupled with the large variety of potential materials and neutron energies that are used. We wish to share our experiences implementing ultra-low temperature noise thermometry at a large-scale facility and discuss possibilities for further developments.

Primary authors: BAIN, Lucy (ISIS Facility); Dr JONES, Alex (ISIS Facility); PRICE, Ross (ISIS Facility); Dr KIRICHEK, Oleg (ISIS Facility); Dr LAWSON, Chris (ISIS)

Presenter: Dr KIRICHEK, Oleg (ISIS Facility) Contribution

Type: Poster

Abstract # 51

HIGH TEMPERATURE SAMPLE ENVIRONMENT REVIEW AT THE ISIS FACILITY.

High temperature sample environment plays a critical part in the scientific research carried out at the ISIS facility, delivering approximately 75 high temperature experiments per year, running in total of circa 240 days, on average these experiments last for 3 to 4 days and have multiple sample changes. Roughly 40% of these experiments involve gas handling which are either catalysis related or in-situ chemical reactions, often requiring the addition of humidity and analysis of the exhausting gases with a residual gas analyser. Of the remaining experiments 15% have temperature requirements between 1000°C and 2000°C and the remaining 45% are standard experiments up to 1000°C.

With improvements to the instruments at ISIS including new neutron guides and improvements to detectors, instruments can collect data faster, resulting in experiments being turned round quicker or give the possibility to run more samples in a shorter time period. With this in mind and trends in experiments currently being run and request received from the user community for future experiments, a review of the current high temperature sample environment equipment and running procedures was carried out to look at what was required for future high temperature experimental support. This poster looks at the findings of the review and highlights the direction of development required to enable the future demands of the ISIS neutron and Muon user community.

Primary authors: MCINTYRE, Paul (STFC ISIS); Mr GOODWAY, Chris (STFC ISIS); Mr SEARS, Adam (STFC ISIS)

Presenters: MCINTYRE, Paul (STFC ISIS); Mr GOODWAY, Chris (STFC ISIS)

Contribution Type: Poster

Abstract # 52

The ISIS Insert

The ISIS Cryogenic team have started developing a Variable Temperature Insert to have the ability to fix leaking/ broken Cryostats without relying on any external manufacturers. The poster will include images taken from a CAD model of the prototype and text explaining the goals and how they will be achieved. It will also include some results from the prototype and how it compares to other options on the market.

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Presenter: Mr BRADBURY, Will (ISIS Neutron and Muon Source, STFC, UKRI)

Contribution Type: Poster

Abstract # 53

CURRENT STATE OF THE ART IN THE LOW TEMPERATURE AND MAGNETS GROUPS AT THE SNS

The current state of the art in the low temperature and magnets groups at the SNS is presented. The ever-expanding needs of the scientific community and the increase in experimental throughput demand the development of novel sample environment capabilities, including developments which enable neutron studies of non-equilibrium systems, combinations of extreme environments, in situ non-neutron measurements, and increased equipment optimization.

This presentation includes the following recently developed or under development sample environments: (1) pump-probes enabling experiments on magnetic systems by either laser excitation or pulsed magnetic fields, two of these providing complimentary in situ non-neutron measurements; (2) a probe for in situ resonant ultrasound spectroscopy. In addition, the unique challenge of mitigating heating due to the beam in an experiment combining a diamond anvil cell and a dilution refrigerator is discussed. Finally, equipment optimization in the form of reducing manpower demands required for operation, such as (a) the liquid helium autofill system, (b) the helium pump-purge system, minimizing cooldown time via (c) liquid nitrogen precooling, and optimizing cooling time and helium consumption through (d) machine learning control of liquid helium cryostats is included.

Primary authors: Mr ELORFI, Saad (Oak Ridge National Laboratory); Dr FANELLI, Victor (Oak Ridge National Laboratory); Dr HUA, Chengyun (Oak Ridge National Laboratory); Dr KISH, Lazar (Brookhaven National Laboratory); Dr KRISHNA, Bhargavi (Oak Ridge National Laboratory); Mr MORGAN, Ryan (Oak Ridge National Laboratory); Mr ROBBINS, Sebastian (Oak Ridge National Laboratory); Mr RUIZ-RODRIGUEZ, Mariano (Oak Ridge National Laboratory); Dr SHERLINE, Todd (Oak Ridge National Laboratory); Mr WENZEL, John (Oak Ridge National Laboratory); Mr WHITE, Tyler (Oak Ridge National Laboratory)

Presenter: Dr SHERLINE, Todd (Oak Ridge National Laboratory)

Contribution Type: Oral Comments:

Abstract # 54

NEW BELT DRIVEN SAMPLE CHANGER FOR A 100mm TOP LOADING CCR

With the neutron sources facilities increasing flux capacity, the use of sample changers has become essential for the efficient use of beam time. We present a new sample changer able to hold up to 23 powder sample cans that fits in a 100mm bore top loading Closed Cycle Refrigerator (CCR). Its temperature range is 5-6 K up to 310 K.

We present data on the cooling performance of this sample changer. Its initial cool down in a warm CCR takes 6.5 hours from room temperature down to 7 K. The nominal base temperature is 6 K. If the host CCR is cold, the cool down time decreases to 3hours approximately. When moving samples in and out from incident beam position, a temperature difference of 0.15 K is measured between consecutive samples and the wait time for temperature stabilization is 4 seconds at base temperature.

A cart to hold two belt driven sample changes with two individual temperature controlled heated compartmentsis included in this presentation. When removing a sample changer from its host, it is stored in one of the cart heated compartments. A second dry sample changer loaded with samples is ready to be mounted in the host CCR as soon as the first one is removed. The compartments have a fan-forced heaters with maximum temperature 311 K (100 F) and positive pressure with a top vent for humidity to escape.

Primary authors: Mr ELORFI, Saad (Oak Ridge National Laboratory); Dr FANELLI, Victor (Oak Ridge National Laboratory); Dr GENG, Xiaosong (Oak Ridge National Laboratory); Mr LOGUILLO, Mark (Oak Ridge National Laboratory); Dr SHERLINE, Todd (Oak Ridge National Laboratory); Mr WENZEL, John (Oak Ridge National Laboratory); Mr WHITE, Tyler (Oak Ridge National Laboratory)

Presenter: Dr SHERLINE, Todd (Oak Ridge National Laboratory)

Contribution Type: Poster Comments:

Abstract # 55

Facility Report from ESS

The Europen Spallation Sourse (ESS) in Lund, Sweden, is 1 year away from its first neutrons and the groups in charge of the sample environment systems are getting ready to support the instruments and the users.

Since January 2023, the MSPS (Materials Science and Physics Support) and the CLS (Chemistry and Life Science) groups share the responsibilities for providing the sample environment systems and taking care of the corresponding labs and workshops. MSPS is in charge of the equipment related to hard condensed matter such as high and low temperatures, high pressure, magnetic and electrical fields and mechanical processing while CLS takes care of the chemistry and soft matter equipment.

New labs and workshops have been installed and are being used and new pieces of equipment have been procured. Some have already been received and tested and others are still in the procurement phase. We also started to enhance our knowledge by creating stronger connections with the other large facilities and sending ESS engineers and technicians to other facilities such as ISIS, SNS and ILL.

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Co-authors: Mr AMMER, Richard (ESS); Mr BORLET, Quentin; CORANI, Alice (ESS); Mr EK-STRÖM, Niklas (ESS); Mr HAGELBERG, Andreas (ESS); HOLMES, Alexander (ESS); Dr HARTL, Monika(ESS); DrPALIWODA,Damian(ESS); MrPAYSSAN,Gael(ESS); MrSAGLIANO,Luca(ESS); Mr SAXTRUP, Lauritz (ESS); Mr SCHNEIDER, Harald (ESS); Dr ZADOROZHKO, Oleksiy (ESS)

Presenter: CURFS, Caroline (ESS)

Contribution Type: Oral

Abstract # 56

Helium Management at HZB

Helium is a limited resource, which has seen a significant increase in price over the last years. Moreover, we saw several times a shortage of Helium on the global market that led to significant problems for the realization of planned experiments at our institute.

The Helmholtz-Zentrum Berlin is operating its own Helium liquefier in a joint organization together with other research institutes and universities in Berlin giving us a high degree of independence from commercial liquid Helium suppliers. However, the rather high losses of Helium at the member institutes and universities are compromising the aspired autonomy. Therefore, in order to realize an undisturbed and rather autonomous supply of liquid Helium, we have to control and finally reduce the losses of Helium.

For this purpose, we are developing hardware and software solutions for Helium Management, which enable us to monitor and control the liquid and gaseous Helium at our institute. These developments include Helium level meters and gas flow monitors with wireless communication, a database system and a visualization software.

This poster will present the current status of our Helium Management System.

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Presenter: KLEMKE, Bastian (Helmholtz-Zentrum Berlin) Contribution

Type: Poster

Abstract # 57

TUM SE@MLZ: WHAT’S THE NEWS?

The Heinz Maier-Leibnitz Zentrum (MLZ) is a leading centre for cutting-edge research with neutrons and positrons. As part of the user operation at the MLZ, TUM Sample Environment (SE) group assists in the installation and operation of complex SE equipment on instruments, providing experimental support to MLZ scientists as well as maintenance and repair of equipment. Beside the support, another key aspect is the constant market research to supply the users with state-ofthe-art equipment. Additionally, we develop new sample environment equipment, if the market cannot provide proper solutions. This involves continuous adoption and improvement of existing experimental techniques and devices as well as the development of novel equipment to cover the requirements of new scientific fields or to go beyond existing limits.
The talk will present the news regarding the new low-temperature and high-pressure equipment acquired in the last two years. In addition, we will present the main characteristics and performance of the three-stage cryostat developed and manufactured in-house, as well as the status of the neutron source and an overview of the activities being carried out for the next year user operation relaunch after a long period of non-operation.

Primary author: Dr SUAREZ ANZORENA, Manuel

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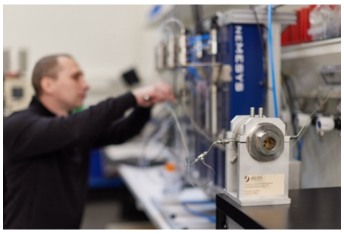
Presenter: Dr SUAREZ ANZORENA, Manuel

Contribution Type: Oral

Abstract # 58

Highlights and newcomers at the MLZ

In this presentation I will introduce our newcomers to our sample environment equipment pool. This will cover soft matter devices, mostly. Among others, I will show you a stopped flow setup for mixing liquids at pressures up to 700 bar within a measurement chamber for SANS applications.



Primary authors: WEBER, Alexander (Forschungszentrum Jülich GmbH); RUBANSKYI, Valentyn (Jülich Centre for Neutron Science JCNS at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH, Garching, Germany)

Presenter: WEBER, Alexander (Forschungszentrum Jülich GmbH) Contribution

Type: Oral

The path to fully automised and standardised complex operando experiments - the ROCK-IT project

This contribution gives an overview over the project ROCK-IT, a collaborative effort of the four German Helmholtz institutes DESY, HZB, HZDR, and KIT. Started in the year 2023 under the impression of the challenges caused by the COVID pandemic, the project aims to establish general procedures for remote controlled operando catalytic experiments. These procedures are realised by fully automated experimental demonstrator setups at different x-ray beamlines of the contributing institutes. The catalytic hydrogenation of CO_2 to methane (methanation) has been identified as the exemplary science case to be investigated in an operando x-ray absorption spectroscopy (XAS) experiment.

ROCK-IT covers all aspects needed for the realisation of fully automised remote experiments: remote user access, cyber security, experiment controls, automation of operando experiments, AI supported real time analysis, and data storage and data management based on F.A.I.R. principles (see fig.1). Whenever possible, unified standards are used so that industrial or scientific users can work in the same environment at any of the involved demonstrator beamlines at BESSY II, PETRA III and KARA.

The operando sample environment is representing the experimental heart of the project. Full remote control of the experiment requires automated (robotic) sample identification, handling and exchange, as well as the control of the catalytic reaction parameters (e.g. gas feed, temperature, gas analysis). The Sample Environment Communication Protocol (SECoP) with its high abstraction and rich metadata plays a crucial role as an interface standard between complex, modular sample environment equipment and the instrument control systems.

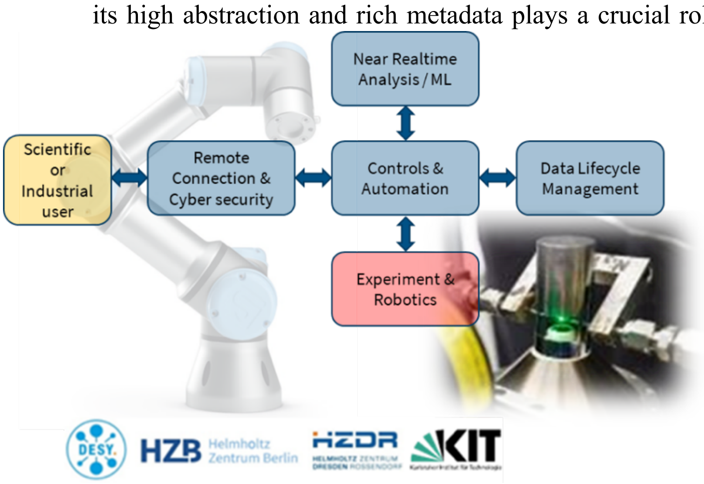


Figure 1: Structure and building blocks of the ROCK-IT project.

The desired autonomy of the experiment requires that all process data and beamline spectra can be automatically interpreted and analyzed near real-time in a collaborative machine learning assisted way. This imposes high standards on data and metadata generation and on electronic lab book usage.

Though focusing presently on catalytic experiments, the building blocks and standards of the ROCK-IT project are portable enough to be applicable to a wide range of other measurements. In this presentation we will give an overview of the ROCK-IT project, its goals and principles.

References

[1] https://www.rock-it-project.de

Primary author: Dr WALLACHER, Dirk (Helmholtz-Zentrum Berlin)

Presenter: Dr WALLACHER, Dirk (Helmholtz-Zentrum Berlin) Contribution

Type: Oral

MULTIPURPOSE CRYO-INSTRUMENTATION FOR IN-SITU TIME-RESOLVED X-RAY IMGAGING AND SPECTROSCOPIC TECHNIQUES

In situ and operando time-resolved macroscopic non-destructive methods such as X-ray tomography or X-ray spectroscopy have become increasingly important, for example to provide insights into mass transfers in porous solids and batteries during charging and discharging processes. However, there are currently few suitable instrumentations for in situ X-ray radiography under controlled temperature and operando conditions.

The input describes the development of an instrumentation (see fig. 1) for time-resolved in- situ X-ray imaging at the BAMline of the BESSY II Synchrotron at the Helmholtz Center Berlin. The cryoenvironment, which is in the test phase, is intended to make the isothermal adsorption of gases or electrolytes in the nanoporous materials studyable by X-ray radiography and to enable operando investigation of porous cathode materials of lithium-sulfur batteries during charging and discharging under temperature changing conditions.

The experimental, methodological requirements to be met are: a precisely controllable temperature range of 100 – 400 K, a 360° rotation of the sample in the beam window with as little distance as possible from the sample to the detector and a spatial stability of the sample position of less than 0.01 mm. The associated challenges in the cryogenic environment require novel construction concepts and a high level of manufacturing precision, which will be described in the poster.

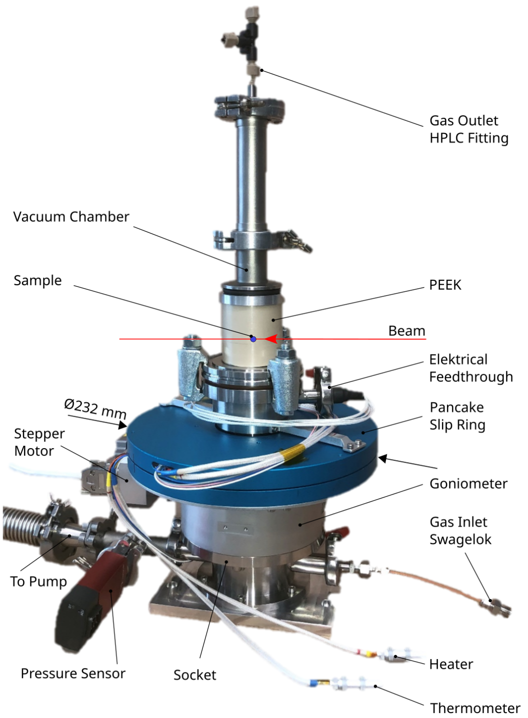


Figure 1: Representation of the cryoenvironment for operando X-ray tomography

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Presenter: THIEL, Hermann (Helmholtz-Zentrum Berlin)

Contribution Type: Poster

Abstract # 61

HOW VARIETY OF WORK COUPLED WITH SPECIALIST KNOWLEDGE STRENGTHENS THE ISIS ELECTRICAL & ELECTRONIC USER SUPPORT GROUP

J.Nutter, M.Williams, S.Cooper, M.Schastny, J.Chandler, K.Allum, T.Canfer, T.Allinson, E.Baker, T.Jenkins

ISIS Neutron and Muon Source, OX110QX, Oxfordshire, United Kingdom

This talk provides an overview of the 'ISIS Electrical & Electronic User Support Group'(EEUSG). The Group, commonly referred to as the Electronics Group, provides essential electrical and electronics work for the ISIS Operations Division.

The Group provides critical support for a wide variety of systems across the ISIS facility including Personnel Protection Systems (PPS), Temperature control systems, Motion control systems, data acquisition, Bespoke software design, Additive Manufacturing, and rapid prototyping.

This presentation will shine a light on the Electronics Group's recent technical accomplishments. It will delve into the diverse skill sets of our technical specialists and explore how their unique expertise contributes to the successful delivery of intricate, multidisciplinary technical solutions.

The talk will highlight:
• An overview of the ISIS Electrical & Electronic User Support Group
• The group's areas of responsibility
• An evaluation of multiskilling & specialising
• Examples of technical solutions

Primary authors: Mr NUTTER, Jamie (STFC); Mr SCHASTNY, Maksim

Presenters: Mr NUTTER, Jamie (STFC); Mr SCHASTNY, Maksim

Contribution Type: Oral

Abstract # 62

HIGH PRESSURE, HIGH TEMPERATURE AND GAS HANDLING DEVELOPMENTS AT THE ISIS NEUTRON AND MUON FACILITY.

The Pressure and Furnace Section at ISIS supports approximately 250 experiments per year involving gas handling, high pressure and high temperature or combinations of these disciplines.

With improvements to the instruments at ISIS including new neutron guides and improvements to detectors, instruments can collect data faster, resulting in experiments being turned round quicker or give the possibility to run more samples in a shorter time period. In addition to these experiments have become more complex pushing existing sample environment equipment and driving development. This has led developments in areas to speed up support as well as new techniques and equipment to meet the new demands. This talk looks at new developments in high pressure and high temperature experiments and looks at what is required going forward to keep up with the improvements in the instruments and the demands this places on the equipment and support staff.

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[2] Low-background materials for high pressure cells used in inelastic neutron scattering, Experiments. Journal of Neutron Research 21 (2019) 105–116, DOI 10.3233/JNR-190115, M. Kibble et al.

Primary authors: GOODWAY, Chris (UKRI - STFC); MCINTYRE, Paul (STFC ISIS); Mr SEARS, Adam (STFC - UKRI)

Presenter: GOODWAY, Chris (UKRI - STFC)

Contribution Type: Oral

NANOPOSITIONING STAGES FOR X-RAY MICROSCOPY AND TOMOGRAPHY

In synchrotron endstations the precise knowledge of the movement of a rotating target is of crucial interest, as for example in X-ray tomography. In particular, the run-out and wobble of a rotating sample need to be measured accurately, and subsequently controlled by feed-back loops, to keep the rotating target on the desired trajectory. Therefore, a prototype nanopositioning system has been developed which combines a precise rotational motion of the sample and a large lateral displacement of a few millimeters. This prototype system, developed within the LEAPS-INNOV initiative 1, will be installed within the X-ray transmission cryo microscope (TXM) at BESSY. The nanopositioning system consists of a stacked arrangement of translation and rotational stages. The 360°-degree rotary axis (Ry) will be measured by an optical encoder grating (Heidenhain, ERA 4400) on a cylindrical surface allowing for a large height variation of several mm in y-direction. The in-plane movement (X, Z) of the rotational encoder with respect to the focusing zone plate of the microscope will be controlled by fiber coupled interferometers using the zero-diffraction order of the grating encoder at 1.55µm. To scan the sample along the x-direction and to adjust the sample after rotation to the focal point of the X-ray beam in z-direction two additional linear stages are mounted upon the rotary stage. These linear stages are controlled by additional interferometers which monitor the motion of plane mirrors attached to the sample holder relative to the cylinder of the rotational encoder surface. To minimize the number of interferometric supply lines we focus on using fiber interferometers from qutools (quDIS) which use the same fiber for supply and detection of the light. These wavelength-scanning interferometers have been tested by our LEAPS-INNOV partners of WG1/WP5.2 to be superior for non-linearity deviations against other fiber interferometers. At all, the design of the setup has the goal for a spatial resolution of 5 nm. The nominal rotational repeatability of the rotary stage (Aerotech Inc., APR150) is 1.5 arcsec. The Heidenhain encoder is specified for a rotary accuracy of 15 µrad.

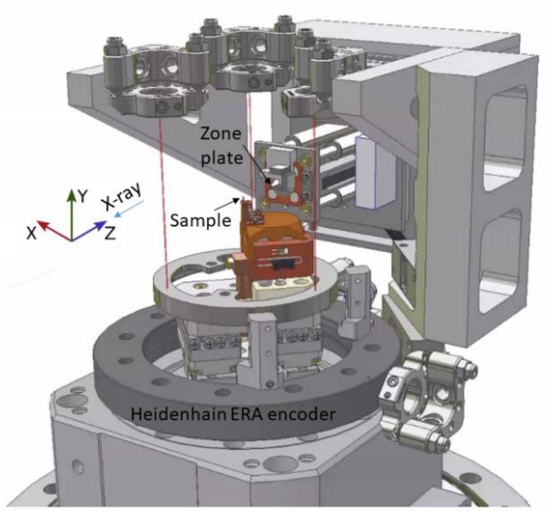

Figure 1: Nanopositioning stage for X-ray tomography at BESSY

1 The LEAPS-INNOV project has received funding from the European Union´s Horizon 2020 research and innovation programme under grant agreement No. 101004728

Primary authors: NEEB, Matthias (Helmholtz-Zentrum Berlin); WALLACHER, Dirk; KLEMKE, Bastian; KIEFER,Klaus; WERNER,Stephan; SCHNEIDER,Gerd; GECKELER,Ralf; FLÜGGE,Jens; WIESEMANN, Urs

Presenter: NEEB, Matthias (Helmholtz-Zentrum Berlin)

Contribution Type: Poster Comments:

Poster presenter M. Neeb

Abstract # 64

LOW-TEMPERATURE ENVIRONMENT AT EUROPEAN SPALLATION SOURCE

The successful operation of a neutron user facility depends heavily on the control and variation of sample environment parameters such as temperature, magnetic or electric field, pressure, or humidity. In particular, low-temperature environments are often used in neutron scattering experiments. TheEuropean Spallation Source(ESS),with its highneutron flux, imposesrequirements on sample environment devices to ensure minimal waiting times for processes like temperature equilibrium or sample changes, thereby optimizing beam time use. The Materials Science and Physics Support (MSPS) group at ESS is implementing advanced low-temperature sample environments, including cryostats utilizing liquid helium or dry cryo-cooler technology, as well as modular low-temperature inserts such as dilution refrigerators and helium-3 (He3) systems. The current capabilities of ESS in providing these essential low-temperature sample environment systems will be presented and the future progress to further enhance experimental efficiency will be outlined.

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Co-authors: Dr HOLMES, Alexander (European Spallation Source); Mr SAGLIANO, Luca (European Spallation Source); Mr SAXTRUP, Lauritz (European Spallation Source); Mr EKSTRÖM, Niklas (European Spallation Source); Mr HAGELBERG, Andreas (European Spallation Source); Dr CURFS, Caroline (European Spallation Source)

Presenter: ZADOROZHKO, Oleksiy (European spallation source ERIC)

Contribution Type: Poster

Abstract # 65

SECoP LIVE DEMONSTRATION: A PRACTICAL LOOK AT THE SECoP SHALL LIBRARY AND THE SECoP TO OPHYD/BLUESKY INTEGRATION

The Sample Environment Communication Protocol (SECoP) is an inclusive, simple and self explaining communication protocol, intended as a common standard for interfacing sample environment equipment and instrument control software [1,2].

In this presentation we will show how to add a SECoP interface to an existing LabVIEW program using the SHALL libraries [3] and how to access a SECoP device by the bluesky experiment control with the help of the SECoP-Ophyd integration [4].

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Primary authors: KIEFER, Klaus (Helmholtz-Zentrum Berlin); BRAUN, Peter (Helmholtz-Zentrum Berlin); CHEN, Zhengyu (Helmholtz-Zentrum Berlin); GRIMM, Nico (HZB); KLEMKE, Bastian (Helmholtz-Zentrum Berlin); ROSSA, Lutz (Helmholtz-Zentrum Berlin)

Presenter: KIEFER, Klaus (Helmholtz-Zentrum Berlin) Contribution

Type: Oral

Abstract # 66

SAMPLE ENVIRONMENT METADATA – PRODECURES AND BEST PRACTICES IN SECoP

For the provision of FAIR compatible data 1, sample environment (SE) equipment should provide a complete set of metadata describing the entire SE setup including the meaning of the provided data and the control parameters.

Using the Sample Environment Communication Protocol (SECoP) as control interface for SE equipment simplifies the procedure to provide SE metadata as SECoP holds the possibility to transport SE metadata in a well-defined and standardised way [3]. In addition, SECoP provides machine readable self-description of the SE equipment which enables a fully automated integration into the instrument control software and into the processes for data storage.

In this presentation will show how SECoP can help to provide a meaningful and complete set of metadata for SE equipment, and how this metadata can be stored in a standardised way.

TheprojectSECoP@HMCissupportedbytheHelmholtzMetadataCollaboration(HMC),anincubatorplatform of the Helmholtz Association within the framework of the Information and Data Science strategic initiative.

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Primary authors: KIEFER, Klaus (Helmholtz-Zentrum Berlin); BRANDL, Georg (Jülich Centre for Neutron Science JCNS at Heinz Maier-Leibnitz Zentrum (MLZ)); BRAUN, Peter (Helmholtz-Zentrum Berlin); Mr EKSTRÖM, Niklas (European Spallation Source); Dr FAULHABER, Enrico (Heinz Maier-LeibnitzZentrum(MLZ),TechnicalUniversityofMunich(TUM)); GÜNTHER,Gerrit(Helmholtz-Zentrum Berlin); KLEMKE, Bastian (Helmholtz-Zentrum Berlin); KOTANSKI, Jan (Deutsches Elektronen-Synchrotron DESY); KRACHT, Thorsten (Deutsches Elektronen-Synchrotron DESY); PETTERSSON, Anders (European Spallation Source); ROSSA, Lutz (Helmholtz-Zentrum Berlin); UHLARZ, Marc (Helmholtz-Zentrum Dresden-Rossendorf); ZAFT, Alexander (Forschungszentrum Jülich); Dr ZOLLIKER, Markus (Paul Scherrer Institut)

Presenter: KIEFER, Klaus (Helmholtz-Zentrum Berlin) Contribution

Type: Oral

Abstract # 67

BULK METALLIC GLASS FOR HIGH PRESSURE GAS CELLS

Bulk metallic glasses (BMG), or amorphous metals, are solid metals with non-crystalline, disordered atomic structures. They are an appealing material choice for sample containment in diffraction based neutronic experiments due to their lack of Bragg peaks; instead, only exhibiting a broad smooth background that is easily subtracted from the data, leaving a clean sample diffraction pattern. Their high strength means that they may be suited to high pressure experiments, where the background of thick-walled vessels can be difficult to completely remove from the neutron data.

ISIS have been researching and developing a Zirconium based BMG high-pressure gas cell for use in HRPD and other diffraction instruments. Cells have been additively manufactured and are currently undergoing testing to validate the stress analysis.

This poster will discuss the challenges of developing both a BMG & additively manufactured cell, current state of play, and the further commissioning work required.

Primary author: MORTAZAVI, Ali (ISIS)

Presenter: MORTAZAVI, Ali (ISIS)

Contribution Type: Poster

Abstract # 68

THE DEVELOPMENT OF ISIS HUMIDITY CELL

Humidity cells are crucial tools in research and industry for studying and understanding the impact of humidity on various materials, products, and environmental processes. Controlling the temperature and relative humidity (RH) surrounding the sample is highly favourable in many biological or soft matter neutron/X-ray scattering/reflectometry experiments. There are existing systems [1-4] being used in several scientific facilities, which required delicate fabrication and temperature control. As the request for temperature and RH control sample environment is increasing in ISIS beamtime proposals, a crossed group team (including engineers from design division, operational group and scientists) is formed to develop and prototype an ISIS humidity cell to bridge the gap. The critical components of the cell were manufactured through Direct Metal Laser Sintering (DMLS) process to near net-shape from AlSi10Mg powder. These parts were completed with post-print 5 -axis CNC machining to meet assembly/sealing requirements. C-axis sapphire windows are used to minimise scattering background from the cell. The internal fluidic heating/cooling channel is integrated within the wall to surround the sample position to optimise temperature homogeneity on this area. RH at the sample is controlled by using partial vapour pressure through altering the temperatures of the sample and water reservoir. In addition, the geometric set-up of the cell is designed to be used in various SANS and reflectometry beamlines in ISIS. By covering the design, manufacture process and some testing data, the presentation aims to share experience and knowledge with colleagues who are interesting to used state-of-the-art metal additive manufacturing process for future work.

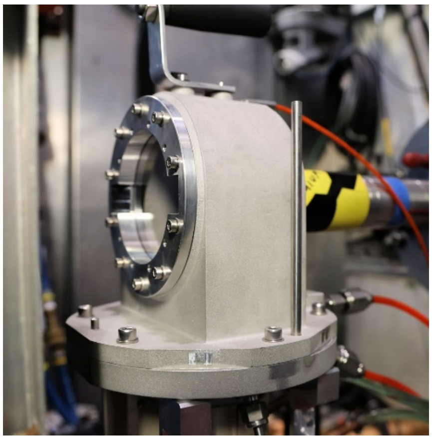

Figure 1: ISIS Humidity cell.

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Primary authors: WANG, Xiao (UKRI, STFC, ISIS neutron and muon Facility); Dr CAVALCANTI, Leide (ISIS Neutron and Muon Source); Dr DALGLIESH, Robert; Mr CHURCH, Andy; ALLUM, Keith (ISIS Neutron and Muon Source); MORTAZAVI, Ali

Presenter: WANG, Xiao (UKRI, STFC, ISIS neutron and muon Facility) Contribution

Type: Oral

SAMPLE ENVIRONMENT AT THE MLF J-PARC

After the last International Society for Sample Environment (ISSE) workshop at Nasu, Japan, the ISSE School was held at Tokai, where Japan Proton Accelerator Research Complex (J-PARC) was, on October 22-26, 2023. We had 18 students, 4 on-site lectures, and 3 on-line lectures. We hope that the participants enjoyed the school.
There were some improvements and problems on the sample environment at the Materials and Life Science Experimental Facility (MLF), J-PARC. For 7 T superconducting magnet, the available instruments were extended to the two chopper spectrometers of BL23 (POLANO) and BL01 (4SEASONS) (for user experiment), in addition to the previously installed instruments of BL14 (AMATERAS), BL15 (TAIKAN), BL17 (SHARAKU), BL18 (SENJU), and BL22 (RADEN). We had a problem to allocate the machine time of the 4He cryostat to the instrument where the sample was strongly radio-activated. We could allocate the next machine time with enough period to cool down the radio-activation and avoid treating the strongly radio-activated material this time. However, this issue will be a serious problem if the machine time is tight. Therefore, we consider installing an additional 4He cryostat. The niobium furnace was used for the high temperature user experiment above 1000℃ on BL01 (4SEASONS) for the first time, in which the temperature reached 1227℃ (1500 K). On BL08 (SuperHRPD), a remote monitoring system for high temperature experiments with the vanadium furnace was installed and the user experiment was performed using the system. Long pulsed magnet system with the pulse width of > 102 ms has been developed, collaborating with the University of Tokyo. Maximum magnetic field is approximately 15 T. Neutron scattering experiments were performed using the system on BL15 (TAIKAN).

Primary authors: Dr WATANABE, Masao (J-PARC Center); Dr TAKAHASHI, Ryuta (J-PARC Center); Dr ISHIKADO, Motoyuki (CROSS); ZHANG, Shuoyuan (CROSS); Mr YAMAUCHI, Yasuhiro (NAT); Mr ISHIMARU, Sora (NAT); Dr ARIMA-OSONOI, Hiroshi (CROSS); Dr SHIN'ICHI, Takata
(J-PARC Center); YUHUA, Su (J-PARC Center); Mr MORIKAWA, Toshiaki (CROSS); Mr KEIICHI, Ohuchi; Dr YAMAUCHI, Sara (J-PARC Center, KEK); Dr OHIRA-KAWAMURA, Seiko (J-PARC Center); SAKAGUCHI, Yoshifumi (Comprehensive Research Organization for Science and Society); Dr OKU, Takayuki (J-PARC Center)

Presenter: Dr WATANABE, Masao (J-PARC Center)

Contribution Type: Oral

Abstract # 71

ESS SAMPLE ENVIRONMENT FOR ELECTROCHEMISTRY

Because of the ability of the neutron to distinguish between small atom as lithium and hydrogen, neutron scattering is an essential tool to analyze, understand and develop electrochemical systems. However, performing neutron scattering experiments brings some constraints to the sample and the cells: The cell material shall be compatible to neutron and have a homogeneous and reproducible background. In addition to the requirements given by the neutron experiment, the ones for electrochemistry have to be added, such as the good contact between the electrode and the sample, the connection of the electrodes etc.

We, the softmatter and chemistry sample environment team (SCSE) at ESS have developed two cells suitable for neutron experiments in collaboration with Tartu University. One is a versatile cell that can be used for inelastic and diffraction measurement for the study of new materials such as solid electrolytes but also batteries. A second cell for battery applications based on the design from Godbole et al.1 was developed to measure coin cell and half cells and to observe the structural change in lithium.

The two cells contribute to the pool of sample environment equipment at ESS and make in situ and in operando measurement on different ESS instruments possible. In addition to the cells, the Chemistry and Life Science Support group at ESS will provide access to appropriate laboratories for the user to perform electrochemistry experiments, to prepare the sample and to assemble the cells.

We will present the equipment already available at ESS to perform electrochemical experiments from the laboratory to the instrument, including the first version of the battery and electrochemical cells, and will show the future needs and potential for developments.

Primaryauthors: CORANI,Alice(ESS); SCHNEIDER,Harald(ESS); HARTL,Monika(ESS)

Presenter: CORANI, Alice (ESS)

Contribution Type: Oral

Abstract # 72

HUMIDITY CELL AND OTHER CHEMISTRY AND SOFTMATTER SAMPLE ENVIRONMENT EQUIPMENT AT ESS

The development of a humidity cell using a humidity generator for SANS experiment has been developed in collaboration with Tartu university.

Using a humidity generator, the cell can reach a desirable temperature and humidity in relative short time allowing a rapid condition change. Because of the size, the cell offers the possibility to develop a small sample changer to optimize the efficiency of a measurement.

Here we present the specificity and the parameters of the new humidity cell and the development to be done.

We will also present some of the equipment from the soft matter and chemistry sample environment group being prepared for commissioning of the instruments.

Primary authors: Mr SCHNEIDER, Harald (ESS); Dr HARTL, Monika (ESS); CORANI, Alice (ESS)

Presenters: Mr SCHNEIDER, Harald (ESS); CORANI, Alice (ESS)

Contribution Type: Poster

Abstract # 73

SOFT MATTER SAMPLE ENVIRONMENT FOR NEUTRON SCATTERING EXPERIMENTS AT ISIS FACILITY

TheISISneutronandmuonsourceattheRutherfordAppletonLaboratoryinOxfordshireisaworldleadi ng center for research in the physical, engineering and life sciences. It is owned and operated by the Science and Technology Facilities Council, UKRI. The popularity of using neutron scattering in Soft Matter (SM) research is growing strongly, thanks to a great extent to opportunities offered by the newly developed Sample Environment (SE). Here I am going to review general tendencies in SM SE and examples of new SE tools used in ISIS neutron scattering experiments. I am also going to discuss ongoing sample environment related research and development projects such as Delft furnace, Coldbox, and Isisstat. I will also present our new experimental operations management tools based on SharePoint and Planner.

Primary author: CHURCH, Andy (STFC)

Co-authors: Mr DALTON, James (STFC); Mr OLIVER, Harry (STFC)

Presenter: Mr DALTON, James (STFC)

Contribution Type: Oral Comments:

Abstract # 74

CRYOBOX DESIGN AND COMMISSIONING

We were tasked with designing a cryogenic cold box that could sustain sub 100K temperatures and provide a method of heating a sample back up to room temperature. This was done to create the sample environment required for diffraction and neutron imaging of the cryo-preserved carp spermatozoa samples that had been shipped to us from the Czech Republic. The box had to hold the samples below 100K to ensure they remained cryogenically preserved. The result was no condensation formed on the windows of the cold box, the heat exchanger controlled the temperature successfully from 100K – 300K. The box allowed us to measure diffraction and imaging data of cryo preserved carp spermatozoa which is currently being analysed. In conclusion we hope that the cryo-box can be used for neutron scattering study of cryogenically preserved bio-medical samples.

Primary author: Mr OLIVER, Harry (STFC)

Presenter: Mr OLIVER, Harry (STFC)

Contribution Type: Poster

Abstract # 75

DELFT FURNACE SAMPLE HOLDER DESIGN AND COMMISSIONING

We were tasked with designing a sample holder for some 12mm diameter steel samples. This was done to create the sample environment required for testing Oxide-dispersed-strengthened steels and welds and their precipitate evolution and optimum tempering temperature. The sample holder had to hold the samples within the neutron beam and withstand the 1000 degree Celsius temperatures. The result was that visiting users were able to run 9/10 of their samples successfully within the DELFT furnace. In conclusion, we hope that the sample holder and modified cores can be used in future experiments involving the DELFT furnace.

Primary author: Mr OLIVER, Harry (STFC)

Presenter: Mr OLIVER, Harry (STFC)

Contribution Type: Poster

Abstract # 76

Operando cells for x-ray investigations in heterogenous catalysis

Heterogeneous catalysis is indispensable for the synthesis of products such as fertilisers, fuels and chemical precursors based on renewable energies. The characterisation of newly developed or optimised thin-film catalysts using X-ray spectroscopic examination methods under industry-relevant operando conditions places high demands on the sample environment. Two operando reactor cells are being developed for this purpose and the status is presented here

<u>Reactor cell for soft-xray XAS-studies:</u>

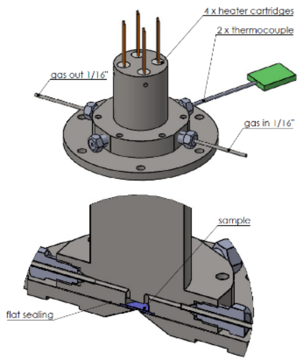

Figure 1: reactor cell for soft-xray XAS

This cell was developed to analyse samples of thin film catalysts spectroscopically in fluorescence under industrially relevant conditions. The samples consist of 1 cm² large and 200 µm thick silicon carriers, coated with a 100 nm thick silicon nitride membrane, which separates the reactor chamber from the vacuum chamber of the beamline at 8*10E-6 mbar in a translucent manner. A 10 - 20 nm thick layer of the catalyst is deposited onto the membrane so that the beam is directed through the silicon nitride membrane and the catalyst layer behind it. The samples are to be analysed in a process gas flow of a few mln/min at temperatures of up to 400 °C and gas pressures of up to 10 bars.

<u>Reactor cell for hard-xray GIXRD and XAS[1]:</u>

A cell for synchrotron-based grazing-incidence x-ray diffraction at ambient pressures and moderate temperatures in a controlled gas atmosphere is presented. The cell is suited for the in-situ study of thin film samples under catalytically relevant conditions. Different domes (glass, high temperature glass and Kapton®) enclosing the sample are available. The sample is deposited onto a 1 cm² large and 600 µm thick silicon carrier and can be studied at up to 500°C and 1 bar gas pressure.

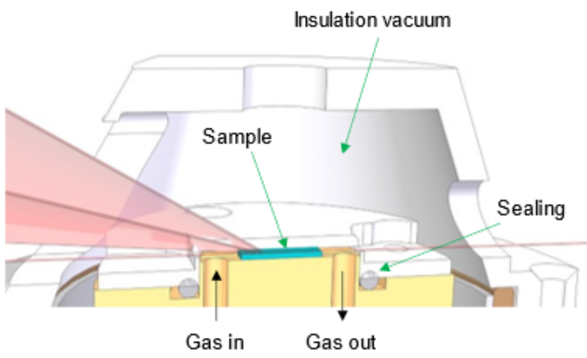

Figure 2: reactor cell for hard-xray GIXRD / XAS

References

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Primary author: BEHRENDT, Frank (German)

Co-authors: GRIMM, Nico (HZB); Dr THUM, Lukas; Dr GILI DE VILLASANTE, Albert; WALLACHER, Dirk (Helmholtz-Zentrum Berlin)

Presenter: BEHRENDT, Frank (German)

Contribution Type: Poster

EUROPEAN SPALLATION SOURCE HIGH-PRESSURE SUIT

High-pressure techniques have broad applications in materials science, fundamental physics, chemistry, and earth and planetary science. The ESS Sample Environment Group for Materials Science and Physics Support aims to facilitate high-pressure experiments across numerous facility instruments while exploring new possibilities in small-angle scattering and imaging.

We support a range of hardware capable of reaching pressures from atmospheric levels up to nearly 100 GPa. The High-Pressure Sample Environment Pool is now equipped with several gas and liquid cells, high-pressure clamp cells, Paris-Edinburgh presses, and screw- and membrane-driven Diamond Anvil Cells, as well as a high-pressure test bunker for testing newly developed high-pressure equipment.

Additionally, high-pressure, low-temperature measurements are possible by combining the highpressure sample environment with low-temperature equipment, such as cryostats. Furthermore, our Diamond Anvil Cell laboratory is equipped with a wide-angle Almax EasyLab Diamond Anvil Cell, suitable for X-ray single crystal diffraction experiments on the 4-circle Xcalibur RigakuAgilent diffractometer available in the Chemistry and Life Science Support (CLS) Laboratory. This setup enables preliminary studies on pressure-induced structural transformations of material.

Primary authors: SAXTRUP, Lauritz (ESS); Dr PALIWODA, Damian (ESS); Dr CURFS, Caroline (ESS)

Presenter: SAXTRUP, Lauritz (ESS)

Contribution Type: Poster

Abstract # 78

News of low-temperature and high-pressure environments at FRM II

In the fisrt part of this poster, we will present the current state of our CCR1K systems, which are design to reach a base temperature of ca. 1.5K. There will be the heat power at different Temperatures below 3K shown and the typical cooldown and operating procedure described.

In the second part of the poster we will present a new 2 kbar high-pressure cell, designed for Grazing Incidence Small Angle Scattering (GISANS) at SANS instruments with a motorized screw press. The pressure cell includes two sapphire windows for optical access, along with a third optical window integrated into the bottom of the cell. This third window enables the use of advanced techniques such as optical microscopy and Raman spectroscopy. The cell is designed to fit samples with a diameter of 60 mm and a height of 10 mm.The motorized 2 kbar screw press allows researchers to remotely control and fine-tune pressure values with precision.

Primary authors: BUCHNER, Andreas (TUM Frm2); VOGL, Lukas (Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II))

Co-authors: SUAREZ ANZORENA, Manuel; BILLER, Regina (Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München)

Presenters: BUCHNER, Andreas (TUM Frm2); VOGL, Lukas (Forschungs-Neutronenquelle Heinz Maier-Leibnitz (FRM II))

Contribution Type: Poster

Abstract # 79

Development of an automatic sample changer for the ROCK-IT project

As part of the ROCK-IT project (**r**emote, **o**perando **c**ontrolled, **k**nowledge-driven, and **IT**-based), we are developing a sample changer for the demonstrator setup that uses a collaborative robot with an individual gripper to pick specially designed sample holders from a magazine and transfer them to the measuring position.

A camera and image recognition are used to determine the grip positions and the set-down positions as well as to identify the sample holders marked with a QR code.

A special holder was designed for the measurement, which has the necessary precision and allows the sample to be heated up to 600°C.

In addition, the gas connections are inserted fully automatically after the sample change.

The entire sample changer system is abstracted via a SECoP interface. The current loading of the magazine is recognized via the camera and the complex process of a sample change can then be easily triggered via the SECoP interface.

References

ROCK-IT: https://www.rock-it-project.de/

SECoP: https://doi.org/10.3233/jnr-190143

Primary authors: GERISCHER, Sebastian (Helmholtz Center Berlin); Mr DONFACK, Bertrand (HZB); KIEFER, Klaus (Helmholtz-Zentrum Berlin); Mr KORKOT, Muhammed (HZB)

Presenters: GERISCHER,Sebastian(HelmholtzCenterBerlin); MrDONFACK,Bertrand(HZB); KIEFER, Klaus (Helmholtz-Zentrum Berlin); Mr KORKOT, Muhammed (HZB)

Contribution Type: Poster

Abstract # 80

THE EUROPEAN SPALLATION SOURCE MAGNET SUITE

Magnets and magnetism will play a major role at ESS, as at all neutron facilities. This is often combined with cryogenic temperatures, sometimes also with polarised neutrons. When planning and prioritising the ESS magnet suite, we took into account the proportion of magnet time expected for each instrument, existing available magnets, detector and instrument geometries, and the science cases to be investigated. The sample environment group worked closely with instrument scientists to come up with a prioritised suite of magnets, which has been updated after the initial set of magnets were known.

Several instruments at ESS are focused primarily on magnetic phenomena, notably MAGiC, ESTIA, and BIFROST, and many others will have magnetic measurements as a major part of their science topics to a greater or lesser extent 1.

We have been fortunate to be able to recover three second hand cryomagnets from HZB after the shutdown of the BER II reactor (the 14.5-15 T magnets VM1(B) and the asymmetric 6.5T VM2). These have been refurbished and tested with up-to-date control electronics and will form part of our suite.

Two new magnets have been procured so far. The first has just been delivered – a 2.1T asymmetric warm bore HTS magnet, primarily for the reflectometry instrument ESTIA. The second is a novel design of asymmetric 8T large aperture vertical field cryomagnet for magnetic diffraction, principally for use on the MAGiC instrument. This is fully designed and ready to go forward for construction.

Regarding the next phase of magnet procurement, a design study has been completed for a 14T spectroscopy magnet adapted for the CSPEC and TREX instruments. Static, and dynamic, magneticforceshavebeencalculated, leadingtosomesurprisingresults(seeabstractbyR.Viznichenko et al). An open tender will follow based on specifications identified by this study. Further, a high horizontal field split pair SANS magnet and a standard electromagnet will be part of the suite. In the longer term, as well as filling out the standard magnet family, ESS is prepared for the next generation of HTS and pulsed field magnets.

With regards to infrastructure, ESS has been designed from the beginning with a helium recovery system, compatible with the HZB helium management system. This allows us to use wet magnets as appropriate. All relevant instruments are subject to strict guidelines about use of magnetic material in the vicinity of the sample position, so unshielded magnets are standard. Nonetheless we have built a force testing device to verify any remaining issues due stray field. Standard mechanical interfaces will help with easy installation and alignment across instruments.

References

1 K.H. Andersen et al, Nuclear Inst. and Methods in Physics Research, A 957 (2020) 163402.

Primary authors: Dr HOLMES, Alexander (ESS); Mr SAXTRUP, Lauritz (European Spallation Source); Mr SAGLIANO, Luca (European Spallation Source); Dr LEE, Wai Tung (European Spallation Source ERIC); Dr CURFS, Caroline (ESS)

Presenter: Dr HOLMES, Alexander (ESS)

Contribution Type: Poster

Abstract # 81

ECS Integration of SECoP at the MLZ

The Sample Environment Communication Protocol (SECoP) is intended to standardize the interface between experiment control systems and sample environment equipment 1. To provide this interface at our institution, an adapter for the Experiment Control System (ECS) has to be written in order to represent hardware connected through SECoP in a canonical way. In case of MLZ, the experiment control system is NICOS [2], which is enhanced with the client provided by the FRAPPY SECoP framework [3] created by MLZ and PSI.
In this talk, I describe the SECoP endpoint of NICOS both in function and intended usage. Additionally, I give a short experience report of gradually moving a legacy driver to a new implementation based on Frappy.

References
1 K. Kiefer et al. "SECoP and SECoP@HMC - Metadata in the Sample Environment Communication Protocol", JACoW, ICALEPCS2023, (2023), THPDP014 https://doi.org/10.18429/JACoW-ICALEPCS2023-THPDP014
[2] https://nicos-controls.org
[3] https://forge.frm2.tum.de/public/doc/frappy/html/

Primaryauthors: ZAFT,Alexander(ForschungszentrumJülich); MrBRANDL,Georg(Forschungszentrum Jülich); Mr BRAUN, Peter (Helmholtz Zentrum Berlin); Dr EKSTRÖM, Nicklas (European SpallationSource); DrFAULHABER,Enrico(MLZ); DrGERRIT,Günther(HelmholtzZentrumBerlin); KIEFER, Klaus; Dr KLEMKE, Bastian (Helmholtz Zentrum Berlin); Dr KOTANSKI, Jan (DESY); Dr KRACHT, Thorsten (DESY); PETTERSSON, Anders (European Spallation Source); Dr ROSSA, Lutz (Helmholtz Zentrum Berlin); Dr UHLARZ, Marc (HZDR); Dr ZOLLIKER, Markus (Paul Scherrer Institut)

Presenter: ZAFT, Alexander (Forschungszentrum Jülich)

Contribution Type: Oral

Abstract # 82

An overview of the Time-Resolved Capabilities and Sample Modularity at CoSAXS

CoSAXS is a multipurpose SAXS instrument located at the 3 GeV ring of MAX IV Laboratory in Sweden. This instrument provides a versatile platform for conducting Small-Angle X-ray scattering (SAXS) experiments on a wide range of samples.
With an extensive array of sample setups, CoSAXS enables the application of multiple techniques on solid and solution samples.
To accommodate the high demand and facilitate the rapid exchange of sample setups, a standardized mounting system has been implemented and additive manufacturing techniques are utilized for efficient prototyping and production of customized sample holders.
Furthermore, CoSAXS is equipped with advanced sample environments, such as the setup for Time-Resolved SAXS-WAXS experiments (TR-XSS). Among other studies it has been used in nonreversible protein reactions after laser activation of caged compounds.

Primary authors: APPIO, Roberto (MAX IV Laboratory); AHN , Byunnam (Max IV Laboratory, Lund University, Lund, Sweden); ANN , Terry (Max IV Laboratory, Lund University, Lund, Sweden); HERRANZ TRILLO, Fatima (Max IV Laboratory, Lund University, Lund, Sweden); MOTA-SANTIAGO, Pablo (Max IV Laboratory, Lund University, Lund, Sweden; Australian Synchrotron, part of ANTSO, Melbourne, Australia); PLIVELIC , Tomás (Max IV Laboratory, Lund University, Lund, Sweden); SILVA, Jackson (Max IV Laboratory, Lund University, Lund, Sweden); SILVA, Vanessa (Max IV Laboratory, Lund University, Lund, Sweden)

Presenter: APPIO, Roberto (MAX IV Laboratory)

Contribution Type: Poster

Abstract #. 83

“Wet or Dry” that’s the question.

With the UKRI STFC goal to be net zero by 2040, the ISIS Cryogenics team have assessed the carbon footprint of their processes.
Cryogenic Sample Environment and Helium are critical for ISIS, Helium and cryogenic equipment enable the cooling of experimental samples to very low temperatures.
Here we describe the basic dry closed cycle refrigerator (CCR) systems, wet helium cryostats, and the equipment that ultimately supports an experiment at the ISIS facility and the power that these devices consume and then assess the carbon footprint these systems have.
We also look at the impact that CCR have had on cooling detectors on instruments within the Molecular Science Group and what carbon emissions the most CCR dependant instrument has.
Helium is a finite resource in need of preservation. In order to reduce our Helium consumption ISIS uses a Helium Recovery (HR) system. This allows us to recover helium used in experiments, store it, and reuse it later on, overall reducing our dependence on external sources.
This system has been in place for 7 years and currently recovers in excess of 90% of the Helium used at ISIS. We revisit the carbon footprint of both our helium recovery process and the production and delivery process from the supplier.
Taking into account the transportation and energy used in each process it was calculated that 500 g of CO_2 is produced per liquid litre of helium (so the carbon footprint is 500g CO_2/l He).
In comparison, the carbon footprint of the gas suppliers process is 710g CO_2/l He, making the output from our system 30% smaller.
We are now about to commission a Liquid Nitrogen (LN_2) precooling system, which will double the helium production from the same energy usage, we have assessed the carbon emission from the LN_2 process and as a further study compare our findings with CCR based liquefaction systems.

Primary author: Mr DOWN, Richard (STFC)

Presenter: Mr DOWN, Richard (STFC)

Contribution Type: Oral

Abstract # 84

HOW CAN WE BENEFIT FROM THE IOT REVOLUTION WHEN CREATING A MODERN AND EFFICIENT SAMPLE ENVIRONMENT CONTROL SYSTEM?

The ESS control system infrastructure is based on EPICS, which is effective for accelerators and similar systems but lacks flexibility in a sample environment context. Thanks to the high flux at ESS, the ESS sample environment team can expect a rapid turnover of sample environment systems, making swift software integration essential.

To efficiently control the sample environment systems and keep up with the high turnover, we developed our own integration tool, Octopy. This tool is based on the following technologies:

- SECoP: Syntax, metadata structure, etc., are defined by SECoP.
- JSON: The Octopy API uses JSON.
- MQTT: Allows the use of IoT tools and libraries, enabling multiple clients to interact with thesame data, add metadata, or modify data ad hoc. It provides flexibility and access to plug-and-play functionality.
- Python: Most of the core functionality is written in Python to allow it to be maintained by asmany people as possible.

Primary author: EKSTRÖM, Niklas (ESS)

Co-authors: CURFS, Caroline (ESS); PETTERSSON, Anders (European Spallation Source); Mr HAGELBERG, Andreas (European Spallation Source); Mr SAXTRUP, Lauritz (European Spallation Source)

Presenter: EKSTRÖM, Niklas (ESS)

Contribution Type: Poster

Abstract # 85

The Sample Environment Communication Protocol (SECoP)

The Sample Environment Communication Protocol (SECoP) serves as an international standard for the communication between sample environment equipment and the experiment control software at scattering facilities. A summary over the standard is presented as well as overview over available implementations and further developments. This includes both sides of the interface: the integration of software and firmware for monitoring and controlling devices on one side, and the integration into the beamline (experiment control) software on the other side.

Primary authors: Dr ZOLLIKER, Markus (Paul Scherrer Institut); BRANDL, Georg (bJülich Centre for Neutron Science JCNS at Heinz Maier-Leibnitz Zentrum (MLZ) Forschungszentrum Jülich GmbH, Garching, Germany); Mr EKSTRÖM, Niklas (European Spallation Source); Dr FAULHABER, Enrico (HeinzMaier-LeibnitzZentrum(MLZ),TechnicalUniversityofMunich(TUM),); KIEFER,Klaus(Helmholtz-Zentrum Berlin); KLEMKE, Bastian (Helmholtz-Zentrum Berlin); PETTERSSON, Anders (European Spallation Source); ROSSA, Lutz (Helmholtz-Zentrum Berlin); Mr WEGMANN, Peter (Helmholtz-Zentrum Berlin); ZAFT, Alexander (Forschungszentrum Jülich)

Presenter: Dr ZOLLIKER, Markus (Paul Scherrer Institut)

Contribution Type: Oral

Abstract # 86

Demonstration of the Implementation of a Simple Example Device into SECoP Using the Frappy Framework

Frappy is a Python framework to implement SECoP servers and clients. Its main working principle is explained, the integration of a simple device using Frappy is shown and the functionality is demonstrated live.

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Presenter: Dr ZOLLIKER, Markus (Paul Scherrer Institut)

Contribution Type: Oral

Abstract # 87

ISIS SAMPLE ENVIRONMENT DESIGN SECTION OVERVIEW

The ISIS Design Division's Sample Environment Section is responsible for delivering specialist equipment for our operations teams, instrument scientists, and visiting users. Specialising in mechanical design and project management, we work together with other operations and design teams across ISIS to deliver our projects. These can range from sample cans and sticks, to full systems incorporating pressure, vacuum, high temperatures, cryogenics, motion, and support & lifting equipment.

This presentation will briefly outline our current portfolio of projects, highlighting recent successes in bespoke technical fields. It will detail how the various scientific and operational staff work together to deliver projects at ISIS.

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Presenter: DUDMAN, Michael (ISIS)

Contribution Type: Oral

RAPID COOLING TECHNOLOGY FOR NEUTRON VACUUM FURNACES

Sample cooling is a major bottleneck in achieving fast throughput for high temperature vacuum neutron furnaces. The standard cooling approach (radiation under vacuum to the water-cooled jacket) takes many hours due to inefficient radiative transfer at sub-500°C temperatures. This process can be expedited by backfilling inert gases [1, 2], but at least 45 minutes is still required, and the cooling gas, usually a precious helium (He), is lost in the process. A new rapid cooling system utilizes a sub-atmospheric pressure He in closed-loop circulation to effectively cool the neutron furnaces without

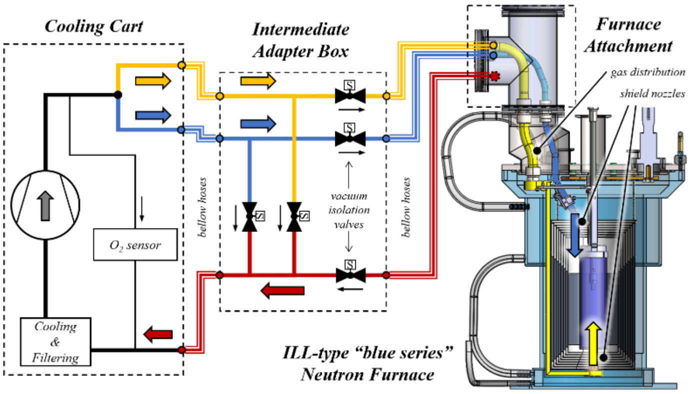

Figure 1: Schematic of rapid cooling system for ILL f...

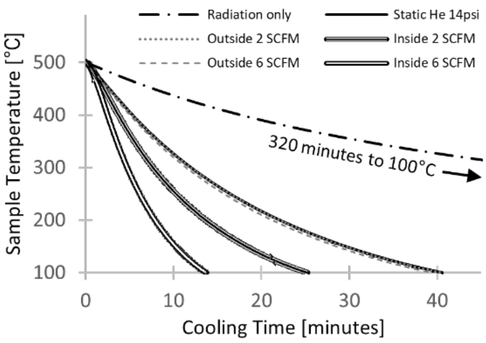

Figure 2: Comparison of cooldown times under vacuum (radiation), static He and active He circulation

safety concerns. The system schematic, Figure 1, highlights major components, including the main cooling cart, adapter-isolation box, and gas distribution nozzles inside the furnace. The gas nozzles installed inside the radiation shields vastly improve the cooling performance. As shown in Figure 2, the 6 SCFM He "inside" flow is able to cool down the furnace from 500°C to 100°C in just 13 minutes, a *23x time improvement over traditional cooldown* and 3x faster than stationary gas. He circulation without gas nozzles, the "outside" case, shows negligible improvement over still He, which agrees with the previous finding by Goodway *et al* [2]. The cooling system has been successfully demonstrated at Oak Ridge National Laboratory (ORNL), including an active beam experiment, Figure 3. The fast cooldown functionality enabled the standard Nb sample to undergo *three complete thermal cycles in just 140 minutes* – half the time it usually takes for a single experiment. Fast and controllable closed-loop cooling with autonomous operation makes this cooling technology attractive for high temperature neutron furnace experiments.

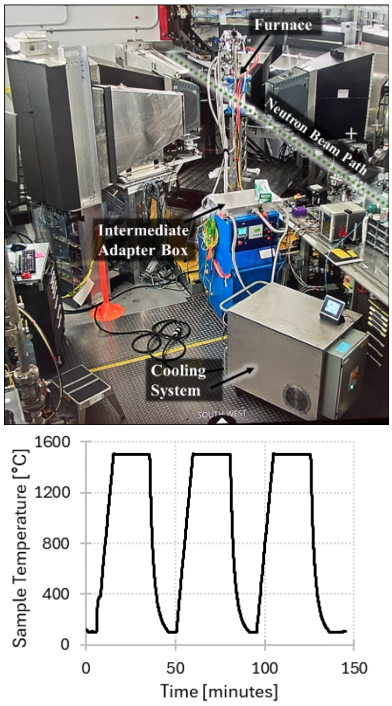

Figure 3: Live beam demonstration at ORNL VULCAN site.

References

[1] J. L. Niedziela *et al*, Rev. Sci. Instrum. 88 (2017), 105116.

[2] C. Goodway *et al*, J. Neutron Res. 21, 3-4 (2019) 137-142.

Primary authors: Dr RADYJOWSKI, Patryk (Advanced Cooling Technologies Inc.); Mr DAVIS, Reese (Advanced Cooling Technologies, Inc.); Mr ADHIKARI, Daksh (Advancec Cooling Technologies, Inc.); Dr CHEN, Chien-Hua (Advanced Cooling Technologies, Inc.); MILLS, Rebecca (Oak Ridge National Laboratory)

Presenter: Dr RADYJOWSKI, Patryk (Advanced Cooling Technologies Inc.)

Contribution Type: Oral

Abstract # 89

NIST CENTER FOR NEUTRON RESEARCH (NCNR) UPDATES!

During the NCNR shutdown, the Sample Environment team and NCNR staff continued working on several upgrade projects and initiatives to support our user program.
This presentation will include an update on the NCNR and the road to restarting, facility instrument improvements, and guides upgrades. We will also discuss the sample environment team's contribution to the Non-Equilibrium Initiative, progress in the helium recovery system, new equipment purchases, development projects, and upgrades to some of our existing in-house equipment.

Primary author: Ms HERNANDEZ, Yamali (NIST/NCNR)

Presenter: Ms HERNANDEZ, Yamali (NIST/NCNR)

Contribution Type: Oral

NIST Center for High-Resolution Neutron Scattering through the shutdown

During the reactor outage, we have been working to enable nonequilibrium capabilities on 3 of the flag-ship instruments within the Center for High-Resolution Neutron Scattering (CHRNS). CHRNS is the heart of the NCNR user program and with cooperation with the National Science Foundation (NSF). We will detail the infrastructure upgrades on these instruments, new software capabilities, point to new sample environments and integrations of the SECoPs protocol, and our extension to time-stamped capabilities. In addition, the NCNRhas a long-standingcommitmenttosharing open data with the community and have been working to uphold the FAIR data principles, aiming to enhance the value of the data collected at our facility for researchers and the scientific community.

Primary authors: BROWN, Craig (NIST); Dr MARANVILLE, Brian (NIST); Mr KIENZLE, Paul (NIST); Dr YE, Qiang (NIST Center for Neutron Research); JOSEPH, Adeniyi (NIST); Mr KRZYWON, Jeff (NIST); Dr MURPHY, Ryan (NIST); Mr CHABOT, Phil (NIST); Dr MALISZEWSKYJ, Nicholas (NIST); Dr YIMING, Qiu (NIST); Dr RODRIGUEZ, Jose (NIST); Mr PHEIFFER, Stephen (NIST); Dr AZUAH, Richard (NIST); Dr DURA, Joseph A. (NIST Center for Neutron Research); Dr GRUTTER, Alex (NIST); Dr HEINRICH, Frank (NIST); Dr HOOGERHEIDE, David (NIST)

Presenter: BROWN, Craig (NIST)

Contribution Type: Oral

4K CRYOSTAT WITH REDUCED SAMPLE CAN TEMPERATURE GRADIENTS

A new 4K cryostat has been developed in-house at the Australian Centre for Neutron Scattering for our backscattering spectrometer, 'Emu'. In the development process, we were able to make several improvements for our needs with respect to commercially available 4K cryostats. One large improvement was the in-house design, with drawing and manufacture of the parts by an Australian company. This will reduce our reliance on sending parts and systems overseas for repair and maintenance. We also designed and manufactured new, robust carbon fibre sample probes, a heat shield and new larger diameter sample cans. We standardized cabling, connectors and temperature controller configurations and characterized the system.

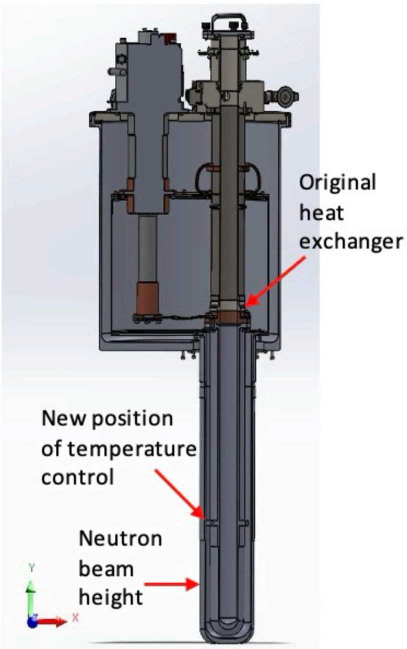

Figure 1: Heat exchanger positioning for reduction of temperature gradients across sample cans

One of the main technical improvements was the reduction of vertical temperature gradients across sample cans installed in the cryostat. These temperature differences can be as large as 10K and have been a long-standing issue across all of our cryostats. The challenge is tuning the temperature of the exchange gas around the sample can to the same temperature as the sample probe puck that the sample can is screwed into. The sample probe puck temperature is controlled by a cartridge heater and sensor in the puck. The exchange gas temperature is normally controlled by a heat exchanger with cartridge heater above the sample space of the cryostat.

Unfortunately, the heat exchanger cannot be positioned at the height of the sample can as it would then be in the neutron beam. In the case of the geometry of the Emu instrument, the problem is compounded by the requirement of a long cryostat tail, meaning that the distance from the heat exchanger to sample can is even longer than usual.

We were able to reduce the temperature differences across the sample cans to the range of 0.2K, by effectively moving the heat exchanger closer to the sample can. We designed a sample space with an added ledge to accommodate either a cartridge heater or ring heater and sensor. The heat exchanger control loop was moved down to this ledge. By using a ring heater we were able to overcome asymmetries in the temperature profile caused by heating on one side of the sample space with a cartridge heater.

Primary authors: SHUMACK, Amy (ANSTO); Mr BALDWIN, Chris (ANSTO); Mr DAVIDSON, Gene (ANSTO); MATEUCCI, R (LAHN, Argentina); Dr PAULL, Oliver (ANSTO); Dr STROH, Joshua (ANSTO); Mr D'ADAM, Tim (ANSTO)

Presenter: SHUMACK, Amy (ANSTO)

Contribution Type: Poster

Abstract # 92

AUSTRALIAN CENTRE FOR NEUTRON SCATTERING: FACILTY AND PROGRESS REPORT

In the two years since the 2022 ISSE workshop in Nasu, Japan, the Australian Centre for Neutron Scattering (ACNS) has continued to host a successful user program. Outlined here is a facility and progress report detailing the status of Sample Environment at ACNS, including operational aspects, new developments, and improvements across the existing suite of equipment. Presented here and in an additional poster, we will report on several key developments including peltier sample changers for SANS beamlines; a modular 4 K top-loading cryofurnace; a 1.3 T horizontal electromagnet with closed-loop control; and the commissioning of a second Kelvinox dilution insert. Other improvements include: the standardisation of hardware; revised infrastructure and the continued reimagining of core equipment across the facility.

In-flight projects will also be discussed, these include, redesigned solid-liquid cells, a suite of new high temperature furnace probes; wide-bore induction furnace, peltier sample tumblers for USANS & SANS and a dry, 1.5K top-loading cryofurnace.

There have also been staffing changes, with two new members joining the ACNS Sample Environment team, Oliver Paull and Joshua Stroh. In the past year we have also hosted two international visitors, Ross Price (ISIS, UK) and Ricardo Mateucci (LAHN, Argentina).

Primary authors: D’ADAM, Tim (ANSTO); Mr BALDWIN, Chris (ANSTO); Mr DAVIDSON, Gene (ANSTO); Dr PAULL, Oliver (ANSTO); SHUMACK, Amy (ANSTO); Dr STROH, Joshua (ANSTO)

Presenter: D’ADAM, Tim (ANSTO)

Contribution Type: Oral

Abstract # 93

Updates from JRR-3 following the restart

Following its restart in 2020 after a decade-long shutdown, JRR-3 has been maintained in stable operation and completed roughly 20 cycles for user programs by July. Alongside the reactor, neutron beam instruments also regained their functionality after overcoming various challenges. The sample environments also saw restoration and then improvements. To facilitate efficient and productive experiments, several instruments were equipped with new sample changers. New materials were applied to achieve a low background for small-angle neutron scattering. For solid-state physics, new dry magnet and top-load cryostats with a shared sample-space design were introduced. These new equipments have been fully operational for over a year, reducing workload, enabling efficient sample change and operation. I will present these advancements since the restart of JRR-3.

Primary author: KANEKO, Koji (Japan Atomic Energy Agency)

Presenter: KANEKO, Koji (Japan Atomic Energy Agency)

Contribution Type: Oral

Abstract # 94

ADVANCED SAMPLE ENVIRONMENT FOR NEUTRON SCATTERING AT JRR-3: A CRYOGEN-FREE HORIZONTAL MAGNETIC FIELD SYSTEM

Neutron scattering is a unique microscopic probe to investigate magnetism. When combined with a magnetic field, it gains further ability to manipulate ordered states, which helps to get insights into magnetic materials. This type of experiment is often carried out on a triple-axis spectrometer with a vertical field magnet, as it gives higher flexibility to explore wide Q-ω space up to high magnetic fields. On the other hand, despite significant demands, the number of horizontal field magnets is limited because of relatively low available maximum fields and limited access in the horizontal scattering plane owing to the shadow of the coils. Recently, there has been growing interest in complex magnetic structures, such as topological spin textures, many of which emerge under magnetic fields— and some of them are accessible only by using horizontal-field magnets. Furthermore, with polarized neutrons, a horizontal field provides unique ability, such as separating nuclear and magnetic components and/or detecting magnetic chirality.

Here, we have introduced a new horizontal-field system for neutron scattering. The system consists of a high-T_c (HTC) superconducting magnet and a variable temperature cryostat (VTC) running without liquid cryogens. The 3T horizontal-field magnet, provided by HTS-110, is specifically designed to accommodate a 3He spin filter; in other words, a low fringe field was realized 1. In addition, a compact coil design enables a wide horizontal window of 60° orthogonal to the field, which is required for measurement under a polarization channel p_x. The VTC is specially designed to fit into the room-temperature bore of the magnet. It shares a common sample space with existing cryogen-free VTCs in JRR-3 [2], enabling sharing of sample sticks and even ³He inserts among these systems. This horizontal-field system introduces new sample environments accessible in JRR-3, offering horizontal fields up to 3 T and low temperatures down to 0.3 K with polarized neutrons. The basic design of this system also fits into the chopper spectrometer at MLF, J-PARC, and will be tested in future.

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Presenter: Dr TABATA, Chihiro (Japan Atomic Energy Agency)

Contribution Type: Poster

Abstract # 95

Automated pumping carts

At ESS we are developing new pumping carts that are partly computer-controlled using Beckhoff PLC:s. This gives us the possibility to not only monitor the cooling process of cryostats and magnets, but also automate processes. We have automated parts of the cool-down and the cooling steady-state, as well as flushing of the sample space. Further automation can be implemented relatively easy with Python-scripts.
The cart uses two Kashiyama NeoDry pumps, for the He4 circuit and sample space respectively. The automated flushing sequence can be used for the entire system during initial preparation for cooling, though the number of automated valves is kept to a minimum. The state of the cart is designed to be easily understood at a glance. Temperature control, LN2 level measurement and refilling and He level measurement (using the HZB monitoring system) are built into the system.

Primary author: HAGELBERG, Andreas (ESS)

Presenter: HAGELBERG, Andreas (ESS)

Contribution Type: Poster

Abstract # 96

3D PRINTED PLASTICS FOR HIGH AND ULTRA-HIGH-VACUUM (UHV): A FEASIBILITY STUDY

The demand for cost and time-effective and customizable components for high-vacuum (HV) and ultra-high-vacuum (UHV) systems has prompted exploration into the application of 3D printing technology. This study investigates the viability of utilizing 3D printed plastics in UHV environments by evaluating their outgassing properties. An extensive evaluation of 3D printing materials was caried out, highlighting the best polymer composition candidates using two of the most common 3D printing techniques, Fused Deposition Manufacturing (FDM) and Stereolithography (SLA). Further experimental investigations are conducted to assess the performance of select 3D printed plastics under UHV conditions, focusing on their ability to maintain structural integrity, minimize outgassing, and withstand baking temperatures. Furthermore, residual gas analysis was used to evaluate the materials compatibility with NEG coated systems and possible presence of other contaminants. The findings suggest that certain 3D printed plastics exhibit promising characteristics for use in HV and UHV systems, with notable examples including Cyclic Olefin Co-polymer (COC) and polyether ether ketone (PEEK). A comparison between machined and 3D printed parts demonstrated that challenges such as porosity and surface roughness showed not to be of great concern.

Primary author: DOMINGUES, Artur (MAX IV Laboratory)

Presenter: DOMINGUES, Artur (MAX IV Laboratory)

Contribution Type: Oral

4 YEARS AFTER MAJOR ESRF-EBS UPGRADE, A VIEW ON THE ESRF EVOLUTION AND THE ROLE OF ITS SAMPLE ENVIRONMENT UNIT.

The European Synchrotron Radiation Facility (ESRF) has undergone a major upgrade, known as the Extremely Brilliant Source (EBS), marking a major leap in synchrotron science. The ESRF-EBS upgrade introduces a new-generation storage ring based on a hybrid multi-bend achromat lattice, which improves the beam brilliance and coherence by up to 100 times compared to previous capabilities. This enhanced performance has opened new possibilities for X-ray-based research, facilitating experiments with unprecedented spatial and temporal resolution. The upgrade also improves energy efficiency and stability, making it a model for future synchrotron sources worldwide that are planning their upgrades.
A selection of beamline having seen a recent upgrade or construction will be highlighted, and links with the sample environment will be emphasized.

In addition to the accelerator improvements, the ESRF-EBS upgrade has had significant consequences for all beamline operations in which the sample environment unit has played an important role in instrumentations for precise temperature and environmental control, enabling more complex and realistic in situ and operando experiments. These upgrades provides researchers with the ability to study samples under more various extreme conditions or in real-time reactions, greatly expanding the scientific capabilities of the facility.
On the other end of the spectrum of subject handled by the sample environment unit, robotic sample changers are continuing evolving toward higher sample change speed, higher conviviality for sample preparation, easy and fast installation and removal on the beamline. Nowadays a complete setup installation plus automatic realignment is done in 3 to 20 minutes, allowing high throughput measurement to be run in case of tight schedule for a couple of hours. Full removal of the equipment allows leaving full space for complex experiment in a matter of minutes.

Primary author: WATIER, Yves (The European Synchrotron Radiation Facility, Grenoble, France.)

Presenter: WATIER, Yves (The European Synchrotron Radiation Facility, Grenoble, France.)

Contribution Type: Oral

Abstract # 98

Sample Environment at Oak Ridge National Laboratory's Spallation Neutron Source and High Flux Isotope Reactor.

With the United States' highest flux reactor-based neutron source for condensed matter research (the High Flux Isotope Reactor) and the world's most intense pulsed accelerator-based neutron source (the Spallation Neutron Source), ORNL is becoming the world's foremost center for neutron science. Research at these facilities encompasses the physical, chemical, materials, biological, and medical sciences and provides opportunities for up to 2000 researchers each year from industry, research facilities, and universities all over the world.

Werecognizethatsampleenvironmentisanintegralcomponenttotheseexperimentsperformedat our facilities. A brief overview of new sample environments and capabilities that we have brought online within the last two years will be presented. Future directions will also be discussed.

Primaryauthor: LYNN,GaryW.(SampleEnvironmentandUserLabs, NeutronSciencesDirectorate, Oak Ridge National Laboratory)

Presenter: LYNN, Gary W. (Sample Environment and User Labs, Neutron Sciences Directorate, Oak Ridge National Laboratory)

Contribution Type: Oral

Abstract # 99

SAMPLE ENVIRONMENT AND CHARACTERIZATION AT THE EUROPEAN XFEL

The Sample Environment and Characterization (SEC) group of the European XFEL supports users and the scientists of our seven soft and hard X-ray instruments in regards of the development and operation of sample environments and sample delivery techniques. Our main focus is on the development of liquid sample delivery for biological and material science samples. In collaboration with several external partners we design devices for multiple delivery techniques for a wide range of samples. Fixed target, cryogenic and magnetic sample environments are also developed and operated in-house.

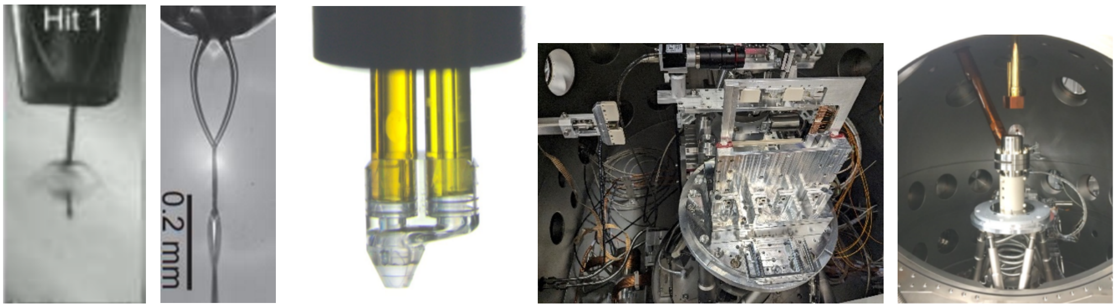

Figure 1: Sample delivery methods

GDVN, Flat Sheet, Mixing Jet, Fast Solid Sample Scanner, PUMA

We run the user laboratories, including biology, chemistry and material science labs, in which the users can prepare and characterize their samples with a wide range of characterization devices.

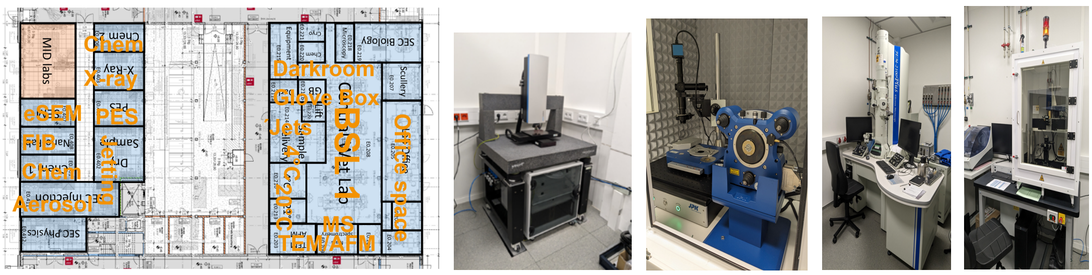

Figure 2: User labs of the European XFEL

Lab plan, Confocal, AFM, TEM, Laue camera

For single-shot experiments, the number of samples can reach thousands per beamtime. Manual handling of the samples and book keeping of all relevant information is not a suitable solution. Therefore, we are implementing a target workflow to automatize these steps for the optimization of the throughput. Starting from the production of a sample, followed by further preparation and characterization steps, the information of the full life cycle will be stored in a database, enabling the scientist to perform the final analysis with all measurements and meta data at hand.

References

[1] https://www.xfel.eu/facility/user_laboratories

Primary author: DEITER, Carsten (EuXFEL)

Presenter: DEITER, Carsten (EuXFEL) Contribution

Type: Oral