# JAPAN PROTON ACCELERATOR RESEARCH COMPLEX





## **MATERIALS AND LIFE SCIENCE EXPERIMENTAL FACILITY (MLF)**

### Contents

MLF neutron and muon instruments ··· 1 Neutron instrument characteristics · · · · · · 2 List of neutron and muon instruments $\cdots$  3 Introduction of each instrument ......... 5 Common sample environment equipments 

## **MLF neutron and muon instruments**



## **Neutron instrument characteristics**

The range covered by each instrument for (left) quasi-elastic / inelastic scattering and (right) diffraction. One could select an optimal instrument for ons's purpose.

Quasi-elastic/inelastic neutron scattering instruments

### Neutron diffractometers





## List of neutron and muon instruments





## **4D-Space Access Neutron Spectrometer (4SEASONS)**

Ouasi-elastic/inelastic neutron scattering

**BL01** 

## High-efficient observation of spin and lattice dynamics over four-dimensional momentum and energy space

#### **Instrument Description**

- •Medium-resolution thermal-neutron spectrometer using a Fermi chopper
- · High efficient measurements through a combination of efficient beam transport, large area detector, and use of multiple incident energies

#### **Specifications**

- Measurable energy: 1 to 300 meV
- Scattering angle range Horizontal: -35° to +130° Vertical  $:-25^{\circ}$  to  $+27^{\circ}$
- Energy resolution: About 5% of incident energy
- Beam size (variable): Max. 45mm  $\times$  45mm Optimum 20mm × 20mm

#### **Sample Environments**

- •Closed-cycle refrigerator (5 to 300 K; up to 600 K with high-temperature option)
- Radial collimator (Please inquire about the appropriate sample size)
- Other MLF-shared sample environment devices

#### **Instrument Configurations**





Beam transport guide tube





Large-area neutron detector



### **4D-Space Access Neutron Spectrometer** (4SEASONS)





#### **Capabilities**

- Observation of motion of spins, atoms, and molecules in the momentum and energy range of 0.1  $\AA$ <sup>-1</sup> to 20  $\AA$ <sup>-1</sup> and 1 meV to several 100 meV
- Determination of the couplings between spins, atoms, and molecules, and their relationship to magnetic and structural properties



Excitation spectra of  $CuGeO<sub>3</sub>$  measured using multiple incident energies  $(E_i)$  simultaneously

#### **Applications**

- Magnetic excitations and lattice vibrations in superconductors
- Novel magnetism in quantum magnets, frustrated magnets  $\frac{1}{1}$ , and itinerant magnets  $\frac{2}{1}$
- I attice vibrations in thermoelectric materials
- Excess excitations in glasses
- Lattice vibrations in catalysts



#### Topological spin excitations in a three-dimensional antiferromagnet



W. Yao et al., Nat. Phys. 14, 1011(2018) (© 2018 Nature Publishing Group).

## **Biomolecular Dynamics Spectrometer (DNA)**

Elucidation of functional origins of materials by measuring atomic and molecular dynamics in nanosecond order

#### **Instrument Description**

 $\bullet$  High energy resolution ( $\neg$ µeV) in a wide dynamic range (meV)

**BL02** 

Ouasi-elastic/inelastic neutron scattering

- Flexible energy resolution and dynamic range by changing pulse-shaping choppers
- . High efficiency and high signal-to-noise ratio  $(-10<sup>5</sup>)$  owing to back coated Si-analyzers

#### Specifications

- $\bullet$  Dynamic range  $\div$  -500 to 1500 µeV : Si(111),  $-2000$  to 6000 µeV : Si(311)
- $0.08 < Q < 1.98$  [Å<sup>-1</sup>] : Si(111),  $\bullet$ Q-range  $1.79 < Q < 3.39$  [Å<sup>-1</sup>] : Si(311)
- Energy resolution: Si111 1 cm slit: 2.2 µeV (300 Hz), 2.4 µeV (225 Hz) Si111 3 cm slit: 3.1 µeV (300 Hz), 3.6 µeV (225 Hz)
- Beam size  $: 3$  cm (vertical)  $\times$  2 cm (horizontal)

#### **Sample Environments**

- ●Top-loading cryofurnace  $(7 K < T < 650 K)$
- •Sample changer (3samples, 20samples)
- Liquid Pressure Cell  $(0.1 < P < 145$ [MPa], room temp.)
- · Humidity control system
- $\bullet$ High electric voltage cell  $(E < 1.5$  kV,  $< 200^{\circ}$ C), High electric current cell  $(I < 5 A, < 200^{\circ}C)$

## **Band chopper (25Hz)** Frame separation chopper (200,150Hz) Pulse shaping chopper (300, 225Hz)

#### **Instrument Configurations**





TOF-type near backscattering spectrometer using perfect Si111 and Si311 crystal analyzers



## **Biomolecular Dynamics Spectrometer**  $(DNA)$





#### **Capabilities**

- Dynamics of atoms, molecules, ions, and spins in wide dynamic range from pico to nano seconds
- ●Translational diffusion, rotation, vibration, spin gap



#### **Applications**

- · Biomacromolecules (proteins, lipids, foods)
- Soft materials (polymers, ionic liquids, hydrated water, rubbers)
- Functional materials (batteries, catalyst, ferroelectrics)
- Magnetism



Reveal the hierarchical dynamics of water in fuel cell catalyst

K. Ito, et al., J. Phys. Chem. C 125, 12645 (2021). (©2021American Chemical Society)

Magnetic anisotropy gap in multiferroic Ba<sub>2</sub>MnGe<sub>2</sub>O<sub>7</sub>



S. Hasegawa et al., Phys. Rev. Research 3, L032023 (2021). (©2021American Physical Society)

#### **BL03 IBARAKI Biological Crystal Diffractometer (iBIX) Neutron diffraction**

Single crystal neutron diffractometer for observation of hydrogen and hydration structure of macromolecules and organic compounds with high efficiency and high resolution

#### **Instrument Description**

- · High measurement efficiency by a wide wavelength range. The development of a high-resolution two-dimensional detector enables highly efficient measurement of protein structure analysis data.
- •The open space around the sample allows for the free placement of various sample environment.

#### **Specifications**

Specification (@1MW)

- $\bullet$  Max. Cell dimension : 135  $\times$  135  $\times$  135 Å<sup>3</sup> (Demonstrated by single crystal structure nalysis of manganese catalase, which has the largest crystal lattice volume)
- $\bullet$ Sample volume  $\div$  < 1 mm<sup>3</sup>
- $\bullet$  Measurement time: 4 days

#### **Instrument Configurations**





#### **Sample Environments**

- Gas flow type cooling system (100 300 K)
- ●Heating device (300 600 K)
- ●Stretching device (Max. load: 200 N, Max. stretching length: 90 mm, Speed:  $1 - 1000 \mu m/sec$



Stretching device

**Heating device** 

 $\sim$ 



Gas flow type cooling system

### **CONTACT** Katsuhiro KLISAKA  $\mathsf{k}$



### **IBARAKI Biological Crystal Diffractometer**  $(iBIX)$





#### **Capabilities**

- •The presence or absence of hydrogen bonds between proteins and inhibitors, and proton tautomerism, based on the positions of hydrogen atoms in proteins
- Positions of hydrogen atoms of OH, NH, and NH<sub>2</sub> in the side chains of amino acid residues
- •The presence of water molecules expected from the existence of the exchangeable hydrogen by heavy water.
- Positions of hydrogen atoms around heavy elements, which are difficult to detect using X-rays
- Protein structure at room temperature with minimal radiation damage
- · Undamaged structure of oxidoreductase that undergoes structural changes in X-rays
- •Information on hydrogen bonds and hydrophobic interactions in protein-compound interactions



TOF diffraction pattrern of a protein crystal

#### **Applications**

- Observation of hydrogen-bond network caused the stabilization of transthyretin tetramer associated with human amyloidosis.
- Visualization of newton's cradle proton relay with amide-imidic acid tautomerization in Hydrolysis process of cellulase that catalyze cellulolysis.
- Two hydrogenation states and structural features of the bilin reductase PcyA that synthesizes photoreceptor pigment and substrate complex.
- Neutron crystal structure of catalase with the largest unit cell volume (135X135X135Å) of all neutrons structure analyses.
- Observation of keto/enolate interconversion of the quinone cofactor and unusual proton sharing between the cofactor and the catalytic base in copper amine oxidase
- Experimental evidence of the electron transfer pathway in the catalytic reaction of the copper-containing nitrite reductase play an important role for the Earth's nitrogen cycle.

The ingenious mechanism of enzyme catalysis revealed by unusual proton sharing by successful high-resolution neutron crystallography of a large protein

#### Background

- Copper amine oxidase : An enzyme that decompose primary amines into aldehydes and ammonia. Containing a Cu<sup>2+</sup> ion and the protein-derived guinone cofactor topa guinone (TPQ). This enzyme in human serum is also involved in the onset of diabetes.
- •Visualization of hydrogen atom positions is necessary to understand the details of the enzyme reaction mechanism of copper amine oxidase.

#### **Breakthrough in this research**

- Successfully performed neutron crystal structure analysis of a large protein (molecular weight: 70.600) far exceeding previous records
- Revealing the precise position of hydrogen ions, essential for elucidating the mechanism of enzyme reactions





#### **Contributions by iBIX**

High-precision, high-resolution neutron structure analysis data for copper amine oxidase could be obtained and successfully determined its three-dimensional structure, including hydrogen atoms

#### Significance of research results

- •The result of this study will greatly expand the scope of application of neutron crystal structure analysis. It is expected that this will lead to the determination of the three-dimensional structures including hydrogen atoms for
- high-molecular-weight proteins and target proteins for Drug Discovery. . This results demonstrate that neutron crystal structure analysis can reveal the
- existence of previously unsuspected structures and reaction mechanism.

The reaction mechanism of an enzyme play an important role for the Earth's nitrogen cycle - High-precision determination of the all-atom structure of copper-containing nitrite reductase (CuNIR) -

#### **Background**

- CuNIR: Copper-containing nitrite reductases (CuNIRs) transform nitrite to gaseous nitric oxide, which is a key process in the global nitrogen cycle.
- To understand the details of the CuNIR chemical reaction (the process in which hydrogen ions are transferred to nitrite ions), it is necessary to determine the hydrogen atom positions.

#### **Breakthrough in this research**

- It is found that a hydroxide ion can exist as a ligand to the catalytic Cu atom in the resting state
- The results of this sutudy indicates the possibility of correctly rewriting the chemical reaction mechanism proposed by previous experiments, and succeeded in obtaining experimental evidence for the pathway through which electrons necessary for the reaction within the protein



**Contributions by iBIX** 

hydrogen atoms



Hydroxide ions bound to copper ions (brown spheres) observed in CuNIR

**Future development** 

Obtained high-precision, high-resolution

neutron structure analysis data from

CuNIR and successfully determined the

three-dimensional structure including

- •Industrial applications: Use of microorganisms with enhanced denitrification activity, development of artificial enzymes
- $\bullet$  Reduction of nitrous oxide gas (greenhouse gas) = Contribution to improving the air quality, reduction of excess nitrogen compounds that flow into the environment as fertilizer = Contribution to improving environmental pollution

enolate and the keto forms of TPQ. Unusual proton sharing It was previously assumed to be an hetween the cofactor enolate form and the catalytic base

structure anion (fully deprotonated  $form<sup>1</sup>$ 

The equilibrium between the

Active-site H-bond network and  $Cu^{2+}$ -coordination His is exist as an imidazolate

#### **Accurate Neutron-Nucleus Reaction Measurement Instrument BL04** (ANNRI) Neutron-nuclear reaction/ Prompt gamma-ray analysis

Instrument precisely measuring prompt  $y$ -rays produced in neutron capture reactions for research related to nuclear science, such as nuclear data measurement, elemental analysis, and astrophysics.

#### **Instrument Description**

● Several y-ray detectors are available for precise measurement:

▶ Ge cluster detectors with high energy resolution, ▶ Large Nal(Tl) detectors with good time resolution, LaBr<sub>3</sub> detector with low dead time.

•Li-glass detectors capable of measuring transmission neutrons.

#### **Specifications**

- Neutron energy range:  $E_n > 0.0015$  eV
- $\bullet$  y-ray detector: Ge spectrometer (Flight path: 21.5 m) :Nal (TI) detector (Flight path: 28 m) :LaBr<sub>3</sub> detector (Flight path: 28.5 m)
- Neutron detector: Li-glass detector (Flight path: 28.5 m)

#### **Instrument Configurations**



**ANNRI** Ge spectrometer (left), and NaI and Li-glass detectors (right)

#### Development of Radiation Detector



Development of Data Acquisition (DAQ) System





DAQ System(CAEN modules) **System Development** 

#### **Peripheral Equipment**

- •Variable Collimator Adjustable beam sizes: Ø 22, 15, 7, 6mm
- Automatic sample changer  $\sim$  Automatic exchange of up to 200 samples
- •Double disk chopper cuts lapsed low-energy neutrons
- Movable neutron filter (Pb, Mn+Co+In+Ag, Cd) Reduction of  $y$ -rays on neutron beam Adjustment of Neutron beam intensity, B.G. estimation



#### **CONTACT**



### **Accurate Neutron-Nucleus Reaction Measurement Instrument (ANNRI)**











#### **Applications**

- Neutron capture cross section studies of Minor actinides (Cm isotopes, Am isotopes, <sup>237</sup>Np, etc.) using the Ge spectrometer and the large Nal spectrometer
- Measurements of total neutron cross sections of Am isotopes using the Li-Glass Detectors
- Development of a new prompt gamma-ray analysis method using the time-of-flight method
- $\bullet$ Study of  $^{112}$ Cd(n,  $v$ ) reaction for astronomical origin of  $^{115}$ Sn
- CP violation in compound nuclear resonance

#### Nuclear data research for nuclear transmutation

Transmutation is a method of convert nuclear waste nuclides such as minor actinides (MAs) and long-lived fission products (LLFPs) into stable nuclides through nuclear reactions.



For the design study of transmutation systems, it is essential to improve the accuracy of the neutron capture reaction cross section, i.e., the ease of neutron capture reaction (red arrow).



A neutron-nuclear reaction measurement instrument (ANNRI) has been installed at MLF, and research is being conducted to improve the accuracy of nuclear data such as neutron capture reaction cross sections.

#### Prompt gamma-ray analysis using the TOF

By using the world's most intense pulsed neutron beam, high-efficiency Ge detectors, high speed DAQ and high-efficiency shielding, we succeeded for the first time in the world in developing a new analytical method that combines two non-destructive elemental analysis methods.

The synergistic effect of the fusion of the new analytical methods has demonstrated that even elements that are difficult to analyze using either method can be analyzed accurately.



## **Neutron Optics and Fundamental Physics (NOP)**

Beamlines for fundamental physics experiments using state-of-the-art neutron optics technology to explore the origins of matter and the universe.

#### **Instrument Description**

- •The beamline is branched into three branches using neutron benders upstream.
- Fast neutrons are shielded upstream and only slow neutrons are available.
- **Each of the three branches has its own** characteristic beam.

**BL05** 

**Fundamental neutron physics** Neutron device development

#### **Specifications**

The beamline is branched into the following three branches. Each has the following characteristics

- · Unpolarised: high intensity
- Polarised: 94 96% polarisation
- Low divergence: low divergence and high luminosity

#### **Auxiliary equipment**

- Position sensitive 2D detector Resolution 0.5 mm
- $\bullet$  Doppler shifter  $3$  type Ultra-cold neutron (UCN) source Pulsed UCNs are available.
- •Multi-layer type neutron interferometer
- Coherent scattering length measurements Sensitivity with 20 mrad/20 min

#### **Instrument Configurations**





#### **CONTACT**



### **Neutron Optics and Fundamental Physics** (NOP)





#### **Capabilities**

- 3 Characteristic beams come in three branches
- Independent experiments can be carried out in parallel on each

Neutron decay

Neutron-matter interactions

Development of UCN devices

#### BL05 Beam conditions for each branch



#### **Applications**

- Precise measurement of neutron lifetime
- Pulsed super-cooled neutron generation using a Doppler shifter
- Neutron scattering of rare gases to search for unknown interactions
- Cold neutron interferometry
- · Ultra-high resolution neutron detector development

#### Muti-layer type neutron interferometer

## Schematic for the

interferometer. The multi-laver neutron mirrors are on beam splitting etalons. Neutron beams are spitted at one etalon and merged on another one.



Setup of the neutron interferometer at



In case one path covered, oscillation disappears.

T. Fujije et al., Phys. Rev. Lett. 132, 023402 (2024) (© 2024 American Physical Society).

Low-divergence beam branch



Oscillation without (top) and with sample (bottom)

#### of a photographic film used (substrate) as a tracking device in particle physics, which features high spatial resolution. We succeeded Cold/UCN in developing a neutron detector which realizes a spatial resolution of 11-99

neutron emulsion detector with

spacial resolution of 100 nm



Nuclear emulsion is a type

Images of photograph by cameras(a&c). microscope(b), and neutron emulsion(d) of the Siemens star test pattern made by Gd. Spacial resolution of less than 1 um is available.

A. Muneem et al., J. Appl. Phys. 133, 054902 (2023) (© 2023 AIP Publishing LLC).

For 3), see Glossary (p.61)

## **Ahenrntinn** point Sputtered by M Hino at KURR

 $10B$ <sub>s</sub>C

Si 0.4 mm

 $~50~\mathrm{nm}$ 

NiC 60 nm Emulsion

C 30 nm 10 um

#### **Village of Neutron Resonance Spin Echo Spectrometers BLO6** (VIN ROSE) **Ouasi-elastic/inelastic** neutron scattering

## Dynamical behaviors of materials from ps to sub-us

#### **Instrument Description**

- Neutron spin echo spectrometers (NRSE and MIEZE) with neutron resonance spin flippers.
- Direct observation of intermediate scattering function is possible from ps to ns to investigate atomic and molecular dynamics.

#### **Specifications**

- $\bullet$  Fourier time : 1 ps 2 ns (Sample to Detector Length: Max. 2 m, Max. MIEZE) Flequncy: 400 kHz)
- Scattering Angle  $2\theta = 0 15^{\circ}$
- $\bullet$  Possible Neutron Reflectivity: R > 1.0<sup>-5</sup>
- MIEZE instrument is currently available for user programs. (Option: Neutron Polarization Analyses) Please contact the responsible persons.

#### **Instrument Configurations**





#### **Sample Environments**

#### ● 4 K GM Cryostat

![](_page_15_Picture_15.jpeg)

## **CONTACT**

![](_page_15_Picture_17.jpeg)

### Village of Neutron Resonance Spin Echo Spectrometers (VIN ROSE)

![](_page_16_Picture_1.jpeg)

![](_page_16_Picture_2.jpeg)

#### **Capabilities**

- · Dynamics of atoms and molecules in nano-second order.
- Neutron polarization analyses are available.

![](_page_16_Picture_75.jpeg)

#### **Applications**

- Slow dynamics of soft matters, such as polymers, proteins, membranes, colloids, etc.
- $\bullet$  Neutron polarization analyses for investigation of super paramagnetism in magnetic nanoparticles  $4$ .
- . Investigation of viscoelastic properties of plastics with molecular scale.
- Study of spin dynamics in magnetic materials.

![](_page_16_Figure_12.jpeg)

M. Hino et al., Physics Procedia 42, 136 (2013) (©2013 Elsevier).

#### Spin dynamics of magnetic Skyrmion in MnSi

![](_page_16_Figure_15.jpeg)

![](_page_16_Picture_16.jpeg)

T. Nakajima et al., Phys. Rev. Res. 2, 043393 (2020). (©2020 American Physical Society).

For 4), see Glossary (p.61)

#### **Super High Resolution Powder Diffractometer BL08** (SuperHRPD) **Neutron diffraction**

## Observation of slight structure distortion with high precision

#### **Instrument Description**

- A number of reflection intensities with distinct peaks can be observed
- A slight peak split with phase transition can be observed
- · Hydrogen atom position can be determined
- Enables the development of new structural analysis methods by complementing X-ray in intensity and resolution

#### **Specifications**

 $\circ$  d –range (standard setting) 0.3 - 3.75 Å (High-angle bank:  $150^{\circ} \le 2\theta \le 175^{\circ}$ )  $0.4 - 5.2 \text{ Å}$  (90 degree bank:  $60^{\circ} \le 2\theta \le 120^{\circ}$ ) 0.6 - 15 Å (Low-angle bank:  $10^{\circ} \le 2\theta \le 40^{\circ}$ )

- $\circ$  Resolution ( $\Delta d/d$ ) Optimal resolution 0.0365% ( $\omega$  2 $\theta$  > 172°)
	- $\geq$  0.08% (High-angle bank)
		- $\geq$  0.35% (90 degree bank)
		- $\geq$  0.7% (Low-angle bank)

#### **Sample Environments**

- Auto sample changer (RT, 10 samples)
- ●4 K closed cycle refrigerator (4 300 K)
- $\bullet$  Cryo furnace (5 800 K)
- $\blacktriangleright$  High temperature furnace ( < 1223 K)
- •1 K closed cycle refrigerator (0.9 300 K)
- ●14 Tesla Magnet

#### **Instrument Configurations**

![](_page_17_Picture_21.jpeg)

![](_page_17_Picture_22.jpeg)

High angle detector bank and high resolution detector unit with 8mm PSD

## **CONTACT** Shuki TORII torij@post.kek.jp Takashi SAITO saitot@post.kek.jp

![](_page_18_Picture_1.jpeg)

![](_page_18_Picture_2.jpeg)

#### **Capabilities**

- High resolution crystal and magnetic structure refinement
- Detection of extremely small structural distortions

![](_page_18_Figure_6.jpeg)

Neutron diffraction data of powder silicon measured by high-resolution mode and crystal structure analysis results using Z-Rietveld

#### **Applications**

- Spin-state transition and giant magnetovolume effect in cobalt oxides<sup>5)</sup>
- High temperature structural analysis of high sodium ion conductors
- Lattice distortion associated with magnetic phase transitions in 4d transition metal oxides
- Negative thermal expansion of perovskite-type cobalt oxides near room temperature<sup>5)</sup>

![](_page_18_Figure_13.jpeg)

We were the first to successfully detect a slight increase in the Ru-O bond length (0.0005 Å) associated with magnetic ordering in the 4d magnet SrRuO<sub>3</sub>.

Lee et al., J. Phys.: Cond. Mat. 25, 465601(2013) (© 2013 **IOP Publishing)** 

![](_page_18_Figure_16.jpeg)

Diffraction patterns of perovskite cobalt oxides showing negative thermal expansion at 0 and 14 Tesla.

The 004 reflection was shifted by 0.05% by the magnetic field and the 200 reflection was reduced in intensity by  $20%$ 

![](_page_18_Figure_19.jpeg)

## **Special Environment Powder Diffractometer (SPICA)**

**Neutron diffraction** 

**BL09** 

Real-time observation of structure and its change of functional materials and non-equilibrium reaction in practical devices

#### **Instrument Description**

- SPICA is dedicated to structural investigations for next-generation rechargeable batteries
- SPICA focuses on neutron diffraction experiments in special environment -particularly in charge-discharge operations (i.e., operando measurements)
- SPICA is used in experiments in various fields, including physics, chemistry, materials science, archeology, environmental science, and battery science

#### **Specifications**

 $\bullet$ Range of d-spacing  $0.3 - 3.7 \text{ Å}$  (High-angle bank)  $0.4 - 5.0 \text{ Å}$  (90-degree bank) 0.5 - 11.0 Å (Low-angle bank)

• Resolution  $(\Delta d/d)$ 0.09% (Optimal resolution) 0.12% (High-angle bank) 0.47% (90-degree bank) 1.27% (Low-angle bank)

#### **Sample Environments**

● Auto sample changer (RT, 40 samples)

●Top-loading 4K-type cryostat (4 - 300 K)

●Top-loading crvo-furnace (30 - 700 K)

● Potentiostat, Temperature control system, etc.

![](_page_19_Picture_15.jpeg)

![](_page_19_Picture_16.jpeg)

![](_page_19_Picture_17.jpeg)

#### **CONTACT**

![](_page_19_Figure_19.jpeg)

## **Instrument Configurations**

![](_page_19_Figure_21.jpeg)

### **Special Environment Powder Diffractometer** (SPICA)

![](_page_20_Picture_1.jpeg)

![](_page_20_Picture_2.jpeg)

#### **Capabilities**

- Neutron diffraction data with high resolution & intensity in the wide  $d$  (or  $Q$ ) range
- Operando neutron diffraction measurements
- Annex building for SPICA is available to prepare samples for neutron diffraction experiments, assemble rechargeable batteries, and perform charge-discharge evaluations before and/or after operando measurements

![](_page_20_Figure_7.jpeg)

Operando neutron diffraction measurements

#### **Applications**

#### Direct observation of battery reactions through operando neutron diffraction measurements

Elucidating the reaction mechanisms that occur during practical operation is crucial for the development of battery technologies. An operando diffraction technique uses high-intensity neutrons to detect reactions in non-equilibrium states driven by high-current operation in commercial batteries. This technique provides valuable information for developing advanced batteries.

![](_page_20_Figure_12.jpeg)

"Real-time observations of lithium battery reactions operando neutron diffraction analysis during practical operation"

S. Taminato et al., Scientific Reports, 6 (2016)28843 (©2016 Nature Publishing Group)

#### Structural investigations of hydride-ion-conducting materials using neutron diffraction

Various hydride-ion-conducting materials have been found and developed nowadays. Neutron diffraction is a powerful tool to determine precisely the atomic positions of hydride ions.

![](_page_20_Figure_18.jpeg)

"Pure H<sup>-</sup> conduction in oxyhydrides" G. Kobayashi et al., Science, 351 (6279), (2016), 1314-1317 (© 2016 AAAS)

#### **NeutrOn Beam-line for Observation and Research Use BL10** (NOBORU) **Fundamental neutron physics/** Neutron device development

Serving a versatile neutron field for characterizing the neutron source as well as for R&D on various devices, irradiation and analysis of materials, etc.

#### **Instrument Description**

- . Robust shielded cave and tough experiment table for massive sample/device testing.
- Closest irradiation position to the source of MLF, which can provide high-energy (above 10 MeV) to low-energy neutrons (below 1 meV).
- Simple and stable beamline device/optics.

#### **Specifications**

- ●L1 (moderator to sample pos.) : 14.0 m
- $\bullet$ Max, beam size : 100 mm  $\times$  100 mm
- $\bullet$  Inside room  $\cdot$  W 2.5 m  $\times$  1.3.5 m  $\times$  H 3.0 m
- $\bullet$  Neutron flux @ 14 m, 1 MW
	- $< 0.4$  eV : 4.8  $\times$  10<sup>7</sup> [n/s/cm<sup>2</sup>]  $> 1$  MeV : 1.2  $\times$  10<sup>7</sup> [n/s/cm<sup>2</sup>] > 10 MeV : 1.2  $\times$  10<sup>6</sup> [n/s/cm<sup>2</sup>]

#### **Sample Environments**

• Fach user builds the environment for each experiment.

![](_page_21_Figure_14.jpeg)

![](_page_21_Picture_15.jpeg)

### NeutrOn Beam-line for Observation and **Research Use (NOBORU)**

![](_page_22_Picture_1.jpeg)

![](_page_22_Picture_2.jpeg)

#### **Capabilities**

- •Measurement and diagnosis of neutron source properties.
- Serves as a test port for neutron optics, detector development, etc.
- Provides a high-intensity, high-energy neutron irradiation field

![](_page_22_Figure_7.jpeg)

![](_page_22_Figure_8.jpeg)

#### **Applications**

- Demonstrated world's highest neutron performance, fixed-point observation
- Element selective studies of local structures around dopants by white neutron holography.
- Development of SEOP<sup>6</sup> -type polarized neutron devices and experiments.
- •Micron-order imaging with high-resolution 2D neutron detector using superconducting device.
- Evaluation of semiconductor device and plant mutagenesis by high-energy neutron irradiation.

#### Spatial distribution of elements using prompt  $y$ -ray analysis

beam dia, of several mm.

 $\Rightarrow$ Identification and

![](_page_22_Figure_16.jpeg)

![](_page_22_Figure_17.jpeg)

#### Cosmic ray tolerance of power semiconductors

![](_page_22_Picture_19.jpeg)

Cross-sectional model of insulated gate bipolar transistor

![](_page_22_Figure_21.jpeg)

For 6), see Glossary (p.61)

#### 22

## **High Pressure Neutron Diffractometer (PLANET)**

**Neutron diffraction** 

**BL11** 

Accurately determine structures of crystalline, liquid, and glass over a wide pressure and temperature range.

#### **Instrument Description**

- High pressure and high temperature experiments up to 14 GPa and 2,000K with the 6-axis press, ATSUHIME
- . High quality diffraction patterns free from contaminant peaks from sample surrounding materials via narrow incident slits pressure and radial collimators

#### **Specifications**

**Diffraction** 

Resolution:  $\Delta d/d = 0.6\%$ , d-range: 0.2-4.2Å Beam size: 1-15mm square. Radial collimator gauge size: 0.5, 1.1, 2.8 mm

#### Radiography

Resolution: 200  $\mu$ m, Field of view: 14 mm square

#### **Sample Environments**

High-pressure presses and available PT range

- Paris-Edinburgh cell: 40 GPa & 300 373 K
- 6-axis press : 14 GPa & 300 - 2,000 K
- Mito System : 5 GPa & 30 - 273 K
- Piston cylinder cell : 2 GPa & 4 300 K

![](_page_23_Figure_17.jpeg)

![](_page_23_Picture_18.jpeg)

#### **CONTACT**

![](_page_23_Picture_20.jpeg)

## **High-Pressure Neutron Diffractometer** (PLANET)

![](_page_24_Picture_1.jpeg)

![](_page_24_Picture_2.jpeg)

#### **Capabilities**

- Atomic and magnetic structures of crystals and liquid/glass under high pressure
- ●Transmission image of the sample under high pressure

![](_page_24_Picture_6.jpeg)

#### H-D interdiffusion of brucite at high PT

![](_page_24_Figure_8.jpeg)

#### **Applications**

- Estimation of light elements in the Earth's core.
- Exploration of new ice polymorphs
- Hydrogen bond symmetrization of OH including minerals
- Pressure induced structural changes in liquid and glasses

#### Discovery of new ice polymorph, XIX

Ice takes various crystal structures depending on the PT condition. This study revealed that the ice VI transformed into the new hydrogen ordered form (XIX) on cooling. The discovery of the new phase is the first in two years, since 2019, and will help in the comprehensive understanding of ice polymorphism in the future.

![](_page_24_Figure_16.jpeg)

stable region of ice XIX discovered in this study. R. Yamane et al., Nature Commun. 12, 1129 (2021)

(©2021 Springer Nature)

#### New graphitic material synthesized by compression of  $CS<sub>2</sub>$

 $CS<sub>2</sub>$  instantly decomposes into sulfur and  $C<sub>2</sub>S$  at about 10 GPa. Annealing of the recovered C<sub>2</sub>S further decomposed into sp<sup>2</sup> graphite sheets bound to sulfur. Since they can be synthesized in large quantities under high pressure, they will be useful for the design of electronic devices based on graphite sheets.

![](_page_24_Figure_21.jpeg)

(Left) Sample recovered from high pressure and its estimated structure. (Right upper) Comparison of diffraction patterns between the recovered sample and layered carbon. (Right lower) Pair distribution function of the recovered sample. C-C interatomic distance characteristic to the carbon sheet are observed.

S. Klotz et al., Carbon 185, 491 (2021) (©2021 Elsevier Ltd.)

## **High Resolution Chopper Spectrometer (HRC)**

HRC enables us to access the wide momentum  $(Q)$  and energy  $(E)$  for conducting forefront researches on dynamical properties in material with the best resolution ever!

#### **Instrument Description**

**BL12** 

**Ouasi-elastic/inelastic** neutron scattering

- High resolution Fermi chopper and high speed T0 chopper for high energy measurements
- Supermirror quide tubes and large-area detectors increase the number of neutrons detected
- Neutron Brillouin scattering measurement is realized by a detector with a minimum scattering angle of 0.6°

#### **Specifications**

Neutron energy :  $3 < E_i < 500$  meV Scattering: Horizontal:  $3^{\circ}$  ~ 62° (L<sub>2</sub> = 4 m),  $-31^{\circ}$   $\sim$   $-13^{\circ}$  ( $\vert \cdot \rangle = 4$  m).  $-5.1^{\circ}$   $\sim$   $-0.6^{\circ}$  (L<sub>2</sub> = 5.2 m) Vertical:  $\pm$  20 ° (L<sub>2</sub> = 4 m),  $\pm 4^{\circ}$  ( $\sqrt{ }$  = 5.2 m) Neutron intensity: (@sample position @1MW)  $1x10^5$  n/s/cm<sup>2</sup> ( $\Delta E/E = 2.5\%)$ Energy resolution:  $\Delta E/E_i \geq 2\%$  ( $\varpi E = 0$  meV)

Maximum sample size:  $4 \times 4$  cm<sup>2</sup>

#### **Sample Environments**

- Closed cycle refrigerator (4-300 K)
- •1 K refrigerator (1-300 K)
- $\bullet$ <sup>3</sup>He refrigerator (0.3-300 K)
- $\bullet$ Superconducting magnet (Max, 5 T, 2 K 300 K)
- Pressure cell (Cylinder type, Max 1.2 GPa)

#### **Instrument Configurations**

![](_page_25_Picture_16.jpeg)

![](_page_25_Picture_17.jpeg)

Small-angle area detectors

![](_page_25_Picture_19.jpeg)

#### **CONTACT**

![](_page_25_Picture_21.jpeg)

![](_page_26_Picture_1.jpeg)

![](_page_26_Picture_2.jpeg)

#### **Capabilities**

- Spin, atoms, and molecular dynamics in solid state
- Spin wave dispersion measurements from ferromagnetic powder samples
- . Phonon dispersion measurements of liquids

![](_page_26_Figure_7.jpeg)

#### **Applications**

- Magnetic excitations in metallic ferromagnets
- Pressure-induced quantum phase transitions and magnetic states
- $\bullet$  Magnetic excitations in multiferroic materials  $\frac{7}{2}$
- Magnetic excitations in one-dimensional quantum spin systems
- •Magnetic excitations in layered transition metal oxides
- $\bullet$  Magnetic excitations in skutterudite systems  $8$
- Magnetic excitations in metallic antiferromagnets
- Lattice vibrations in liquid and polycrystalline systems

#### Neutron Brillouin scattering study measurement with a detector system down to 0.6° of scattering angle

![](_page_26_Figure_18.jpeg)

S. Itoh et al., Nature Commun. 7, 11788 (2016). (© 2016 **SPringer Nature)** 

#### Novel excitations near quantum criticality in geometrically frustrated antiferromagnet<sup>1)</sup> CsFeCl<sub>3</sub>

![](_page_26_Figure_21.jpeg)

## **Cold-neutron disk-chopper spectrometer (AMATERAS)**

A low-energy dedicated inelastic/quasi-elastic scattering instrument designed to offer fine and tunable energy resolution and high neutron flux meeting the needs of a wide<br>range of research fields, such as magnetic systems, amorphous substances, and liquids

#### **Instrument Description**

**BL14** 

Ouasi-elastic/inelastic neutron scattering

- Dynamics measurements in the energy range from  $10^{-2}$  to  $10^{2}$  meV
- · High-efficiency beam transport, detectors with a large solid angle, and multi-Ei technique are available
- · Highly efficient measurement detects weak signals from dynamical motions of atoms and spins that could not be captured before

#### **Specifications**

- $\bullet$  Incident energy :  $1 < 80$  meV
- $\bullet$  Energy resolution :  $\Delta E/E_i > 1\%$  ( $E_i < 3$  meV)  $\Delta E/E_i > 2 - 3\%$  ( $E_i < 20$  meV)  $\Delta E/E_i$  > 4 - 5% ( $E_i$  < 80 meV)
- Detector coverage: 3.4° 116° (horizontal),  $-16^\circ - 23^\circ$  (vertical)
- Optimal sample size  $\varnothing$ 1 ×2 cm<sup>3</sup>

#### **Instrument Configurations**

![](_page_27_Picture_12.jpeg)

![](_page_27_Picture_13.jpeg)

#### **Sample Environments**

- Bottom-loading closed-cycle refrigerator (5 300 K)
- ●Top-loading closed-cycle refrigerator (7 500 K)
- $\bullet$ High-temperature stick  $(T < 680 \text{ K})$
- •Other equipment is also available. Some of equipment cannot be used in full-spec. Please inquire of the BL14 staff members about more detail

![](_page_27_Picture_19.jpeg)

![](_page_27_Picture_20.jpeg)

Fask disk-chopper

Vacuum scattering chamber and detectors

<b>CONTACT</b>		
	Seiko OHIRA-KAWAMURA seiko.kawamura@j-parc.jp	(AEA))
	Maiko KOFU maiko.kofu@j-parc.jp	$(\overline{(\text{AEA})})$

### **Cold-Neutron Disk-Chopper Spectrometer** (AMATERAS)

![](_page_28_Picture_1.jpeg)

![](_page_28_Picture_2.jpeg)

#### **Capabilities**

- Observation of lattice vibrations and magnetic excitations from 0.1 meV to several 10 meV on spatial scales on the order of Å
- . Investigation of diffusion and relaxation of atoms/molecules moving in the time range of 0.05 - 50 ps and spatial range of 2 - 20 Å, and their relationship to structure

![](_page_28_Figure_6.jpeg)

#### **Applications**

- Magnetic excitations and lattice vibrations in strongly correlated electron systems and quantum spin systems
- Vibration and diffusion phenomena in liquids and low-energy dynamics in amorphous materials
- Lattice dynamics of functional materials such as thermoelectric materials
- Molecular motion and flexibility in polymers and biomaterials

![](_page_28_Figure_12.jpeg)

For 9), see Glossary (p.62)

#### **Small and Wide Angle Neutron Scattering Instrument BL15 (TAIKAN)** Small angle neutron scattering

Structural information with a wide spatial range, from sub-nanometer to micron scales, is obtained for analyzing the structures and non-equilibrium phenomena of various materials such as metals, magnetic materials, superconductors, soft matter, biopolymers, and composites

#### **Instrument Description**

High-resolution and high-efficiency measurements over a wide range of spatial scales, both small-angle and wide-angle, enable hierarchical structural analysis. In-situ observation measurements using a variety of sample environment devices are possible.

#### **Specifications**

Range of wavelength: 0.08 - 0.78 nm (unpolarized) 0.25 - 0.78 nm (polarized) Range of  $q$ :  $7\times10^{-3}$  - 170 nm<sup>-1</sup> (unpolarized)  $7\times10^{-3}$  - 25 nm<sup>-1</sup> ( polarized )

#### **Instrument Configurations**

![](_page_29_Figure_7.jpeg)

![](_page_29_Picture_8.jpeg)

Sample changer

#### **Sample Environments**

Sample changer, 3 K/4 K refrigerator, 1 T magnet, 4 T cryo magnet, 10 T magnet, Rheometer, tensile tester, humidity controller, laser furnace, gas/vapor adsorption equipment, etc.

![](_page_29_Picture_12.jpeg)

1 T magnet and 4K refrigerator

![](_page_29_Picture_13.jpeg)

generator

Laser heating furnace (chamber section) and heating image

#### **CONTACT**

![](_page_29_Picture_111.jpeg)

### **Small and Wide Angle Neutron Scattering Instrument (TAIKAN)**

![](_page_30_Picture_1.jpeg)

![](_page_30_Picture_2.jpeg)

#### **Capabilities**

- Structural information from sub-nanometer to micron scale
- Structural analysis of nanomagnetic materials, biomaterials, and composites
- Analysis of non-equilibrium phenomena

![](_page_30_Figure_7.jpeg)

Measurement g-range of TAIKAN

H. Iwase, Oleoscience 16, 10 (2016) (© 2016 Japan oil chemists' society)

#### **Applications**

- Analysis of the hierarchical structure of gluten in flour products
- Heterogeneous structure analysis of phenol resin during gelation process
- Nanostructural Analysis of Tire Rubber Material (Hydrogen Nuclear Spin Polarization)
- Study of solid polymer materials for fuel cell electrodes
- Analysis of micro precipitates in steel materials
- Magnetic-structural analysis in nano-scale

![](_page_30_Figure_17.jpeg)

The magnetic structure is determined by measuring spin-flip and non-spin-flip scattering, because the spin state of the scattered neutrons depends on the direction of the magnetic moment of the material.

R. Takagi et al., Nat. Commun. 13, 1472 (2022). (©2022 SPringer Nature).

#### Structural analysis of sulfonated hydrocarbon films in humid environments

Study of proton conductivity trends ( $QP > MP > BP$ ) and structural connectivity of water clusters

![](_page_30_Figure_22.jpeg)

SANS profiles of (a) SPP-MP, (b) SPP-BP, and (c) SPP-QP films under 0 to 80% RH in D2O humidity at 80° C. Solid lines show the results of fitting with model functions.

![](_page_30_Picture_24.jpeg)

Morphology model of SPP-MP, SPP-BP, and SPP-OP membranes at 80° C and 80% RH

K. Shiino, et al., ACS Appl. Polym. Mater. 2020, 2 (12), 5558. (©2020 American Chemical Society).

## **Soft Interface Analyzer (SOFIA)**

**BL16** 

## Horizontal sample geometry Neutron Reflectometer suitable for soft condensed matters

#### **Instrument Description**

- •The high penetrability of neutrons and their ability to distinguish isotopes are used to investigate nanostructures at interfaces that cannot be observed by conventional methods.
- By injecting the beam downward, free surfaces such as gas/liquid and liquid/liquid interfaces as well as the solid interface can be investigated.

#### **Instrument Setup**

![](_page_31_Picture_7.jpeg)

©HAYASHI Yuki

#### **Specifications**

- Rapid measurements by using the high-intensity incident neutron beam
- $\bullet$ Wavelength range: 0.2 0.88 nm (single frame) 0.2-1.76 nm (double frame)
- $\bullet$  Incident angle: < 3.5 degrees
- $\bullet$  Sample size < 50mm<sup>w</sup> x 100mm<sup>D</sup> (typically 2 to 3 inch wafers)
- $\bullet$ low background:  $R > 10^{-7}$
- Simultaneous time-slice measurements at wide-Q range possible:  $\Delta t$ ~s to 10 min
- Estimated measurement time for all-Q region (at beam power 850kW, as of June 2024) ○Gas/solid: 30 min (Ø 50 mm) ○Liquid/solid: 30 min (Ø75 mm)  $\circ$ Gas/liquid: 10 min (40×40 mm<sup>2</sup>) OLiquid/liquid: 3 hrs (20×40 mm<sup>2</sup>)

![](_page_31_Picture_17.jpeg)

#### **Sample Environments**

- ●Temperature control: RT-250°C/0 100°C/  $RT - 300^{\circ}C$  (Rapid heating by laser irradiation)
- ·Solid/Liquid interface measurement cells (Remote injection available)
- Gas/Liquid interface measurement cells (Langmuir trough available)
- ·Liquid/Liquid interface measurement cells

![](_page_31_Picture_23.jpeg)

#### **CONTACT**

![](_page_31_Picture_127.jpeg)

![](_page_32_Picture_1.jpeg)

![](_page_32_Picture_2.jpeg)

#### **Capabilities**

- Evaluation of film thickness and roughness at various interfaces such as solid/solid, solid/liquid and gas/liquid.
- The average structure can be observed over a scale of a few nanometers to submicrometers in depth.

![](_page_32_Figure_6.jpeg)

was investigated. Soft Matter, 18, 545 (2022). ©2022 Royal Society of Chemistry

![](_page_32_Figure_8.jpeg)

The effect of fhe friction-reduction of automotive engine oil was elucidated.

Tribol. Int., 167 (2022) 107365. ©2022 Elsevier.

#### **Applications**

- Swelling behavior of functional polymers in solvents.
- Observation of interfaces of organic devices with stacked structures.
- Structural changes in polymer thin films in annealing processes.
- Electrode interface of lithium-ion batteries.

![](_page_32_Picture_16.jpeg)

Phase separation behavior of organic solar cell thin films by complementary use of neutron and X-ray. Soft Matter 7 (2011) 9276-9282. ©2010 Royal Society of Chemistry

![](_page_32_Figure_18.jpeg)

Time-lapse measurements of the process of network structure formation on swelling of proton conducting membranes.

Langmuir 34 (2018) 15483-15489. ©2018 American Chemical Society

#### **Battery** Carbon electrode Electrolyte charging Si  $\overline{\mathbf{3}}$ 100 Height from Substrate (nm

Operando measurement of film formation process on anode surface of Li-ion batteries on charging and discharging.

ACS Appl. Mater. Interfaces 8 (2016) 9540-9544. ©2016 American Chemical Society

![](_page_32_Picture_24.jpeg)

Differences in luminescence performance depending on the deposition process of OLEDs are explained by differences in the interface structure. Adv. Mater. Interfaces 1 (2014) 1400097. ©2014 Wiley-VCH GmbH, Weinheim

## Polarized neutrons reveal the magnetic structure in a thin film.

#### **Instrument Description**

**BL17** 

**Neutron reflectometry** 

- •The nanometric structure of the surface and interfaces in a thin film is examined by the reflection profile of a neutron beam incident at a small angle less than a few degrees.
- •The direction and spatial distribution of the magnetic moment is analyzed by the reflection profiles dependent on the spin direction of the polarized neutrons.

#### **Specifications**

#### 1.Specification

- $\bullet$  Observed g range 0.05 17.9 nm<sup>-1</sup> (unpolarized)  $0.1 - 8.19$  nm<sup>-1</sup> (polarized)
- Polarization ratio up to 97 %
- $\bullet$ <sup>3</sup>He point detector
- ·MWPC-type two dimensional detector (detection area of 128 x 128 mm<sup>2</sup>)

#### 2. Measurement conditions

- •Magnetic field up to 7 T
- ●Temperature from 4 to 500 K
- •Measurement in vacuum, air, and liquids

#### **Sample Environments**

- Electro-magnets (1, 4, and 7 T)
- ●4K-refrigerator
- Heater stage up to 500 K
- Electro-chemical cell
- Liquid immersion cell
- ●Temperature and humidity control chamber (Temperature 5 - 85℃, Humidity: 0 < 85%RH)

![](_page_33_Picture_22.jpeg)

#### **Instrument Configurations**

![](_page_33_Picture_24.jpeg)

![](_page_34_Picture_1.jpeg)

![](_page_34_Picture_2.jpeg)

#### **Capabilities**

- Spatial distribution of chemicals normal to the interfaces of materials at length scales from nanometer to micrometer.
- Depth distribution of the magnetic moment in a magnetic thin film

#### **Applications**

- Analysis of moisture absorption process in polymer films
- Elucidation of proton-conduction pathway in polymer electrolytes
- Magnetic structure analysis for topological insulators and interfaces in ferromagnetic materials
- Structure analysis of electronic double layers at the interface of electrodes

![](_page_34_Figure_11.jpeg)

![](_page_34_Figure_12.jpeg)

thin film in humid atmosphere

![](_page_34_Figure_14.jpeg)

Magnetic field dependence of the depth distribution of the magnetic moment in a multi-layer thin film of  $Cu/Ni/Fe/Mn/Cu$ 

#### **Extreme Environment Single-Crystal Diffractometer BL18** (SENJU) **Neutron diffraction**

## Precise and reliable determination of crystal/magnetic structure

#### **Instrument Description**

- Determination of hydrogen positions and complicated magnetic structures by highly efficient diffraction measurements
- Diffraction measurements under multiple extreme sample environments such as low temperature and magnetic field.

#### **Specifications**

- •Maximum cell length: 50 Å
- $\bullet$  Typical sample size: 0.5  $\times$  0.5  $\times$  0.5 mm<sup>3</sup> (crystal structure)  $1 \times 1 \times 1$  mm<sup>3</sup> (magnetic structure)

incident neutro

acuum chamb

● Detector coverage: -167° to -13°  $+58^{\circ}$  to  $+167^{\circ}$ 

detector

collimator

#### **Sample Environments**

- 1-axis/2-axes goniometers (Room temperature)
- $\bullet$  2-axes cryostat ( $>$  4 K)
- $\bullet$  2K cryostat (> 2 K, > 100 mK with dilution unit)
- $\bullet$ <sup>3</sup>He cryostat ( $>$  300 mK)
- $\bullet$  Magnet (< 7 T, > 2 K, dilution unit is available)
- $\bullet$  Furnace (< 1500 K)

#### **Instrument Configurations**

![](_page_35_Picture_17.jpeg)

#### Outer view of SENJU

![](_page_35_Picture_19.jpeg)

Typical diffraction image obtained by SENJU

![](_page_35_Picture_115.jpeg)

### **Extreme Environment Single-Crystal** Diffractometer (SENJU)

![](_page_36_Picture_1.jpeg)

![](_page_36_Picture_2.jpeg)

#### **Capabilities**

- Crystal/magnetic structure determination of a small single-crystal
- Elucidation of structure-function relations under extreme sample environments

![](_page_36_Figure_6.jpeg)

![](_page_36_Figure_7.jpeg)

## Double proton transfer coupled spin<br>transition system

Successfully determined accurate positions of hydrogen atoms in hydrogen-bonds of a Fe complex showed the complex is the first temperature-dependent double proton transfer coupled spin transition compound.

![](_page_36_Figure_10.jpeg)

The high spin state phase is the double proton transferred state

T. Nakanishi et al., Journal of the American Chemical Society 145 19177-19181 (2023).

(©2023 American Chemical Society)

#### **Applications**

## Magnetic structure of a material showing<br>topological Hall effect

Theoretically, the Hall effect manifests itself under a special arrangement of magnetic moments (topological Hall effect). However, no experimental evidence had been presented.

![](_page_36_Picture_17.jpeg)

Magnetic structure of  $CoM_3S_6$  (M = Nb, Ta) was elucidated to clarify the origin of the Hall effect observed in those compounds by using SENJU.

![](_page_36_Figure_19.jpeg)

The so-called "all-in-all-out" spin arrangement generates the fictitious magnetic field. The Hall effect in the CoM3S6 is induced by the fictitious field and is the "topological Hall effect".

H. Takagi et al., Nature Physics 19 961-968 (2023).

(©2023 American Chemical Society)

## **Engineering Materials Diffractometer (TAKUMI)**

**Neutron diffraction** 

**BL19** 

## Solving various problems related to stresses and microstructures of various engineering materials

#### **Instrument Description**

- Simultaneous meas, of scat, vectors (strains) in two orthogonal dir. or more
- Short time meas. with high accuracy
- Meas. the distribution of lattice strain, etc. by narrowing the gauge and scanning the sample,
- · In situ meas. (abundant sample environments: load, temp.)

#### **Specifications**

- $\bullet$  Resolution  $\Lambda d/d \cdot 0.17 0.4\%$
- d-range: 0.05 to 0.29 nm (standard), 0.05 - 0.50 nm (wide range)
- Sample stage:  $700 \times 700$  mm<sup>2</sup>, load capacity < 1 t
- Radial collimator: 1 mm, 2 mm, 5 mm
- Data reduction during or after meas.

#### **Sample Environments**

- ●Standard loading mach. (50 kN, ten, comp)
- ●Induction furnace for high temp loading (1373 K)
- Cryogenic system for loading mach. (15 K 280 K)
- ●Hydraulic fatique mach. (60 kN, <30 Hz)
- High temp loading mach. (30 kN, 1273 K) ●Fulerian Cradle
- Electric field application device, etc.
- Options: dilatation meas., digital image corelation

![](_page_37_Figure_22.jpeg)

### **Engineering Materials Diffractometer** (TAKUMI)

![](_page_38_Picture_1.jpeg)

![](_page_38_Picture_2.jpeg)

#### **Capabilities**

• Lattice strain (stress), crystal defects (dislocations, crystallite size, etc.), texture, phase fraction, phase transformation

![](_page_38_Figure_5.jpeg)

![](_page_38_Figure_6.jpeg)

#### **Applications**

- Deformation mechanism, function expression mechanism by in situ analysis during deformation of metallic materials (RT, low temp, high temp)
- Microstructure control process by in situ analysis during thermo-mech process
- Measurement of residual strain and stress distribution of mechanical parts
- Evaluations of lattice strain and microstructures of non-metallic materials such as rocks, concrete, and ceramics

Cooling

Compression

Austenitization

Heating

![](_page_38_Figure_12.jpeg)

sos

 $+10C/$ 

 $950C$ 

 $-30C/s$ 

 $10s$ holding

Applying compression promotes

the  $v \rightarrow a$  transformation

straining

22000 24000  $rac{1}{100}$  $28000$ 

A Shibata et al., Scripta Materialia 165 (2019) 44-49 (@2019 Elsevier

![](_page_38_Figure_13.jpeg)

![](_page_38_Figure_14.jpeg)

## Ibaraki Material Design Diffractometer(iMATERIA)

**Neutron diffraction** 

**BL20** 

General-purpose neutron scattering instrument for quick and high-resolution measurements of diffraction and small-angle scattering in the wide d (lattice spacing) region

#### **Instrument Description**

· High-resolution powder diffraction Crystal structure analysis of powder materials Continuous rapid measurement by automatic sample exchange system

· Wide-angle detector arrangement Multiscale structural analysis Crystal orientation (texture) analysis by subdivided banks Local structure (pair distribution function) analysis by total scattering measurements

• Small-angle scattering measurements Nanostructure analysis of multicomponent materials by dynamic nuclear spin polarization

#### **Specifications**

- Measurement range:  $0.07 < d < 3,000$  Å
- Resolution:  $\Delta d/d \sim 0.16\%$  (High Resolution Bank)
- ●Typical measurement time: Powder diffraction of cathode materials for LIB:  $5 \sim 10$  min at 900 kW

Texture measurement of steel material: 4 min at 900 kW

Small-angle scattering of polymer material: 3 min at 500 kW

(depending on the composition of the sample, the purpose of measurement, etc.)

#### **Sample Environments**

- Sample exchange mechanism (RT, vacuum)
- ●Vacuum high temperature furnace (RT 1173 K)
- ●Cryo-furnace (3 K RT, RT 673 K)
- Rapid heating and cooling device (RT 1273 K,  $+10$  K/s, -20 K/s)
- High temperature universal deformation apparatus (RT - 1273 K, maximum load: 50 kN)
- Sample exchanger for small-angle scattering
- ●Superconducting magnet for nuclear spin polarization (1.2 K, 7 T)

![](_page_39_Picture_22.jpeg)

## Ibaraki Material Design Diffractometer (iMATERIA)

![](_page_40_Picture_1.jpeg)

![](_page_40_Picture_2.jpeg)

#### **Capabilities**

- Crystal structure
- local structure of crystalline and amorphous
- Nanostructure and microstructure
- Crystal orientation (texture)

![](_page_40_Figure_8.jpeg)

#### lonic conduction paths in new solid electrolytes for all-solid-state lithium-ion batteries

![](_page_40_Figure_10.jpeg)

#### In-operando neutron diffraction measurement during charging and discharging of laminate cell type lithium-ion battery.

![](_page_40_Figure_12.jpeg)

In situ neutron diffraction measurement under an environment simulating the microstructure control process of steel materials Measurement of phase transformation behavior using rapid heating and cooling device

 $-40$ 

 $\frac{1}{6}30$ 

 $rac{8}{6}$  20

![](_page_40_Figure_14.jpeg)

Due to the bainite transformation that occurs during constant temperature holding at 673 K, austenite with a low carbon concentration is gradually consumed, and austenite with a high carbon concentration appears.

![](_page_40_Figure_16.jpeg)

473  $210 -$ V<sub>2</sub> flow carbon V<sub>22</sub> (high carbon 4000 4500 5000 3500 By treating austenite with different carbon concentrations as separate

phases, it is possible to quantitatively determine the abundance ratio of both.

#### Texture changes using a high temperature universal deformation apparatus

Measurement of changes in (011) and (111) orientation due to tensile deformation at room temperature (a) and high temperature (b)

Observation of texture changes corresponding to stress oscillations observed during high-temperature tensile deformation

#### **Applications**

- Crystal structure of solid electrolyte for all-solid-state lithium-ion batteries
- Crystal structure changes of electrode materials for lithium-ion batteries by in-operando charging and discharging measurement
- Atomic arrangements of photocatalyst coating liquid
- Crystal structure of platinum alternative catalyst for fuel cells
- Structure of high-performance rubber materials with crosslinked network
- Collapse process of detergent foam with surfactants
- **•Texture of steel materials**

1073

 $873$ 

 $\frac{8}{673}$ 

- Residual austenite components in steel materials
- Dynamic microstructural changes of steel materials in heat treatment process

#### In situ small-angle neutron scattering measurements of the collapse process of detergent foam

Utilizes the characteristics of time-of-flight measurement using pulsed<br>neutrons to instantaneously measure neutron scattering in the wide-q range

#### Development of new surfactants to create detergents with appropriate foam generation and removal properties

Analysis of foam structure and collapse process with different surfactants

There are surfactant micelles in the foam similar to those in the solution

- change significantly • Observing the drainage process of
- water in foam

![](_page_40_Figure_39.jpeg)

Foaming the surfactant solution with an air pump Then, the bubbles are irradiated with a neutron beam and the collanse process is measured using time-resolved small-angle scattering method.

![](_page_40_Figure_41.jpeg)

## **High Intensity Total Diffractometer (NOVA)**

**Neutron diffraction** 

**BL21** 

## Neutron total diffractometer for structural analysis from crystals to liquids and amorphous materials.

#### **Instrument Description**

- Neutron total scattering and powder diffraction measurements using the world's highest intensity
- Measurements over a wide momentum transition space (high real space resolution)

#### **Specifications**

- Q range: 0.03 100 Å<sup>-1</sup> (d range: 0.06 200Å)
- $\bullet$  O resolution:  $\Delta$ O/O ~ 0.35 %
- Scattering angle  $2\theta$ : 0.7 170 $^{\circ}$
- ●Standard sample vol.: Ø6 mm×20 mmh  $(0.6 \text{ cc approx.})$

#### **Sample Environments**

● Auto sample changer (SC)

No. of sample: 40 (room temperature)

- Auto sample changer with closed cycle refrigerator (TSC) No. of sample: 18, Temp.: 20 - 700 K
- ●Top-Loading cryo-furnace (TLC) Temp.: 5 - 700 K
- Fermi chopper for Inelastic scattering exp. Reso.: 5 - 20%
- •2K cryostat (MLF common SE) Temp.: 1.8 - 300 K
- ●<sup>3</sup>He cryostat (MLF common SE) Temp.: 0.3 - 300 K

![](_page_41_Figure_19.jpeg)

**Instrument Configurations** 

#### **CONTACT**

![](_page_41_Picture_21.jpeg)

## **High Intensity Total Diffractometer** (NOVA)

![](_page_42_Picture_1.jpeg)

![](_page_42_Picture_2.jpeg)

#### **Capabilities**

- Atomic positions, such as hydrogen, and magnetic structure, and changes in these
- Structural changes from the nearest neighbor atom distance to about nm
- Structural analysis of fluctuation systems such as amorphous and liquids

## **Examples of high-speed measurements DOLL**  $QIA$

(Upper left) 1sec measurement of  $SiO<sub>2</sub>$  glass (bottom left) 40msec measurement profile of Si powder (Right) Deuterium absorption process of Pd (10 sec)

#### **Applications**

- Crystal and local structure analysis using small amounts of sample
- Short-time observation of structural changes by in situ measurements
- Observation of structural changes by in situ measurements at low/high temperatures under (gas) pressure
- Local structure analysis of solids · liquids using isotopic substitution

![](_page_42_Figure_14.jpeg)

![](_page_42_Figure_15.jpeg)

42

## **ENERGY RESOLVED NEUTRON IMAGING SYSTEM - RADEN-**

Neutron imaging

**BL22** 

Visualization of internal sample shape, crystallographic, elemental, and magnetic information by analyzing the energy dependence of neutron transmission spectra with position resolution

#### **Instrument Description**

- Energy-resolved neutron imaging Bragg edge imaging Resonance absorption imaging Polarized neutron imaging Phase contrast/dark field imaging
- •Neutron radiography, tomography

#### **Specifications**

- Wavelength range:  $\lambda$  < 8.8 Å (L=18 m, 25 Hz)
- Wavelength resolution  $(\Delta \lambda / \lambda)$ : 0.2%
- $\bullet$  Neutron flux @1 MW:  $1.1 \times 10^8$  n/s/cm ( $F < 1$  MeV,  $I/D = 180$ )
- $\bullet$  Beam size: < 300 $\times$ 300 mm<sup>2</sup>
- Spatial resolution: 10 300 µm
- Collimator ratio (L/D): 180 7500

#### **Sample Environments**

#### ●Filter (Pb, Bi, BK7)

- •Sample stage
- (Large: capacity 1.0 t. Middle: capacity 650 kg)
- · Heater (resistive heating, Infra red lamp)
- •ToF 3D polarimetry apparatus
- •Neutron Talbot-Lau interferometer
- ·Hydrogen gas supply/dilution unit

![](_page_43_Figure_21.jpeg)

T. Shinohara et al., Rev. Sci. Instrum. 91, 043302 (2020). (@2020 AIP Publishing LLC)

#### **CONTACT** Takenao SHINOHARA takenao.shinohara@j-parc.jp Tetsuya KAI tetsuya.kai@j-parc.jp Hirotoshi HAYASHIDA *RICROSS* h\_hayashida@cross.or.jp

## **Energy Resolved Neutron Imaging System** (RADEN)

![](_page_44_Picture_1.jpeg)

![](_page_44_Picture_2.jpeg)

#### **Capabilities**

- Conventional radiography/tomography Non-destructive observation, defect inspection, liquid flow
- Bragg edge imaging Crystallographic information, strain distribution
- Polarimetric imaging Magnetic field information

• Resonance absorption imaging Elemental information, temperature distribution

#### Neutron CT images of a Japanese sword

![](_page_44_Picture_9.jpeg)

Y. Matsumoto, et al, Materials Research Proceedings 15 (2020) 221-226. (© 2020 Materials Research Forum) Neutron CT images of automobile parts

![](_page_44_Picture_11.jpeg)

### **Applications**

- •Neutron radiography/tomography: Observation of energy-related devices (Li ion batteries, fuel cells, water electrolysis, etc.), grease in mechanical bearing, concrete, cultural heritage/archeology (Japanese swords, meteorite, etc.), nuclear engineering.
- •Bragg edge imaging: steels, metallic alloys, single crystals, cultural heritage/archeology (Japanese swords, meteorite, etc.)
- Resonance absorption: Li ion batteries, scintillator materials, nuclear engineering
- Polarimetric imaging: electric motors, electric transes, magnetic materials (electric steels, ferritic magnets, etc.)

#### 3D distribution of magnetic field inside a solenoid analyzed by neutron polarimetric tomography

![](_page_44_Figure_18.jpeg)

M. Sales et al., J. Phys. D: Appl. Phys. 52, 205001 (2019) (© 2019 IOP Publishing).

#### Spatial distribution of crystallographic information ofinduction heated gear obtained by Bragg edge imaging

![](_page_44_Picture_21.jpeg)

![](_page_44_Figure_22.jpeg)

![](_page_44_Figure_23.jpeg)

![](_page_44_Figure_24.jpeg)

Neutron Radiograph

Plane Spacing

**Projected Density** 

Crystallite size

## **Polarized Neutron Spectrometer (POLANO)**

POLANO is a direct geometry chopper spectrometer with polarization options for the study of dynamics in materials such as magnets, superconductors, functional materials, and even liquids, leading to ability to separately observe various degrees of freedom in materials.

#### **Instrument Description**

- Reasonable resolution viewing a decoupled moderator
- Wide accessible momentum space
- Relatively higher energy polarization analysis
- Application example

**BL23** 

**Ouasi-elastic/inelastic** neutron scattering

- Magnetism Strongly correlated electron systems Quantum fluids
- Vibration of atoms or molecules

#### **Specifications**

- •Moderator: Decoupled moderator
- $\bullet$  Detectors: (hor.) -25° 125°, (ver.)  $\pm$  8°
- $\bullet$  Incident energy: 10 1000 meV
- $\bullet$  Energy resolution:  $\Delta E/E_i \geq 2 4\%$  @E=0
- •Momentum resolution ( $\Delta Q/k_i$ ): 1 2%
- Beam cross section:  $20x20$  mm<sup>2</sup> (optimum)  $50x50$  mm<sup>2</sup>
- Polarization energy: 10 meV  $\lt E_i \lt 40$  meV

#### Sample Environments

- Top-loading GM cryostat  $(T = 4 - 300 \text{ K})$
- Bottom-loading GM cryostat  $(T = 7 - 300 \text{ K})$
- $\bullet$ <sup>3</sup>He sorption refrigerator  $(T = 0.3 \text{ K})$
- (MLF) 7 T-magnet with ORC
- •10mm, 20mm, 30mm, 40mm, 50mm collimators
- $\bullet$  SEOP polarizer and 5.50. bending super mirror analyzer (20° of scattering angles)

![](_page_45_Picture_24.jpeg)

**Instrument Configurations** 

## **CONTACT**

Tetsuya Rex YOKOO tetsuya.yokoo@kek.jp

Shinichi ITOH shinichi.itoh@kek.jp

![](_page_45_Picture_28.jpeg)

### **Polarized Neutron Spectrometer** (POLANO)

![](_page_46_Picture_1.jpeg)

![](_page_46_Picture_2.jpeg)

#### **Capabilities**

- . POLANO is optimized for the measurement of spectroscopies.
- ●Variety of so-called degrees of freedom in materials, such as atoms, molecules, spins, charges, and orbitals can be detected as a dynamic properties by the POLANO.
- Cross-correlations of the degrees of freedom can be separately observed including coherent/incoherent cross section.

#### **Applications**

- Transverse/longitudinal modes separation in quantum spin systems
- Right/left-handedness in chiral materials
- Lattice vibration and spin waves in high temperature superconductivity
- Elementary excitations and orbital waves in orbital-ordered electronic states
- $\bullet$  Spin-lattice interactions in multiferroic materials  $\frac{7}{2}$
- Multipole ordering and hierarchical structures in f-electron systems

![](_page_46_Figure_14.jpeg)

## Muon experiment instrument for materials and life science (Muon D1) rotation/relaxation/resonance

Detecting local magnetic fields in materials by using muon. Muons with a high momentum are available, allowing high-pressure experiments because of the high transmission<br>capability. Negative muon can observe local magnetic fields near nuclei in materials.

#### **Instrument Description**

• µSR experimental using pulsed positive or negative muons with variable-momentum.

**Muon spin** 

• Measurements under various complex conditions, such as high pressure and low temperatures, to cover a wide variety of phenomena.

#### **Specifications**

- $\bullet$  Beam property:  $\mu^{+}/\mu^{-}$  27 90 MeV/c single-pulse (surface muon) or double-pulse Temp: 0.05 - 1000 K  $\bullet$ Sample
- Environment Mag. Field: 0 0.4 T(along the beam direction)  $0 - 0.01$  T (perpendicular to the

beam direction) Pulsed light-irradiation by a flash lamp

#### **Sample Environments**

● Oxford top-loading dilution refrigerator  $(T = 0.05 - 30 K)$ 

 $\bullet$  Oxford microstat  $(T = 4 - 500 \text{ K})$ 

 $\bullet$ high-T furnace ( $T = 300 - 1073$  K)

#### **Instrument Configurations**

![](_page_47_Picture_14.jpeg)

![](_page_47_Picture_15.jpeg)

#### High-temperature Furnace

**Dilution Refrigerator** 

![](_page_47_Picture_18.jpeg)

### **Muon Apparatus for Materials and Life Science Experiments**

![](_page_48_Picture_1.jpeg)

![](_page_48_Picture_2.jpeg)

#### **Capabilities**

- · Magnetic and electronic states in solids and liquids by observations of internal magnetic field.
- Electronic state of dilute hydrogen in matter.
- Electron, hydrogen and ion dynamics.

![](_page_48_Figure_7.jpeg)

#### **Applications**

- Cooper pairing symmetry in superconductors.
- Flux-line lattice in superconductors.
- Electronic structure of hydrogen in semiconductors.
- Magnetism near the quantum critical point.
- · Ion diffusion in solids.
- $\bullet$  lon dynamics by negative  $\mu$ SR.

![](_page_48_Figure_15.jpeg)

#### Ferromagnetic fluctuation in the high-Tc cuprate  $[(BiPb)_{2}Sr_{2}CuO_{6+d}]$

![](_page_48_Figure_17.jpeg)

Phys. Rev. Lett. 121, 057002 (2018). (© 2018 Amerian **Physical Society)** 

## **Muon Experimental Area for Basic Science (Muon D2)**

Experimental area for research on properties of muons as a particle, structure and reaction of muonic atoms, and non-destructive elemental analysis on archeological or rare specimens using the world's strongest pulsed muon

#### **Instrument Description**

• Momentum-changeable positive and negative muons with high intensity are available

**Fundamental muon physics** Negative muon X-ray elemental analysis

- Users can bring their equipment for experiments
- Non-destructive elemental analysis with muonic X-rays can be performed

#### **Specifications**

- $\bullet$ Type Decay positive · negative muon Surface positive muon
- Momentum 5 50 MeV/c (changeable)
- $\bullet$  Intensity  $5.0 \times 10^6 \mu^{\pm}$ /s@50 MeV/c at 1 MW
- Double bunch 25 Hz  $\bullet$  Structure Bunch Width 100 ns  $600 \text{ ns}$ Interval (Single bunch available using kicker)

#### **Sample Environments**

- Open space
- $\bullet$  Under vaccuum
- $\bullet$  He, N<sub>2</sub>, or other inert gasses using a kapton window

#### **Instrument Configurations**

![](_page_49_Picture_16.jpeg)

D<sub>2</sub> Experimental Area

Experimental chamber equipped with Ge detectors featuring a gold coin

## **CONTACT** Izumi UMFGAKI umegaki@post.kek.jp Patrick STRASSER patrick.strasser@kek.jp

![](_page_50_Picture_1.jpeg)

## D<sub>2</sub>

#### **Capabilities**

- Elemental Analysis
- Soft error in semiconductor
- Physical properties of muon

![](_page_50_Figure_7.jpeg)

Schematic representation of a muonic atom

#### **Applications**

- Reproduction of soft error in semiconductor due to cosmic muon irradiation using muon beam
- Non-destructive elemental analysis (Archeologyical specimens meteorites batteries, etc.)
- Observation of muon rare decay phenomena

![](_page_50_Figure_13.jpeg)

![](_page_50_Figure_14.jpeg)

## High-intensity muon beam for general use (Muon H1)

Various experimental studies such as fundamental physics are performed with high precision and sensitivity using high-intensity pulsed muon beams

#### **Instrument Description**

- Positive and negative pulsed muon beams with high intensity and momentum tunability are available.
- User equipment can be brought in for experiments requiring high statistics with long beamtime
- Open space, 6.2m(D) $\times$ 5.7m(W) $\times$ 3.5m(H)
- Surrounded by a magnetic shielding

**Fundamental muon physics** Negative muon X-ray elemental analysis

> • Area temperature is controlled by packaged air conditioning.

#### **Specifications**

- Type of beam: Surface muon or cloud positive/negative muon
- Momentum:  $10 \sim 120$  MeV/c
- Intensity: 10<sup>8</sup>  $\mu$ <sup>+</sup>/s@28 MeV/c,  $4 \times 10^7$   $\mu$ <sup>-</sup>/s@50 MeV/c
- Beam structure: Double bunches, 25 Hz

Bunch width 100 ns, bunch separation 600 ns

● Equipment: Users can install their own equipment.

**Instrument Configurations** 

#### **Sample Environments**

● Open space. Users can install their own equipment.

![](_page_51_Picture_17.jpeg)

![](_page_51_Picture_18.jpeg)

#### Beam port

### **CONTACT** Takayuki YAMAZAKI takayuki@post.kek.jp Naritoshi KAWAMURA nari.kawamura@kek.jp

## High-intensity muon beam for general use

![](_page_52_Picture_1.jpeg)

![](_page_52_Picture_2.jpeg)

#### **Capabilities**

- Fundamental physical constants of muon (w/ user-prepared equipment)
- New physics beyond the Standard Model of particle physics (w/ user-prepared equipment)

#### **Applications**

- Precise measurement of the muonium hyperfine structure
- Search for muon-to-electron conversion

![](_page_52_Figure_9.jpeg)

![](_page_52_Figure_10.jpeg)

![](_page_52_Figure_11.jpeg)

![](_page_52_Picture_12.jpeg)

## General purpose µSR spectrometer (ARTEMIS)

**Muon spin** rotation/relaxation/resonance

> Muon spin rotation/relaxation/resonance ( $\mu$ SR) measurements for electronic properties of materials and/or electronic state of hydrogen introduced to the material.

#### **Instrument Description**

- Positive muons  $(\mu^+)$  with 27-MeV/c momentum (Surface muon)
- Single pulse- or double pulse-operation can be chosen by using the beam kicker.
- Spectrometer: ARTEMIS (1280 ch detectors)

#### **Specifications**

- $\bullet$   $\mu$  SR signal count rates with a flypast chamber @800kW
	- $\triangle$ Sample with  $\oslash$  25 mm : 180 M events/hour (Double pulse) 90 M events/hour (Single pulse)
	- $\triangle$ Sample with  $\oslash$  10 mm : 60 M events/hour (Double pulse)
		- 30 M events/hour (Single pulse)
- Longitudinal magnetic field: max 0.4 T
- Transverse magnetic field: max 12.5 mT (40 mT with  $\mu$  TC magnet)

#### **Sample Environments**

- $\bullet$  Oxford <sup>3</sup>He cryostat ( $T = 0.3 30$  K)
- $\bullet$  Oxford Microstat (T = 4 420 K)
- Furnace  $(T = 300 1073 \text{ K})$

#### **Instrument Configurations**

![](_page_53_Picture_19.jpeg)

![](_page_53_Picture_20.jpeg)

<sup>3</sup>He cryostat

#### **CONTACT**

![](_page_53_Picture_23.jpeg)

## General purpose µSR spectrometer (ARTEMIS)

![](_page_54_Picture_1.jpeg)

![](_page_54_Picture_2.jpeg)

#### **Capabilities**

- Magnetic and electronic states in solids observed by local magnetic field
- Electronic states of dilute hydrogen in materials
- · Dynamics of electrons, hydrogen, and ions

#### **Applications**

- Synthesis and reaction processes of magnetic materials
- . Ion diffusion behavior in solids
- Analysis of reaction mechanisms in operando measurements

![](_page_54_Figure_11.jpeg)

#### storage materials (MgH<sub>2</sub>) w/ milling w/o milling PRESSURE (hPa) PRESSURE (hPa)  $\Delta$  ( $\mu$ s'<sup>1</sup>)  $\binom{\text{us}^2}{\text{0}}$ 500 600 700 800  $300$ 400 500 600 700 800 300 400 TEMPERATURE (K) TEMPERATURE (K)

 $\triangle$  is nuclear field distribution width determined by uSR

Sustainable Energy & Fuels 3, 956 (2019). (© 2019 Royal Society of Chemistry)

#### **Electronic state of hydrogen** in semiconductors (InGaZnO<sub>4</sub>) 0.30  $Ga<sup>3</sup>Zn<sup>1</sup>O<sup>6</sup>$  $- Ga<sup>3</sup>O$  $(a)$  $0.25$  $50.20$  $3a^3c$  $Ga<sup>3</sup>Zn<sup>1</sup>O<sup>6</sup>$  $\overline{a}$  $0.15$  $0.10$  $Ga<sup>2</sup>7n<sup>2</sup>O<sup>4</sup>$  $Ga<sup>1</sup>Zn<sup>3</sup>O$ 0.05  $0.00$  $-4.0$  $-3.5$  $-3.0$  $-2.5$  $-2.0 -1.5$  $-1.0$  $-4.5$ **Formation Energy**  $E_f(eV)$  $(b)$   $Zn<sup>4</sup>O<sup>4</sup>$  $(c)$  Ga<sup>3</sup>Zn<sup>1</sup>O<sup>6</sup>

![](_page_54_Figure_16.jpeg)

Appl. Phys. Lett. 115, 122104 (2019). (© 2019 AIP Publishing)

![](_page_54_Figure_18.jpeg)

• Local spin correlations in materials

• Flux-line lattice in superconductors

• Cooper pair symmetry in superconductors

● Electronic state of hydrogen in semiconductors

## **Muonium Laser Physics Apparatus (Muon S2)**

Precise measurement of muonium energy levels using lasers, or ultra-slow muon production by three-photon ionization of muonium

#### **Instrument Description**

**Fundamental muon physics** Negative muon X-ray elemental analysis

- Pulsed surface muon (positive) beam available
- Possibility to bring in user equipment and conduct experiments that require high statistics over long beamtimes.
- •Completely enclosed area available for laser experiments

#### **Specifications**

•Beam performance 27 MeV/c positive muon both single and double pulse modes available (requires coordination with S1 area)

• Beam intensity

 $\sim$  3×10<sup>6</sup>  $\mu$ <sup>+</sup>/s (single pulse) at 760 kW

#### **Sample Environments**

#### •Open space

 $\bullet$ Area size Length:  $5.0 \text{ m}$ Width:  $3.8 \text{ m}$ Height: 2.8 m

#### $\bullet$ Utilities

Cooling water Flectrical power Compressed air & exhaust

#### **Instrument Configurations**

![](_page_55_Picture_16.jpeg)

![](_page_55_Figure_17.jpeg)

## **CONTACT**

![](_page_55_Picture_19.jpeg)

![](_page_56_Picture_1.jpeg)

![](_page_56_Picture_2.jpeg)

#### **Capabilities**

- Generate muonium (an exotic atom consisting of a positive muon and an electron) from slow muons pulled out from an accelerator.
- Irradiate with ionizing lasers (resonant ionization dissociation method) to strip off electrons.
- Generate ultra-slow muons with thermal energy (approximately 0.03 eV).

### **Applications**

•Muonium precision laser spectroscopy experiment

Precise measurement of the 1s-2s energy

• Acceleration of ultra-slow muons using radio-frequency quadrupole linac (RFQ)

![](_page_56_Picture_10.jpeg)

![](_page_56_Figure_11.jpeg)

S. Uetake et al., High Energy News 39 (4), 170 (2021) (in Japanese) (©2021 Japan Association of High Energy Physicists)

Acceleration of ultra-slow muons by radio-frequency quadrupole linac (RFQ)

![](_page_56_Picture_15.jpeg)

![](_page_56_Figure_16.jpeg)

Preliminary experimental results in S2 area (2024)

![](_page_56_Figure_18.jpeg)

## Ultra-Slow Muon Microscope (Muon U1A)

A high time-resolution, depth-resolved probe that elucidates surface and interface magnetism with extremely small compasses

#### **Instrument Description**

J1A

**Muon spin** rotation/relaxation/resonance

- · Ultra-slow muons generated via laser ionization of thermal muonium
- Low- and tunable implantation energies 1/1000 of that of surface muons
- . High time resolution comparable to that of continuous beams
- Small beam size that is 1/10 of a typical surface muon

#### **Specifications**

- $\bullet$  Low-energy positive muons  $(\mu^+)$
- Energy: from 0.5 keV to 30 keV
- Energy spread: approx. 50 eV (FWHM)
- Time resolution: 1 ns (Std. dev.)
- Beam size: 1.6 mm (Std. Dev.)
- •512 channels positron detector

#### **Sample Environments**

- Helium-flow cryostat (4.2 K 300 K)
- Static magnetic fields up to 0.14 T
- Current introduction
- Voltage application
- A load-lock chamber for sample transport
- $\bullet$  RHFFD

#### **Instrument Configurations**

![](_page_57_Picture_22.jpeg)

The high-voltage platform

The muon spin spectrometer

## **CONTACT** Sohtaro KANDA kanda@post.kek.jp Yutaka IKFDO ikedo@post.kek.jp

![](_page_58_Picture_1.jpeg)

#### **Capabilities**

- Magnetic and quantum states within solid samples and their depth dependence based on internal magnetic field observations
- Electronic state of trace hydrogen within a material
- Dynamics of electrons, hydrogen, and ions within a material
- Interactions between low-energy muons and materials

![](_page_58_Figure_7.jpeg)

Implantation depth in Cu  $(\AA)$ 

Calculated implantation depth profiles of ultra-slow muons in copper.

#### **Applications**

- $\bullet$ Magnetic properties of thin-films and micro samples that are difficult to study with conventional  $\mu$ SR.
- $\bullet$   $\mu$ SR measurements that require depth resolution, such as determination of the magnetic field penetration length of a superconductor.
- $\bullet$   $\mu$ SR measurements for selective observation of the interior of materials, such as the study of superconductivity at interfaces.
- Study of elementary processes involving low-energy muons and muonium atoms

![](_page_58_Figure_15.jpeg)

A typical result of USM- $\mu$  SR with SiO2/Pt/SiO2/Pt multilayer thin-film

## For neutron instruments

![](_page_59_Picture_10.jpeg)

![](_page_60_Picture_0.jpeg)

## For neutron instruments

![](_page_60_Picture_72.jpeg)

### For muon instruments

Name/description Specification Temperature range: 5 - 370 K Mini-Cryo Microstat Temperature range: 4 - 450 K Minimum temperature: 90 mK Low Dilution refrigerator temperature Sample space: 040 mm Vertical cryostat Temperature range: 2 - 300 K <sup>3</sup>He refrigerator Temperature range: 0.3 - 30 K High Temperature range: 300 - 1000 K Furnace temperature Longitudinal field magnet Magnetic field range: 0 - 0.35 T Transverse field magnet Magnetic field range: 0 - 0.0125 T Magnetic field Magnetic field range: 0 - 0.04 T Small transverse field magnet Magnetic field range: 0 - 0.12 T U1A magnet

 $*$  in preparation

## Glossary

#### 1) Frustrated quantum magnet

BL01 / page 6 BL12 / page 26

BL01 / page 6

**BL05 / page 13** 

![](_page_61_Picture_3.jpeg)

J-PARC press release https://www.j-parc.jp/c/press-release/2019/10/19000342.html

#### 2) Itinerant magnet

In general, electrons in solids are classified into two types: "itinerant states" in which they spread throughout the crystal and move freely, and "localized states" in which they are bound around atoms.

The f-electrons in rare earth and actinide compounds have properties intermediate between these "itinerant states" and "localized states" and exhibit extremely complex and strange behavior.

SPring-8 press release http://www.spring8.or.ip/ia/news publications/press release /2007/070901/

#### 3) Doppler shifter

The BL05 - NOP beamline is equipped with a ultra-cold neutron (UCN) doppler shifter for research on neutron optical devices using UCN and the development of UCN detectors. A UCN doppler shifter is a device that moves a neutron supermirror with high reflectance in the direction of propagation of incident neutrons at 1/2 of the velocity of incident neutrons, causing the neutrons reflected by the supermirror to lose velocity and become UCN.

KENS monthly report https://www2.kek.jp/imss/kens/topics/2014/04/111232.html

#### 4) Superparamagnetism

![](_page_61_Picture_13.jpeg)

The magnetic energy of nanoparticles is very small, and the direction of magnetization is easily disturbed by surrounding thermal energy. Therefore, even if each nanoparticle has ferromagnetic magnetization, the direction of magnetization tends to vary among different particles. This state is called superparamagnetism.

SPring-8 press release http://www.spring8.or.jp/ja/news publications/press release/2004/040909/

#### 5) Giant magnetovolume effect and negative thermal expansion BL08/page 18

The origin of negative thermal expansion is volume contraction accompanying the disappearance of magnetic order, the so-called magnetovolume effect.

Proceedings of 7th Japanese society Meeting for Neutron Science

#### 6) SEOP (spin-exchange optical pumping)

**BL10 / page 22** 

The <sup>3</sup>He spin filter is able to polarize neutrons ranging from the cold neutron to the thermal neutron, and because it is a gas, it can be formed into any shape and cover a wide range of neutron scattering angles. In the SEOP (spin-exchange optical pumping) method, the outermost shell electrons of alkali metal atoms such as rubidium are polarized using optical pumping, and 3He nuclear polarization is achieved by spin exchange between these electrons and the <sup>3</sup>He atomic nucleus.

KENS monthly report https://www2.kek.jp/imss/kens/topics/2012/12/071914.html

#### 7) Multiferroic material

**BL12 / page 26 BL23 / page 46** 

It is a material in which dielectricity and magnetism are combined through its structure, and magnetic and electrical properties are interconnected, and it is expected to be applied to a variety of electronic devices.

J-PARC reaserch result https://j-parc.jp/researcher/MatLife/ja/result/2012 05.html

#### 8) Skutterudite

**BL12 / page 26** 

The name comes from the discovery of CoAs3, a compound with this structure, in a place (Skutterud) in northwest Oslo, Norway. The chemical formula is RxT4X12 (R: metal, T: transition metal, X: pnicogen). It is known that depending on the combination of elements, materials exhibit various properties such as superconductivity, semiconductivity, and valence fluctuation.

RIKEN press release https://www.riken.jp/press/2011/20111102/index.html

#### 9) Barocaloric effect = pressure caloric effect

**BL14 / page 28** 

The calorific effect is when a material generates heat or absorbs heat due to external factors, such as when a magnetic material undergoes a phase transition from ferromagnetic to paramagnetic due to a magnetic field, or when a dielectric material undergoes a phase transition from ferroelectric to paraelectric due to an electric field. The pressure-caloric effect refers to a phenomenon in which a phase transition accompanied by heat absorption or heat generation is induced by pressure.

J-PARC press release https://j-parc.jp/c/press-release/2019/03/2900

#### Before applying a proposal to each instrument, please contact the "J-PARC Center User's Office" by email  $(i$  proposal@ml.j-parc.jp).

OMI F web page: https://mlfinfo.ip/en/

**User's Guide** 

![](_page_62_Picture_14.jpeg)

OJ-JOIN web page: https://www.j-neutron.com/i-ioin.html

![](_page_62_Picture_16.jpeg)

○ J-PARC Center Users Office  $\overline{7}319-1106$ 162-1 Shirakata, Tokai, Naka, Ibaraki 1st floor Aya's Quantum Beam Research Center

![](_page_62_Picture_18.jpeg)

#### **(J-PARC Materials and Life Science Experimental Facility (MLF)>**

#### **Neutron instruments**

Muon instruments

![](_page_63_Picture_3.jpeg)

![](_page_63_Picture_4.jpeg)

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162-1 Shirakata, Tokai, Naka, Ibaraki, 319-1106 Aya's Quantum Beam Research Center Tel. 029-219-5300 (Representative) URL https://neutron.cross.or.jp/en/

![](_page_63_Picture_7.jpeg)

**J-PARC** 

![](_page_63_Picture_9.jpeg)