Workshop reports

Sept 19-21, 2022

Oak Ridge National Laboratory

Neutron Scattering on continuous sources – future developments. US-Japan workshop

Charge:

After limited exchange during the pandemic, this workshop aims to:

- Review recent developments
- Identify common fields of interest
- Initiate close collaborations in the field of hard and soft condensed matter and instrumentation/development

Focus Sessions were supposed to introduce key topic points and identify possible fields of collaboration. Three Sessions were held on Hard Condensed Matter, Soft Condensed Matter and Instrumentation.

Hard Condensed Matter:

- 4-circle diffractometer FONDER - Hiroyuki Kimura (Tohoku University): Four-circle diffractometer FONDER summary
  - Ge(311) monochromator with vertical focus (lambda= 1.24 Å), sin(theta)/lambda ≤ 0.79 Å-1
  - Four-circle diffractometer is currently operated.
  - Crystal and magnetic structure analysis under
  - 2.5 K ≤ T ≤ 700 K, H ≤ 0.6 T, p ≤ 1.4 GPa, strain ≤ 200 MPa, electric field, and simultaneous measurement of neutron diffraction, P-E loop, and permittivity, can be realized.

Curved 2D-PSD project. Originally developed in HANARO. It will be installed in near future. Radius of curvature = 550 mm, Δ2η/X = 0.332 deg./pixel, Δχd/L/Y = 0.0057 deg./pixel. Shortage of 3He gas is serious. -> Transfer from old 3He detector used in AGNES. Radial collimator for 2D-PSD can be effective to reduce background. Development member for prototype 2D-PSD (Prof. Y. Noda and Dr. C.-H. Lee) had already retired. The inheritance of technology from them is crucial. The technology of 2D-PSD has been lost even at HANARO

- DEMAND upgrades and future developments – Huibo Cao (ORNL): DEMAND is a single crystal neutron diffractometer, comparable to FONDER Hiroyuki Kimura presented. DEMAND can cover single crystal neutron diffraction under various sample environment conditions. A few key items were asked and discussed including Position sensitive 2D detector (scintillator neutron camera versus He-gas detector), collimator for complex sample environment, Polarizer options (3He filter, Heusler monochromator, super-mirror polarizer), multi-mode operation (four-circle and two-axis for different sample environment setups). HFIR's DEMAND and JRR-3's FOUNDER can benefit from the

communication and collaborations on Detector, polarizer, sample-environment development. I am looking forward to possible experiments at JRR-3 in future to get some onsite experience with JRR-3 instruments.

- STS CHESS – Gabriele Sala (ORNL): A preliminary concept of the new STS direct geometry chopper spectrometer optimized for small sample analysis, CHESS, was presented at US-Japan workshop. I presented a general Layout of the instrument, paying particular attention to explain how the guide system and the chopper system have been optimized for flux and Repetition Rate Multiplication Mode Respectively. The talk also showed predicted gains over current worldwide direct geometry Instruments calculated using Monte Carlo Techniques.
  Questions arose from the audience were mainly connected to the cost of octagonal guide System and the choice of just limiting the RRM mode to eight energies hitting the sample Per pulse to minimize the background noise.

- SANS for hard matter - Hazuki Furukawa (Ochanomizu Univ. / RIKEN CEMS)

She introduced Hard Matter projects studied by SANS technique. The topics include Skyrmion matters and Vortex ones of super conductors. The latter focused on physical features that SANS measurements proved. She also introduced her ongoing project on Helical vortex state, especially how to detect the phase.

- Development of pressure cell - Yoshiya Uwatoko (ISSP):

We introduced a pressure apparatus for neutron diffraction experiments developed in a US-JP cooperative project. I introduced the piston-cylinder type pressure generator, the mini-MacWhan cell, and the multi-anvil type pressure device, and explained their advantages and disadvantages. BeCu or Zr-BMG material piston-cylinder cell is generated up to 1.8 GPa, which is very convenient and helpful. Hybrid NiCrAl piston-cylinder cell operates up to 2.5 GPa. But the transmission is only10%. Improved McWhan type cell designed by Prof. Onodera operates up to 3 GPa. But not convenient for use due to big body size. Then I newly designed a min McWhan cell that operates up to 2.5 GPa. Due to measuring over 3 GPa, I design the Palm Cubic Anvil Cell, which operates up to 8 GPa. Its transmission is 30%. Also, Uni-axial Pressure Cells by Duralumin 7075 are developed. We Could operate an easily applicable multi-extreme condition high-pressure cell for neutron diffraction.

- Educational use of neutron triple axis spectrometer - Kazuaki Iwasa (Ibaraki University): The educational use of the triple-axis spectrometer T1-1 (HQR) installed in JRR-3 for Major in Quantum Beam Science (QBS) of Ibaraki University was reported. Such an educational activity at JRR-3 etc. is expected to contribute human resource development in QBS field.

- Teaching students TAS techniques using vTAS – Songxue Chi (ORNL): The triple axis spectrometry (TAS) is a versatile technique in the measurement of the scattering function in energy and momentum space. The difficulty in using a TAS instrument lies in the constant need to translate reciprocal space parameters to real space motor positions and vice versa. It is easy to demonstrate how to carry out a TAS experiment using vTAS (virtual Triple Axis Spectrometer), which is an interactive software that shows both the TAS geometry and the momentum space. In the last two NXS (Neutron

and X-ray School) events, we teach graduate students using this software how to align crystals and how to make TAS experimental plans.

Soft Condensed Matter:

- Soft Matter Sample environments for neutron scattering – Lilin He (ORNL)
- Understanding the arrangement of lipid/surfactant molecules in Bicelles using neutron scattering with contrast variation and deuteration – Wellington Leite (ORNL)
- The role of disordered protein in maintaining cellular structure – Viswanathan Gurumoorthy (ORNL)
- Utilize neutron imaging to study polymer compatibilization – Yuxuan Zhang (ORNL)
- Spin echo (Oda, instrumental scientist of iNSE): The neutron spin echo spectroscopy can reach a high energy resolution less than micro eV, that are difficult to cover with other spectroscopic methods. Because of such unique feature, efforts are being made to reoperate the spin echo spectrometer at JRR-3, iNSE even after a 10-year shutdown. Although iNSE's performance is moderate one comparing with the upgraded spin echo instruments around the world, iNSE is still expected to play an important role in the study of soft matter dynamics. Commissioning of iNSE is in progress to partly accept external users in 2023. In ORNL, due to the great demand from the fields of soft matter physics and biophysics, a new spin echo instrument in the guide hall of HFIR is planned.
- Application of machine learning for SANS data analysis - Wei-Ren Chen (ORNL)
- Deconvoluting hierarchical plant cell wall structure using X-rays and Neutrons – Manju S. Mudiyanselage (ORNL)
- SANS for soft matter - Rintaro Inoue (Kyoto University)
- *In-vivo* environment is highly concentrated due to the presence of various bio-molecules such as proteins, nucleic acids, lipids and so on. Under such concentrated environment, both entropic and enthalpic effects, which are essentially negligible under dilute environment, play a significant role for the structure and dynamics of bio-molecules. Then, it is strongly expected that the structural and dynamical information derived from dilute environment are not necessarily applicable for those under concentrated environment. One of the fascinating properties in neutron scattering is discernment of hydrogen (H) from deuterium (D) through the difference in neutron scattering length between them. Taking advantage of this property, we have established the inverse-contrast matching small-angle neutron scattering (iCM-SANS). Main technical point associated with iCM-SANS is the usage of 75 % deuterated protein. Since the scattering length density of 75 % deuterated protein is equal to that of D_2O, 75 % deuterated protein can be treated as an “invisible” component through SANS measurements. We then try to extend this technique for studying proteins under concentrated environment and prepared concentrated 75% deuterated protein in D_2O. We experimentally confirmed that concentrated 75% deuterated protein is nearly contrast-matched in D_2O, supporting the applicability of this for mimicking in-vivo environment.

- STS Centaur – Shuo Qian (ORNL)

Instrumentation:

- Sample environment in PONTA - Taro Nakajima/ Hiraku Saito (ISSP)
- Double-focusing technology - Taku Sato (IMRAM Tohoku University)
  Development of doubly focusing analyzer and monochromator of GPTAS at JRR-3 was introduced. First the doubly focusing analyzer was installed at GPTAS in 2007. This analyzer system consists of 9 horizontally rotating blades with fixed vertical focusing. Positions of the 9 blades can be translated manually. Second, the doubly focusing monochrometer was installed. The monochromator mechanism consists of 5 horizontally rotating blades with one-motor-controlled vertical focusing. Several tricks, including thin and holed Al backing plates that support PG crystals, or inserting Gd2O3 containing film behind PG crystals, were introduced for the background and suprious peak reduction.
- Implementation of the EPICS control system for GPTAS at JRR-3 – Taku Sato (IMRAM Tohoku University)
  An effort to make transition to the EPICS-based controlling system was introduced. Presently, GPTAS uses EPICS for the temperature controller and analyzer motor drivers, and will soon make an full transition to EPICS IOCs. Several discussion on the focusing, as well as EPICS transition were done. Possible future collaborations were also discussed.
- AGNES at JRR-3 – Osamu Yamamuro (ISSP) AGNES is a time-of-flight type chopper spectrometer which was installed by ISSP at the C3 cold guide of JRR-3 (JAEA). The energy resolution is 120 μeV (standard mode) or 50 μeV (high resolution mode). During the long shut down period due the earthquake in 2011, the neutron guide tube and collimator of AGNES were changed from a normal Ni type to 3Qc supermirror one, which increased the neutron intensity 6.8 times. The merit of AGNES is wide temperature range (3-800 K) and gas/liquid high pressure (< 500 MPa). The uses of a 1 K cryostat (T > 0.7 K) and vapor-deposition cryostat are also possible. These enable us to make various experiments mainly in soft matter science. ISSP really welcomes US people coming to Japan to use AGNES through the US-Japan collaboration program.

- The Latest Development in Spherical Neutron Polarimetry at ORNL – Jacob Tosada (ORNL)
- ^{3}He-polarizer program at HFIR – Chenyang Jiang (ORNL)
- Collaboration on Polarized Neutron Scattering (topic from US-Japan meeting)

- HFIR Beryllium Reflector Replacement (HBRR) project – Matthias Frontzek: The HFIR Beryllium Reflector Replacement (HBRR) project has been presented. The scope and timeline of the project has been discussed and the opportunities of this project for instrument upgrades have been outlined. Due to funding the upgrade possibilities are limited, but future upgrades on all instruments will be enabled.
- Sample environment developments, possibility for complimentary use (topic from US-Japan meeting)

Plenary talk "hard condensed matter science using multiplexed detectors and polarization analysis"
Igor Zalyzniak: Zaliznyak overviewed early developments in multianalyzer setup on SPINS cold neutron triple axis spectrometer at NCNR, which led Harriger and Zaliznyak to propose the Inverse Rowland Inelastic Spectrometer (IRIS) design. In the two early working setups, either a large flat analyzer, or a consequential set of 9 see-through analyzer blades were used, resulting in a polychromatic image of a sample which was placed at the focal point of a radial collimator in front of the position sensitive

detector (PSD). Those early studies have shown that using the radial collimator in such a multi-analyzer setup is absolutely crucial, as otherwise very large increase in background associated with exposing large analyzer array to each detector element makes signal to background ratio low and measurement impractical. These polychromatic configurations result in different coupled (Q,E) scans across the PSD for each sample setting, resulting in a coupled slice through the phase space resulting from a triple axis scan. This complicates data analysis and optimization of the experiment. Nevertheless, for one-dimensional systems where directions perpendicular to chains are of no importance, these configurations are advantageous and were successfully used to detect single magnon to continuum crossover in Haldane chain system [I. A. Zaliznyak, S.-H. Lee, S. V. Petrov, Phys. Rev. Lett. 86 (2001); I. A. Zaliznyak. J. Appl. Phys. 91, 8390 (2002)].

An Inverse Rowland setup, on the other hand, allows to place a monochromatic sample image in the focus of a radial collimator and thus allows performing constant-energy slices across the detector array. Such capability is similar to MACS spectrometer at NCNR but allows to cover the scattering angle range more continuously. Analyzer configurations which allow to concentrate all reflecting power of the crystal analyzer array optimized to accept large range of scattering angles on collecting a narrow-energy slice of scattering covering broad range of reciprocal space is advantageous for continuous source instruments. This is distinct from CAMEA setup, where the analyzer power is distributed over a band of energies thus decreasing intensity within each narrow energy band, similarly to time-of-flight instruments at a spallation source. As illustrated by the example of quantum-critical magnetic scattering from itinerant magnet YFe2Al10 [W. Gannon, et. al, PNAS 115, 6995 (2018)], this can become a critical advantage of continuous source instruments compared to time-of-flight instruments at pulsed spallation sources, which would make possible measurements that are impossible at a spallation source of a smaller power which relies on efficiency of data collection within a large energy band.

IRIS, MACS, and other multi-analyzer continuous source setups are also easily amenable to neutron polarization analysis option using supermirrors, which is a significant advantage, and which should not be overlooked.

Plenary talk "Inelastic Spectrometers at JRR-3" Takatsugu Masuda: Takatsugu Masuda explains the status of inelastic spectrometers and the upgrade project of cold TAS in JRR-3. The new multiplex spectrometer HODACA is now under commissioning, and science experiment will start in 2023. Scientists in ORNL are welcome to use it.

Summary of Talking points/Possible fields of collaboration:

- Use of EPICS for instrument control. Mutual benefit for common TAS Control System Studio
- Much of the discussion focused on advanced polarization capabilities, including the development of spherical neutron polarimetry at ORNL. In the past, JRR-3 had the capability to use cryopad for measuring off-diagonal polarization tensor elements, but this has not been used in many years. There is an interest in developing spherical neutron polarimetry at JRR-3 and this may present an opportunity to partner with scientists at HFIR.

- Under the US-Japan collaboration, exchange of researchers from Japan to US is possible. It is desirable to find a way to sponsor exchange for researcher from the US to visit Japan for an extended period (1month).
- Exchange between the 4-circle instruments FONDER and DEMAND
- The sample environment and data analysis for SANS measurements of polymers. Mayumi, Oda, Wei-Ren and Chanwoo will collaborate to develop stretching device for polymer gels and perform in-situ SANS experiments on their tough polymer gels under mechanical stress.
- Neutron spin echo (NSE). Mayumi, Oda, Piotr, and Laura exchange the information of the instrumentation of NSE. Mayumi and Oca are interested in carrying out a NSE experiment on their polymer samples at SNS.