ISRD-RCN Workshop:

Exploring Dynamic Properties of Earth and Planetary Materials Using Neutron Scattering and Imaging

SCIENTIFIC PROGRAM

Session I: Exploring Dynamic Material Properties Using Neutrons

(Chair: Ryan Hurley)

8:00 AM - 8:30 AM: Opening remarks (Daniel Hussey and Wenlu Zhu)

For an overview of using neutron methods for studying earth and planetary materials, see the June, 2021 Elements issue: https://www.elementsmagazine.org/exploring-with-neutrons/

8:30 AM - 9:30 AM: **Larry Anovitz (ORNL)**

Applications of Neutron Scattering to Geochemistry

Abstract: Geosciences and its cousin, Planetary Sciences are broad fields encompassing aspects of physics, chemistry, materials science and biology. Neutron scattering experiments provide means to study a wide range of materials properties, providing advantages in sample penetration, and hydrogen, magnetism, and isotopic sensitivity. Thus, neutron scattering has a broad potential application to a wide range of problems in the geosciences. The connection between the two has been growing in recent years, although many opportunities remain. This talk will begin with an overview of neutron scattering studies, their applications and limitations and comparisons with X-ray approaches, then continue to examples of where neutron analyses, coupled with other approaches, played a critical role in understanding geological processes. These will include the results of an experimental study of volume-reducing limestone dissolution and reprecipitation reactions in the model systems calcite-dolomite (CaCO3 - CaMg(CO3)2 and calcite-fluorite (CaCO3-CaF2) analyzed through the combination of X-ray and neutron (ultra) small angle scattering ((U)SAXS & (U)SANS), and fluids under "ultraconfimenement," where only a single fluid molecule can fit in a given space.

See: DOI: 10.1021/acs.jpclett.0c02026

9:30 AM -10:00 AM: Coffee Break

10:00 AM - 11:00 AM: **Brian Kirby (NIST)**

Neutron sources, accessing user programs, the NCNR CHRNS program

Abstract: Highly penetrating and non-destructive, with sensitivity to light elements and magnetic fields, neutron beams provide information about the microscopic structure and dynamics of materials that is difficult or impossible to obtain via other techniques. State-of-the-art neutron measurements require a facility-scale source, such as a nuclear reactor or proton accelerator / target system, as well as sophisticated, custom-built instrumentation. As such, researcher access to neutron techniques is generally limited to user programs at centralized facilities. Operating 30 beam instruments and serving more than 3000 researchers from industry, academia, and government annually, the NIST Center for Neutron Research in Gaithersburg, Maryland is one of the world's premier centers for neutron science. In my presentation, I'll provide an overview of this key component of the Nation's scientific infrastructure and discuss the utility of intense neutron beams for understanding a wide range of materials systems, including soft matter, quantum materials, and industrial components.

See: https://www.nist.gov/ncnr/chrns/science-highlights

The workshop is supported by NSF EAR-1926627

11:00 AM - 12:00 PM: **Alessandro Tengattini (ILL / UGA)**

Simultaneous neutron and X-ray tomography at NeXT-Grenoble to explore coupled processes in porous media

Abstract: Porous media are a broad class of naturally occurring and engineered materials, ranging from concrete to food material and from rocks to bio-materials. In common they share a complex microstructure and a tendency to undergo coupled hydro-chemo-thermo-mechanical processes.
Historically X-ray imaging has been the main approach to study their full-field response, but in a growing number of cases neutron imaging has proven equally essential to study processes, e.g., to explore the role played by hydrogen-rich substances, such as water, within them. Recent developments have significantly pushed the spatio-temporal resolution of neutron imaging as well as allowed for the acquisition of truly simultaneous neutron and x-ray tomographies. Their combined use is uniquely powerful, thanks to the high complementarity of their contrast. It allows not only to study different aspects of processes (e.g., the interdependence between the opening of cracks and water penetration) but even aids in the identification of the different phases comprising a sample as highlighted in Fig. 1
This contribution will propose an overview of recent developments in neutron imaging including the combined use of x-ray imaging, focusing on recent discoveries allowed by- and new venues opened in- the study of porous media. It will also detail the new venues opened thanks to the recently achieved major upgrade of the instrument NeXT-Grenoble.
See: DOI: 10.1016/j.gete.2020.100206

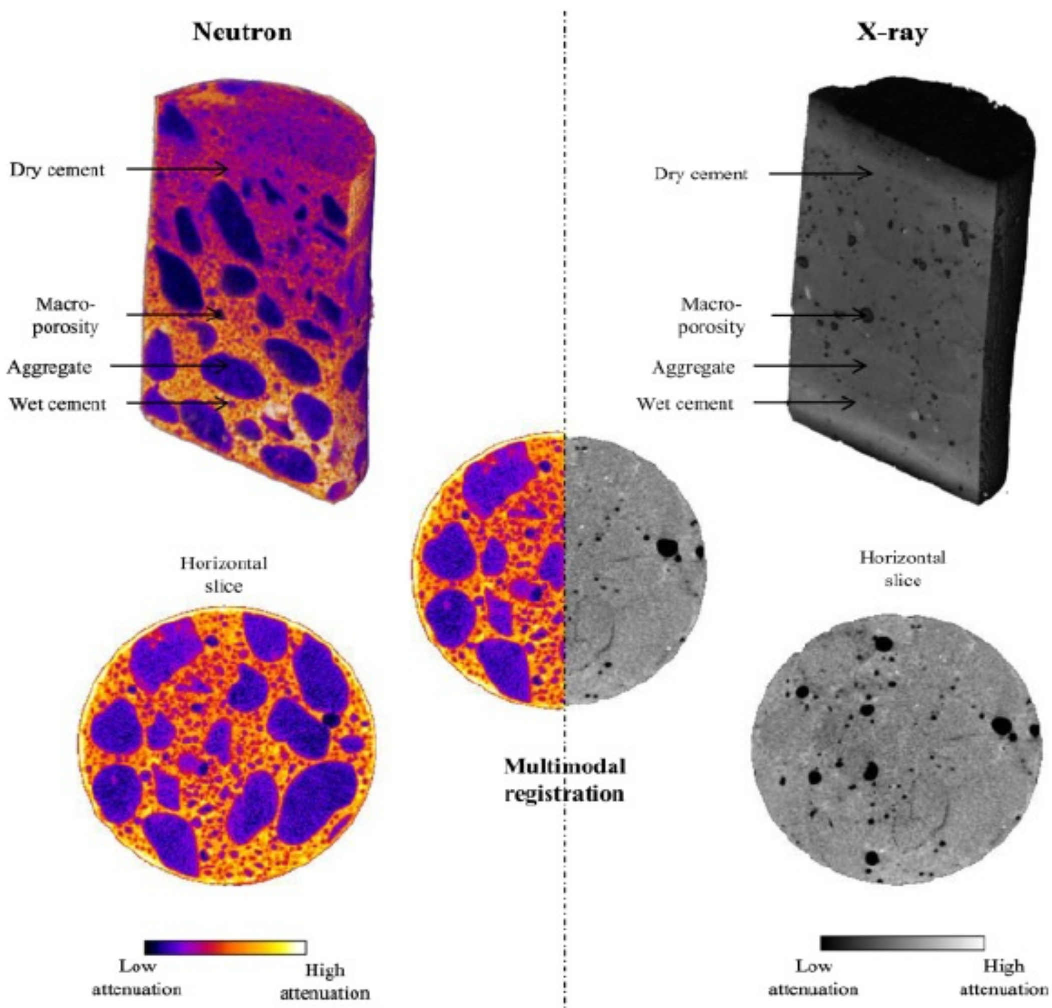

Fig. 1: Example of the unique complementarity of information provided by neutrons and X-rays in the study of concrete.

The workshop is supported by NSF EAR-1926627

1:30 PM - 4:30 PM: **Visiting NIST Center for Neutron Research**

Lobby, NCNR overview discussion with Ron Jones (5 minutes)
Break into 3 groups, and visit 6 instruments, 15 minutes at each instrument.
Guide Hall:
HFBS, Madhu Tyagi, https://www.nist.gov/ncnr/high-flux-backscattering-spectrometer
30 m SANS, Yun Liu, http://www.ncnr.nist.gov/programs/sans/sans_inst.html
CANDOR, David Hoogerheide, https://www.nist.gov/ncnr/chrns-candor-white-beam-reflectometer
Neutron Imaging, David Jacobson, https://www.nist.gov/programs-projects/imaging-neutron-and-x-ray-tomography-next-system

Confinement:
USANS, Ryan Murphy, https://www.nist.gov/ncnr/bt5-usans-ultra-small-angle-neutron-scattering
DARTS, Thomas Gnaeupel-Herold, https://www.ncnr.nist.gov/instruments/darts/

Session II: Neutron Small Angle Neutron Scattering
(Chair: Jennifer Girard)

7:30 PM - 8:30 PM: **Yun Liu (NIST / UD)**

Studying the properties of porous structures and confined guest molecules using SANS

Abstract: Small angle neutron scattering is a powerful tool to study the properties of porous structures and the behavior of confined guest molecules. For many porous materials, they have heterogenous surface properties such as different chemical compositions. The heterogenous distribution of the pore surface composition is difficult to be measured. With the contrast variation method, we demonstrate an approach to measure the heterogenous distribution of pore surfaces using kerogens as examples. I will also discuss methane gas adsorption in model porous materials and show that the surface roughness and pore size play important roles for the gas storage.
See: DOI:10.1021/acs.langmuir.6b02291

July 26 (Wednesday): Day 2
Session III: Neutron Diffraction and Extreme Environments
(Chair: Doug Schmitt)

8:00 AM - 9:00 AM: **Javier Campo (INMA / ILL)**

Neutron scattering at high pressures: opportunities in geology sciences

Abstract: Neutron scattering techniques can provide fundamental insight into the different behaviors shown by matter. In this lecture, a short introduction of the properties of the neutron–matter interactions and the fundamentals of neutron scattering will be presented in order to facilitate an understanding of the peculiarities of this probe. Special attention will be paid to show the complementarity of the neutron scattering techniques with other scattering techniques. Also the extreme conditions diffractometer "XtremeD" built at the Institut Laue Langevin will be introduced, the landscape of similar instruments around the globe, and several scientific examples related with geology or mineralogy under high pressure or at high temperatures will be explained.
See: DOI:10.1088/1742-6596/325/1/012010

9:00 AM - 10:00 AM: **Juscelino Leao (NIST)**

High Pressure Capabilities and Challenges at the NIST Center for Neutron Research

Abstract: The NIST Center for Neutron Research currently provides a variety of pressure apparatus

ranging from 20 MPa to as high as 2.5 GPa that are specially designed for neutron spectroscopy. Most of the pressure equipment can be mounted in a variety of instruments throughout NCNR's facility allowing for experimental flexibility and maximizing beam time use. The available NIST Center for Neutron Research pressure equipment, as well as challenges in developing pressure apparatus designs to exploring dynamic properties of earth and planetary materials using neutron scattering and imaging is presented here.
See: https://www.nist.gov/ncnr/sample-environment/equipment/high-pressure

10:00 AM -10:30 AM: Coffee Break

10:30 AM - 11:30 AM: **Bianca Haberl (ORNL)**

Understanding the role of hydrogen in phase transformations through high pressure neutron diffraction

Abstract: High pressure neutron diffraction is a powerful technique for the characterization of materials and minerals containing light elements. Neutron diffraction can be used to directly determine elemental stoichiometries and atomic positions of light elements, even of hydrogen or deuterium, can further determine how stoichiometries and positions change under high pressure, and can thus yield a new understanding of the impact of these light elements on a given pressure-temperature phase diagram. Such insights clearly are very useful for understanding the phase behavior of minerals naturally containing hydrogen or hydroxide groups, or also of deep mantle hydrous minerals.
However, while corresponding X-ray diffraction techniques have long enabled in situ studies at several Mbar and several thousand K, neutron diffraction has suffered from a need for large sample volumes due to (relatively) low neutron flux even at the strongest sources. This has long limited the available pressure (and temperature) conditions for in situ studies. While many successful studies have been conducted at neutron sources worldwide, concerted efforts are underway at to address these shortcomings and enable in situ diffraction under even higher pressure and temperature conditions.
In this presentation, I will first introduce the current state-of-the art of neutron diffraction studies on minerals under high pressure, high temperature conditions as well as of studies on recovered minerals at Oak Ridge National Laboratory (ORNL) and beyond. I will then narrow in on activities at ORNL that aim to address the shortcomings in necessary pressure technology. Specifically, I will highlight the recent developments of a new neutron diamond anvil cell that has finally enabled studies above the Mbar [1] as well as its application to hydrogen-containing materials [2]. I will conclude with an outlook on necessary future developments for simultaneous extreme conditions such as, for example, high pressure, high temperature in a laser-heated neutron diamond cell [3].
See: [1] DOI: 10.1038/s41598-023-31295-3; [2] DOI: 10.1063/5.0069425; [3] DOI: 10.1063/5.0093065

Session IV (Posters): Study of Earth and Planetary Materials (Chair: Wen-lu Zhu)

July 27 (Thursday): Day 3

Session V: Recent Advances at Neutron Beamlines (Chair: Haiyan Chen)

8:00 AM - 9:00 AM: **Heloisa Bordallo (UCPH / ESS)**

Shedding light on the Muddy World of Clays with Neutrons: Water Dynamics in a Broad Time Scale

Abstract: Hydrous clay minerals (smectites) have complex interactions with water that both and separate them from other layer silicates (e.g., micas). The hydration energies of cations held within the interlayer space of montmorillonite have long been viewed as key to clay mineral hydration. Many experiments have shown that the charge and hydration energy of the interlayer cation imparts differences in the way the clay mineral hydrates, orders and interacts with its surroundings. Additionally, water adsorption to the surfaces of clay minerals cannot be discounted as important. As fine-grained materials, the structure of the pore-network within a particle of clay mineral (containing perhaps many thousands of individual layers) can be shown to control water uptake and release. Various approaches can be used to study these processes and this presentation I will focus on the applications of neutron spectroscopy,

The workshop is supported by NSF EAR-1926627

particularly quasi-elastic neutron scattering. Here I will detail how this amazing experimental technique allows for a full description of the water mobility within bulk pores, inside the interlayer space, and in the surrounding interlayer cation of montmorillonite. If time allows, I will also discuss on the driving force for water filling pores in the two chalk samples, one from an outcrop in northwestern Denmark and the other from a drill core from the Danish North Sea Basin.
See: DOI: 10.1021/jp803274j and 10.1016/j.clay.2020.105928

9:00 AM - 10:00 AM: **Hassina Bilheux (ORNL)**

VENUS: Time-of-flight imaging capabilities for Earth and planetary materials

Abstract: Neutron radiography and computed tomography are established transmission techniques used to study geomaterials (soil, rocks, etc.). These techniques provide 2 and 3-dimensional attenuation-based maps of an object. Beyond transmission, advanced techniques such as Bragg edge and resonance imaging can correspondingly provide unique information based on the crystalline and elemental/isotopic properties of the sample. Oak Ridge National Laboratory is currently building the VENUS imaging beamline at the Spallation Neutron Source. VENUS is optimized for time-of-flight imaging techniques such as Bragg edge and resonance imaging. Currently under construction, the beamline will start commissioning during fall 2024. This presentation focuses on the application of time-of-flight imaging capabilities for Earth and planetary materials. A brief update on the status of the VENUS construction project and means to access the beamline after construction and commissioning have ended will also be discussed.
See: DOI: 10.1063/1.5013676

10:00 AM - 10:30 AM: Coffee Break

10:30 AM - 11:30 AM: **Jacob LaManna (NIST)**

Emerging Neutron Imaging Methods

Abstract: Rock deformation research poses some challenges and opportunities to neutron imaging. The sample environment, pressure vessels, and/or load frames can be bulky, minerology can have poor contrast variation in neutrons, and important microstructure can often be at a scale below imaging resolution. Multiple new neutron imaging techniques are currently underdevelopment that can address these difficulties for rock deformation studies. Neutron imaging requires the sample be placed as close to the detector as possible to achieve the best resolution which is not feasible with large sample environment. Wolter optics will allow the neutron beam to be focused down removing the limitations of pinhole optics and will place the sample several meters from the detector with ample room for large load frames and other test equipment. Poor contrast of minerology due to materials being similar in neutron attenuation or high amounts of scattering obscuring small differences can be overcome with simultaneous neutron and X-ray tomography (NeXT). X-rays interact with materials differently than neutrons and provides an additional source of contrast that can separate two material peaks in a histogram better than either mode independently. New reconstruction methods are being pursued to use the X-ray information to improve neutron tomography resolution and scatter reduction. Neutron imaging has demonstrated a best-case pixel resolution of 1.5 μm but many structures in rocks, such as porosity and grains, can be much smaller than 1.5 μm. A new neutron far-field interferometer technique currently being developed will simultaneously acquire 3 images. One of these images is the dark field image which is equivalent to small angle neutron scattering (SANS). Since the technique is based on tomography, this will give spatially resolved SANS where every voxel will have a SANS curve that can be fit to determine the structure inside down to the nm. This talk will give an overview of current developments for Wolter optics, NeXT, far field interferometry, and other techniques and will discuss the relevance of these techniques to rock deformation studies.
See: DOI: 10.1063/1.4989642 and 10.2138/gselements.17.3.189

The workshop is supported by NSF EAR-1926627