

# WCNR-11 - 11th World Conference on Neutron Radiography

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## Book of Abstracts



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## Combined neutron and x-ray imaging on concrete cores

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Neutron imaging at ANSTO (DINGO) and x-ray imaging (commercial instrumentation) has allowed the dual investigation of the same concrete samples highlighting a unique ability to take advantage of the best of these complimentary techniques to visualize the pores, flaws, cracks and even effects of bad mixing practice. It has become evident to us that the combination of the two techniques can enable unique insight into concrete.

Concrete service life is directly related to its durability or the ability of concrete to resist the intrusion of fluids that lead to corrosion. While good quality concrete can have a service life measured in centuries, poor quality concrete may need expensive repairs within a year of placement. As the cost of repairs to concrete infrastructure is measured worldwide in tens of billions of dollars per year it is obviously of some economic importance to find out why a concrete is flawed.

Concrete is made by mixing cement, aggregate, sand, and water together and after placement is compacted and cured. Poor mixing technique using the wrong composition (commonly too much water), inadequate compaction or inadequate curing, can lead to flawed concrete with a short service life. Other problems such as placement of the reinforcement tie bar into the concrete or the alkali silica reaction in the concrete can also limit the service life.

Optical microscopy has often been used as to determine which flaws in concrete have been the cause of short service life. We have found that both neutron and x-ray imaging (with or without tomography) can be used as a cost effective tool to visualize the flaws in concrete. In particular it is easy to see the relationship between flaws, voids, and emerging cracks.

X-ray imaging of the same sample at the same resolution as the neutron imaging shows beam hardening and strong reflection/interference near the reinforcement makes analysis of the concrete (within the X-ray beam cone) difficult. However, where the reinforcement was outside the X-ray cone, X-ray imaging provided high contrast of pores and cracks within the concrete and at a greater resolution.

The advantage of these imaging methods is that the only sample preparation needed is cutting the sample and the removal of water by heating. Neutrons can typically give pixel resolutions of 50  $\mu\text{m}$  for samples 35 mm in radius while X-ray imaging for much smaller samples ( $\sim 10$  mm) can give pixel resolution of less than 0.1  $\mu\text{m}$ . This combination of neutron and x-ray imaging gave added value as both methods are complementary in terms of resolution, penetration and contrast.

The visualisation of the images gives insights into the flaws that limit service life of concrete that no other technique can attain.

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## Development of Fast Neutron Imaging as a Non-Destructive Evaluation Tool in the Nuclear Industry

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The use of fast neutron imaging and tomography as a non-destructive evaluation (NDE) tool for the inspection of high density components in support of their integrity is assessed. We aim to demonstrate its validity within the nuclear industry, primarily in support of component life extension and consider the potential for a compact advanced source imaging system onsite at AWE. Results from a series of neutron imaging experiments on calibrated image quality indicators (IQIs) using the WNR Facility 60R fast neutron imaging flight path (Geanie) on LANSCE are presented where shielding behind 3 inches of uranium still allows for a resolution of close to 300 microns using a lens coupled CCD neutron imaging camera. A modular variable opacity IQI will be discussed with the purpose of replicating a number of engineered features at various locations within a component. Data will be presented for ¼ scale manufactured versions of this conceptual IQI having been imaged on the IMAT cold neutron beam line at the STFC in the UK. Finally initial imaging results from the CHIP IR fast neutron beam line will also be discussed; whilst the beam line is not designed for imaging it could serve as a useful test bed for novel scintillator development and model validation using the GEANT 4 particle transport code for forward modeling of component imaging. Future work will include assessing alternative panel based imaging systems, further optimisation of the lens coupled camera, scintillator development and validation using IQIs.

**Speaker Sessions and Seminars / 110**

## **Neutron micro-CT as a non-destructive tool for Palaeontology in Australia**

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The physical extraction of fossilised remains from rocks enables quantitative physiological investigation of bone-dimensions, volume, and porosity, however leads to the destruction of valuable contextual information and soft-tissue remains within the matrix.

Conventional and synchrotron-based X-ray computed tomography (XCT) have been utilised for many years as critical tools in uncovering valuable 3-D internal and surface renderings of scientifically important fossils, however poor contrast and X-ray penetration often prevents thorough tomographic analysis.

DINGO, Australia's first and only neutron micro-computed tomography (nCT) instrument, located at the OPAL nuclear research reactor, is being used to obtain unprecedented reconstructions of extraordinary fossilised anatomical features not visible with conventional imaging techniques. This presentation will outline the physical capabilities of DINGO, the limitations and results to-date in the field of palaeontology. Drawing upon specimens scanned from across Australia, North America, New Zealand, and China, this presentation will demonstrate the complementarity of nCT to classic XCT methods for certain geological formations and fossil localities.

nCT has yielded unprecedented contrast and detailed-reconstructions of fossilised soft tissue in a Jurassic cynodont. The stomach contents and digestive function of herbivorous and carnivorous dinosaurs, and a Cretaceous Australian crocodylian have been revealed, providing insights into ancient environments and food chains. In this way, a new species of Australian dinosaur has been discovered.

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## Polar wildfires and fire-adapted seed dispersal during the Cretaceous global hothouse

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Several highly effective fire-adaptive traits first evolved among modern plants during the mid-Cretaceous, in response to the widespread wildfires promoted by anomalously high atmospheric oxygen (O<sub>2</sub>) and extreme temperatures. Serotiny, or long-term canopy seed storage, is a fire-adaptive strategy common among plants living in fire-prone areas today, but evidence of this strategy has been lacking from the fossil record. Deposits of abundant fossil charcoal from sedimentary successions of the Chatham Islands, New Zealand, record wildfires in the south polar regions (75°–80°S) during the mid-Cretaceous (ca 99–90 Ma). A new species of fossil conifer reproductive structure, *Protodammara reimatamiori*, was consistently associated with these charcoal-rich deposits. Neutron tomography has been crucial in revealing the morphology and internal anatomy of these fossils, which exhibit a range of serotiny-associated characters. Complementary Fourier-transform infrared spectroscopy has chemotaxonomically constrained the phylogenetic placement of these fossils, demonstrating that fire adaptations evolved independently in at least two conifer families during the mid-Cretaceous. Numerous fossils from similar, contemporaneous deposits of the Northern Hemisphere suggest that serotiny was a key adaptive strategy during the high-fire world of the Cretaceous.

### Speaker Sessions and Seminars / 157

## Imaging at the Spallation Neutron Source: Opportunities and Challenges

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Over the past few years, several wavelength-dependent neutron imaging capabilities have been developed at spallation neutron sources such as RADEN at J-PARC and IMAT at ISIS. At the Spallation Neutron Source of Oak Ridge National Laboratory, wavelength-dependent experiments are ongoing, and a temporary imaging capability is being planned at the Spallation Neutrons and Pressure Diffractometer (SNAP), beamline 3 instrument. A design of this new imaging capability is presented. The facility will be equipped with exchangeable apertures optimized for cold, thermal and epithermal neutrons, respectively. A dedicated sample area (for 2D and 3D data acquisition) and in-house event mode microchannel plate (MCP) detector are currently being developed as part of this project. Recently, the team has measured crystalline structures (using cold neutrons) and isotopic content (using epithermal neutrons) in superalloys and nuclear fuel material, respectively. We present the characterization of additively manufactured (AM) Inconel 718 using wavelength-dependent radiography, the so-called Bragg edge imaging technique, diffraction and modeling. This dual-modality capability combined with modeling provides unique information about the crystalline

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## Water Migration in Engineered Barrier Materials for Radioactive Waste Disposal

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The US Department of Energy is evaluating various strategies for deep geological repositories for the permanent disposition of spent nuclear fuel. The development and evaluation of engineered barrier system design concepts and their potential interactions with natural barriers or with other engineered interfaces are important to the long-term (i.e., tens of thousands of years) safety and performance of geological repositories. Current understanding of barrier behavior and water migration is dependent on simulation modeling. Codes such as TOUGH2 are used to characterize behavior of natural and engineered barriers. These codes have been used to simulate the observed progress of hydration phenomena through various media in the laboratory. There are, however, fundamental limitations to these simulations.

Both natural and engineered barriers are needed to limit the flow and movement of water or adsorb contaminants by chemical means. Media selection for barriers includes the use of materials that have very low permeability. However, the complete elimination of water movement through even low permeability material is not viewed as practical. Rather, what is needed for predictive modeling is a full understanding of the mineralogical behavior and pore structures of the material under saturation conditions and the transport characteristics of water through potential engineered barrier materials.

Neutron imaging was used to understand the transport of fluids through dense solids. The experiments simulated wetting–drying–rewetting cycles to ascertain if changes in mineral hydration are temporary or permanent. Bentonite used in these experiments has undergone extensive characterization as it is being considered for barrier material. The samples were subjected to hydrothermal treatments that included contact with deionized water or with saturated NaCl and saturated KCl solutions.

Neutron imaging at the Oak Ridge National Laboratory High Flux Isotope Reactor and the National Institute of Standards and Technology provided radiographs of water within bentonite samples that were prepared with different sized aggregates packed to varying bulk densities. The images were processed using Python scripts, and the resulting water uptake profiles were compared using a forward-averaging algorithm. Uptakes were fit to polynomials. Water saturation before and after uptake was determined gravimetrically.

Because of the possibility of direct observation, neutron imaging results can be used to answer fundamental questions associated with ground water transport in clays. Results from these experiments will contribute to the development of well-calibrated models that can be extended beyond laboratory scale, which is essential for the proper selection and evaluation of barriers to radionuclide transport.

Speaker Sessions and Seminars / 103

## Jupyter Notebooks for Neutron Radiography Data Processing Analysis

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The High Flux Isotope Reactor (HFIR) CG-1D neutron imaging facility accommodates a broad range of research applications such as materials science, engineering, energy, physics, biology and plant physiology. This instrument is equipped with a modern data acquisition system that helps users to acquire data in a semi-automated fashion. Until now, raw data were processed using MatLab and/or ImageJ, which required extensive training by beamline staff. In order to improve user experience and to allow live feedback processing of the raw data, the imaging software team has developed tools such as semi-automated reconstruction and Jupyter Notebooks that can be adapted to the specific scientific questions from the research team. One of the advantages of the notebooks is that facility users do not need to be advanced image processing scientists, nor do they need expertise in Python programming. Another advantage is that an existing notebook can be readily adapted for a new experiment without a tremendous time commitment from the imaging software team. Using a few research examples, this talk will present the tools developed and used by the the scientific community coming to CG-1D.

**Speaker Sessions and Seminars / 127**

## Neutron Imaging for Fuel Cells: Yesterday, Today and Tomorrow

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Neutron imaging has been applied since nearly two decades to visualize the water distribution in operating fuel cells, and has largely contributed to unravel the mysteries of water management in these devices. Two key characteristics make neutron imaging particularly attractive for fuel cell research: the high penetration of neutrons through dense structural materials such as aluminum and steel, and the strong contrast provided by liquid water. This combination makes neutron imaging fully non-invasive, in the sense that little adaptations have to be done on fuel cells, if any.

Here, a brief overview of the contributions brought in the past by neutron imaging to the field of fuel cell research (at PSI and worldwide) will be given first. Following this, the application of neutron imaging to our latest research, focusing on our developments in novel porous materials [1] and in innovative fuel cell designs based on evaporative cooling [2,3] will be presented. Finally, I will give an outlook focused on how advanced neutron imaging techniques such as neutron grating interferometry (nGI) and time-of-flight (TOF) imaging can solve problems beyond the reach of conventional imaging.

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## Sample Environment for Neutron Radiography at the Australian Centre for Neutron Scattering

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The sample environment team at the Australian Centre for Neutron Scattering (ACNS) works in conjunction with instrument scientists and users to produce innovative equipment to allow a broad range of experiments to be carried out on DINGO, the ACNS radiography instrument.

These include equipment to control humidity while observing paint dry, observe a single grain of rice cook, and observing starch behaviour under heat and shear. Or to put it another way this presentation will give details of our vapour delivery system (Hiden Isochema), sample temperature control and a modified Rapid Visco Analyser (Perten RVA). Along with our standard sample environment equipment including closed cycle cryostat (6 – 300K), high temperature vacuum furnace (400- 1700K), temperature controlled sample holder for tomography (255 – 375K).

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## Commissioning the of NDDL-40 Neutron Detection System at Oregon State University

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The Neutron radiography facility (NRF) at Oregon State University (OSU) has been modified to begin working on the non-destructive evaluation of concrete materials to study the early stages of shrinkage, cracking, and water transport of concrete during the curing process. The objective of this work is to investigate the efficiency and spatial resolution of the Neutron Detection-Delay Line (NDDL) 40 Micro-channel plate (MCP) detector for the use of neutron radiography and tomography with the eventual goal of concrete imaging. Working in collaboration with the School of Civil and Construction Engineering, the NRF at OSU has added a NDDL 40 vacuum sealed neutron imaging detection system with a delay line system readout developed by Nova Scientific Inc. The Study showed the system was capable of a maximum spatial resolution of ~200 µm with a detector efficiency of 6%.

**Speaker Sessions and Seminars / 135**

## Design and Construction of Grating-Based Interferometers for the Oak Ridge National Laboratory, High-Flux Isotope Reactor, CG-1D Tomography Beamline

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The ORNL HFIR CG-1D neutron tomography beamline will be the future site of grating-based interferometry/tomography. This presentation will give a work-in-progress report describing construction activities and results commencing in late spring, 2018.

Two interferometer designs will be developed: Talbot-Lau and far-field. Talbot-Lau has the advantage of considerable operational experience at several facilities, particularly at the PSI ICON beamline. The far-field interferometer is relatively new to X-ray and neutron imaging and may offer more access to dark-field imaging as a function of interferometer autocorrelation scattering length. In addition, neutron flux through the far-field interferometer should be 2-fold greater than the Talbot-Lau design due to one fewer absorption gratings.

The CG-1D neutron tomography is well suited for the addition of grating interferometry. The beamline is currently operated with a high-flux, polychromatic cold neutron beam offering useful flux in the wavelength range 1.8 to 6 Å. Beam divergence is usually set at  $L/D = 400$ . The distance from pinhole collimator to detector is 5 m. The neutron path is protected with helium-filled flight tubes having thin aluminum windows. The first grating will be mounted near the pinhole optics, thus sharing the the same radiation enclosure. The other two gratings will be more easily accessible.

The presentation is expected to cover of some these topics:

- Construction of a Talbot-Lau interferometer;
- Construction of a far-field interferometer;
- ☒• Optical simulations;
- Fabrication of extremely small period neutron phase gratings;
- ☒• A motor control system based on Python and EPICS; and
- ☒• Planned applications of interferometry to laser sinter additive manufacturing.

## Speaker Sessions and Seminars / 50

### Fission Neutron Tomography of a 280-L Waste Package

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For the non-destructive characterization of radioactive waste packages for the declaration or verification of their radioactive inventory, well-established passive and active methods are applied. These are mainly based on gamma-spectroscopic emission measurements (segmented gamma scanning), gamma-transmission measurements (e.g. radiography and tomography) using an external Co-60 source or accelerator, neutron emission counting with time correlation analysis to distinguish between neutrons originating from spontaneous fission or (alpha,n) events, respectively, and neutron interrogation techniques inducing fission events. Tomography using fission neutrons, both in transmission and emission mode, is not applied on waste packages, yet.

In a recent feasibility study [2] it was demonstrated that fission neutron radiography of 200-l (radioactive) waste drums is possible at NECTAR [1]. In a subsequent step, the study is extended on tomographic investigation of 200-l and one 280-l mock-up waste drums. The latter contained a 200-l

drum with a mixture of supercompacted waste in the bottom and raw waste in the upper part. The result of this 3D-tomography is compared with the corresponding one using an external Co-60 transmission source.

In further experiments at NECTAR, the influence on the resulting images in radiographic measurements were investigated for additional strong AmBe-neutron sources being present in the waste packages. These results will give information on possible artefacts in tomographic reconstructions caused by internal neutron sources in the radioactive waste packages.

Results of these measurements will be presented and discussed. In a final conclusion, the applicability of fission neutron tomography, its specific characteristics, the limitations and a critical comparison with the well-established Co-60 gamma-transmission tomography for the non-destructive characterization of radioactive waste packages will be presented.

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## Parameter determination for image alignment in fission neutron tomography

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For an accurate tomographic reconstruction, the correct alignment of particular radiographic projections relative to the detector plane is crucial. Without alignment image blur or other artefacts corrupt the reconstruction quality. Applying mechanical corrections and image post-processing may correct potential misalignment.

For a typical neutron CT set-up used at NECTAR [1] the mechanical alignment affects the adjustment of the manipulator stage and the detector system. Hence, the sample's rotational axis is centered and aligned parallel to the scintillator screen. However, minor misalignments may remain making additional, i.e. software-based corrections necessary.

A best practice procedure implemented in many software packages is the matching of two measured mirrored images with an angular distance of exactly 180° [2]. The assessment yields the tilting angle and the translational offset which are in turn used for the software-based correction of the remaining images. Other methods are based on evaluating complete tomographic data sets for measurements on 360° (e.g. [3]). However, it has been overlooked that mirrored radiographs are translated and rotated samples of the same object. Therefore, rigid image registration methods are applicable for the simultaneous determination of lateral shift and tilting angle.

At NECTAR, tomographic data sets for three samples having different morphological structures were measured by means of fission neutrons using the golden section scan strategy [4]. Each data set consists of more than 900 images at different angular positions.

The aforementioned methods for correction of misalignments are applied and the quality investigated by the resulting tomograms. Although the focus is on fission neutrons, the results are applicable on other neutron energies, too.

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## Characterization of commercially available PP/ZnS scintillators for fission neutron imaging

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NECTAR (neutron computed tomography and radiography) is a neutron imaging instrument available at the FRM II neutron source at Heinz Maier-Leibnitz Zentrum (MLZ). It is a unique instrument, at which neutron imaging can be performed either with thermal or fission neutron spectra [1]. The later is obtained via fission reactions taking place in the so-called converter plates consisting of highly enriched uranium silicide, which are placed in front of the beam tube window. The penetration depth of fission neutrons is much higher as compared to cold or thermal neutrons, thus fission neutron imaging gives more insight in large objects and samples containing strongly attenuating elements. Therefore, this technique has a great potential as a nondestructive technique for investigation of inner structure of large objects e.g. archaeological or paleontological samples, which cannot be penetrated with low energy neutrons [2]. While detection technology for cold/thermal neutron imaging is already well established, spatially resolved detectors for fission neutrons offer relatively poor image resolution and low detection efficiencies, both. Namely, spatial resolution of images taken with fission neutrons is about 2 orders of magnitude lower, while exposure times are about one order of magnitude longer as compared to thermal neutron imaging. Therefore, research on development of scintillators for fast neutron imaging is of high importance.

In this work we present the results of characterization studies of commercially available scintillator screens composed of PP/ZnS:Cu and PP/ZnS:Ag. The scintillators of different thicknesses were provided by RC Tritec AG company and were tested for the light output, gamma radiation sensitivity and spatial resolution at the NECTAR facility. As expected, it was observed that light output increases and spatial resolution decreases with the scintillator thickness. Moreover, it was demonstrated that both compositions of the scintillating material provide similar light output, while the gamma sensitivity of PP/ZnS:Cu is significantly higher as compared to PP/ZnS:Ag based scintillators [3].

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## A preliminary experimental study on neutron holography technique at CMRR

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Neutron holography is an imaging technique permitting the three-dimensional reconstruction of the micro-structure of the original sample by using monochrome neutron beam. Neutron holography is able to penetrate deeply into matter with high resolution, and is applicable to the micro-structure investigation of a wide variety of hydrogen-containing compounds and neutron-absorbing isotopes doping crystals. In contrast to X-ray and electron holography techniques which are based on similar principles, the limited intensity of neutron source and the difficulties on beam modulating create obstacle in neutron holography experiments, thus the hardware and reconstruction methods need to be improved for wider applications. Neutron holography in China has not been studied yet limited by the experimental condition.

A systematic primary research of neutron holography according to China Mianyang Research Reactor (CMRR) condition has been carried out, including numerical simulation, reconstruction approaches, critical experiment parameters. Recently a holography experiment of a Pd-H single crystal was carried out by using high resolution neutron diffractometer at CMRR. The results reveal the position of atomic Pd nucleus in accordance with numerical simulations. Since the reconstructed image quality is worse than expected due to limited efficiency and recording time, ways to improve the holographic image are discussed.

These results will be helpful for future works on instrument construction and applications of neutron holography.

### Speaker Sessions and Seminars / 123

## An experimental approach for quantitative scattering correction in neutron imaging.

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### Introduction

Quantitative neutron imaging is hampered by different sources of nonlinearity: polyenergetic beam, beam hardening, detector uneven light distribution and neutron scattering. The correction of these effects is necessary to approximate the log-attenuation as a linear function of the sample density. We focus here on the scattering component, caused by the neutron interaction with the sample and with the experimental apparatus. We recently proposed a fully empirical method for scattering correction without the need of prior knowledge of the neutron spectrum or of the sample composition [1]. We describe here its implementation: a scattering correction term is included into the post-processing image normalization procedure, which usually only includes open beam and dark current images. We show the performance of the proposed approach in removing scattering related cupping artifacts in a test CT acquired at the NEUTRA beamline at PSI.

### Methods

An aluminum frame containing neutron absorbing cylinders made of 10B4C, called black bodies (BBs) evenly distributed over the field of view has been constructed. As the BBs are opaque to neutrons, the measured neutron intensity behind them can be interpreted as the scattered neutrons component.

During the experiments, two more sets of images are acquired using the reference frame for the estimation of the scattered neutrons contribution to the image intensity: with and without the sample in the beam. These images are used to correct for the scattering contributions from the sample and the background, respectively.

The scattering component is estimated from the BB measurements by segmenting the BBs and interpolating the underlying values with a 2D second order polynomial scheme. A dedicated dose

correction scheme is also implemented to compensate for beam fluctuations and the decrease in transmission due to the presence of the BB grid. The computed scattering components are subtracted from projections and open beam images, before image normalization.

The necessary image processing and implementation of image normalization is integrated in MuhRec, an open source CT reconstruction software [2].

#### *Results and discussions*

A cylindrical aluminum container (10 mm external diameter, 2 mm thick) filled with water was imaged at NEUTRA measuring position II with 625 tomographic projections uniformly distributed over a full rotation of 360deg (100  $\mu\text{m}$  thick 6LiF/ZnS scintillator). BB images were taken without the sample and with the sample according to a sparse CT scheme (25 equally distributed projections over 360deg) and linear interpolation was applied to estimate sample scattering in missing angles. With the proposed approach, the cupping artifacts in the reconstructed CT were successfully compensated. Mean attenuation coefficients were around 3.57  $\text{cm}^{-1}$  for corrected vs. 2.84  $\text{cm}^{-1}$  for the non-corrected CT (expected value 3.6  $\text{cm}^{-1}$ ).

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## MuhRec, an open source tool for CT reconstruction

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We describe in this contribution the open source release of the computed tomography (CT) reconstruction software, MuhRec. The software computes CT reconstruction based on filtered back projection from raw projection data and allows for a flexible configuration of an arbitrary set of pre-processing modules. For neutron imaging experiments, the most common configuration includes: i) computation of attenuation images from raw data with normalization to reference images, ii) spot cleaning iii) ring cleaning, iv) projection filter and v) back projection for parallel beam geometry. Both command line and GUI modes are available, to facilitate users to set up the reconstructor and execute the processing. It is developed at Paul Scherrer Institut (PSI) with the needs of neutron imaging users in mind [1] and can be used on multiple operating systems. Its development is ongoing and was chosen in the framework of the European project SINE2020 [2], to provide to the neutron imaging community a general tool for tomographic image reconstruction. Its development is furthermore supported within the PSI and European Spallation Source (ESS) in-kind contribution for the image analysis software of the imaging instrument, ODIN.

New features of the latest release include a redesigned graphical user interface for better workflow and improved user experience. Reading NeXus files [3] is now supported. On the algorithm side we have added support for cone beam geometry to allow reconstruction of X-ray CT and neutron CT in case of divergent beam. This is relevant for simultaneous X- and neutron CT acquisition, which is now available at several neutron imaging beamlines worldwide. Furthermore, a new algorithm for scattering correction is included as a module [4]. The module uses reference data to estimate the scattering component which is subtracted from the projections. Using the correction module makes it possible to obtain reconstructed attenuation coefficients close to values given in literature [5]. The source code is accessible together with wiki documentation in the git repository [6]. Future development aims at adding many more new features, and in particular the inclusion of different reconstruction techniques, such as iterative, support for different image format and further pre-processing modules.

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## Total variation iterative reconstruction from few projections for the indirect foil-film radiography

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Computed tomography (CT) has been considered as an important tool for evaluating the structural integrity of samples non-destructively, which requires many projections to be acquired from different angles. The indirect foil-film transfer technique has been developed for acquiring neutron projections of irradiated components released from the nuclear reactors. This technique is insensitive to gamma radiation. However, it costs significant amount of time for producing projections, which is prohibitive for acquiring many projections needed for CT reconstruction. Thus, it is difficult to obtain three dimensional (3D) reconstruction with high quality for this technique by using conventional Fourier reconstruction methods. In this paper, the total variation (TV) iterative algorithm and equally sloped tomography (EST) based on total variation (TV) algorithm are developed for limited-angle and sparse-view CT, respectively. The performance of the two algorithms is assessed with simulated data firstly. This can help us to find the optimal imaging parameters for reconstructions of the fuel assemblies from few projections.

**Speaker Sessions and Seminars / 67**

## Implementation of thermal neutron radiography at medium and low power research reactors in Iran

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Research reactors have been used as good neutron sources for neutron radiography systems during last decades. Although these reactors have many disadvantages, such as lack of portability, high

cost and high waste production, these sources can provide high and stable neutron flux and also have some equipments such as beam tubes in order to extract neutron beams through the biological shield. In recent years, a thermal neutron beam was designed and implemented in the radial “E” beam tube of Tehran Research Reactor (TRR). TRR is a 5 megawatt research reactor and equipped with seven beam tubes. Characterization of this thermal neutron radiography beam was done using the Image Quality Indicators (IQI) of American Standard and Testing Materials (ASTM). Besides that, during the past year, another thermal neutron beam is implemented at the Miniature Neutron Source Reactor (MNSR). MNSR is a 30 kilowatt research reactor and compared to the TRR, it has not external beam tube. Therefore, in this case an external beam tube is designed and constructed in order to achieve an appropriate neutron radiography beam. Some samples like IQIs, fresh nuclear fuel rods, ancient pottery, plant roots and soil, graphite box are studied using these two neutron radiography beamlines. In this paper, the design details of these neutron radiography beamlines, the parameters of these beamlines, the result of beam characterizations and some experiments that are done at these facilities are presented.

### Speaker Sessions and Seminars / 128

## Evaluation of fast neutron imaging scintillators

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As neutron imaging emerges as a complement to current nondestructive testing techniques such as X-Ray imaging, more research is focusing on fast neutron imaging. Fast neutrons offer excellent penetration through heavily shielded materials due to their low probability of collision interactions, however this also makes their detection difficult. This work applies lens-coupled imaging to measure several different scintillator screens' aptitude for fast neutron imaging. The experimental apparatus consists of an electron-multiplying charged coupled device (EMCCD) camera and a lithium doped front-surface mirror. We evaluate fast neutron imagers constructed at Lawrence Livermore National Laboratory (LLNL) that consist of Polyvinyltoluene (PVT) scintillators loaded with different dopants and given different backing materials. The PVT scintillators were irradiated both at The Ohio State University's Research Reactor (OSURR), which has a fast neutron flux of approximately  $104n/(\text{cm}^2 \text{ s})$  and at Idaho National Laboratory's (INL) Neutron Radiography Reactor (NRAD), with a fast neutron flux of  $107n/(\text{cm}^2 \text{ s})$ . Grayscale values of the fast neutron images are utilized to determine the relative light output, while the Modulation Transfer Function (MTF), derived from the images is used to calculate the spatial resolution. These two properties are used to determine the optimum fast neutron imaging configuration. Additional scintillator materials were also subjected to NRAD's fast neutron beam and their imaging properties compared with PVT. Various phantoms are also imaged to demonstrate fast neutron imaging's practical advantages in real-world scenarios.

### Speaker Sessions and Seminars / 155

## Permeability changes when using waste materials to generate acid resistant mortars, neutron investigations.

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Cements (mortars and concrete) are, despite their wide spread use, susceptible to chemical attack and weathering. Cements are particularly susceptible to acid attack, which leaches Ca and Al from the cement paste and lowers paste pH, thereby weakening the cement matrix. Similarly, sulfate in waters can also interact with the cement matrix [1,2], where several reactions occur. Firstly, sulfate reacts with Ca in the system to precipitate gypsum, which removes the Ca from the primary role of forming Calcium-aluminates, and -silicates that provide the cement strength. Secondly, sulfate may interact with the aluminate in the cement to form a Calcium-alumino-sulfate (ettringite) [1,2]. Ettringite, is a low-density mineral (1.8 g/cm<sup>3</sup>), hence when higher-density cement minerals (2.2 g/cm<sup>3</sup>) are mobilised to form a low-density ettringite, cement expansion and cracking occurs. Therefore, ettringite formation weakens the cement paste, and rapidly deteriorates cement (mortar and concrete) performance and life expectancy. Consequently, in sulfate-rich areas such as acid sulfate soils, seawaters, and many saline soil environments, specialist cements are often required that circumvent the sulfate attack [1,2].

However, waste materials like coal fly-ashes, high-pH bauxite refinery residues, blast furnace slags, have been suggested for incorporation often substituting cementing (pozzolanic) materials, or have been suggested as pore fillers, such as meta-kaolin, to prevent sulfate penetration [2]. We looked at two large volume waste materials, seawater neutralised bauxite refinery residues (Bauxsol™) and high temperature co-generation sugar cane bagasse ash (SCBA), in mortars. Both waste materials when used as sand replacements in the mortar, improved the acid resistance, including strength retention, and decreased spalling. However, neutron imaging indicates that while Bauxsol™ decreased permeability, SCBA increased permeability consistent with chloride ingress testing [3]. Laser ablation inductively coupled plasma mass spectrometry showed limited sulfate penetration to mortars containing Bauxsol™, while XRD and visual inspections showed surface depositions of acidic sulfato-salt (e.g., alunogen, Jarosite-like minerals, and iron hydroxy sulfates [green-rusts]) [1,2]. Data collected would therefore suggest that Bauxsol™ incorporations within the mortars actively worked to decrease sulfate ingress, restricting gypsum and/or ettringite formation through pore-blocking, which may or may not have been accompanied by shifts in cement paste chemistry. Whereas, SCBA inclusion within mortar mixes, although increasing permeability, increased sulfate resistance most likely through a shift in cement chemistry away from Ca-aluminates (e.g., tricalcium aluminate; C3A) toward Ca-silicates (e.g., di-calcium silicate; C2S), despite the deactivation of silica within the SCBA from the high burn temperatures [3,4]. Unfortunately, for both SCBA and Bauxsol™ the XRD evidence on cement chemistry shifts remain inconclusive.

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**Speaker Sessions and Seminars / 81**

## **Development of a Line-Pair Gauge and Standard Test Method for Measuring Basic Spatial Resolution of Neutron Imaging Systems**

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Standards are required for commercial and quality-controlled processes. All current standards for neutron radiography are intended for use with film, but there are currently no standards that technically apply to modern digital neutron radiography systems. Trends in the neutron imaging community show a move towards digital systems that have many advantages compared to traditional film neutron radiography methods. Digital neutron imaging systems are widely used for research applications with great success. Unfortunately, the lack of applicable standards has hindered use of modern digital neutron imaging systems for industrial applications. Standards that apply to digital systems would allow use of advanced digital systems for commercial applications that require quality control with standards.

Members of the American Society for Testing and Materials (ASTM) E07.05 Committee for Neutron Radiography are developing a standard test method and device for measuring basic spatial resolution and total image unsharpness that would apply to any neutron imaging system. Line pair gauges, such as the duplex-wire gauge described in ASTM E2002, are image quality indicators frequently employed in x-ray and gamma radiography to establish basic spatial resolution. The ability to discern two closely spaced lines on the images of the device is related to the image unsharpness and basic spatial resolution of the imaging setup. Current efforts to develop a line-pair gauge are based on the same approach used in ASTM E2002, but with materials suitable for use with neutrons instead of x-rays.

The E07.05 Committee composed an initial testing procedure, and prototype line-pair gauges were designed and fabricated for validation studies to determine the suitability of this device as a new ASTM standard. The proposed method accommodates neutron images produced with any neutron image acquisition method using neutron beam lines with cold or thermal neutron spectra. It would cost nearly the same as the sensitivity indicator and beam purity indicator devices described in ASTM E545, which are already in wide use in the neutron imaging community. The gauge is small (25 mm by 50 mm) to maximize the field-of-view available for objects being examined. The gauge uses gadolinium to absorb neutrons on a 3-mm thick substrate of relatively neutron-transparent glass, with the line pairs laser etched from the gadolinium. Measurements using the gauge are easy and straightforward to perform, yet provide meaningful image quality information.

The committee has completed the first set of round robin testing, which included multiple facilities with a wide range of imaging systems. Each facility acquired images using the test procedure and provided the resulting radiographs to the committee along with comments and input for improvements. Overall, the approach seems promising, and a second prototype is being designed based on lessons learned in the first round robin tests.

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### **First neutron computed tomography with digital neutron imaging systems in a high-radiation environment at the 250 kW Neutron Radiography Reactor at Idaho National Laboratory**

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The Neutron Radiography Reactor (NRAD) at Idaho National Laboratory (INL) was designed for epithermal neutron radiography for examination of highly-radioactive irradiated nuclear fuel elements. Radioactive samples are remotely lowered into the East and North Radiography Stations (ERS and NRS, respectively), and a rail transfer system remotely positions radiography cassettes into the detector position for indirect radiography. The indirect transfer method with film has been used at NRAD for around forty years, but recent efforts seek to develop digital camera-based neutron imaging systems. Two initial camera detector systems were built using an inexpensive but high-quality scientific CMOS camera with robust shielding, and tests were performed in collaboration with Heinz Maier-Leibnitz Institut of Technische Universität München.

The first tests were performed in 2017 in the NRAD's ERS using a 10 cm field-of-view camera-based system with an inexpensive scientific CMOS camera shielded by lead bricks and borated polyethylene plates. The camera and motor stages were controlled by a Raspberry Pi computer. The first series of digital neutron images was successfully acquired, but radiation field was so high that the Raspberry Pi computer crashed after acquiring only 44 images despite being shielded behind a 10 cm layer of lead bricks.

A much improved camera box was designed based on lessons learned from the efforts in 2017, which was constructed and installed in NRAD's NRS in 2018. This imaging system included a two-mirror architecture with a longer optical path to reduce scattered radiation to the camera, more robust radiation shielding, and a translation stage for remote focusing of the camera lens. A downscaled version of the ANTARES instrument control at MLZ was installed using a Laptop and three Raspberry Pi computers to control the imaging system components. The very first digital neutron computed tomography at INL was successfully acquired, consisting of 420 neutron radiographs acquired in 4 hours. These first tests with camera-based neutron imaging systems have demonstrated the potential to both increase the throughput of radiography by an order of magnitude and provide higher quality spatial information with three-dimensional tomographic reconstructions compared to two-dimensional radiographic projections, which represent a significant improvement compared to current film radiography capabilities.

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## **Gamma Discriminating Scintillation Screens for Digital Transfer Method Neutron Imaging with Camera-Based Systems**

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Trends in the neutron imaging community show a move towards digital systems that have many advantages compared to traditional radiography, including higher detection efficiency, position stability and flexibility in the field-of-view, high spatial resolution, and potentially real-time imaging capabilities. The most common camera-based neutron imaging systems observe light from <sup>6</sup>LiF/<sup>65</sup>ZnS scintillator screens placed directly in the neutron beam. Unfortunately, these screens are sensitive to gamma radiation, which may preclude their use for examining highly-radioactive objects such as irradiated nuclear fuel.

A collaborative project between Idaho National Laboratory (INL) and Paul-Scherrer Institute (PSI) is investigating a new type of scintillation screen that uses ZnS with a dysprosium converter instead of Li-6. While Li-6 decays promptly upon absorption of a neutron, Dy-165 decays over time according to a half-life. Neutron absorption in Dy-164 produces Dy-165 (T<sub>1/2</sub>=2.33 h) and Dy-165m (T<sub>1/2</sub>=1.26 m) which both decay by beta emission. Such a screen exposed to a neutron beam creates a latent image by neutron activation of the dysprosium in the scintillator screen. The activated screen is

then transported into a camera box that is shielded from the gamma radiation from the beam and radioactive specimen. The scintillator screen continues emitting light from decay of the activated dysprosium and a camera integrates the digital image over time. Such an imaging system combines modern camera-based system architecture with the approach of traditional indirect transfer method radiography.

A set of 23 scintillator screens were fabricated with various material ratios, thicknesses ranging between 30-225  $\mu\text{m}$ , substrate material (aluminum or dysprosium metal), and phosphor granularity. Experiments performed at PSI's NEUTRA beam line measured the light output of each screen for exposure times ranging between 1-20 minutes and decay times up to 20 minutes. The resulting data is processed to calculate each screen's doubling time during exposure, signal decay half-life, and total integrated light output during decay. Additionally, images were acquired of a Siemens star test pattern for spatial resolution measurements.

The results show for the first time that the combination of dysprosium with a scintillation material like ZnS can produce light which is measurable under common camera-based detection conditions and that neutron radiographic images of reasonable quality can be produced. The light signal decay half-lives were on the order of 1 minute, shorter than expected based on the half-lives of Dy-165, which may be caused by unidentified short-lived radionuclides. The resolution was also poorer than expected at 350  $\mu\text{m}$ . Potential improvements and additional converter materials may be investigated in the future that could increase the light output and spatial resolution.

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## Digital neutron radiography of irradiated nuclear fuel with an MCP/Timepix detector

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The Neutron Radiography Reactor (NRAD) beneath the main hot cell of the Hot Fuels Examination Facility (HFEF) at Idaho National Laboratory (INL) provides neutron radiography capabilities for evaluation of irradiated nuclear fuel. Neutron radiographs are currently acquired using the transfer method in which activated converter foils are coupled to x-ray film or phosphor image plates. While this process produces high-quality neutron radiographs with a large field of view, it is expensive and time consuming. Furthermore, volumetric and other advanced neutron imaging methods (e.g. element-specific, phase contrast, Bragg edge imaging) are nearly impossible without modern digital imaging techniques that would significantly expand and improve the quality of data that can be extracted from neutron beamline examinations. A collaborative project between INL and UC-Berkeley is investigating the possibility of performing neutron radiography of spent fuel assemblies at INL with a high-resolution, neutron-sensitive microchannel plate (MCP) detector.

Initial scoping tests were performed to determine whether imaging of irradiated fuel would be possible before subjecting the detector to such a rigorous test. The stability of the detector in the high radiation environment of a radial neutron beamline, demonstrating that the achievable neutron contrast should be sufficient despite the high gamma radiation background (>2 Sv/hr) and epithermal neutron content of the neutron beam. Detector response and stability under intense 662 keV and 1.2 MeV gamma radiation (exceeding dose rates of 6.5 Sv/hr at the detector position) were also calibrated with bright Cs-137 and Co-60 isotopic sources. At the same time it is verified experimentally that the optimization of the achievable contrast will be possible by implementation of lead filters and calibration of detector parameters.

The MCP detector was then installed in the East Radiography Station (ERS) behind an elevator that remotely positions radioactive samples from the HFEF main cell above into the ERS neutron beam

for radiography. An irradiated fuel pin lowered into position was measured to produce a dose rate of 5.5 Sv/hr to the MCP. Despite this high dose rate, neutron radiographic images were successfully acquired that exhibited high spatial resolution and signal-to-noise ratio. Additionally, a real-time neutron video was acquired at 20 fps showing the fuel pin moving vertically in the elevator. These tests demonstrate that MCP detector systems can examine irradiated nuclear fuel samples. Much higher dose rates (>100 Sv/hr) emitted by other available samples would diminish the signal-to-noise ratio, but examination of such samples should be possible. These promising results are not limited to the studies of spent fuel assemblies, but are also relevant to any experiment where imaging needs to be performed in a high radiation environment.

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## Reactivation of the Transient Reactor Test (TREAT) Facility Neutron Radiography Program

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The Transient Reactor Test (TREAT) Facility is a 19,000 MW reactor located at the Idaho National Laboratory designed to perform transient testing on nuclear fuel experiments and materials. A neutron radiography facility was installed with the first radiograph being produced in 1967 that utilized the indirect (or transfer) method of neutron radiography to examine irradiated nuclear fuels and experiment test loops. The TREAT neutron radiography facility performed approximately 5,000 radiographs by the spring of 1977. The original radiography facility was upgraded in 1974-1975 to correct previous deficiencies and to improve capabilities. In 1986, an upgraded aperture was installed to increase the resolution of images followed by a major reactor upgrade completed in 1988. Overall, the existing neutron radiography system was in good working order. Originally built in 1958, the TREAT Facility was in operation until it was placed in a shutdown status in 1994. Following the Fukushima disaster and seeing a need for enhanced accident tolerant fuels, the United States Department of Energy decided to restart the TREAT facility and resume transient operations. In November 2017, the TREAT reactor was successfully restarted and is currently performing operational testing in preparation for initial experiment irradiations and transient testing.

This paper reports on efforts to reactivate the TREAT neutron radiography station. A few minor shielding and system improvement upgrades have been completed along with the installation of a dedicated room for computed radiography equipment. Detailed beam characterization work was not performed following the last reactor upgrade. To characterize the neutron beam, gold foil activation measurements were made to determine an average neutron flux and flux profile. Additional multi-foil activation measurements will be made to calculate the energy spectrum. An open beam image will be taken that will provide the information about variations in the beam profile. A series of system qualification shots will also take place to accurately determine the effective image quality and image acquisition parameters. Full operational status of the TREAT neutron radiography system is scheduled for late summer of 2018.

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## Test Abstract

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test abstract

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## **Applications of Fast Neutron Radiography to Fluid Flow and Tumbling Media**

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This investigation highlights the use of fast neutron radiography (FNR) as a technique to determine the intrinsic properties of dynamic media.

The inherent property of sand, the hydraulic conductivity, is determined using the constant head method. Through the attenuation of the fast neutrons by water, we see the evolution of the water front with time and determine important parameters from the radiographs. These parameters are employed into Darcy's law and Gardner's equation for the calculation of the hydraulic conductivity which shows how fast neutron radiography can yield unique information of the live process of water absorption through sand.

The high penetrability of fast neutrons is also used to determine the steady state of dynamic flow of grinding media within a tumbling mill. Tumbling mills are a pivotal part of the comminution process, enabling one to increase the surface area of materials as well as releasing entrapped materials from the crush casing. The shape of the internal mill charge during its dynamic flow can be used to calculate important mill parameters, which are used to infer the optimal speed for the best comminution of the mill charge. Key aspects of the motion of the mill charge in a rotation phase, help one obtain the optimal rotation speed required for maximum comminution. Fast neutron radiography (FNR) is used to obtain the parameters related to the best grinding conditions

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## **Overview of the Conceptual Design of the Upgraded Neutron Radiography Facility (INDLOVU) at the SAFARI-1 Research Reactor in South Africa.**

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The value added by neutron beam line facilities through research of is evident from the number of new facilities planned and commissioned worldwide. In order to provide local and international researchers with world-class capabilities, Necsa embarked on the upgrade of the neutron beam line instruments at the SAFARI-1 nuclear research reactor, which entails inter alia a complete functional neutron diffraction facility. The concept design of an upgraded neutron radiography (NRAD) beam

line named INDLOVU (Zulu name for Elephant – one of the “Big Five”) (“Imaging Neutron Device to Locate the Obscure and Visualise the Unknown”) has been finalised and the documentation for legal requirements, safety, electronic and control systems are in the approval stage, thereafter facility assembly will commence.

The upgraded NRAD facility will be unique in its application format as it can perform, through selective filtering, not only thermal neutron radiography but also utilise individually, the full radiation beam, the intermediate or fast neutron spectrum as well as the gamma-ray component of the radiation beam. As the beam port is positioned axial to the reactor core, a maximum radiation flux of  $1 \times 10^9$  neutrons.cm<sup>-2</sup>.s<sup>-1</sup> is envisaged, without filtering and when utilising the full radiation beam. The traditional scintillator-mirror-CCD camera concept, mounted inside a light tight box as detection system, is adopted. The CCD camera will be able to focus on an interchangeable field of view from  $5 \times 5$  cm<sup>2</sup> to  $35 \times 35$  cm<sup>2</sup> on the back of the scintillator screen. The detection system comprises of application specific (as determined by radiation sensitivity) exchangeable scintillation screens and the CCD camera is equipped with an automatic focusing capability.

INDLOVU comprises of a number of subsystems and components inter alia such as the processing systems (e.g. shutters, collimators, radiation filters, beam and flight tubes, experimental), safety systems (e.g. shielding, conventional and radiation safety), control system (e.g. DACS, PLC), utilities (e.g. electrical, HVAC) and sample management system (e.g. sample receiving, storage, dispatch and data management). This presentation will describe the design of the South African INDLOVU NRAD facility with respect to each of the subsystems in terms of their design, functionality, importance and operational interconnection with each other. In addition, to evaluate the performance of the facility in terms of expected radiation beam intensity and quality, neutron ray tracing simulations of the attainable flat field at the detector plane, for each of the 4 different L/D ratios (125, 250, 400 and 800), will be compared to theoretical design calculations.

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## Upgrade of the Neutron Radiography Set-Up at the JEEP-II reactor

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The JEEP II research reactor (2 MW thermal power) at the Institute for Energy Technology (IFE) in Kjeller (Norway) is presently the only neutron source in the Nordic Countries. A national infrastructure project, funded by Research Council of Norway, to upgrade the neutron scattering facilities, NcNeutron, is currently running (2016-2020). NcNeutron aims at establishing a neutron research and technology exchange center and being a Norwegian and regional home-laboratory in neutron-based science.

The upgrade of the facilities includes the reconstruction and modernization of the neutron radiography instrumentation which was built in the mid-1970s. The current setup is based on a traditional Dy-foil technique with a relatively high spatial resolution (40-50  $\mu$ m) and an L/D ratio of about 240. It is limited by a low maximum number of recorded images per day and a man-hour intensive image processing. The facility is presently dedicated to the analysis of post-irradiation examination data from safety and integrity tests of nuclear fuels performed within reactor safety research projects.

The neutron radiography upgrade consists of three main tasks: (i) beam optimization (new sapphire and bismuth filter, new aperture with diameter of 20 mm, and new in-pile collimator); (ii) modification of the current radiography cell, implementing different configurations for the analysis of radioactive and non-radioactive samples; (iii) installation of new sample stage and new digital neutron imaging detector.

The new neutron imaging instrument (NIMRA) is designed to have an L/D ratio of about 300 and a FOV of 15 cm x 15 cm. It will be intended for studying energy materials (e.g. hydrogen storage systems, fuel cells and electrolyzers, heat storage units) as well as flow in porous media (e.g. clays, concrete). Applications within other material classes and systems will be actively pursued.

## Functional 3D structures made by additive manufacturing

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Additive manufacturing (3D printing) provides a new freedom in materials design. In our recent research work, metallic and ceramic structures have been prepared by different 3D printing techniques including selective laser melting, extrusion and digital light projection. X-ray CT and Neutron CT has been used for structural examination. In combination of wet-chemical processes (thermal decomposition and electroplating), catalyst materials have been coated on the surface of 3D structures for various applications. High oxygen evolution reaction (OER) performance has been observed indicating the great potential of 3D printing in fabrication of highly efficient catalyst electrode.

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## Large area MCP-based neutron imagers

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Neutron imaging detectors based on neutron-sensitive microchannel plates (MCPs) were constructed and tested at beamlines of thermal and cold neutrons. The MCPs are made of a glass mixture containing enriched boron and natural gadolinium, which makes the bulk of the MCP an efficient neutron converter. Contrary to the neutron-sensitive scintillator screens normally used in neutron imaging, spatial resolution is not traded off with detection efficiency. While the best neutron imaging scintillators have a detection efficiency around a percent, a detection efficiency of around 50% for thermal neutrons and 70% for cold neutrons has been demonstrated with these MCPs earlier.

In our tests we coupled a neutron-sensitive MCP to a phosphor screen which was read by a low-noise CMOS camera. Images of a gadolinium test mask designed for this purpose show a limiting resolution of about 50  $\mu\text{m}$ . We will show images and tomographic reconstructions made with thermal and cold neutrons.

A first prototype of this concept had a modest size of 40 mm active diameter. A new unit is now available with a 100 $\times$ 100 mm<sup>2</sup> active area. This detector does not have the limitations in rate capability and active area coverage that are seen in imaging detectors with electronic readout structures, while being orders of magnitude more sensitive than other detectors with optical readout like scintillators. Also the afterglow known from neutron imaging scintillation screens is completely absent.

The phenomenal detection efficiency over the large active area will change the field of neutron tomography. Where nowadays it is common to acquire ~800 projections in about a day of exposure, our detector can complete this in about an hour. The fact that many times less neutron flux is integrated to attain a certain image quality also means that samples activate less, proportionally to exposure time. Rare artifacts and valuable museum pieces can be imaged and still return to their owner. Small, low power nuclear reactors running on conventional low-enriched uranium become suitable neutron sources for imaging. The images in this study taken at the research reactor in Delft are a case in point. We are exploring the possibility of neutron imaging with neutron generators, which may take neutron imaging from large scale user facilities to labs in academia and industry.

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# INVESTIGATION OF HYDROMECHANICAL PROCESSES IN POROUS ROCK USING 4D NEUTRON IMAGING

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## INTRODUCTION

The characterization of localized deformations and its effects on the permeability of rocks is fundamental to a number of resource engineering challenges, e.g., hydrocarbon and water production and CO<sub>2</sub> sequestration. However, the complexity of performing conclusive experimental campaigns to analyze the hydro-mechanical behavior of porous subsurface rocks leads to a lack of necessary ground truth to develop analytical and numerical models.

In this work the coupling of triaxial deformation and the evolution of fluid flow in porous rocks (in particular sandstone) is explored using high-speed neutron imaging. Neutrons are highly sensitive to hydrogen, providing the ideal probe for detecting fluids (e.g., water and oils) in dense porous materials such as rocks [B1]. Furthermore the property of neutrons to distinguish between isotopes allows to use deuterated (heavy) water, which attenuates the beam less than the light water, as contrast agent. In this way it is possible to track the front between two fluids which have similar flow properties but very different neutron interactions.

## EXPERIMENTAL METHOD

The experimental campaign was performed at the Cold Neutron Tomography and Radiography (CONRAD-2) [B2] instrument at Helmholtz Zentrum Berlin (HZB) where it was possible to acquire fast tomographies in 1 minute.

The samples were deformed ex-situ in a triaxial apparatus in Laboratoire Sols, Solides, Structures, Risques (3SR, Grenoble). X-ray tomographies were acquired before and after the triaxial loading to obtain the strain fields through Digital Volume Correlation (DVC) [B3]. During the experiment light water was flushed through the sample while fast tomographies were acquired. Therefore, a relation between deformation and changes in permeability field can be analyzed.

In order to be able to image the advancing fluid front and quantify its velocity in 3D, the experimental setup controls the pressure on the top of the sample, the confining pressure and the water flow rate while measuring the volume of the water leaving the sample and the pressure on the bottom of the sample.

## RESULTS

Five samples of Vosges sandstone were deformed under triaxial conditions at 30 and 40 MPa confining pressure and loaded to different levels of axial strain to be able to study the changes in permeability with different degrees of deformation. An example of the strain fields determined using DVC analysis of the x-ray tomographies acquired before and after the loading is shown in Figure 1. Figure 2 shows an example of a neutron tomography image of the light water advancing into the heavy-water saturated sample; the image has been thresholded to show just the light-water in the sample.



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## Imaging investigation of Chinese bimetallic sword fragment from 2nd-1st century BCE

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Scientific investigations and archaeometric studies have played a major role in the field of archaeology, especially with regard to materials transformed through human activity, like metals. Metals are generally investigated through metallography and Scanning Electron Microscopy (SEM), which required sampling or surface preparation. Neutron techniques instead are able to provide the bulk properties of metals in a non-invasive way.

In this work we present a neutron imaging study of a Chinese bimetallic sword fragment from 2nd-1st century BCE. In particular, white beam Neutron Tomography (NT) and Neutron Resonance Transmission Analysis (NRTA) have been applied, using the IMAT and the INES beamlines of the ISIS pulsed neutron source in the UK, respectively.

The earliest example of bimetallic weapons in China dates as early as the Shang Dynasty (1600–1100 BCE), where meteoric iron and bronze were combined to forge weapons 1. With the discovery of iron smelting technology during the Spring and Autumn Period (770–473 BCE), bimetallic swords with bloomery iron and bronze became more common 2. They have been found in many parts of central China.

The sword fragment investigated has an iron blade mounted on a studded bronze grip (probably for a twine binding) and a ricasso with three long spikes protruding on each side. The object resembles two published examples with similar form of hilts [3, 4] listed as originating from burials investigated in the mountainous regions of Longpaozhai, in the Min River Valley (Central Sichuan), dating from the 2nd or 1st century BCE. Similar swords are also found further north and may have been introduced from further west.

NT allowed us to study the inner morphology of the sword, revealing details of its conservation status and the forging and/or casting of the different components. NRTA provided a 2D map of the elemental composition of the artefact, indicating the nature of the bronze alloy of the grip (whether tin bronze, leaded tin bronze, or arsenical tin bronze) and of the iron blade.

The study presented was complemented by Neutron Diffraction, Neutron Resonance Capture Analysis (NRCA), and negative muons, providing a full characterisation of the object in terms of alloy composition, microstructural characterisation and elemental information, in a non-destructive way.

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### Speaker Sessions and Seminars / 156

## Performance and resolution upgrade on DINGO at OPAL

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The neutron radiography / tomography / imaging instrument DINGO is operational since October 2014 to support research at ANSTO 1. DINGO had a high subscription rate from a broad national and international scientific user community and for routine quality control for defense, industrial, cultural heritage and archaeology applications. DINGO provides a useful tool to give a different insight into objects because of different contrast compared to X-rays and high sensitivity to light elements. In the field of industrial application it has shown promising results for studying cracking and defects in concrete or other structural material. A major part of applications from both sides of the community, research and industrial user, was demanding the high resolution setup on DINGO. In the original design DINGO could provide a minimum pixel size of 27  $\mu\text{m}$ . The neutron beam size can be adjusted to the sample size from 50 x 50 mm<sup>2</sup> to 200 x 200 mm<sup>2</sup> with a resulting pixel size from 27 $\mu\text{m}$  to ~100 $\mu\text{m}$ . The measured flux (using gold foil) at this high resolution setup for an L/D of approximately 1000 at HB-2 is  $1.1 \cdot 10^7$  [n/cm<sup>2</sup>s], which is in a similar range to other facilities. Depending on the sample composition a full tomography has been taken in 24 – 36 hours with a 50  $\mu\text{m}$  thin ZnS/6LiF-screen and the CCD (Andor IKON-L) camera. In a two stage upgrade the background radiation has been reduced by an additional slit system adjusting the beam size more flexible and further down to 0.5 x 0.5 mm<sup>2</sup>. The new system allows minimizing the beam according to the sample size. In combination with the Andor IKON SCMOS and Kenko distance rings, to increase the focal length of the existing 100mm lens the pixel size was reduced to 7 $\mu\text{m}$ . The scintillator was a 10  $\mu\text{m}$  thick Gadox screen and for each projection we have taken 3 – 6 images for better white spot correction. We would like to present first radiography and tomography results using the new setup [2,3]. A full tomography under these conditions can be taken in 2 -4 days depending on the nature of the sample.

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### Speaker Sessions and Seminars / 83

## Designing a Fast-Gated Scintillator-Based Neutron and Gamma Imaging System

Verena Geppert-Kleinrath<sup>1</sup>; Matthew Freeman<sup>1</sup>; Frank Merrill<sup>1</sup>; Petr Volegov<sup>1</sup>; Carl Wilde<sup>1</sup>

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The Los Alamos National Laboratory Advanced Imaging Team is designing two novel neutron and gamma imaging systems being built to image inertial confinement fusion processes at the National Ignition Facility. While the immediate application of the design is in fusion diagnostics, the lessons learned will be transferable to any fast-gated radiographic imaging system. The stringent requirements for the detectors include sub-millimeter spatial resolution, sufficient cross section to allow neutron imaging at  $10^6$  neutrons/cm<sup>2</sup> in total, efficient light collection, and stable noise properties. Since the systems will be gated to allow the collection of frames at different neutron energies, fast scintillator timing characteristics in the nanosecond range and minimal secondary decay are a must. A comprehensive study of scintillator materials at two different neutron sources, the Los Alamos Neutron Science Center and the OMEGA laser facility in Rochester, NY, have influenced key design decisions. The recently concluded experimental campaigns have shown the benefits of lens-coupled monolithic scintillator systems over pixelated fiber arrays. Ongoing work includes the custom design of telecentric large aperture lenses required for the novel systems.

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## Neutron Beam Flux and Energy Spectrum Characterization at the Neutron Radiography Reactor at the Idaho National Laboratory

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The Neutron Radiography Reactor (NRAD) is a 250 kW TRIGA reactor that includes two radial neutron beamlines designed for neutron radiography of irradiated nuclear fuel at Idaho National Laboratory (INL) in the United States. Previous efforts have characterized these neutron beams to inform instrument scientists and users about the neutron beamline. However, reactor operations have since changed the way the control rods are withdrawn from the reactor to control reactor power, potentially changing the power profile of the reactor and, thus, the neutron flux and beam tilt of the neutron beamlines. Therefore, the neutron beams must be re-characterized for neutron flux, flux profile, cadmium ratio, and neutron energy spectrum. The resulting image quality of neutron radiographs is also measured because these beams are primarily used for neutron imaging applications and these beamline characteristics impact the radiographs produced. This paper reports the completed measurements made at the East Radiography Station (ERS) neutron beam.

The thermal neutron flux of the ERS beam measured using gold foil activation is  $9.61E6 \pm 1.47E5$  n/cm<sup>2</sup>/s with a relatively uniform profile across the image plane. This neutron flux is 58% higher than measured values before the changes in control rod withdrawal approach. The cadmium ratio measured using bare and cadmium-covered gold foils is  $2.04 \pm 2\%$ , which is considerably lower than other major thermal neutron imaging facilities and indicates very high epithermal neutron content. The ERS neutron radiography system is a Category I facility as determined by American Society for Testing and Materials (ASTM) standards.

The neutron energy spectrum was determined using multi-foil activation measurements and subsequent unfolding of the measured results using the Unfolding with MAXED and GRAVEL (UMG) software package. An energy spectrum calculated using the Monte-Carlo N-Particle (MCNP) radiation

transport code provided an initial spectrum for the unfolding algorithms in the UMG software. The neutron energy spectrum calculated with UMG and MCNP based on foil activation measurements is then implemented into an MCNP model of the ERS neutron beam, which provides a valuable tool for future studies such as beamline modifications, expected behaviors of materials in the beam, beam conditioning approaches, radiation shielding considerations, and neutron imaging system design.

#### Speaker Sessions and Seminars / 90

### Bragg-edge Neutron Strain Tomography

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Bragg-edge residual strain tomography has been achieved for the first time in general two-dimensional systems. This approach allows the reconstruction of detailed stress and strain distributions within polycrystalline solids from sets of Bragg-edge transmission strain images.

In contrast with traditional scalar tomography, this problem is ill-posed due to an issue surrounding the uniqueness of solutions - infinitely many strain fields can give rise to the same set of Bragg-edge images. Work over the last decade has provided some solutions to this problem for a limited number of special cases. Our approach to this problem was to develop a reconstruction algorithm for arbitrary systems based on a least squares process constrained by equilibrium.

This presentation will outline this approach and provide details of an experimental demonstration on two samples using data from the RADEN instrument at the J-PARC spallation neutron source in Japan. Validation of the resulting reconstructions is provided through a comparison to conventional constant wavelength strain measurements carried out on the KOWARI engineering diffractometer within ANSTO in Australia.

#### Speaker Sessions and Seminars / 34

### Experimental Validation of the Model Connecting Time, Contrast Wavelength and Spatial Resolution

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A model describing the connection between time, spatial, wavelength and contrast resolution was presented at ITMNR-8 in 2016 1. Resolution limits caused by the sample contrast were derived from the model. The resolution of neutron imaging measurements is limited by practically available illumination time. The time needed for radiography and tomography measurements depends on the fifth and sixth power of the spatial resolution for radiography and tomography measurements, respectively. A general limitation is reached if illumination times of several days per image or tomogram are reached.

Neutron radiographs and a neutron tomography were measured at the BOA and POLDI beamline at SINQ (Paul Scherrer Institut, Switzerland) as well as at the ANTARES beamline at FRM-2 (TU Munich, Germany) to validate the model. Test specimens consisting of an aluminium frame with gold ( $\Sigma_{\text{total}} = 6.28 \text{ cm}^{-1}$ ) and hafnium ( $\Sigma_{\text{total}} = 5.12 \text{ cm}^{-1}$ ) wires with a thicknesses of 75 and 125  $\mu\text{m}$ , respectively, as well as copper ( $\Sigma_{\text{total}} = 1.00 \text{ cm}^{-1}$ ) wires with thicknesses of 20, 50, 75 and 125  $\mu\text{m}$  were illuminated at various times and collimations. The effective pixel size of the detectors applied was adapted by pixel rebining to fit the detector resolution to the wire thickness.

The results will be compared with the predictions of the model. Whereas the hafnium wire becomes visible after few seconds, The 20  $\mu\text{m}$  thick copper wire was not visible even after 6 h illumination time at ANTARES with a collimation of 800 in terms of L/d. The gold, hafnium and the copper wires with 125 and 75  $\mu\text{m}$  thicknesses are visible in the reconstruction of a tomography measured at Antares with 420 projections each 40 s illuminated.

The validity of the model premises are checked. The differences between visual and numerical analysis as well as the practically reachable resolution limits depending on the sample contrast will be discussed.

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## Feasibility Tests to Measure the Correlation between Hydrogen Concentration and Elastic Strain in Zircaloy-4

Mirco Grosse<sup>1</sup> ; Douglas Syzdek<sup>2</sup> ; Marco Conte<sup>2</sup> ; Mario Walter<sup>3</sup> ; Burkhard Schillinger<sup>4</sup>

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Mechanical properties of zirconium alloys are affected by absorbed hydrogen. One of the negative processes which can occur is the so-called delayed hydride cracking (DHC). DHC describes hydrogen diffusion to the strained region ahead of a crack tip due to the lower chemical potential in the strained lattice compare to the unstrained one. The hydrogen becomes enriched ahead of the crack tip and embrittles this region. The crack grows into this region even the mechanical load keeps constant. The crack growth results in a stress/strain release of the region formerly ahead of the crack tip. Therefore, the chemical potential at this position increases and the hydrogen distribution is no longer in equilibrium. To establish new equilibrium, the hydrogen has to diffuse to positions ahead of the new crack tip position. The process is repeated until rupture of the component. To model this phenomenon the dependence of the hydrogen chemical potential on the elastic strain has to be known.

Neutron radiography and tomography experiments were performed at the ANTARES beamline at FRM-2 (TU Munich, Germany) to check the feasibility of determining this dependence. The hydrogen distributions were measured at four 3-point-bending specimens pre-loaded with hydrogen and bended until the onset of plastic deformation at two temperatures at which the  $\beta$ -phase of zirconium is stable. In one specimen a clear gradient in the hydrogen concentration was detected. The hydrogen distribution was correlated to the estimated elastic strain distribution.

The results of the feasibility measurement open the door to investigate the dependence of the hydrogen chemical potential on strain and temperature.

## Neutron Radiography at SARAF: from Reactor to Accelerator

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Soreq Applied Research Accelerator Facility (SARAF) will be a user facility for basic and applied nuclear physics, upon expected completion at the beginning of the next decade. SARAF is based on a 40 MeV, 5 mA CW proton/deuteron superconducting linear accelerator. A high intensity accelerator-based Thermal Neutron Source (TNS) will be a major application of SARAF within its higher goal to enhance and back-up Soreq IRR-1 5 MW nuclear research reactor, mainly for neutron imaging and neutron diffraction research. The current thermal neutron radiography system of IRR-1 was characterized at the imaging plane in order to determine the neutron flux, beam profile, cadmium ratio and gamma background. The image quality was examined based on American Society for Testing and Materials (ASTM) standards. The main characteristics found: neutron flux is  $6-9 \times 10^5$  n/s/cm<sup>2</sup> and cadmium ratio of 10-15, with collimation ratio (L/D) of 250. SARAF TNS is designated to provide an accelerator based neutron radiography system with equivalent or upgraded capabilities compared to IRR-1. The TNS will be based on a liquid lithium conversion target, generating a fast neutron yield of up to  $2 \times 10^{15}$  n/s when irradiated with a 40 MeV, 5 mA (0.2 MW) deuteron beam. The produced fast neutrons will be moderated to the thermal energy range by heavy water that surrounds the conversion target, along with a beryllium multiplier which enhances the number of neutrons, and a peripherals neutron reflector. Extraction tube toward the radiography systems will be positioned at backward angles with respect to the incident deuteron beam in order to diminish the contribution of fast neutrons. The dimensions of the moderator, multiplier and the tubes position are investigated by detailed Monte-Carlo simulations and a preliminary design of the radiography system has been established. The simulation results will be presented and they indicate that the neutron beam characteristics at the imaging plane will be improved compare to those of IRR-1 facility.

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## Demonstration of a biaxial tensile machine for a neutron Bragg-edge imaging measurement

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A biaxial tensile machine reproduces the stress state close to actual press molding and is effective for studying the material properties under biaxial stress. Thus it greatly contributes to the reduction of press molding failure 1. The relation between a moldability of a material and microstructure, such as strain, phase distribution, crystallite size, texture, dislocation density, and so on, has been noted, and studies by the neutron scattering method are proceeded. However, in the neutron scattering method, it is difficult to visualize two-dimensional distribution of the microstructure, so we have developed a new technique combining a neutron Bragg-edge imaging technique which enables visualization of the two-dimensional distribution of the microstructure with the biaxial tensile testing machine. For development of this method, we developed a vertical-type biaxial tensile machine and installed it in J-PARC BL22 RADEN which can provides the neutron Bragg-edge imaging experiment 2. In the demonstration measurement at RADEN, a cruciform test specimen, high-tensile steel plate with thickness of 3.2 mm, yield point of 460 MPa, tensile strength of 600 MPa, was used as a sample [3]. Bragg-edge imaging measurements of no-load state, elastic deformation state, plastic deformation state, after unloaded state were carried out on the observation face about 30 mm square at the

center of the cruciform specimen. In the elastic and plastic deformation state, the biaxial stress ratio  $X:Y = 1:1$ , where the rolling direction of the sample taken as the X direction, was applied. From this profile, the two-dimensional distribution of the lattice spacing and the width of the Bragg-edge was obtained. In the loaded state, the compressive strain distribution was observed in the thickness direction of the specimen. The broadening of the width of the Bragg-edge which is thought to be caused by the increase of dislocation density was also observed.

The variation of the compressive strain distribution was larger than the designed value 3% defined in Ref. 3. This is probably because the slippage of crystal surface occurred because the load was held for about 20 hours in order to obtain sufficient statistical precision in the Bragg-edge imaging measurement. These details will be discussed in the presentation.

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## Speaker Sessions and Seminars / 1

# 1” CCD CAMERAS FOR NEUTRON & X-RAY IMAGING

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## Introduction

We will describe three proven applications of 1-inch CCD cameras to neutron and x-ray imaging, as recently provided for Indonesia, Thailand & Malaysia. The 1-inch ICX694ALG is Sony's largest CCD, with high efficiency and exceptionally low noise.

### 250x200 mm neutron/x-ray imaging camera

Our ICX694ALG camera can be compared to the excellent, but much more expensive, Andor and PCO cameras using sCMOS detectors [1,2]. sCMOS, like CMOS, has lower read noise but higher dark current, making it better for fast data acquisition on high flux sources. But there is little advantage for the many users in Universities or Institutes with low flux reactors or generators, where the lower dark current of the CCD, with similar efficiency, is an advantage. Fig.1 shows our camera, a neutron image obtained on a 100 kW Triga reactor and an x-ray image obtained on a 120 kV x-ray source; low flux neutron images were obtained in as little as 5 seconds.

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### 1:1 macro & Laue backscatter cameras

Fig.2 shows our 1:1 macro imaging and Laue cameras. with a backscattered x-ray pattern (center). We use the ICX694ALG for all these types of cameras.

Indico rendering error

Could not include image: Problem downloading image (<http://neutronoptics.com/WCNR11/fig2.jpg>)

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3. We thank the IAEA Vienna for support for developing laboratories

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## PaleoChemistry using Neutron Tomography

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WCNR-11 Abstract

PaleoChemistry using Neutron Tomography

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PaleoChemistry is not just a newly created paleontological field. It concentrates on exploring

We are using multimodal available ways and means to examine the fossils, including such as μ-

It was found thru many neutron tomography scans I did in the past several years, it's an exce-

## Speaker Sessions and Seminars / 40

### HIGH RESOLUTION HIGH ENERGY NEUTRON COMPUTED TOMOGRAPHY AT LANSCE-WNR

James Hunter<sup>1</sup> ; Ronald Nelson<sup>1</sup> ; Michelle Espy<sup>1</sup> ; Doald C. Gautier<sup>1</sup> ; Amanda Madden<sup>1</sup> ; Nicola Winch<sup>1</sup> ; Alicia Swift<sup>2</sup> ; Ray Edward<sup>3</sup> ; Chris Aedy<sup>3</sup> ; Nicholas James Bazin<sup>3</sup> ; Giles Aldrich-Smith<sup>3</sup>

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It has long been recognized that neutrons can complement x-rays for imaging. This is due to their very different attenuation characteristics based on nuclear cross-section, which allows imaging of low Z materials through higher Z materials. Additionally one can use energy dependent Time of Flight (ToF) imaging to exploit phenomenon like nuclear resonances for isotope and element specific imaging. The Los Alamos Neutron Science Center (LANSCE) accelerator is an 800 MeV proton linear accelerator which supplies protons to a range of missions including two spallation neutron targets, one moderated (water and liquid hydrogen) and one unmoderated. This combination of targets provides flight paths which have cold, thermal to epi-thermal and fast neutron energy ranges. In addition the proton pulse structure of the LANSCE accelerator provides neutron pulse lengths of < 270ns for the thermal/cold flight paths and < 1ns for the fast flight paths. These pulse lengths allow for energy discrimination from eV to ~100 MeV.

Over the last 6 years there has been significant renewed interest in utilizing this source for neutron imaging as a complement to existing x-ray and proton imaging capabilities at LANL. To this end



thermal to epi-thermal integrated and ToF imaging (2D radiography and Computed Tomography or CT) have been established and cold neutron propagation based phase contrast imaging has been demonstrated. Finally, significant work has been put into developing a fast neutron imaging capability with the goal of reaching sub mm resolution on objects with an integrated density > 200 g/cm<sup>2</sup> and a CT scan time of less than 12 hrs. Fast neutron imaging at high resolution is an area with relatively sparse development due to a lack of available high intensity sources. This talk focuses on advances made in fast neutron imaging at LANSCE-WNR over the last 4 years including flight path modifications, scintillator development and detector testing. Results are shown for a range of scintillators, flat panel detectors and lens coupled camera systems. In addition energy discriminating Time of Flight images from 2 to 60 MeV are shown. Imaging results are shown on imaging quality indicators, a range of industrial parts (cracking, casting voids, etc) and on fossils of various sizes. Where available x-ray CT results are shown on the same parts to demonstrate the pros and cons of fast neutron imaging. Finally, ongoing work and outlook for continued improvement in fast neutron imaging will be discussed.

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## Simultaneous Neutron and X-Ray Quantitative Computed Tomography of Concrete Deterioration

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The NIST simultaneous neutron and X-ray tomography (NeXT) system is being applied to measure nondestructively the growth over time of ettringite, Ca<sub>6</sub>Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>(OH)<sub>12</sub>·26H<sub>2</sub>O, crystals in concrete specimens to investigate the mechanisms and kinetics of this deterioration process. Neutron tomography is especially suited for this topic because of the large H content of ettringite which produces a prominent attenuation factor. The simultaneous X-ray tomography is useful for distinguishing among the other hydrogen-bearing phases present (cement paste, calcium hydroxide and water-filled pores) on the basis of their densities. Three specimens of concrete were cast under different conditions designed to promote or inhibit delayed ettringite formation. They were scanned by NeXT at approximately one month intervals for a year after casting. The scanning time was approximately 12 hours. The voxel size of the reconstructions was 25µm. Due to the very strong attenuation of the hydrogen-bearing phases the specimen diameter was limited to 5 cm. To prevent pore water loss during the scanning period the specimens were sealed in Teflon. Internal standards consisting of microspheres of known attenuation were embedded in the specimens during casting to calibrate the grayscale histograms and to provide reference points for beam hardening corrections, fiducial marks were engraved in the stainless steel bases of the specimens for registration of images from successive scans. Classification of each voxel to a cement phase involves segmentation of the 2-dimensional gray scale histogram of the neutron and X-ray attenuation factors.

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## Neutron and X-Ray Radiographic/Tomographic Analysis of Limestone Dissolution

Lawrence Anovitz<sup>1</sup>; David Cole<sup>2</sup>; Daniel S. Hussey<sup>3</sup>; Jacob LaManna<sup>4</sup>; Alexander Swift<sup>2</sup>; David L. Jacobson<sup>3</sup>

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Carbon dioxide capture and sequestration in deep geological formations is an important option for reducing greenhouse gas emissions. While the importance of porosity and pore-evolution has long been recognized, quantitative assessment of how porosity and permeability evolve in reactive carbonates exposed to CO<sub>2</sub>-loaded brines have not been well constrained. A typical pH range for CO<sub>2</sub>-acidified brine is 3 to 4.5 depending on alkalinity. This represents a substantial perturbation of typical brines that range from pH 6 to 8. The key questions include how accessible are the pores to fluid transport and how does the pore network evolve as the matrix reacts with the acidic solution? Limestones and dolostones contain nano- to macroscale porosity comprised of cracks, grain boundaries, fluid inclusions, single pores, vugs and networks of pores of random shapes and orientations. Accessible, interconnected pores are arguably a critical part of the entire pore system. They act as pore throats, constraining overall flow and are the most likely locations for extensive rock alteration. Neutron imaging is well suited to studies of fluid flow as using neutron tomography/radiography we can directly image water or hydrocarbons without the aid of an added contrast medium that could modify interfacial tension and fluid/fluid interactions. Thus, neutron radiography and tomography have the potential to significantly advance our understanding of fluid flow in a wide variety of subsurface research applications. In order to understand the reaction of acidified fluids we have used a combination of simultaneous neutron and X-ray tomography to study the uptake and reaction of water and an acidic fluid (pH 2,4 HCl) with two samples of Indiana limestone, one with a permeability of 2-4 mD, the other with a permeability of 70 mD. Water and acid were allowed to permeate the sample based solely on capillary uptake, and the latter was also introduced under forced flow conditions of 1 and 10 ml/min. In wormhole formation was observed for faster flow rates and more acidic conditions, and tomographic images were obtained after the reaction. The results clearly show that the grain boundaries and other parts of the porous network initially present in a rock plays an important role in controlling the dissolution process.

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## Determining the evaporation and condensation coefficients of cryogenic propellants

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The control of propellant boil-off is essential in long-term space missions. However, a clear understanding of propellant cryogenic condensation/evaporation coefficients is lacking. A method to determine accommodation coefficients using a combination of neutron imaging, thin film evaporation modeling and CFD modeling has been established. Phase change experiments were conducted in the BT-2 Neutron Imaging Facility at the National Institute of Standards and Technology (NIST) by introducing cryogenic vapor (H<sub>2</sub> and CH<sub>4</sub>) at a set pressure into Al6061 and SS316L test cells placed inside a 70mm cryostat. Condensation is achieved by lowering the cryostat temperature below the saturation condition and vice versa for evaporation. Neutron imaging is used to visualize the liquid-vapor interface inside metallic containers due to the difference in attenuation between the cryogen and the metal. Phase change tests are conducted using liquid hydrogen and methane at a range of saturation points between 80 and 230 kPa and corresponding phase change rates were determined. The contact resistances and other transient heat transfer properties of the cryostat setup is

determined from the combination of a CFD thermal transport model and a “dry” thermal cycling test. The calibrated CFD model then allows for the determination of the inner wall temperature profile. Results from neutron imaging and the thermal model serve as boundary conditions to a multiscale evaporation model. A macroscale 2D FEA model is used to compute evaporation flux in the bulk meniscus while a thin film evaporation model is used to account for enhanced evaporation near the contact line. Using a combination of neutron imaging, CFD thermal model and a multiscale evaporation model, there is a possibility to extract the accommodation coefficient while accounting for the curvature, disjoining pressure and a variable interface temperature. The accommodation coefficient of H<sub>2</sub> decreases from  $0.65 \pm 0.12$  at 88 kPa to  $0.22 \pm 0.1$  at 226 kPa and is independent of container material/geometry. The error is dominated by the uncertainty in the temperature measurements ( $\pm 0.25$ K).

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## In operando visualization of oxygen gas propagation in a polymer electrolyte membrane electrolyzer via neutron radiography

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As the world population and energy consumption are growing rapidly, implementing a sustainable energy infrastructure with lower carbon footprints has become an urgent need. In order to lower greenhouse gas emissions, local electricity grids must be able to store the excess power from renewable sources such as solar and wind power, since these energy sources are intermittent in nature. Polymer electrolyte membrane (PEM) electrolyzers enable storage of electrical energy in the form of hydrogen gas, providing a promising solution to overcome the intermittent behaviour of renewable energy 1. Currently, the undesirable accumulation of the by-product oxygen gas in the anode components of PEM electrolyzers interrupts the liquid water transport to the reaction site, leading up to 25% loss in efficiency 2. The transport phenomena of water and gas in PEM electrolyzers stands to be improved, since PEM electrolyzers are made of opaque metal components and elude direct visual observation. Therefore, a viable visualization method is required to further our understanding of the transport phenomena, which could enable the development of improved materials for PEM electrolyzers.

Neutron radiography imaging is a promising technique for visualizing the transport behaviour in PEM electrolyzers, since metals do not significantly attenuate neutron beams 3. In this work, neutron radiography was employed to quantify oxygen gas in an operating PEM electrolyzer. With the advanced imaging capabilities provided by the Neutron Imaging Facility at the National Institute of Standards and Technology, we were able to image oxygen gas travelling in the anode flow channels. Specifically, we observed a linear growth in gas concentration with increasing position along the anode flow channel, which has been assumed [4], but has not been previously visualized in the literature. The results demonstrate the feasibility of using neutron radiography for in operando visualization to gain a deeper insight into understanding the two-phase flow phenomena in PEM electrolyzers.

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## Evaluation of areal densities of two elements in a composite by epithermal neutron transmission spectroscopy

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Neutron radiography techniques have been widely used to visualize the spatial distribution of a light element taking full advantages of its high sensitivity for them. Although quantitative measurements for one element in an object have been performed by obtaining calibration curves or calculations, it is essentially impossible to evaluate several elements simultaneously by conventional neutron radiography. A neutron resonance absorption radiography technique is used to obtain enhanced images of several elements at the same time, however, most of light elements are not available due to the absence of resonance in the available neutron energy region. For such elements, we have been developed a new method to quantify areal densities of several elements in a composite by epithermal neutron transmission spectroscopy<sup>1</sup>. In this method, a measured neutron energy-dependent transmission curve is fitted by a calculated transmission curve from the neutron total cross-sections of respective elements and their areal densities with a least-square method and thus, the areal densities are obtained by the fitting analysis. This method has possibility to visualize spatial distributions of several elements in a composite by using a two-dimensional spectroscopy detector. In the previous study, this method was applied to a pure iron plate, a pure carbon plate and a stack of them at the Hokkaido University neutron source. The evaluated areal densities agreed with those derived by the sample's weight, volume and thickness within 10%<sup>1</sup>.

In this study, we applied this method to various kinds of elements and composites of two elements to investigate its scope of applicable elements. The samples were selected in terms of the difference of the energy dependences of neutron total cross-sections. In the energy range of epithermal neutrons, the energy-dependences of total cross-sections are roughly categorized into two types, (a) constant (potential scattering is dominant) and (b) not constant (due to the effect of other interactions, e.g., absorption). We selected carbon (C) and lead (Pb) from the type (a), boron (B), iron (Fe) and titanium (Ti) from the type (b), and prepared five composites by combining Pb and C, Pb and B, Pb and Ti, Ti and B, Ti and Fe, where B was replaced by BN due to the chemical stability. The selected elements of the type (b) have the different energy dependences each other. Measurements were carried out at the NOBORU instrument located at beam port 10 of the pulsed neutron source of the Materials and Life Science Experimental Facility at J-PARC. A time-analyzing neutron imaging detector, GEM 2, was used in terms of future applications of this method for neutron imaging. From the results, except for the case of the composite of Pb and C (both are the type (a)), evaluated areal densities of elements were reasonably obtained, compared from those derived by their weight and dimensions. We discuss the applicable scope of the proposed method in the one- or two-element case.

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### Speaker Sessions and Seminars / 73

## High-frame rate neutron imaging of bubble behavior in air-water two-phase flow

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Gas-liquid two-phase flow appears in nuclear power reactors and is one of the important phenomena for the safety analysis of the reactor. Especially, the transient behavior of the two-phase flow structure is very complicated and has to be understood in detail by experiments. For that purpose, flow measurement method with high temporal resolution is required. Previously, a lot of methods have been developed and applied. Neutron imaging can visualize the flow in metallic pipe and the spatial flow structure can be understood. Therefore, it is very useful tool for two-phase flow measurement. However, the improvement of the temporal resolution was not easy because of the limitation of the neutron source and the imaging system. The authors have been developed high-frame rate neutron imaging system, which consists of a high-speed camera, an optical image intensifier, a high-sensitivity lens, a scintillator and a dark box, previously. In the present study, the system was upgraded by using a high sensitive high-speed camera and an ultrahigh-sensitivity lens. As a result, the frame rate of 10,000 Hz could be achieved at B-4 neutron guide tube facility in Kyoto University Research Reactor. The current system was applied to air-water two-phase flow measurement in a circular pipe, and the bubble behavior was observed. In addition, the simultaneous measurements with high-speed X-ray radiography were carried out to compare the imaging results. The X-ray was irradiated to the test section in a direction perpendicular to the neutron beam. From these results, the possibility of the 3-D visualization of bubbles using neutron and X-ray radiographs were also investigated.

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## Visualization of structure of molten lead bismuth eutectic

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Lead Bismuth eutectic (LBE) is a promising candidate of the coolant for accelerator driven system (ADS) and fast breeder reactor (FBR). Energy-resolved neutron transmission imaging is a useful means to visualize the behavior of the LBE in flow channel. A recent study has reported that the spatial distribution of the crystallographic information in the LBE can be obtained from Bragg-edge transmission, which is the decrease in neutron transmission due to Bragg diffraction from the crystalline solid phase. However, the molten state of the LBE was not characterized, even though it plays a principal role in thermophysical properties. The molten LBE also shows the neutron diffraction caused by the structure of liquid, which has to decrease the neutron transmission similar to the Bragg edge transmission from the solid structure. If the diffraction contribution from the liquid structure in the neutron transmission spectrum can be observed, it can provide useful information about the atomic structure in the molten LBE. Therefore, the energy-resolved neutron transmission imaging measurement of the molten LBE was carried out to observe and analyze the diffraction contribution from the structure of the liquid.

The experiment was conducted at the energy-resolved neutron imaging instrument BL22 RADEN and at the neutron beamline for observation and research use BL10 NOBORU at J-PARC. The LBE sample was sealed in rectangular steel pipes and heated up to 300 degree using cartridge heaters. The neutron transmission spectra of the molten LBE show significant decrease compared to those estimated from the absorption coefficient. This difference is attributed to the diffraction contribution from the structure of the liquid. The calculated neutron transmission spectrum from the neutron diffraction profile reported in a previous work well explains the features of the observed neutron transmission spectra such as humps. This indicates that the information about liquid structure can be obtained from the neutron transmission spectra. Furthermore, it enables to visualize the spatial distribution of the structure in the molten LBE. This will be a powerful technique to investigate the behavior of the molten LBE in the flow channel.

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## Simultaneous measurements of water distribution and electro-chemical characteristics in polymer electrolyte fuel cell

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Simultaneous measurements of water distribution in the through-plane direction and the electrical impedance were carried out by using a neutron radiography and electro-chemical impedance spectroscopy for evaluating the influence of water transport phenomena in a polymer electrolyte fuel cell (PEFC) on the power generation performance. The water distributions were obtained every 60 sec after the power generation. The resistance of proton exchange membrane (PEM) decreased with increasing the water saturation in the membrane. Reaction resistance increased with the liquid water in the PEFC. Effects of the water saturation on the PEM resistance was much smaller than that on the reaction resistance. Therefore, the reaction resistance was dominant effect for the degradation of the power generation if much water accumulates in the membrane.

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## Preliminary Evaluation of Wettability Enhancement in Capillary Tube due to Radiation Induced Surface Activation

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Supercritical water reactor (SCWR) is one of the generation IV reactor concepts. The SCWR is light water cooled reactor which is operated at supercritical pressure. Owing to its high outlet temperature, high thermal efficiency can be achieved. The design of the SCWR employs great knowledges acquired thorough thermal power plants. Stainless steel is utilized for cladding in the SCWR to realize required corrosion resistance at such a high temperature. Characteristics of this steel are of significant important such as corrosion resistance, heat transfer property, etc. Unlike thermal power plants, many additional criteria should be incorporated in the SCWR. One of the biggest differences is the radiation. The Radiation Induced Surface Activation (RISA) effect is known for its unique characteristics. We are currently investigating the RISA effects on stainless steel under high temperature and high pressure condition. In this presentation, wettability enhancement due to the RISA is evaluated. Water levels in stainless capillary tubes are visualized by means of the neutron radiography. Gamma-ray irradiated tube and non-irradiated tube are filmed side-by-side. Wettability enhancement was analyzed with the difference of the water levels. Some preliminary results will be presented in the conference.

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## New insights into fracture mechanics of sedimentary rocks using neutron tomography and mechanical tests

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Sedimentary rock is a widely used natural resource in civil, mining and hydrocarbons engineering applications. These materials exhibit discontinuities in the form of mineral composition, crack/fractures (natural or induced) at different spatial scales. However, the complexity of the material constitution and lack of experimental data acquired from direct observations limited the understanding of the mechanical behaviour of these materials especially crack initiation and propagation. This work addresses this limitation by acquiring 3D images of fracture propagation in sedimentary rocks under prescribed boundary conditions using neutron imaging at BT2, the Neutron Imaging Facility at the NIST Center for Neutron Research.

This paper discusses the crack propagation in a low strength Kirby sandstone sample (25 mm in diameter and 63 mm long) under triaxial stress conditions ( $S_{max}=0-40$  MPa applied on the flat surface and  $S_{min}=0$  on the cylindrical surface), where  $S_{min}$  and  $S_{max}$  are minimum and maximum principal stresses during the experiment. The data acquired from the tomograms at  $S_{max}=30$  MPa, revealed a single fracture that spanned 88% of the length of the rock core sample. With the increase in  $S_{max}$  to 40 MPa, the fracture width increased by 0.3%. Assuming brittle fracture and using the modulus of elasticity,  $E=5.16$  MPa, obtained from uniaxial compression test carried out on a similar Kirby sandstone sample and the crack length from the tomograms, it was found that the energy required to create two new surfaces in Kirby sandstone is 0.343 J/sq.m. These results demonstrate that combined neutron imaging with mechanical tests for macroscopic parameters provides new insights into the mechanics of fracture in geological materials.

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## Conceptual design of the test neutron imaging system at CSNS

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A test neutron imaging (NI) system is recently approved to be built on day-one instrument general purpose powder diffractometer (GPPD) at the China Spallation Neutron Source (CSNS). The conceptual design and neutron beam characteristics of the NI system have been studied. Decoupled poisoned hydrogen moderator as neutron source for GPPD and a flight path of 30 m from moderator to sample will provide wavelength resolution better than 0.2% for Bragg edge imaging. The neutron flux at the imaging sample position will be  $\sim 106$  n/s/cm<sup>2</sup>. The best spatial resolution will be 50  $\mu$ m and 2 mm by using CCD based detector and GEM (Gas electron multiplier) detector, respectively. The NI system will provide experimental test capabilities of instrument concept, imaging methods and applications.

Speaker Sessions and Seminars / 65

## Construction of a Quasi-Monoenergetic Neutron Source for Fast-Neutron Imaging

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Lawrence Livermore National Laboratory is developing a high-brightness, quasi-monoenergetic neutron source for fast-neutron imaging. Past and on-going image quality index (IQI) measurements of various objects show that there is great promise for fast-neutron imaging, specifically for imaging structural and material integrity of low-density materials within high density enclosures. Simulations, calculations, and measurements show that discerning detail in the low-density materials as well as interfaces between low- and high-density materials is greatly improved using fast-neutron imaging compared to X-rays and has high potential for seeing corrosion between different materials. The intensity of the neutron source is expected to be 1011 n/s/sr with a fixed energy at 10 MeV with 5% bandwidth at 0-degrees. A 7-MeV pulsed linear accelerator will drive the neutron source. The accelerator will deliver a 300-uA average current deuteron beam onto a pulsed deuterium windowless gas target. The gas target is necessary because of the combined beam power and the requirement for a small source spot size. We will present the results of measurements of fast-neutron imaging we have made with different source types. We will discuss our source construction and plan forward for fast-neutron imaging.

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## Speaker Sessions and Seminars / 96

### Studying early stage pedogenesis using on-the-fly bimodal tomography

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Urbanization and increasing sealing of the landscape by impervious surfaces lead to fast water runoff in the cities. In urban areas, rain water is often channeled towards swales where it is left to infiltrate through recently engineered soil. As the runoff water is prone to carrying dissolved and colloidal contaminants, it is important to investigate the water infiltration process through soil at swales, estimating the soil's water filtering capabilities. Similarly, green roofs of buildings contain specifically designed engineered soils. It is expected that soil properties are gradually changing during first months and years after plants introduction. This may result in changed water retention capacity, evaporation rates, runoff amount and water filtration. The inner soil surface areas exposed to water flow paths need to be quantified for this purpose. Here it is necessary to involve non-invasive imaging techniques because soil is one of the most complex porous media that is known. We have combined neutron and X-Ray tomography (NX) to elucidate the complex water flow through the organic matter rich engineered surface soil in the early stage of pedogenesis. Soils under study involved mixture of 20% topsoil, 50% sand, and 30% compost as well as green roof growing media based mainly on crushed expanded clay and spongolite stone. One set of samples was prepared by packing of fresh material into aluminum cylinders, while the second set of samples was collected from experimental plots after two months of pedogenesis. The complexity of the sample composition requires the information from a second imaging modality to reduce ambiguity in the interpretation. The challenge of the investigation was that the water flow through the sample is relatively high which required



on-the-fly acquisition using both modalities. This provided bimodal volume pairs acquired simultaneously at the rate of one per 180s using the NX installation 1 at ICON, Paul Scherrer Institut, 2. We will illustrate the image processing chain and show results from the preliminary analysis. The reconstruction was performed using our open-source CT reconstruction software 3 and includes correction for scattering correction [4].

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### Speaker Sessions and Seminars / 124

## Analyzing neutron imaging data – an open-source collaboration

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It is well-known that a neutron imaging experiment is not finished with the acquisition of the data, but merely is the starting point for data processing and evaluation in order and to extract the information that is needed to draw conclusions about the sample or the observed process. This can be very time consuming depending on the amount of data and complexity of the information it contains 1. Imaging at pulsed neutron sources enables efficient acquisition of wavelength resolved image data sets, but typically leads to an increased complexity of data analysis. In the majority of cases, the analysis of imaging data includes a sequence of similar operations, at least for the first steps. Later in the process, analysis steps will be more specific with regards to method and sample, but they in general still benefit from available building blocks to solve subtasks. While novel methods, in particular wavelength resolved techniques, still require significant development of software and analyses tools, conventional experiments would profit from unification and interoperability of analyses tools across available instrumentation at different sites. The authors represent software development initiatives for neutron imaging like 2 at four major neutron sources and have decided to join forces to develop corresponding open source analysis tools for neutron imaging [4].

The neutron imaging user community is very heterogeneous, which is typically reflected in the requirements for software tools. All stakeholders agree that a full understanding of particular tools

should not be a hurdle or prerequisite for users to analyze their data. This shall also be reflected in flexibility with regards to that some users might require graphical user interfaces while others prefer scripting tools that allows flexible handling of multiple data sets. With this in mind, we aim at making different aspects of energy resolved imaging [5] available to a wider user community and allowing scientists to produce more high quality scientific results in shorter time. The presentation will provide an overview of the project, its objectives as well as an outline of developments and progress.

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## Methods to combine multiple images to improve quality

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Noise and artifacts have a negative impact on the image quality and the resulting image analysis. The signal to noise ratio (SNR) caused by the neutron flux and the light conversion efficiency is one component in this. Here, we are more concerned about the effect of outliers, which frequently appear in neutron images. There are two approaches to reduce the impact of spots (1) by applying spatial outlier rejection filters and (2) by acquiring multiple images which are combined into a single image a total neutron dose similar to the dose of option (1). Here, we focus on the second option where we will show the importance of the choice of combination method. The impact is demonstrated to show the ability to reject outliers but also that the SNR can be improved. The combination approach has the advantage that it does not affect neighbor pixels. The tested methods are the arithmetic average, median, and a new weighted average. The weighted average show very promising results compared to the other two.

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## Feasibility study of two-dimensional neutron-resonance thermometry using molybdenum in 316 stainless steel

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A two-dimensional thermometry technique based on neutron resonance reactions derives the temperature of specified elements in an object by analyzing the Doppler broadening of a neutron resonance measured by a time-analyzing neutron imaging-detector. This technique is expected to be one of the important applications of the energy-resolved neutron imaging system, RADEN, at J-PARC. As a part of ongoing feasibility studies, the authors discussed the reliability of this technique using tantalum and tungsten from 26 to 285 degrees Celsius in a previous study <sup>1</sup>. Tantalum is the most commonly used sensor material for neutron resonance thermometry due to its suitable resonance properties (width, energy and magnitude), however, a more widely-available material has an advantage to promote applications of this technique. The authors focused on molybdenum contained in 316 stainless-steel (with a weight fraction of 2-3 wt%), which is a well-known and widely-used material in various fields. One molybdenum isotope, molybdenum-95, exhibits resonances of several kilo-barns at a neutron energy of 44.75 eV. The magnitude of this resonance cross section is large enough for resonance absorption imaging, and the thinner relative width of the resonance than that of tantalum may bring about higher sensitivity for temperature, while the resonance energy is about 10 times higher. This consideration of the resonance properties and a calculation using the REFIT code [2,3] indicated that molybdenum could be used for neutron resonance thermometry. On the other hand, the temperature effect might not be the same between pure molybdenum (used in the REFIT calculation) and molybdenum contained within an alloy since the Doppler broadening caused by the increase of thermal vibrations of the molybdenum atoms can be affected by the crystallographic structure of the composite material. Thus, the authors were motivated to measure the broadening of the molybdenum resonance in 316 stainless steel.

Transmitted neutrons were measured through a 3 mm thick 316 stainless-steel plate placed in a heater at temperatures between 23 and 500 degrees Celsius at RADEN. A copper plate (1 mm in thickness) was attached to the stainless-steel plate to ensure a homogeneous temperature distribution. For the measurement, the proton beam power was 400 kW, and the energy-dependent neutron transmission was imaged using a gas-electron multiplier (GEM) detector. Thermal and cold neutrons were eliminated from the incident beam by a cadmium filter, and the neutron beam size was collimated to 50 by 50 mm<sup>2</sup> using stainless-steel and boron-impregnated polyethylene blocks. The sample area within the neutron beam was 30 by 50 mm<sup>2</sup>. In this presentation, the feasibility of neutron resonance thermometry using molybdenum in 316 stainless steel is discussed, specifically in regard to the reliability of the technique.

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### Speaker Sessions and Seminars / 126

## An Experimental Trial of 3D Synergy Modeling from X-ray CT and Neutron Radiograms

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The synergy imaging is an imaging technique which obtains a nuclide distribution image with higher spatial resolution using the differences between cross sections of neutron and X-ray <sup>1</sup>. The concept of the synergy imaging is developed from the image alignment technique using the mutual information (MI). In our previous computer simulation study, the procedure extended to the three-dimensional (3D) nuclide mapping. For making a 3D volume model, we assumed the use of X-ray computer

tomography (CT) technique, and the neutron radiograms which were taken along only orthogonal three directions. The 2D synergy imaging were carried on along the proper three directions between neutron radiograms and reconstructed X-ray radiograms from the CT model. Obtained nuclide distribution results for three directions were reconstructed by the back projection method and obtained the 3D nuclide distribution by analyzing the voxel data after the back projection. This procedure has a great advantage for the 3D model construction with neutron, because the number of the neutron radiogram measurements is reduced greatly.

In this study, we applied this 3D synergy imaging method to an actual object and demonstrated the 3D voxel model reconstruction of the nuclide distribution using the procedure. The sample object was an aluminum cylinder of 20 mm diameter and 10 mm height including metal wires of Ta, W, Pb, In and Ag. The X-ray CT measurement carried on the laboratory system with 150 keV micro focus generator. The neutron imaging was taken at Hokkaido University Neutron Source (HUNS), Japan. The neutron detector was GEM type with spatial distribution of 0.8 mm. The sample was set on just before the detector window to eliminate blurring. For the case the neutron radiogram size has only 25 x 25 pixels. From the X-ray CT measurement we pulled out the appropriate images which coincided with the neutron measurement directions, and proceeded the synergy imaging to obtain the nuclide distributions. The resulted 2D nuclide distributions were very coarse because of the statistics of the neutron radiograms, then we averaged the each area where the shadow of each wire was recognized. The results obtained were able to distinguish each nuclide. Finally, we reconstructed the 3D voxel model by the back projection of three nuclide distribution images from the three orthogonal directions. The obtained 3D model has the higher spatial resolution equal to the X-ray CT voxel and correct nuclide information of wires. That is, it can be said that 3D synergy imaging was successful.

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## Improvement of Neutron Color Image Intensifier Detector using an Industrial Digital Camera

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Neutron radiography has advantageous in the non-destructive inspection field because of its high penetrating power, and mutual use with X-ray radiography provides useful information through the synergy imaging 1 and others. One of the unique imaging devices for the neutron radiography is the neutron color image intensifier (CNII) 2. It consists of a vacuum tube installed with electromagnetic lenses, and has an input window with a boron or gadolinium compound film and an output fluorescent screen. The projected image on the output screen is recorded by a digital camera. The typical spatial resolution of CNII is under 0.1 mm.

In the lineup of the CNII detector system developed until now, a commercially available single-lens reflex digital camera or a high-speed digital camera has been selected as cameras to be combined. Since these cameras have relatively large volumes, the shielding boxes are large and it becomes difficult to align on the beam line easily. Also, for neutron radiography under low flux, it is necessary to apply the long exposure time or accumulate the many number of shot frames. In the case of the long exposure time or high sensitivity setting of the digital camera, the long-time and high-sensitivity noises have to be noticed in addition to the radiation noise. Therefore, in this study, we developed a compact CNII detector system with the long-time stability under less noise so that it can be used for mobile use.

The newly developed system equips the CNII of 4 or 9 inches. The combined camera is an industrial one. The advantage of industrial cameras is that they have linearity of output brightness, which makes easy to ensure quantitative measurement. Considering the circular output screen of CNII,

in order to ensure the maximum effective field of view, selection criteria of the camera is that the aspect ratio of the image sensor is nearly 1: 1, the transfer image rate is more than 60 fps and the pixel number is more than current high vision standard. We selected a compact 5M pixel camera with USB3.0 connection. The optical path between the CNII output screen and the camera is shorten in order to reduce the size of the detector system. The camera is equipped the thermostatic device to decrease noises and improve its stability. Finally, we succeeded in compacting the system to 30 and 60 kg for 4 and 9-inch CNII system, respectively. The systems were tested on the several beam lines and taken radiograms such as an image of nuclear fuel materials successfully.

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## Speaker Sessions and Seminars / 146

### Highlights from the CONRAD-2 beamline at HZB

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The material characterization by neutron imaging reached a new level after developing innovative techniques using different contrast mechanisms than the common beam attenuation. In this way properties of materials and complex systems can be resolved by position sensitive mapping of diffraction, small-angle scattering and refraction signals. In addition the improved spatial and time resolution of the detector systems allow for micro tomography studies and 3D dynamic investigations. Applications related to 2D and 3D visualization of material phase heterogeneities, texture, fluid dynamics, magnetic structures and phase transitions in applied materials from the CONRAD-2 neutron imaging instrument at the Helmholtz-Zentrum-Berlin (HZB) will be presented.

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### NEUTRON MICROSCOPE ARRANGEMENT USING MACRO LENSES

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Neutron imaging is becoming an increasingly important diagnostic technique, providing complementary information to X-ray imaging for a wide range of applications in science and industry. Up to now the complementarity was related mainly to the different contrast provided by neutrons. Recently a high-efficient optical arrangement based on macro lenses was proposed for achieving high spatial resolution in neutron imaging experiments which is comparable to the one used in the X-ray imaging. The advantages of the macro lens detector system are its flexibility where different effective pixel sizes and Field-of-Views (FOV) can be setup but also its high light efficiency which allows for short exposure times resulting in tomography experiments.

The improvement of the spatial resolution in neutron tomography experiments opens new fields of applications where volumetric data obtained by using of neutrons and X-rays can be compared and even registered.

Results of 3D high-resolution complementary investigations of rock samples, plants and Li-Ion batteries will be presented.

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## **Recent progress of neutron imaging development at TRIGA MARK II PUSPATI research reactor**

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Neutron radiography facility at TRIGA MARK II PUSPATI research reactor(RTP) was built at one of the radial beam ports since 1984 but was stagnant for a long period. The facility is known as NUR and has low thermal neutron intensity at the sample position. The NUR has recently been re-developed to enhance the radiation safety and to facilitate digital radiography and tomography operation. In addition, the facility consists of a new collimator design and new imaging system using CCD camera. The new imaging parameters and recent neutron imaging development of this refurbished neutron radiography facility will be discussed in this paper.

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## **The Radiography using Am-241 / Be neutron source for industrial applications**

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Am241 / Be is a representative neutron source with Cf252. In this paper, we have studied neutron radiography using Am241 / Be source. The Am241 / Be source has a neutron energy distribution of up to 11 MeV and an average energy of 5.4 MeV. In addition, gamma rays of 0.033 MeV are generated and have a mixed radiation field, which is good for radiographic imaging of composite materials. Half-life is 433 year, and long time is available, and when enough flux is provided, it can be used for shooting thick objects because of high energy.

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## Fabrication of neutron gratings for high sensitivity grating interferometer

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Symmetric neutron grating interferometer has several advantages such as high sensitivity and easy grating fabrication by Gadox powder filling method. Symmetric neutron grating interferometer using 50um period gratings was prepared for high phase contrast image, and the performance test was conducted at NG6 beam-line of National Institute of Standards and Technology (NIST). As a result, neutron phase contrast and dark-field images were obtained. After obtaining preliminary results of symmetric neutron grating interferometer, new neutron gratings have been preparing to get higher phase sensitivity. The source grating using Gadox powder filling method is a neutron absorption grating with 0.25 opening ratio, 36um period and 100um height. The phase grating with 0.5 opening ratio, 36um period and 34.39um height is fabricated using silicon etching method. The analyzer grating with 0.5 opening ratio, 36um period and 100um height is fabricated using Gadox powder filling method. The performance of symmetric neutron grating interferometer using these newly fabricated gratings will be evaluated at NG6 beam-line of NIST and discussed at this conference.

### Speaker Sessions and Seminars / 116

## Development of Quantitative Crack Analysis Techniques Using Neutron-Absorbing Liquid Penetrants

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Contrast agents for neutron radiography have been demonstrated for industrial applications; however, quantitative evaluations of these contrast agents are scarce in the published literature. This project will develop a quantitative tool to determine crack extent using processed neutron radiographs. This quantitative tool will be valuable for analyzing cracks in irradiated materials such as higher burnup fuels and advanced cladding materials where conventional crack measuring techniques are not possible

The East Radiography Station of the Neutron RADiography (NRAD) reactor at Idaho National Laboratory, imaged aluminum alloy crack test blocks prepared per American Society for Testing and Materials (ASTM) standards. Image processing extracted quantitative measurements of the crack area from the resulting film radiographs. A digital image threshold segregation process segregated the crack area was to black and the background to white. Testing different contrast agent solutions and varying the methods of infiltration provided data on the most effective infiltration and washing methods.

While the initial round of neutron radiography proved that digital image processing of gadolinium-enhanced crack radiographs could yield a quantitative measurement of crack extent, the resulting pixel counts were not clearly correlated to the amount of gadolinium in the crack. Neutron activation analysis (NAA) of the infiltrated cracks can provide a quantitative measurement of the amount of infiltrant in each crack; however, the low thermal neutron cross-sections of gadolinium-158 and

gadolinium-160 make NAA difficult. Dysprosium is well-suited for NAA because it is chemically similar to gadolinium, possesses a high thermal neutron cross-section, and has a daughter product (dysprosium-165) with a 2.33 hour half-life.

The Geologic Survey TRIGA Reactor (GSTR) at the Denver Federal Center in Lakewood, Colorado irradiated small (<10g) aluminum alloy blocks containing dysprosium-infiltrated cracks. The resulting activity of the irradiated dysprosium provides a quantitative measurement of the amount of infiltrant in each crack, which can be compared to the crack extent measured using image processing technique.

Once the most effective infiltration and wash methods have been determined, infiltration solutions containing both a contrast agent for radiography and an isotope for NAA can be developed. Combining information from both NAA and neutron radiography provides a relationship between the total crack area in a digitally processed image and the mass of infiltrant in the crack(s). This relationship makes it possible to determine mass of crack extent solely from neutron radiography, allowing for crack size analysis of irradiated materials where NAA is not possible.

## Speaker Sessions and Seminars / 51

### Development of a compact accelerator-based pulsed neutron source and simulation of the neutron beam performance and Bragg edge imaging

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We have been developing a compact accelerator-based pulsed neutron source at the National Institute of Advanced Industrial Science and Technology (AIST) in Tsukuba, Japan. The main purpose of this neutron source is to analyze structural materials of automobiles and other transportation vehicles nondestructively by means of the high penetration power of neutron beams. We plan to focus on Bragg edge imaging because it can provide images of crystalline strain, phase, size, orientation etc. which will be useful for the development of innovative materials and their joining techniques. The key parameters required for using Bragg edge imaging effectively are the neutron flux and wavelength resolution at a sample position. In order to optimize the flux and resolution to the highest values possible for a compact neutron source, we designed a dedicated accelerator, neutron source, and beam line. The flight path length of the neutron beam is 8 m. A solid methane decoupled neutron moderator was chosen. A linear electron accelerator was adopted and the pulse width of the electron beam is less than 10 microseconds. These choices make possible a neutron wavelength resolution of about 0.6 %. To obtain a high neutron flux, the repetition rate of the electron accelerator is 100 Hz and the maximum power of the electron beam is about 10 kW. We are performing Monte-Carlo simulations to estimate the performance of the neutron beam for these parameters. The simulations suggest a neutron flux of about 11,000 1/cm<sup>2</sup>/s for thermal neutrons and a neutron wavelength resolution of about 0.6 % at the sample position is possible. In this presentation, we will introduce the compact accelerator-based pulsed neutron source at AIST, which is now under construction, and our estimates of the neutron beam performance and Bragg edge imaging examples obtained by Monte-Carlo simulations.

This presentation is based on results obtained from a project commissioned by the New Energy and Industrial Technology Development Organization (NEDO).



## Speaker Sessions and Seminars / 68

## Comparison of crystallographic structures of Japanese swords in Muromachi and modern periods by using pulsed neutron imaging

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Japanese swords are interesting cultural heritage from metallurgical point of view due to its peculiar characteristics. Its making process is not fully understood even now. Crystallographic information will be useful to understand metallurgical characteristics and to know making process. Non-destructive analysis is desired to obtain the crystallographic information for such valuable samples. Neutrons are powerful tool to study metallic cultural heritages due to their high penetrating power and capability to give crystallographic information 1. Bragg edge imaging gives real-space distributions of bulk information in a crystalline material. In addition, by analyzing position dependent Bragg edge spectra, quantitative crystallographic information can be obtained 2.

There were five traditional styles (Gokaden) of Japanese sword-making in the Koto (old sword) age; A.D. 987–1596. The crystallographic characteristics will depend on areas and ages of the swords. Therefore, systematic study is recommended for comprehensive understanding. As one of such researches, we performed pulsed neutron imaging measurements on three swords in Muromachi period (14–16 centuries), and one sword in modern period as a reference.

The experiments were performed at the Energy-Resolved Neutron Imaging System, RADEN at J-PARC 3. Each sword was measured at three places with a counting-type 2D detector. The transmission data were analyzed using RITS code [4]. Quenching area was more clearly observed in the modern sword than in the old ones. There was difference in distributions of lattice spacing. Detailed analysis results will be presented.

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## Pulsed neutron imaging

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Pulsed neutron sources are useful for imaging since they give different information compared with steady reactor sources [1,2]. At the pulsed neutron source, we can easily use the time-of-flight method to analyze the neutron energy and it enables us to obtain position dependent transmission data as a function of energy with high energy resolution. The transmission spectrum is affected by neutron interaction cross section, and by analyzing the energy dependency of the transmission we can deduce quantitative information of an object. 'Quantitative evaluation' is one of the most important characteristics of the pulsed neutron imaging.

There are various kinds of neutron interactions with matters. Coherent scattering is most important interaction to deduce the crystallographic information. We can get information on crystal phase, crystallite size, preferred orientation (texture) [3], and strain [4,5]. Micro-strain information will be included in the Bragg edge transmission [6]. Incoherent scattering does not have strong energy dependency. One of applications is to use the increase of low energy cross section of hydrogen, and the hump of cross section due to oscillation of hydrogen atom in a metal hydride [7]. Another feature of the neutron cross section is resonance peaks at higher energy. Information on dynamics of a peculiar element was studied [8], temperature was measured [9] and elemental distribution in a sample was obtained [10]. Other special feature of neutrons is magnetic interaction. By using the magnetic interaction combined with energy dependent transmission, we can evaluate the absolute value of the magnetic field [7, 11].

Material analysis and cultural heritage researches are performed by using pulsed neutron sources, and further development of data analysis is still ongoing. In the presentation, the pulsed neutron imaging method and its applications are presented.

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**Speaker Sessions and Seminars / 121**

## Wavelength-resolved neutron imaging on IMAT

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The 'IMAT' instrument, which specializes in Imaging and MATerials science, is now well into its commissioning phase. The basic performance parameters for white-beam tomography and energy-dispersive neutron imaging have been determined [ 1 ] and the instrument is currently being prepared for user operation [ 2 ]. Here we report on the evaluation of the wavelength-resolving imaging options on IMAT, including pink-beam imaging using disk choppers and energy-dispersive Bragg edge imaging using time-resolving detectors. These time-of-flight techniques enable image contrast enhancement and mapping of structure properties. We will review the recent infrastructure installations and software developments that have been undertaken to take advantage of these techniques, making the facility ready for applications in a diverse range of disciplines such as engineering material science, battery research, earth science and cultural heritage.

[ 1 ] T. Minniti, et al., Nucl. Instr. Methods A 888 (2018) 184.

[ 2 ] W. Kockelmann et al., J. Imaging 4 (2018) 47.

## Speaker Sessions and Seminars / 58

### New insights into the tooth structure of pelycosaurs by means of neutron tomography

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Pelycosaurs are the most primitive members of the Synapsida, which is the clade that includes mammals. Consequently, pelycosaurs are of special interest with respect to our early evolution. We investigated a skull of *Varanosaurus acustirostris* for the first time by means of neutron tomography at the facility ANTARES at FRM II in Munich. *Varanosaurus acustirostris* was a representative of the primitive pelycosaur group Varanopseidae. It derives from Early Permian deposits of Texas.

As the most remarkable result we found that *Varanosaurus* possessed plicidentine, i.e. infolded dentine at the base of the tooth roots. With the exception of the sphenacodontid pelycosaur *Dimetrodon*, plicidentine is unknown in Synapsida (Brink et al., 2014). Hitherto, plicidentine has been observed only in fishes (sarcopterygians and actinopterygians) and some basal tetrapod groups.

Our results suggest that plicidentine was more widespread among basal synapsids than previously thought. Functionally, the infolded dentine layer provided an increased area for attachment for the shallow tooth roots in the pulp cavities of the jaw. Now, neutron tomography allows non-destructive investigation of the tooth structure of these valuable fossils.

References:

Brink, K.S., LeBlanc, A.R.H. & Reisz, R.R. 2014. First record of plicidentine in Synapsida and patterns of tooth root shape change in Early Permian sphenacodontians. *Naturwissenschaften*, DOI 10.1007/s00114-014-1228-5.

## Speaker Sessions and Seminars / 137

## Time-resolved in-operando neutron imaging of lithiation and delithiation process in custom-built rechargeable Li-ion batteries

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We are reporting an in-operando study of time-resolved neutron imaging on cycled Li-ion batteries to analyze the phase changes during lithiation and delithiation.

Rechargeable lithium-ion (Li-ion) batteries are used to power up our daily portable appliances. They are electrochemical cells consisting of a positive electrode separated from a negative electrode, in electrolyte solution, which allows only Li-ions to move between the electrodes.

The most common material used for the negative electrode in rechargeable commercial Li-ion batteries is graphite, due to its mechanical stability and good electrical conductivity. Li-ions are intercalated in between and within the graphene layers in the graphite structure, creating crystallographic phase changes. The charge-discharge process is accompanied by the challenge of (de)intercalating Li-ions into/from the crystalline structure, thus forcing the lattice to distort and create defects, which contributes to transport-related structural damage upon fast cycling, thus shortening the lifetime. 1 Kinetic behaviour of Li-ions and phase transformation mechanisms are poorly understood due to difficulties in isolating these factors experimentally. However, these changes can be observed using neutrons 2. Due to the large neutron cross-section of Li and due to their penetration of bulk samples they are well suited for in-operando studies of Li-ion batteries.

We present the results of in-operando time-resolved Bragg-edge transmission neutron experiments of charge-discharge cycles of a custom-built Li-ion half-cell performed at RADEN@J-PARC, Japan. The measurements were performed on a custom-made battery cell with graphite:carbon black:polyvinylidene fluoride (8:1:1) as the working electrode and metallic Li as the counter electrode. They were charged and discharged at different C-rates, the current rate normalized to the maximum battery capacity. The first cell was discharged at two C-rates: C/34 and C/68, with a short period of relaxation between the discharges, and charged at C/34. The second cell was discharged at C/20 and at C/34 until the potential reached 0.001 V, with a relaxation period in between, and charged at C/34 until 3 V. Results of the neutron radiography experiments show phase changes in the working electrode and lithium intercalation and deintercalation during cycling. The phase changes are reflected in the variations of the graphite, LiC<sub>12</sub> and LiC<sub>6</sub> characteristic Bragg edges.

1 K. Persson et al., *J. Phys. Chem. Lett.*, 2010, 1 (8), 1176-1180

2 M. Kamata et al., *J. Electrochem. Soc.*, 1996, 143 (6), 1866-1870

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## Examination of the Spontaneous Imbibition of Fluids into the Eagle Ford Shale Formation using Neutron Imaging

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Hydraulic fracturing is the process of injecting high pressure fluids into shale hydrocarbon reservoirs to increase permeability and liberate oil and gas reserves. The majority of this injected fluid is never recovered from the reservoir and is termed leak off. The leak off fluid that enters a shale formation can decrease the productivity of a well by blocking the escape pathway of the oil and gas, thus preventing recovery. One possible mechanism for the escape of this fluid into a reservoir is the spontaneous imbibition of fluids through fractures caused by capillary forces. This study quantitatively examines the spontaneous imbibition of saturated fluids through fractures in the Eagle Ford Shale using neutron imaging. This method collects images in which the height of the water front through time can be measured. The uptake of water into the shale should vary linearly with the square root of time, however, this relationship is not always observed. By examining the factors that control imbibition, such as lithology and fracture width, better quantitative models can be designed to predict the outcome of the fluids used in hydraulic fracturing.

**Speaker Sessions and Seminars / 138**

## **Current developments and research applications of the NIST NeXT system**

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The NIST Neutron and X-ray Tomography (NeXT) system provides simultaneous complimentary multimodal information for the characterization of materials. Neutrons and X-rays provide complementary non-destructive probes due to the contrast differences that arise from the differences in interaction with matter for the two modes. NIST's NeXT system was initially commissioned in 2015 and has been operating fully in the Center for Neutron Research facility user program with robust demand. The system works by orienting at 90 keV microfocus X-ray tube orthogonally to the thermal neutron beam. With the truly simultaneous capture of the two modalities, it is possible to perform multimodal tomography of dynamic or stochastic samples while penetrating through sample environment equipment such as pressure and flow vessels. Through volume registration and data fusion of the two reconstructed volumes, improvements to image segmentation and phase identification can be made with 2D histograms that leverage the strengths of each mode. Current research applications using the NeXT system range from oil and gas recovery, strength of concrete and building materials, electrochemical energy storage and conversion, geophysics and geochemistry, and cultural heritage, among others. This talk will give an overview of the NeXT system, discuss several recent results obtained on the instrument, and detail future directions for improving the measurement method.

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## **Investigation of material with low-Z inside a thick steel box by Fast-Neutron Radiography and Tomography Techniques**

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Fast-neutron imaging is a very attractive nondestructive technique for industrial applications, because of its high penetration capability. For this work, an experiment for finding defects on the surface of material with low-Z inside a thick steel box has been performed by means of fast-neutron 2-D and 3-D imaging techniques at the NECTAR facility at the Research Neutron Source Heinz Maier-Leibnitz (FRM II) in Garching (Germany).

The work is a feasibility study of distinguishing abilities of fast-neutron imaging of different features in complex objects composed of a mixture of high Z and low Z materials.

The result of this study will be presented in the conference.

#### Speaker Sessions and Seminars / 4

### What Future in Neutron Imaging?

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The usage of neutron beams for non-destructive material studies has a long tradition since suitable sources were available. Meanwhile, neutron imaging has been developed towards a routine method at many places with basic (radiography, tomography) and more advanced (grating interferometry, polarized and diffractive imaging, data fusion) features. This development was only possible after the introduction of digital detection systems which mostly replaced analogue (film based) detection methods.

A generic neutron imaging facility consists of the following components: primary source, beam tuning devices, sample environment and a neutron imaging detector. There is no real standard how to tune and to compose these different pieces in the best way: each neutron imaging facility is built uniquely, taking into account the specific properties, mainly those of the neutron source.

Most of the powerful neutron sources in use for neutron imaging are based on research reactors. IAEA is providing a useful tool for a survey about the situation of research reactors world-wide 1. It gives the following status: operational: 223, usage for neutron radiography: 72, under construction: 8, planned: 14. In addition to these sources there are projects planned and realized for neutron imaging stations at spallation sources. Other accelerator driven sources, based on D-D or D-T reactions are available and used partly for some imaging activities.

In general, the number of sources will be more reduced than increased, given by the reactor age and the public acceptance in several countries. Therefore, the way to increase further the capabilities for neutron imaging is to access the underutilized sources and to equip them with best-performing infra-structure. Fortunately, some of the new source projects take neutron imaging options into account from the beginning and a best performing facility can be built. To install neutron imaging stations at an already equipped source needs special considerations and often compromises.

Another important aspect is the introduction of neutron imaging methods into practice either of scientific or practical applications. Since X-ray methods are much more common and increasingly used for research and in industry, a direct competition is not possible although some technical details are similar. We have to focus more on the inherently strength of neutrons and to perform related investigations under highly professional and best performing conditions. Therefore, the access for scientific users and industrial partners to neutron imaging facilities has to be enabled easily. Due to the limited number of high performing beam lines and the different shut-down phases a dialogue between the facility operators will help to increase the utilization on highest level.

There is still much potential for further methodical development and technical improvements. In addition, a focus has to be given to the data treatment and evaluation while the image data volume is increasing dramatically. A link to neutron diffraction and scattering enables a deeper insight to material properties and their modifications. New aspects like additive manufacturing and the study of materials for energy conversion and storing are handled very efficiently using neutron imaging techniques.

1 <https://nucleus.iaea.org/RRDB/RR/ReactorSearch.aspx?rf=1>

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## Investigation of SINQ (lead/zircaloy) target structures by means of neutron imaging techniques

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The Paul Scherrer Institut (PSI, Switzerland) operates the spallation neutron source SINQ since 1997 with great success. It is the most powerful national neutron source and basis for neutron research, also accessible by the international user community via annual open calls for scientific proposals.

The continuous proton beam of 590 MeV from the high intensity proton accelerator (HIPA) 1 has reached a power level of more than 1 MW demanding a corresponding continuous development of target technology which provides the most effective neutron conversion together with guaranteed reliable operation under extremely harsh irradiation conditions. Lead was found the most useful material for the spallation process with its high neutron yield and the low neutron absorption cross section.

In order to avoid a totally liquid target (with the drawback of permanent heating above the melting point), the SINQ targets consists of Pb filled Zircaloy-2 tubes of about 10 mm diameter and 20 cm length arranged in a hexagonal rod bundle perpendicular to the proton beam direction; colloquially the Pb filled Zircaloy-2 tubes are called "Cannelloni". The rod bundle is cool via a forced cross flow of heavy water, D2O. At the highest power level, the target operation has to allow for local melting of the Pb inside the Zircaloy-2 cladding. In cases of proton beam trips or other shutdown of the heating power the Pb freezing and shrinking starts immediately. For these cases of this thermal cycling a reduced filling of the Cannelloni's by 10% is foreseen during the installation process. Quality assurance of the filling factor by neutron radiography inspection is done in advance to the final target assembly.

In the last years SINQ has been operated in competitive mode with a second spallation target station (UCN source); the full proton beam power is diverted to the UCN source in for a few seconds only with a duty cycle of up to 3%. This short period of missing beam power is enough to result in solidification of the molten Pb for the time of the beam kick to the UCN source.

The consequences for the target reliability have been studied in different manners. Next to thermodynamic simulations we performed two kinds of neutron imaging studies: inspection of samples from spent targets with the NEURAP technique 2; in-situ studies for the melting/freezing process with heating/cooling elements and a mock-up target rod setup.

These investigations got high relevance after a severe target failure in 2016. Several Cannelloni's cracked open and liquid Pb was washed out and blocked coolant flow channels. Both types of neutron imaging studies will help to identify reasons for that material damage/failure in order to avoid further incidents and to find the operation limits of the current target technology.

1 M. Seidel et al., Production of a 1.3 MW proton beam at PSI Proc. 1st Int. Particle Accelerator Conf. (IPAC'10) Japan TUYRA03 pp. 1309–13

2 Lehmann, E.H., Vontobel, P., Hermann, A., Non-destructive analysis of nuclear fuel by means of thermal and cold neutrons, NIM A Volume 515, Issue 3, 11 December 2003, Pages 745-759

## The ODIN Project at the European Spallation Source

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ODIN (Optical and Diffraction Imaging with Neutrons) is a beamline project at the European Spallation Source (ESS). It is a collaboration between the ESS, the Paul Scherrer Institut (PSI) in Switzerland and the Technical University Munich (TUM) in Germany, with TUM as lead institution.

ODIN will provide a multi-purpose imaging capability with spatial resolutions down to the  $\mu\text{m}$  range. The pulsed nature of the ESS source - combined with a versatile neutron chopper system - will give access to wavelength-resolved information with variable resolution and bandwidths. Different imaging techniques, from traditional attenuation-based imaging to advanced dark field, polarized neutron or Bragg edge imaging, will be available within the full scope of ODIN with unprecedented efficiency and resolution. A summary of the technical full scope and its science application will be given and the updated conceptual instrument design including its challenges will be presented.

### Speaker Sessions and Seminars / 7

## Development of scintillator for a compact fast neutron imaging equipment at INPC of CAEP

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Fast-neutron imaging (FNR) is a nondestructive testing technology using fast neutrons as probes. The key problem of improving the quality of fast-neutron imaging is developing a suitable detector, which can convert the invisible fast-neutron image into a visible light image effectively and distinguishably.

The researchers in Institute of Nuclear Physics and Chemistry (INPC) of Chinese Academy of Engineering Physics (CAEP) are focusing on fast neutron imaging promotion and application. Now a transportable neutron imaging equipment has been installed based a compact accelerator neutron source using D-T reaction. In order to improve the quality of FNR, two kinds of fast neutron scintillators are developed at INPC. One is made of ZnS particles, resin and wavelength-shifting fibers (WSF), and the other is made of ZnS particles and polypropylene (PP). The appropriate parameters of the scintillators such as fibers arrangement, distance between fibers are optimized theoretically and the facture of the scintillators is also optimized. The scintillators are tested with 14 MeV neutrons at INPC and with fission neutrons at NECTAR, FRM II. The light output results show that all the scintillators are sensitive to 14 MeV neutrons and fission neutrons. The imaging results also matched the calculations, shown that the scintillators resolution is better than 1 mm.

## Neutron imaging study of ancient russian cultural objects



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Archaeological and cultural heritage objects have a special value in connection with their uniqueness and antiquity, being the main source of information about the past of humanity. Therefore, the attraction of modern methods of nondestructive testing for their research is the most reasonable. One of the progressive methods of non-destructive testing and researching of structural features and internal macro-inhomogeneities of archaeological objects is the method of neutron radiography and tomography.

Recently, there has been a significant increase in successful cooperation between the Joint Institute for Nuclear Research and the Institute of Archeology of the Russian Academy of Sciences. The complementary archaeological and physical research makes it possible to study a large number of valuable objects from various large-scale archaeological excavations in the territory of the Russian Federation. Several interesting results of such neutron studies at the neutron radiography and tomography facility on beamline 14 of the IBR-2 the high-flux pulsed reactor are presented.

## Speaker Sessions and Seminars / 21

### **Influence of varnish materials on the spatial and time-dependent moisture sorption dynamics of wood used for musical instruments studied by neutron imaging**

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The hygroscopicity of wood influences wooden musical instruments in various ways. On the one hand, the moisture content (MC) affects mechanical and acoustical properties via density, stiffness and damping. On the other hand, changes in MC result in swelling and shrinkage. Moreover, spatial MC gradients can lead to high internal stresses, which may result in cracks and fracture.

Varnishes act as a retarding barrier for moisture diffusion. Hitherto, the effect of varnish has been noted in terms of structural deformations (i.e. board cupping due to the one-sided varnish application) or as altered mass changes. However, more detailed studies on the impact of varnishes on the dynamics of the spatial MC distribution are scarce. Furthermore, old instruments commonly show a typical wear pattern. Areas that are regularly exposed to contact, sweat and/or breath, suffer from varnish deterioration. This raises the question whether the remaining varnish in worn off areas, mainly consisting of grounding or sealer materials, can still effectively protect against humidity changes.

Neutron imaging has proven to be a suitable technique to investigate moisture transport in wood. As neutrons are very sensitive to hydrogen, it is possible to determine and localise MC changes. In order to assess and characterize the moisture barrier performance of various varnish materials as well as worn off and intact varnish systems, an investigation with differently varnished wood samples was conducted.

The study was performed at the thermal neutron imaging beamline NEUTRA at the PSI. Imitating the conditions of musical instruments, the lateral sides were sealed, thus allowing sorption only at the upper and lower surfaces. The samples were preconditioned (35% RH and 20°C), ensuring equilibrated and known reference conditions. In total, 80 samples (10 runs with 8 samples each) were investigated, enabling a five-time repetition of 16 different wood and varnish material combinations. The samples were put in a climate chamber, allowing for an in-situ measurement of the MC changes while controlling temperature and RH. Based on a comparison of the reference radiograph to the radiographs taken over time, the spatial MC distribution and its time evolution were determined. For the time span studied (5h at high and low RH), no moisture sorption was observed for the completely varnished surfaces. The results revealed that the sorption occurs homogeneously across the surfaces and that pretreatments decelerate the moisture uptake. Interestingly, a grounding consisting of clear oil varnish and pumice powder displays a low barrier and a pretreatment mainly consisting of albumen and gum arabic did not lead to a protection at all.

The study has proven the applicability of neutron imaging for the investigation of spatial and time dependent changes in wood MC, enabling the examination of varnish influences. The results reveal the effectiveness of different varnishes and allow for an assessment of their influences on dimensional and acoustical properties of wooden musical instruments. The results can likewise be used for validations of material and sorption models, being relevant for e.g. coatings on wood in general (i.e. wood as building material) or in wood conservation science.

### Speaker Sessions and Seminars / 143

## Materials Research at CONRAD-2/HZB: Recent Developments and Outlook

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In recent years, the rapid development of neutron imaging methods by the operators of neutron sources and their users has triggered a tremendous improvement of both spatial and time resolution and furthermore the implementation of techniques that utilise new contrast mechanisms. Such developments have now become standard methods for many research fields in materials science. The range of current and potential applications is broad, including general materials research – with a particular emphasis on the area of materials and systems related to the generation and use of renewable energy – but also examples from biology, palaeontology, and cultural heritage and specific engineering materials. One important catalyst for the further improvement of neutron imaging techniques is the rapidly increasing demand for non-destructive and non-invasive in-situ and operando investigations of materials and devices that are used for energy supply, such as batteries and fuel cells. Here, the properties and the operation characteristics of the related materials and devices are often closely connected to the distribution and movement of light elements such as lithium and hydrogen. Due to their intrinsic properties, neutrons penetrate deeply into most common metallic materials while they have a high sensitivity to light elements such as hydrogen, hydrogenous substances or lithium. This makes neutrons perfectly suited probes for research on materials that are used for energy storage and conversion. In this contribution an overview to recent developments and activities at the CONRAD-2/V7 facility at Helmholtz Centre Berlin (HZB) will be provided. Technical developments on various fields will be presented, e.g. methods based on Bragg-edge imaging and dual-mode imaging, and data quantification techniques. Applications on energy-related materials research, employing in-situ techniques will be shown. Finally an outlook on the future of these activities at Helmholtz Centre Berlin will be provided.

## Speaker Sessions and Seminars / 131

## Diffusion coefficients of H in Zirconium alloys at operating temperatures by neutron imaging

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Zirconium based alloys are widely used in the nuclear industry, mostly as tubes and claddings operating in high-pressure water at temperatures between 250°C-350°C. Hydrogen (H) or deuterium (D) ingress due to waterside corrosion, and subsequently precipitates as a brittle hydride phase. Degradation mechanisms involve the accumulation of these brittle hydrides at cold spots or crack tips, as a result of H diffusion in response to thermal and stress gradients, respectively. In both cases, the diffusion coefficient of H at operating temperatures determines the crack growth velocity. Here, we have adapted a traditional method to determine the diffusion coefficient of H in Zirconium based alloys, in order to apply it to smaller specimens and significantly reduce experimental times. The method involves the formation of a surface hydride layer on a small specimen machined out of a plate or tube, and the determination of the H concentration profile obtained after an annealing treatment at the temperature of interest. The innovation of the present work is the non-destructive determination of these low H concentration profiles by neutron imaging, achieving ~5 wt ppm H concentration and a spatial resolution of ~25  $\mu\text{m}$  x 5mm x 10 mm. Experiments have been performed on specimens produced from Zircaloy-2 and Zr2.5%Nb rolled plates having different metallurgical conditions. Diffusion coefficients have been measured along the rolling and transverse directions of the plates at temperatures of 250°C, 300°C, and 350°C. Zircaloy-2 results agree well with literature values within typical uncertainties reported in the literature (~30%), and presented little variation with direction and metallurgical condition. On the other hand, Zr2.5%Nb shows larger diffusion coefficients, with considerable variations depending on the metallurgical condition of the plate and the direction of H diffusion.

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## Current status of neutron radiography and tomography at RADEN

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The pulsed neutron imaging instrument RADEN located at BL22 in the Materials and Life Science Experimental Facility (MLF) of J-PARC is designed to take full advantage of the high-intensity short pulsed neutron beam to conduct energy-resolved neutron imaging experiments 1. We are continually working on not only development of energy-resolved neutron imaging techniques and related devices such as state-of-the-art two-dimensional detectors (e.g.  $\mu\text{NID}$  2) but also improvement of experimental environments and techniques for conventional radiography and tomography. In RADEN, we can select several detector systems and imaging conditions according to the experimental purpose. In terms of conventional imaging, we can select the field-of-view (FOV) from 30 x 30 mm<sup>2</sup> to

300 x 300 mm<sup>2</sup>, the pixel size and number, the frame rate, and so on. For neutron radiography, it is possible to obtain transmission images with a spatial resolution of 0.05 and 0.3 mm for the minimum and maximum FOV, respectively, by using a back-illuminated CCD camera (2048 x 2048 pixels) in combination with a 6LiF + ZnS scintillator with the thickness of 0.05 or 0.1 mm. Furthermore, stroboscopic imaging at 25Hz, which allows the observation of dynamic process, was achieved by synchronizing the exposure time of an electron-multiplying CCD camera (1024 x 1024 pixels) with the timing of the neutron pulse generation at J-PARC. With this technique, we have succeeded in visualizing boiling refrigerant inside an automotive heat exchanger with time resolution on the order of tens of milliseconds. For neutron tomography, a new measurement system specific to pulsed neutron tomography was developed, in which both the camera-type detector and the rotation stage are controlled simultaneously by the software framework IROHA2, developed at J-PARC MLF, and the acquisition time is automatically adjusted so as to make the number of neutron pulses the same for each projection. Then we can obtain images with the same exposure condition and avoid the failure of data taking due to accidental beam stoppage or the change in beam destination. Three-dimensional image and its tomograms can be reconstructed from the acquired projection images by the filtered back projection (FBP) method or the simultaneous algebraic reconstruction technique (SART) with in-house reconstruction software utilizing a GPGPU computing system. The current spatial resolution of the tomograms, evaluated using a line-pair sample, was about 0.3 mm in the case that the projection images were taken with the spatial resolution of 0.3 mm. This number can be improved by optimizing the experimental conditions such as the distance between rotation center of the object and scintillator, the number of projection images, and so on.

In this presentation, we will report the current status of neutron radiography and tomography at RADEN together with recent results using commercial products and biological specimens, and also report initial results of a new trial using a quasi-monochromatic neutron beam created using a double-disc chopper installed in the optical system of RADEN.

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## Comparative study of ancient and modern Japanese swords using neutron tomography

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Japanese swords have superior characteristics in strength and toughness, giving them important value not only as a historical work of art but also from a metallurgical point of view. It is known that Japanese swords differ in the making process depending on the era and place of manufacture. Modern Japanese swords are produced based on the making processes developed after the Edo period (17th - 19th century). On the other hand, with regard to ancient Japanese swords 'Koto' manufactured from the Kamakura period to the Muromachi period (12th - 16th century), there are still many unknown points in the materials and making processes used due to lack of historical records. Therefore, evaluating the internal crystallographic structure of ancient Japanese swords and comparing them with modern Japanese swords are important footholds for understanding the historical change of the Japanese sword making processes. From the above viewpoints, we have investigated the three-dimensional internal structure and crystallographic structure of ancient and modern Japanese swords non-destructively using neutron tomography and energy-resolved neutron imaging (Bragg-edge imaging). In this study, we report on the results of the neutron tomography measurement. All

experiments were carried out at the pulsed neutron imaging instrument RADEN located at BL22 of J-PARC MLF 1.

In the resulting neutron tomograms, it was observed that the neutron transmittance was different between steels around the cutting-edge and steels inside the blade in each of two ancient Japanese swords manufactured in the Muromachi period (named Morikage and Sukemasa) and a third modern Japanese sword (named Masamitsu) manufactured in the mid-20th century. This corresponds to the formation of the martensitic phase around the cutting-edge by quenching process, referred to as 'Yaki-ire', during sword making. Furthermore, in the tomograms of the blade edge, it was observed that the neutron transmittance was distributed corresponding to the wavy pattern, referred to as 'Hamon', particular to Japanese swords. Additionally, several line-like structures presumed to have been produced in the folding and forging processes were observed only in the modern Japanese sword. A detailed comparison of the results for the ancient and modern swords will be presented.

This work partially includes the result of 'Collaborative Important Researches' organized by JAEA, QST and U. Tokyo.

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## High-resolution Detector for Neutron Diffraction and Quantification of Subsurface Residual Stress

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We have developed a new high-resolution large-area detector for neutron diffraction imaging, specifically for the measurement of subsurface residual stress in engine components. Neutron diffraction typically requires monochromatic thermal or cold neutrons and neutron flux at the detector is orders of magnitude lower than for standard neutron radiography. Therefore the detection efficiency for incident neutrons must be maximized in order to reduce acquisition times. Here we use the high absorption cross section of LNI (6Li:NaI-xI:Eu,Tl) scintillators coupled to an Anger camera consisting of an array of silicon photomultiplier (SiPM) detectors.

The LNI scintillator is derived from the well-known NaI scintillator and comes in two formats for this application, a vapor-deposited film and a crystal sliced into 1-2 mm thick layers for imaging. The vapor-deposited films have a columnar structure, providing the advantage of enhancing spatial resolution. The sliced crystals allow for a thicker layer of LNI, and hence higher efficiency, than the films.

The LNI scintillator has demonstrated a high neutron yield, up to 153,000 photons/neutron, as well as a very high gamma equivalent energy up to 4.7 MeV, which is approximately the theoretical maximum. These attributes make it a good candidate for pulse height discrimination (PHD) to remove gamma events. A 1 mm slice of LNI:Tl,Eu produced 41,000 photons/MeV, which is ~20% greater than NaI:Tl. Columnar films have demonstrated the highest spatial resolution, up to 500  $\mu\text{m}$  coupled to the Anger camera and up to 7 lp/mm coupled to an EMCCD detector.

Use of spatially resolved detectors for diffraction signals from single and polycrystalline materials is realizing strong interest in the neutron community based from experience at advanced light sources

which have plenty of photons to work with. Since diffraction signals from polycrystalline materials are inherently weak, the use of this approach at neutron facilities, which typically provide a low flux of neutrons in the thermal or cold energy ranges, poses unique problems. This paper presents important aspects of utilizing high-resolution and high efficiency neutron detectors for obtaining important engineering measurements such as residual stress considering the target materials of interest. We present for the first time measurements associated with diffraction signals captured using an Anger camera with high spatial resolution for an example scattering polycrystalline powder appropriate for the incident mono-energetic cold neutrons at the CG1 (Cold Guide Hall) beamline of the High Flux Isotope Reactor at the Oak Ridge National Laboratory.

## Speaker Sessions and Seminars / 108

### Fibre-optics taper for high resolution neutron imaging

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The increased demand of high-resolution neutron imaging has not been followed by a correspondingly increased availability of high-resolution options, due to the technical challenges and high costs of designing and manufacturing such systems. Neutron flux limitations are also a key factor that hinders the adoption of traditional high resolution solutions.

To overcome this situation and to open up the possibility to perform high resolution investigations to a larger number of facilities (thus widening the pool of potential users by this increased availability), we propose the use of a fibre optics taper as add-on to existing standard-resolution systems 1. A fibre optics taper is a bundle of tapering optical fibres that are bunched together to preserve their relative arrangement. Such a device can transport light from one end to the other very efficiently, while providing a substantial magnification of the incoming image.

By constructing a suitable holder that attaches to the existing imaging setup to one end and to a high-resolution scintillator to the other (figure 1), one can achieve spatial resolutions of 20  $\mu\text{m}$  with relative ease, while keeping the counting time low due to the high transport efficiency.

Sub-20  $\mu\text{m}$  resolutions have also been achieved with such a system by using zoom lenses and, by employing a specially designed 157-Gd enriched scintillator, resolutions approaching 10  $\mu\text{m}$  have been measured.

In this presentation we will show the results of our systematic investigations regarding achievable resolution, conformality of the recorded images and light transport efficiency and we will discuss about shortcomings and advantages of such a setup.

In the second part of the presentation, we will show a use case of such a setup, outlining the reasons why the taper was used and presenting the results obtained by such investigation.

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1 M. Morgano, et. al., "Unlocking high spatial resolution in neutron imaging through an add-on fibre optics taper," *Opt. Express* 26, 1809-1816 (2018)

## Speaker Sessions and Seminars / 43

### MODERN FACILITY FOR NEUTRON RADIOGRAPHY AND TOMOGRAPHY FOR APPLIED RESEARCH ON THE BASE OF THE VVR-K REACTOR

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#### MODERN FACILITY FOR NEUTRON RADIOGRAPHY AND TOMOGRAPHY FOR APPLIED RESEARCH ON THE BASE OF THE VVR-K REACTOR

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At the basin-type reactor on thermal neutrons VVR-K, an experimental facility is setting up to conduct researches using neutron radiography and tomography. A neutron beam with a cross section of 20 x 20 cm forms a system collimator, for which the value of the characteristic parameter  $L / D$  can vary from 350 to 2000.

#### INTRODUCTION

The neutron radiography method consists in obtaining neutron images of the investigated objects. Due to the different degree of attenuation of the neutron beam during the passage through materials of different chemical composition, density and thickness of the components of the investigated sample, the information on the internal structure of the materials with spatial resolution at the micron level is provided. This method of nondestructive control is characterized by a deeper penetration into the thickness of the material compared with complement x-ray introscopy method and is advantageous in studying samples with both light (for example, hydrogen or lithium) and heavy elements. All modern and newly created neutron sources are equipped with neutron radiography and tomography facilities. Methods of neutron radiography now is widely applied for material investigations and products for nuclear technologies, paleontological and geophysical objects, unique objects of cultural heritage. It should be noted that now, much attention is also paid to unique research of physical and chemical processes in fuel cells and batteries, processes associated with the penetration of hydrogen or water into the thickness of various materials. Functional development of the invention of neutron radiography is made by neutron tomography. In this method the volumetric reconstruction of the internal structure of the investigated object is performed from a set of individual radiographic projections, i.e. for different angular positions of the sample relative to the direction of the neutron beam.

The presented work describes in detail the design and main parameters of the new experimental facility for investigations using neutron radiography and tomography, created on the 1st channel of the VVR-K reactor.

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## Using Neutron Imaging to Understand Water Dynamics in Anion Exchange Membrane Fuel Cells

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#### Using Neutron Imaging to Understand Water Dynamics in Anion Exchange Membrane Fuel Cells

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Polymer electrolyte membrane fuel cells are considered to be one of the most promising alternatives to internal combustion engines as power sources for vehicles due to their environmental friendly features and high energy conversion efficiency<sup>1,2</sup>. Water management is of vital importance to achieve maximum performance and durability for polymer exchange membrane fuel cells. On one hand, to maintain good ionic conductivity, the membrane and ionomer in the catalyst layers (CLs) have to remain hydrated. On the other hand, if there is too much liquid water present, the CLs and gas diffusion layers (GDLs) will be heavily flooded, resulting in high mass transport resistance. Compared to the proton exchange membrane fuel cell (PEMFC), the anion exchange membrane fuel cell (AEMFC) is desired for its widely accepted potential to reduce the cost of the catalysts, membrane and stack components. However, many researchers have failed to achieve high power in AEMFC because of poorly controlled and poorly understood water phenomena.

One aspect of the operation of AEMFCs that has only recently been appreciated is that water management is potentially much more complex in AEMFCs than PEMFCs<sup>3,5</sup>. In PEMFCs, no water is consumed by either reaction and 2 water molecules are produced per oxygen molecule consumed at the cathode. Hence, there is only a mild concern of cathode flooding at high current density. In AEMFCs, water is both produced (4 H<sub>2</sub>O at the anode for every O<sub>2</sub> consumed at the cathode) and consumed (2 H<sub>2</sub>O molecules per O<sub>2</sub>). This means that AEMFC engineers need to not only watch for flooding at the anode, but also make sure that liquid water is continuously available in the membrane and at the cathode to avoid dry out – while also avoiding excess cathode water. It also means that the optimum water content for the overall cell is a dynamic function of the current density – this is not at all the case for PEMFCs. Thus, it is urgent to develop experiments to better understand the water distribution in operating AEMFCs and take steps to actively control the water content and balance. .

In this work, neutron imaging experiments were conducted at the NIST Center for Neutron Research and for the first time the amount and distribution of water and its impact on AEMFC performance was measured. The results show that liquid water is present in the membrane and not gas phase water from humidified air. The neutron imaging results also led to a redesign of the cell.

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### Speaker Sessions and Seminars / 10

## Energy-resolved Neutron Imaging of Materials for Nuclear Energy

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Nuclear energy technologies are used to produce a significant portion of the world's electricity, and this will continue to be true as numerous countries build or expand their nuclear power plant fleets. The continued use and growth of nuclear energy globally, however, faces significant materials science and engineering challenges. These include the development of advanced nuclear fuel materials with accident-tolerant properties, structural materials with high corrosion resistances, and waste forms appropriate for geological disposition. Energy-resolved neutron imaging techniques such as neutron energy resonance imaging and Bragg edge imaging offer the capability to nondestructively characterize, understand, and explore materials for nuclear energy. Development of these techniques has grown exponentially as pulsed neutron sources and neutron detectors continue to advance. Information that can be obtained and spatially resolved includes isotopic composition, temperature, strain, and stress, as well as crystallographic phase and orientation. Efforts are underway at Oak Ridge National Laboratory to develop neutron imaging capabilities to study materials for nuclear energy. This discussion will focus on the recent progress to develop and leverage energy-resolved neutron imaging techniques to study materials with applications in the nuclear energy sector and will include recent experimental results. For example, we have mapped the three-dimensional spatial distribution of uranium and gadolinium in UO<sub>3</sub>-Gd<sub>2</sub>O<sub>3</sub> spheres using neutron energy resonance imaging to understand the chemical process used to produce them. Neutrons in the epithermal energy region (roughly 0.1 eV to 1 keV) were used. Results from preliminary studies using Bragg edge imaging to understand the conversion of spherical uranium-containing kernels from oxide to either carbide or nitride, which are of interest for use in several proposed advanced nuclear fuel forms, will also be addressed.

**Speaker Sessions and Seminars / 167**

## **The upgraded neutron grating interferometer at ANTARES – Design, Performance and Applications -**

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Neutron grating interferometry (nGI) is a relatively new neutron imaging technique which is the adaption of a Talbot-Lau Interferometer for neutrons 1. It simultaneously delivers information about the transmission (TI), phase shift (DPC) and the scattering (DFI) inside a sample 1.

In particular the DFI has generated high interest, due to its ultra-small-angle neutron scattering (USANS) contrast mechanism, allowing to indirectly resolve structures which cannot be directly resolved by an imaging instrument 2,3.

For instance, nGI is sensitive to magnetic domain walls and consequently allows to measure the effect of induced stress in a sample onto the mobility of its magnetic domains [4]. Moreover, the distribution of flux domains within type-I and type-II superconductors has recently been visualized [5],[6]. Also there have been strong efforts to use nGI and particularly the DFI as tools for quantitative measurements of microstructures in materials. A theory has been proposed, which directly links the DFI contrast within the material to a Fourier back transform of its scattering function evaluated at a correlation length  $\xi_{GI} \sim \lambda$  [7].

A prerequisite for such quantitative measurements is a high signal-to-noise-ratio (SNR). For DFI measurements it has been shown that the main reasons for statistical uncertainties are (i) low DFI signal and (ii) low visibility [8]. Here, the visibility is the quotient between the amplitude and the mean value of the oscillation during an nGI scan and is an indicator for the performance of an nGI setup.

While the DFI signal is, as mentioned above, connected to the correlation length which can be tuned during the experiment, the visibility is strongly dependent on the quality of the gratings. Especially the quality (absorptivity) of the analyzer grating (G2) is a great concern here, as it is generally the grating with the smallest period (several  $\mu\text{m}$ ). Current fabrication techniques cause the grating to strongly deviate from an ideal binary absorption profile. As has been shown in [9] this strongly degenerates the visibility. Furthermore, tuning the correlation length either lowers the achievable real space resolution or results in a change in neutron wavelength which also causes a decrease in visibility.

Hence a high visibility is an essential basis for quantitative measurements. In our contribution we will present the upgraded nGI setup at the ANTARES beamline at FRM II. This nGI setup has been heavily redesigned, compared to its precursor [10]. The redesign allowed to optimize the distances between the gratings, as well as the grating periods. In particular, the source and analyzer gratings, which are both absorption gratings, have been improved towards binary absorption profiles. With these changes in the improved ANTARES nGI we have achieved a visibility of 75% over the whole detector area (76mm x 76mm) at the design wavelength of 4 Å. It is worth noting that this visibility is very close to the theoretical limit imposed by the spatial coherence generated by the used G0 grating.

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## Internal stress in electrical steel sheets – Effects and Applications

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Electrical steel sheets are used in transformers as well as in rotors and stators of electrical motors to guide the magnetic field. The efficiency of an electrical steel sheet strongly depends on the amount of energy lost during the reversal of magnetization, which is dependent on the mobility of the magnetic domains. The mobility of the magnetic domains is in turn influenced by the treatment of the electrical steel sheet during fabrication. Especially internal stress induced by manufacturing (e.g. by cutting the sheets) causes a degradation of the mobility of the magnetic domains, due to the magneto-elastic effect 1,2. As a result the overall efficiency decreases, which is evident in larger hysteresis losses in classical magnetization measurements. However, there is a lack of spatially resolved techniques which probe the magnetic domain constellation in bulk samples of technically relevant dimensions.

In this context, neutron grating interferometry (nGI) is the technique of choice to analyze the local effect of induced stress on the magnetic properties of a material, as nGI does not only allow to analyze the surface but also the bulk local magnetic properties, which is not possible with most other techniques 3.

nGI simultaneously provides information about the neutron transmission (TI), the phase shift of the neutron wave inside a material (DPC) and the amount of ultra-small-angle-neutron scattering (USANS) inside a sample 3. Especially the contrast provided by USANS has aroused high interest, as the resulting image (DFI) is sensitive to the distribution of magnetic domain walls,

which serve as possible scattering centers. Hence the DFI signal is related to the local distribution and size of magnetic domains inside a sample.

In our contribution we will show how stress induced by the manufacturing process locally changes the mobility of the magnetic domains. Here, we will focus on two cases: First, the unintended degradation of the mobility due to blanking, which induces a high amount of stress close to the cutting area, therefore limiting the mobility of the magnetic domains and resulting in a change in the magnetic hysteresis of the material. Measuring the hysteresis close to the cutting area with high resolution has recently become possible due to upgrades to the nGI used at the ANTARES beamline.

The second case is the intended degradation via imprinting on the electrical steel sheets which allows to guide the magnetic field without having to resort to cutting out parts into the sheet and therefore lowering the mechanical stability. This allows to use electrical steel sheets which have less losses during the reversal of magnetization due to their material composition, but as a trade off have a lower yield strength. This in turn increases the efficiency of electrical engines.

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#### Speaker Sessions and Seminars / 54

### Pareto Optimal Solutions for a Neutron Radiography Collimator

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Neutron radiography is a non-destructive technique extensively used in the investigation of materials. The integrity of the investigation depends in part on the quality of the radiographic image produced by a neutron radiography system. A neutron collimator is one of the components that contribute to the quality of radiograph. Optimization of a neutron collimator entails finding the balance between two conflicting objectives, namely the size of homogeneous (flat flux) region and the intensity of the neutron beam flux. The diameter and the position of the collimator aperture are among the parameters that determine the homogeneity and the intensity of the neutron beam flux. It is desirable to find the best parameters for a neutron collimator design. A collimator optimizer based on ray tracing and multi-objective particle swarm optimization techniques was designed, implemented and tested to provide design parameters in the form of Pareto optimal solutions. The desired optimal solutions for the aperture diameter and position can be chosen from the set of Pareto optimal front graphs, to suite the conditions of a particular neutron radiography system. The test results showed that the Pareto optimal front graph has a linear form, and all Pareto optimal solutions were found to be at the closest position from the neutron source.

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### Crystallographic structure study of a modern Japanese sword Masamitsu using pulsed neutron imaging

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#### Abstract:

The metallographic characteristics are necessary information to clarify making processes of Japanese swords, since the processes were not clearly handed on. Japanese swords have been produced in various ages and areas since ancient times. Therefore, in order to deduce characteristics of Japanese swords depending on the area and the age, we need to analyze many Japanese swords. However, It is necessary to analyze them with a nondestructive method since Japanese swords are valuable. Neutron experiments are powerful tools to study metallic cultural heritages because of their high penetrating power and capability to give crystallographic information 1. By analyzing position dependent Bragg edge spectra obtained with a pulsed neutron source, quantitative visualization of the crystallographic information of a sword can be achieved 2.

Although we would finally like to investigate old Japanese swords, it is important to know the characteristics of modern Japanese swords in order to understand crystallographic characteristics of a Japanese swords. In this work, we investigated crystallographic information of the modern Japanese sword made by Masamitsu in 1969. The experiments have been performed at the Energy-Resolved Neutron Imaging System, RADEN at J-PARC 3. A tip, middle and tang regions of the Masamitsu sword were measured with a counting-type 2D detector, GEM [4]. We analyze the measured 2D-transmission spectra using RITS code [5] to obtain spatial distribution of the crystallographic information. The results suggest that the cutting edge of the modern sword Masamitsu is strongly quenched to produce a hard martensite phase in this region.

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## Crystallographic structure study of a Japanese sword Sukemasa in Muromachi period using pulsed neutron imaging

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Japanese swords are very attractive not only as a work of art but also a metallurgical point of view. There were five traditional styles (Gokaden) of Japanese sword-making in the Koto (old sword) age; A.D. 987–1596. Since Japanese vintage swords became valuable, it is indispensable to establish non-destructive analysis method to identify some peculiar characteristics, related to the sword making processes. Neutron experiments are powerful tool to study metallic cultural heritages due to their high penetrating power and capability to give crystallographic information 1. Bragg edge imaging, in particular, gives real-space distributions of bulk information in a crystalline material as well as neutron tomography. In addition, by analyzing position dependent Bragg edge spectra obtained at the pulsed neutron source, quantitative visualization of the crystallographic information of a sword can be achieved 2.

In this work, we investigated crystallographic information of a Japanese sword made by Sukemasa in Izumi province (out of Gokaden) in the first quarter of the 16th century. The experiments have been performed at the Energy-Resolved Neutron Imaging System, RADEN at J-PARC 3. The Sukemasa sword was measured at the point of blade, its middle and tang with a counting-type 2D detector and at middle part with a CCD camera. We are now analyzing the measured 2D-transmission spectra using RITS code [4] to obtain spatial distribution of the crystallite size, the texture variation, the d110 shift and its broadening for each sample. Complementary data analysis using white beam tomography is also on going. Detailed analysis results will be presented.

#### **Acknowledgement**

This work partially includes the result of ‘Collaborative Important Researches’ organized by JAEA, QST and U. Tokyo.

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### **Speaker Sessions and Seminars / 46**

## **Offical or Illegal? Tomographic analysis of plated silver coins from Ancient Greece.**

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The first coins were made of electrum and were minted during the 7th century BC in Lydia (Asia Minor). Plated electrum coins began to appear soon after, and these have usually been identified as privately manufactured 'fakes'. But it is possible that they were in fact produced in the state's own mint. The art of plating coins required a very high skill level. Attaching a thin piece of electrum over another metal (silver was the preferred core at this time) required a high degree of metallurgical knowledge and practical skills. The Australian Centre for Neutron Scattering has been involved in a study with the Australian Centre for Ancient Numismatic Studies at Macquarie University since 2014. A number of plated coins have been studied using a combination of Neutron Tomography, Diffraction and Texture Measurement, as well as SEM and X-Ray Tomography. Our study also includes later ancient silver that can now be shown to be plated. The project has explored the thickness of the plating layer, porosity in the metals, and the presence of intermediate layers. Silver plating layers of 0.4mm are common and gold leaf layers of less than 0.1mm over a silver core have been studied.

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## **Conversion from Film Based Transfer Method Neutron Radiography to Computed Radiography for Post Irradiation Examination of Nuclear Fuels**

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The Neutron Radiography Reactor (NRAD) located at the Idaho National Laboratory (INL) in Southeast Idaho in the United States provides researchers with neutron radiography nondestructive examination images of irradiated nuclear fuels for post irradiation examination (PIE). Past, present, and future reactor fuel designs of varying geometry are routinely examined for behavioral characteristics after irradiation. Typical behaviors of interest include cracking, swelling, migration, voids, and changes in density.

Transfer method neutron radiography is performed at NRAD for irradiated fuels. Dysprosium and cadmium-filtered indium conversion screens (foils) are activated, then coupled to industrial X-ray film and the decay radiation from the foils exposes the film and provides radiographs from thermal and epithermal neutron energies, respectfully. The film is developed with a commercial photo processor, interpreted by a certified neutron radiographer, and then digitized with a high resolution flatbed scanner. The digital images are transmitted to the researcher as the record copy.

Although effective, the process is slow, labor intensive, and expensive. Several types of film previously used are now obsolete, and the cost of film has increased significantly over the years. In an effort to improve imaging efficiency and capability, NRAD has initiated an effort to convert from film to computed transfer method neutron radiography utilizing photo-stimulable phosphorus (PSP) imaging plates (IP). The transfer method remains the same, however the PSP is scanned with a dedicated computed radiography (CR) system and the resultant image is interpreted by a certified neutron radiographer. This technique eliminates film, processing, chemicals, silver recovery, and film storage. The images are immediately available for post processing utilizing open source algorithm software. This paper describes the characterization and qualification process for converting from film to computed neutron radiography using the transfer method, and also compares the performance of CR with the existing film-based process. CR has proven to reduce the time required to produce an image and has improved the efficiency of neutron imaging operations at NRAD. Image resolution between film and CR was determined from a variety of image quality indicators, and has proven to be acceptable for programmatic use. NRAD is currently running both film and CR for comparison purposes to establish a baseline resolution.

## Speaker Sessions and Seminars / 66

## Development of event-type neutron imaging detectors at the energy-resolved neutron imaging system RADEN at J-PARC

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At the RADEN instrument 1, located at beam port 22 of the high-intensity, pulsed neutron source at the Materials and Life Science Experimental Facility at J-PARC in Japan, we take advantage of the accurate measurement of neutron energy by time-of-flight to perform *energy-resolved neutron imaging*. By analyzing the two-dimensionally resolved, energy-dependent neutron transmission, these techniques can image macroscopic distributions of microscopic properties for bulk materials *in situ*, including crystallographic structure (Bragg-edge transmission), nuclide-specific density and temperature distributions (resonance absorption), and internal/external magnetic fields (polarized neutron imaging). At RADEN, we use advanced neutron imaging detectors based on cutting-edge technologies, such as micropattern detectors and fast, all-digital data acquisition systems with Field Programmable Gate Arrays (FPGAs), to provide event-by-event timing information with sub- $\mu$ s resolution.

To better perform these measurements at RADEN, we are continually working to improve our event-type neutron imaging detectors for better spatial resolution and shorter measurement times and, as a user facility, to improve the ease-of-use of their control and analysis software. In particular, we are actively developing a micropattern detector known as the Micropixel chamber based Neutron Imaging Detector ( $\mu$ NID) 2. The  $\mu$ NID uses a gaseous time projection chamber (TPC) with a micropixel chamber ( $\mu$ PIC) micropattern readout. This 400- $\mu$ m pitch, two-dimensional strip readout is coupled to an FPGA-based data acquisition system designed for high-rate operation. Absorption on  $^3\text{He}$  in the gas mixture facilitates neutron detection, and the detailed tracking and analysis of the reaction products in the TPC enables a fine spatial resolution. The  $\mu$ NID currently provides 100  $\mu$ m spatial resolution with a 10 cm  $\times$  10 cm field of view, 0.25  $\mu$ s time resolution, 26% detection efficiency for thermal neutrons, ultra low gamma sensitivity, and an effective peak count rate of 1 Mcps<sup>3</sup>. We have recently redesigned the  $\mu$ NID control software to allow full integration into the automated experiment control system at RADEN, and we are carrying out optimization of the analysis algorithms for improved image quality and rate performance. We are also developing a new 215- $\mu$ m pitch  $\mu$ PIC readout for improved spatial resolution, and a  $\mu$ NID with boron-based converter for increased count rate via a much-reduced event size.

In this presentation, we will give an overview of our detector development activities at RADEN and discuss in detail the present status of the  $\mu$ NID system. Demonstration measurements for energy-resolved neutron imaging and preliminary results for the small-pitch  $\mu$ PIC and  $\mu$ NID with boron converter will also be shown.

This work was partially supported by the Momose Quantum Beam Phase Imaging Project, ERATO, JST (Grant No. JPMJER1403).

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## Speaker Sessions and Seminars / 153

**Neutron imaging of hydrogen diffusion in polycrystalline forsterite aggregates**Sarith Patabendigedara<sup>1</sup> ; Filomena Salvemini<sup>2</sup> ; Simon Clark<sup>3</sup><sup>1</sup> *Department of Earth and Planetary Sciences, Macquarie University*<sup>2</sup> *ANSTO*<sup>3</sup> *Macquarie University***Corresponding Author(s):** sarath-kumara.samaya-manthr@hdr.mq.edu.au

An understanding of hydrogen diffusion in nominally anhydrous minerals (NAMs) is an essential context of correct interpretation of conductivity dissimilarity in Earth mantle. The mechanism of hydrogen diffusion in dominant mantle minerals was described by Demouchy (2010) and Demouchy and Casanova (2016) using a defect model in crystalline materials. This concept is well-known and well documented in the material science community (Nowick 2012) where the effects of in-grain and grain boundary (gb) diffusion are separated using the bricklayer model and other associated derivatives of this model (Tuller 2000). Separation of the two components of the proton conductivity in olivine will substantially improve current proton conduction model for Earth mantle. Finally, it will help to interpret magnetotelluric conductivity data and will give prospects to find new mineral sources and explain other sub-surface geological phenomena such as volcanism and plate tectonics. (Demouchy and Bolfan-Casanova 2016)

A recent insight is that the high conductivities determined from proton conduction measurements at low temperatures are mainly due to conduction along grain boundaries (Demouchy 2010). Demouchy (2010) was the first, and to date only experimental work on hydrogen grain-boundary diffusion in olivine, the dominant upper mantle mineral phase. We have repeated Demouchy's experiment with neutron imaging which is a most promising in-situ technique to image hydrogen diffusion profile. Neutrons can penetrate through the capsule while providing information about contents and they are highly sensitive to hydrogen in the sample. Therefore, neutron imaging allows measuring time and temperature dependent gb hydrogen diffusion rates in mantle minerals.

We carried out a series of experiments where we diffused water (H) through a forsterite polycrystalline matrix at high-pressure and temperature. The capsules and their contents were imaged using the DINGO neutron tomography instrument at the Australian Centre for Neutron Scattering. The results indicate hydrogen transport inside the forsterite polycrystalline matrix as changing neutron attenuation along the diffusion direction of the polycrystalline block and it correlates with temperature dependent hydrogen diffusion in this mineral. This study revealed the ability of neutron imaging technique to find the proton diffusion coefficient of NAMs. We are sharing these results in this conference.

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The Anger Camera developed by the detector group at the Oak Ridge National Laboratory was utilized for the present work for its unique advantage of employing multiple modules to obtain desired/large active measurement area for detecting diffracted/scattered thermal neutrons. Considering the relatively small flux associated with diffracted/scattered neutrons available in the detector view area possible for structural materials as scattering specimens, suitable efficiency with high spatial resolution is a requirement for utilizing imaging type detectors. The potential to implement pulse shape (in addition to pulse height) discrimination based scintillators further enhances the ability to detect diffracted neutrons with enhanced signal to noise ratio. In this paper, initial results associated with <sup>6</sup>Li glass based scintillator will be presented. Plans are underway for replacing it with a recently manufactured and highly efficient Lithium Sodium Iodide (LNI) based large area scintillator yielding a spatial resolution of 500  $\mu\text{m}$  while maintaining very high neutron detection efficiency. The Anger camera system implemented in our study has three major sub-assemblies: the optical front-end, the preamp and the digital processing board assembly. The optical front-end consists scintillator that is optically coupled to an array of photomultiplier tubes (PMTs). The Anger camera is neutron detecting system consisting of a scintillating film, and a set of multi-anode photomultiplier tubes (PMT). Each PMT consists of an 8 x 8 anode array. In the reported experiments, the authors explored the feasibility of using this system to detect and quantify diffraction peaks and peak shifts at the Neutron Residual Stress Facility (NRSF2), High Flux Isotope Reactor (HFIR) in the Oak Ridge National Laboratory (ORNL). The imaging based detector was attached to the existing Helium based detector system using a custom-built frame to ensure consistent relative angles with the Anger camera detector during measurements. ABS plastic shielding mechanism was developed consisting of two separate components: a slit-like structure close to the sample and a shield around the camera. Due to the two-dimensional nature of the pixelated detector, acquired data will be in the form of an image such that a diffraction peak appears as vertical high intensity line of pixels with finite thickness (corresponding to a part of the Debye-Scherrer diffraction cone) indicative of measurement parameters. The image data was vertically integrated, and a peak fit algorithm was implemented using a custom developed software. Reference measurements using polycrystalline powders with known atomic planar spacing will be discussed along with measurement settings associated with expected resolution for peak shift measurements. Initial results are promising and demonstrate that a suitable scintillation-based neutron detecting system is viable for residual stress based diffraction measurements. Small area detectors are also feasible with suitable consideration to scattering volume and distance to detector.

## Speaker Sessions and Seminars / 64

### Energy resolved imaging using the GP2 detector: progress in instrumentation, methods and data analysis

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We report on the continued development of the 'GP2' detector [ 1 ], highlighting a selection of energy resolved measurements and associated methodology. GP2 is a 100k pixel time-of-flight (ToF)

neutron camera, which combines a gadolinium converter film and a CMOS (Complementary Metal Oxide Semiconductor) readout sensor [ 2 ]. This paper is separated into three categories; (1) detector optimization and integration into the Imaging and Materials diffractometer (IMAT) [ 3 ] (2) method development using sample environment and (3) the ensuing data reduction and analysis.

The process of taking an R&D detector into the user program of IMAT is briefly described. Recently the GP2 detector has been integrated with the IMAT control software, achieved via the 'Experimental Physics and Industrial Control System' (EPICS) [ 4 ], which means that the detector is controlled and operated from the ISIS 'IBEX' environment [ 5 ]. This ensures that the experimental run-time is synchronized, all instrument parameters are recorded (such as beamline monitors) and that data is archived. IMAT changes imaging detectors via a robot arm, for which bespoke mechanics have been commissioned. Improvements to the detector neutron efficiency via isotopically enriched gadolinium will also be discussed.

GP2 has been used to perform the first low temperature imaging study on IMAT. Characterization measurements of the CCR (closed cycle refrigeration) sample environment and energy resolved measurements from samples which undergo phase changes at low temperatures will be reported.

The ToF spectrum recorded in each pixel of the detector provides much more information in addition to the macroscopic cross section. Unique physical parameters can be extracted via feature parameterization; fitting a Bragg edge for texture or strain for example. A complete parameterization, a Rietveld refinement in transmission, is usually an under-determined problem and requires good prior knowledge of the sample. Here we highlight methods of contrast enhancement and feature extraction that do not require prior knowledge of the composition of sample or extensive fitting. These methods of contrast-enhancement simply require the existence of unique features in the ToF spectra. The effectiveness of methods like principal component analysis and energy-band division are of course limited by 'how different' the ToF is across the pixels. However, these methods offer a simple 'online' analysis. Their immediate benefit is to distinguish features that in a white-beam image (integrated in ToF/energy) would 'accidentally' have had the same grey value due to their combination of path length, density and cross section being similar despite their ToF being different. One example is shown in figure (left), where the white beam image does not discriminate between the alternating austenite/martensite nuts, on a martensitic bolt. By choosing an appropriate weighting scheme the materials can be separated resulting in the three distinct grey-values shown in figure (right).

Indico rendering error

Could not include image: Problem downloading image (<https://s20.postimg.cc/xq15e8ki5/Nut>)

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## Speaker Sessions and Seminars / 94

### Laue Multi-Grain Indexing with Neutrons

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Polycrystalline materials undergoing thermal or mechanical loading suffer deformations and damage which can modify their grain size, orientation and texture. To obtain multigrain information from crystalline samples, 3D grain mapping is performed using x-ray diffraction at synchrotron radiation sources. However, the scope of this technique is limited due to the lack of penetration power inside of bulky metallic samples. Neutrons have usually a higher penetration depth in comparison with x-rays, and some grain maps have already been reconstructed from neutron data. However these methods have so far been dependent on the use of energy-resolved neutron imaging techniques, either with a velocity selector or a time-of-flight approach.

We are developing a new method to obtain the position, orientation and shape of grains from polycrystalline samples without initial wavelength resolution needs. So far the technique has been validated for grain sizes in the range of hundreds of microns and samples up to 2 cm diameter. The novelty of the reconstruction approach, enabling white beam measurements, lies in the use of a forward model to predict diffraction patterns being fitted to the position of the experimental diffraction spots and hence revealing number, position and orientation of individual grains. This is very different from common energy resolved crystal diffraction where the wavelength is typically used to solve Bragg's law.

The approach utilizes the knowledge of the beamline setup and crystal composition to predict the geometry of the Laue pattern measured during the experiment on the diffractometer. The code compares and optimizes the predicted pattern with respect to the measured diffraction patterns concerning grain positions and orientations until the match is satisfactory. As a result the positions and orientations of contributing grains are retrieved. The process of search and optimization is first done for individual grains and repeated until no additional grains are found with statistically significant anymore.

Experiments were performed at the E11 thermal beamline of the BERII neutron source at the Helmholtz Zentrum Berlin in Germany. The detection system of the installed instrument FALCON is a scintillator-camera based neutron imaging set of two detectors with a field of view of 400x400 mm each and a pixel sizes of 100  $\mu\text{m}$ . For our experiments we typically set one detector in forward diffraction direction and the other one in backward diffraction mode.

The current version of the code has already been proven capable of indexing 18 grains from an annealed  $\alpha$ -Fe cylindrical sample with 5 mm diameter and 5 mm height. In addition 8 grains have been indexed from a YBaCuFeO5 multiferroic oligo-crystal using only forward diffraction data.

With the current version of our code white beam Laue neutron multi-grain indexing becomes possible. However, this is only the first step towards retrieving a full 3D grain map including the morphology. Our next steps are focused on advancing these capabilities, finding new applications and bringing the code to a user-friendly level.

## Speaker Sessions and Seminars / 144

### Comparative study: X-ray and neutron CT on a mummified votive offering

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This study involved investigation of an unusual Egyptian votive mummy (IA.2402) of unknown age and provenance, generously loaned by the Australian Institute of Archaeology (AIA) in Melbourne, Australia. The AIA was interested to learn more about the authenticity and contents of the mummified bundle, while preserving the physical integrity of the object and causing as little damage as possible. The application of 3D imaging techniques was ideal to non-destructively study the object and still discover as much as possible about its contents. Using a combination of established and novel techniques: X-ray computed tomography (CT) and neutron CT provided valuable insight, both individually and collectively, revealing a partial animal skeleton, and several layers of textile and padding. Use of both techniques allowed for complementary study of bones, soft tissue, and textile components. Collaboration with a zooarchaeologist confirmed the animal remains to be a small, juvenile feline. Neutron CT, not yet routinely applied to archaeometric studies of mummified remains, provided insight into wrapping techniques used in the mummification process of votive animal offerings. In addition to these imaging studies, pigment analysis was also performed on the coloured markings on the wrappings. This was done using a scanning electron microscope (SEM) and Raman spectroscopy in order to determine their composition, and to verify their authenticity. Radiocarbon dates were acquired on samples taken from the external wrapping and the internal contents, revealing an age discrepancy between the two. This as a result is an example of recycling votive offerings, and sheds some light on the economic and religious climate in which the mummy was made and traded.

#### Speaker Sessions and Seminars / 142

### Comparison of porosity in coke like materials determined using traditional techniques and neutron tomography

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Metallurgical coke is an important raw material used in the ironmaking blast furnace as a reducing agent and structural component of the furnace burden. One of the factors effecting coke performance is porosity. Traditional methods of determining coke porosity involve metallurgical techniques that assess two dimensional cross-sections of a given coke. In this work we discuss the limitations in this approach in terms of the inter-connectivity of the porosity present in metallurgical coke and a laboratory designed coke analogue as assessed via traditional techniques and neutron tomography.

#### Speaker Sessions and Seminars / 76

### Modern Detector Concepts for Fast-Neutron Radiography

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The presented topic is part project PERTINaX (periodic testing by imaging with neutrons in addition to X-rays) which has started in November 2016. The project is funded by the German Federal Ministry of Economic Affairs and Energy (BMWi) under the funding code 1501534 and continues work

and research done in the project NISRA (neutron imaging system for radioactive waste analysis) 1. Aim of the PERTINaX project is the development of a mobile fast-neutron radiography system which can be combined with neutron activation analysis for non-destructive testing of high density and shielded components. A neutron generator from Adelphi Technology, Inc., which emits fast neutrons (2.45 MeV neutrons) with a neutron yield of  $1E9$  neutrons/sec will be used in combination with a detector system that is currently under development.

### **Detector system - scintillator materials**

The main task of PERTINaX is the development of a detector system that offers sufficient spatial resolution. Different scintillator materials in combination with Silicon Photomultipliers (SiPMs) for read out will be used.

In an environment where  $\gamma$ -radiation is present,  $\gamma$ -fogging of taken neutron radiographs is a known problem due to the fact that most scintillator materials are also sensitive to  $\gamma$ -radiation. Organic scintillators like trans-stilbene, plastic scintillators like EJ-276 or liquid scintillators, e.g. EJ-301 from Eljen Technology 2, allow pulse-shape-discrimination (PSD) which can be used to distinguish between  $\gamma$ - and neutron radiation and therefore to reduce  $\gamma$ -fogging. Stilbene-compound scintillators (investigated by Seung Kyu Lee et al. 3) or liquid scintillators filled in matrices of thin glass capillaries represent another alternatives.

### **Scintillator readout via SiPM**

Applying PSD requires detectors which can provide timing information. Furthermore the detector should have a spatial resolution in the range of  $\text{mm}^2$ . Therefore, SiPM arrays which are combining these properties can be used for the scintillator read out. Such arrays are currently used in positron emission tomographs for instance. Appropriate analogue and digital electronics for signal read out, especially for digitizing the signals and applying PSD to a large number of cells resp. pixels is under development.

### **Neutron radiography and neutron activation analysis**

Neutron radiography can be used to specify the geometry/homogeneity of specimen such as closed barrels for radioactive waste that often contain radiation shields or hydrogen containing materials. The presence of shielding components leads to large uncertainties for neutron activation analysis. These components can be identified (structural information) with neutron radiography. Their influence on parameters such as neutron- self- shielding factors or neutron flux could then be used to improve the results of the neutron activation analysis.

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2 <http://eljentechnology.com/products/liquid-scintillators/ej-301-ej-309> (Apr 2018)

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## **Speaker Sessions and Seminars / 139**

### **289 Million year old terrestrial vertebrate community revealed**

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The Dolese Brothers Limestone Quarry, near Richards Spur, Oklahoma, USA, preserves an Early Permian (298 million years old) infill in a series of Ordovician limestone and dolostone karst fissures.

Speleothems intimately associated with the site indicate that Richards Spur is a cave system, suggesting a unique preservational environment for vertebrates, one that is distinct from those of more typical Early Permian lowland deltaic/fluvial localities. The locality is unique in the preservation of exclusively terrestrial vertebrates, with the vast majority of fossil material found at this site during the last 8 decades of excavations being completely disarticulated. However, recent collecting activities have yielded articulated material, indicating that many of these recently discovered animals were likely washed in before being disarticulated or probably fell into the caves during monsoonal rains. The fossil materials are also unique preservationally because they have been impregnated with hydrocarbons derived from the underlying Woodford oils of Oklahoma. Fossilization has resulted in dark colored skeletal elements preserved in gray clays and limestones, making them easily recognizable, but the process likely occurred under conditions that facilitated the formation of abundant pyrite around and inside the bones. This unique combination makes the fossils from this vast cave system difficult to image using x-ray, but ideally suited for imaging using the quasi-parallel collimated beam of neutrons, as provided by the OPAL reactor at ANSTO. The superior image quality provided by this method has provided unprecedented access to the detailed anatomy and structure of both unprepared fossil materials, and to the internal anatomy of numerous new or little-known taxa from this locality, the richest and taxonomically most diverse assemblage of terrestrial vertebrates for the Paleozoic Era. The fossil materials examined using the DINGO facility include several small and medium sized amphibians, a stem amniote, several eureptiles and parareptiles, and a synapsid. The anatomical details of the skulls of these terrestrial vertebrates provided by neutron computed tomography have opened up new avenues for the study of the conquest of land by amniotes, the distant ancestors of living reptiles, birds and mammals, and by the amphibians that also were apparently able to compete with them for a relatively short time, 300-270 million years ago, during the Early Permian. Most significantly, the internal braincase anatomy revealed by this method is allowing us to examine in detail the evolutionary changes in the brain and some of the sense organs housed in the cranium across major transitions, from amphibians to amniotes, and through the dichotomy of amniotes into the reptilian and mammalian neural and sensory systems.

#### Speaker Sessions and Seminars / 5

### Development of Neutron Imaging Facility at Dhruva research reactor, India

Tushar Roy<sup>1</sup> ; Yogesh Kashyap<sup>1</sup> ; Shefali Shukla<sup>1</sup> ; Mayank Shukla<sup>1</sup> ; Ravi Baribadalla<sup>1</sup> ; S.C. Gadkari<sup>1</sup>

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A neutron imaging beamline has been set-up at Dhruva research reactor, India. The techniques currently implemented are Neutron Tomography, Neutron Phase Contrast Imaging and Real-Time Neutron Radiography. Combinations of sapphire and bismuth single crystals have been used as filters at the collimator input to reduce the epithermal neutron and gamma contribution respectively. The maximum beam size is restricted to ~ 120mm diameter at the sample position. A cadmium ratio of ~ 250 with L/d ratio of 160 and thermal neutron flux of  $4 \times 10^7$  n/s-cm<sup>2</sup> at the sample position has been achieved. The conventional Neutron imaging is carried out with a lens coupled CCD camera and neutron scintillator, while high resolution neutron image plates (25 $\mu$ m pixel) have been used for carrying out Phase sensitive experiments. Moreover, different scintillator and lens combinations are available to user to select large field of view with moderate resolution or high resolution with small field of view. Operation and control of sample manipulator, Detector, monitoring cameras etc can be remotely carried out from shielded experimental hutch. Different applications in the fields of reactor engineering, material science studies, archaeology, etc. shall be discussed.

We have carried out neutron tomography on Zr-2.5Nb samples containing different amount of hydrogen ingress. This test was used to validate minimum detectable limits for the same at our facility. Further studies on the diffusion of hydrogen in Zr-2.5Nb are underway. Neutron tomography studies on the metallic foam samples were carried out and its mechanical properties were simulated using volume data obtained from tomography experiments. This approach provides a powerful alternative to compare the model manufactured materials mechanical properties and for detection flaws either in the manufacturing or during different stages of its operation. In continuation with our previous work, we have set-up study lead solidification using neutron imaging technique and derived important properties in an accidental scenario.

## Speaker Sessions and Seminars / 145

### 3D Velocity Vector Measurements in a Liquid-metal by Using Image Unsharpness in Neutron Transmission Images

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To investigate liquid metal flow has critical importance in many industrial applications like metallurgy and Nuclear engineering. However, it is still difficult to measure the liquid metal flow at high temperature at present. Recently ultrasonic velocity measurement becomes one of the important measurement methods in such liquid metal flow, but its applicable temperature range is still limited to relatively low temperature level. Neutron Imaging can be applied to the velocity field measurements of liquid metal two-phase flow, which has been studied by the present author. Using only one neutron source, two-dimensional behavior of tracer particles dispersed in liquid metal flows can be visualized by traditional neutron imaging. In this study, the image unsharpness of the tracer particles was analyzed to obtain the 3-dimensional positions of the tracer particles in the liquid-metal flow. The purpose of this study is to investigate the accuracy of the 3-D velocity vector measurements in a liquid-metal single phase flow. Experiments have been performed at the Kyoto University Research Reactor by using low-melting-point Liquid-metal (Newton alloy, 97 deg. C melting point) to with AuCd3 particles, which have almost the same density as the liquid-metal.

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### Visualization and Measurement of Boiling Flow Behaviors in Parallel Mini-channel Heat Exchanger by Neutron Radiography

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Compactness and reduction in temperature difference between fluids are still strongly required for heat exchanger used in binary power cycles for waste heat utilization and refrigeration/heat pump systems. One of the way is to increase heat transfer area density. Microchannel compact heat exchanger manufactured by diffusion bonding process is developed. Since smaller diameter leads to larger pressure loss, micro channel heat exchanger has many parallel channels. Therefore, in the case that the heat exchanger is used for evaporator, refrigerant flow distribution often causes a deterioration in heat transfer performance. On the aspect of heat exchanger, the temperature of heating medium will decrease by heat exchange in an evaporator. The temperature change means the decrease in temperature difference between fluids, and lead to the change in heat flux. It is important to understand the boiling flow distribution in parallel refrigerant channel. In this study, boiling flow behaviors in a single layer microchannel heat exchanger had been visualized by neutron radiography. The heat exchanger made in stainless steel has 21 refrigerant parallel channels with semicircle cross-section, and also has 20 parallel channels for the heating medium. HFC-134a was used as the refrigerant, and a fluoro-carbon FC-3283 was used as the heating medium. The refrigerant channels are placed in crossed arrangement with the channels of the heating medium. The neutron radiography system in the research reactor of Kyoto University. Radiographs on a scintillation converter were recorded by a cooled CCD camera with the exposure time of 30 seconds. From the obtained radiographs, void fraction distributions defined as the ratio of vapor volume occupied in the vapor

channel were measured, and the effects of refrigerant mass flux, channel configuration of refrigerant, and temperature of heating medium were evaluated.

### Speaker Sessions and Seminars / 118

## Neutron imaging, a key scientific analytical tool for the Cultural Heritage project at ANSTO

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A strategic scientific research project Cultural Heritage has been initiated at the Australian Nuclear Science and Technology Organisation (ANSTO). The project aims to promote the access to the suite of nuclear methods available across the organisation, and the use of a non-invasive analytical approach in the field of cultural-heritage, archaeology, and conservation science. The latest scientific analytical tools, which are available under the operation of ANSTO, including neutron-, synchrotron- and accelerator-based techniques, have been increasingly demanded for a wide range of applications to heritage materials.

Neutron Imaging (NI), in particular, has become a valuable means for research in these fields. The fundamental properties of the neutron – no electric charge, deep penetration power into matter, and interaction with the nucleus of an atom rather than with the diffuse electron cloud – make this sub-atomic particle the ideal probe to survey the bulk of a variety of heritage materials, such as metals, pottery, paintings, etc.

In collaboration with Australian museum institutions and universities, and international experts, a series of forensic studies involving the neutron imaging beamline DINGO1 at the Australian Centre for Neutron Scattering (ACNS) will be showcased. NI was successfully used to characterise the structure, morphology and composition of cultural heritage objects without the need for sampling or invasive procedures. When integrated by complementary methods, NI data were able to shed light on the most advanced manufacturing processes developed by different cultures over time, determine the authenticity of work of art or provide information on the conservation status.

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## Archaeometric analysis of Samurai's swords

Filomena Salvemini<sup>1</sup> ; Vladimir Luzin<sup>2</sup> ; Max Avdeev<sup>3</sup> ; Anton Tremsin<sup>4</sup> ; Anna Sokolova<sup>5</sup> ; Sue Gatenby<sup>6</sup> ; Min-Jung Kim<sup>6</sup> ; Francesco Grazzi<sup>7</sup>

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In the current study, Neutron Imaging (NI) has been combined with Neutron Diffraction (ND) techniques to non-destructively characterize the laminated structure of a set of Samurai's swords, part of the East Asian Collection of the Museum of Applied Arts and Sciences (MAAS) in Sydney.

Since ancient time, distinctive carbon steels were shaped and specifically arranged for different parts of the blade to optimize their mechanical performance, forged and heat-treated accordingly to a specific school tradition which were orally transmitted and peculiarly evolved, frequently without historical records <sup>1</sup>.

NI and ND have been demonstrated to be a valuable tool to qualitatively and quantitatively characterize, from macroscopic to atomic scale, material properties of such metal artifacts in a non-destructive way <sup>2</sup>

In particular, neutron tomography, diffraction, residual stress and Bragg-edge transmission analyses were performed on samples of well-known origin, time period and authorship to create a reference database on the main lamination methods developed by Japanese swordsmiths. These benchmark data were cross-matched with results obtained from a group of mumei (no-signature) blades in the attempt to attribute their manufacturing tradition basing on qualitative and quantitative data rather than stylistic criteria.

Analysis and comparison of all collected results allows drawing conclusion about variability or similarity of the actual production techniques of the Japanese swords.

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## Speaker Sessions and Seminars / 61

### Phase Grating Moire Interferometry

Dusan Sarenac<sup>1</sup> ; Dmitry Pushin<sup>1</sup> ; David Cory<sup>1</sup> ; Michael Huber<sup>2</sup> ; Muhammad Arif<sup>2</sup> ; Benjamin Heacock<sup>3</sup> ; Daniel Hussey<sup>2</sup> ; David Jacobson<sup>2</sup> ; Jacob LaManna<sup>2</sup> ; Alex Cronin<sup>4</sup> ; Houxin Miao<sup>5</sup> ; Han Wen<sup>5</sup>

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In this talk I will present our work on developing far-field moire neutron interferometry at the National Institute of Standards and Technology's Center for Neutron Research. We have successfully built a two phase-grating moire interferometer and employed it for phase contrast imaging. This novel technique allows for broad wavelength acceptance and relaxed requirements related to fabrication and alignment, circumventing the main obstacles associated with perfect crystal neutron interferometry. In addition we provide the first demonstration that a neutron far-field interferometer can be employed to measure the microstructure of a sample. It is possible to measure the microstructure in the length scale range of 100 nm to 100 um by varying the grating spacing. Lastly, I will talk about our demonstration of a three phase-grating neutron interferometer and its promising application to accurately measure big G, the Newtonian constant of gravitation.

**Speaker Sessions and Seminars / 60****Holography with a neutron interferometer**

Dmitry Pushin<sup>1</sup> ; Dusan Sarenac<sup>1</sup> ; Michael Huber<sup>2</sup> ; Benjamin Heacock<sup>3</sup> ; Muhammad Arif<sup>2</sup> ; Charles Clark<sup>2</sup> ; Chandra Shahi<sup>4</sup> ; David Cory<sup>1</sup>

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In 1948 Dennis Gabor introduced the technique of “holography” where an image of an object is reconstructed by using a far-field electron micrograph of the object as a transmission mask for visible light. The development of coherent laser light sources in the 1960s vitalized the field to a degree that optical security holograms are now a standard feature of many paper currencies, credit cards, and identification documents. We have reported the first demonstration of holography using neutron beams and macroscopic objects. The high penetrating ability of neutrons allows our holograms to provide details about the inner structure of objects which ordinary laser light-based visual holograms cannot. Neutron holography is a new enabling tool for interferometric testing of materials, with a unique usefulness in the analysis of buried interfaces. In addition, the same experimental configuration can be used for the characterization of coherence of neutron beams.

**Speaker Sessions and Seminars / 56****Evaluation of micro-strain, dislocation density and crystallite size from broadening of multiple Bragg-edges observed by pulsed neutron transmission imaging**

Hiroataka Sato<sup>1</sup> ; Kenji Iwase<sup>2</sup> ; Takashi Kamiyama<sup>1</sup> ; Yoshiaki Kiyonagi<sup>3</sup>

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It is recognized that Bragg-edge neutron transmission method can deduce crystal structure, crystalline phase, crystallographic texture, crystallite size (from the primary extinction effect) and macro-strain in the imaging mode. In this study, further material information, micro-strain, dislocation density and crystallite size, were deduced by broadening analysis of multiple Bragg-edges.

So far, we have investigated that Bragg-edge broadening (FWHM of d-spacing distribution) is same as diffraction peak FWHM 1, and proportional to ferrite/martensite ratio and the Vickers hardness 2. However, the FWHM can be separated to the crystallite size component and the micro-strain component relating to dislocation density 3. In addition, the dislocation density is very important information for material strength characterizations. For this reason, we tried to separate these broadening components, and deduce micro-strain, crystallite size and dislocation density by using the Williamson-Hall (WH) method. The WH method needs line-broadening information of various diffraction indices.

Pulsed neutron transmission and diffraction experiment [1,4] was performed at J-PARC MLF BL19 “TAKUMI”. During a tensile test of a low-carbon ferritic steel plate, both data of transmission (by 256-pixels Li-6 glass-scintillator detector) and diffraction (by TAKUMI) were measured. As a result, Bragg-edges and diffraction peaks of various diffraction indices were obtained.

We firstly checked the classical WH (cWH) plots 3 of both transmission data and diffraction data.

This shows relation between Bragg-edge broadenings and diffraction peak broadenings for various diffraction indices. As a result, it was confirmed that Bragg-edge broadenings corresponded to diffraction peak broadenings. In addition, it was correctly observed that the cWH plots did not have linearity due to the anisotropic elasticity. Thus, Bragg-edge broadening is consistent with diffraction peak broadening for multiple diffraction indices. This means that the same data analysis procedure as the diffraction method can be applied to the Bragg-edge transmission method.

For dislocation density analysis, various high-reliability methods have been proposed in X-ray/neutron diffractometry; modified WH plot, modified Warren-Averbach method, CMWP fitting etc. For a low-carbon steel (only ferrite phase) under cold deformation like this experiment, Akama et al. found the best method; the corrected cWH plot and a dislocation density estimation method using a slope of the plot [5]. By using this method, the Bragg-edge neutron transmission imaging method can quantitatively deduce the dislocation density. As a result, it was found that the dislocation density after the tensile test was about  $2\sim 3 \times 10^{14} \text{ m}^{-2}$ , and this value was consistent with a similar X-ray diffraction study [5]. Since the corrected cWH model is usable, we are now developing a new fitting program for Bragg-edge neutron transmission spectra by using this model. Owing to this, it is expected that reduction of the analytical error is achieved in the imaging mode.

The authors are thankful to Dr. S. Harjo, Dr. S. Takata, Dr. T. Ito and Dr. K. Aizawa for experimental assistances at TAKUMI.

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## Crystallographic structure study of a Japanese sword Noritsuna in Muromachi period using pulsed neutron imaging

Hiroataka Sato<sup>1</sup>; Yoshiaki Kiyanagi<sup>2</sup>; Kenichi Oikawa<sup>3</sup>; Kazuma Ohmae<sup>2</sup>; Anh Hoang Pham<sup>4</sup>; Kenichi Watanabe<sup>2</sup>; Yoshihiro Matsumoto<sup>5</sup>; Takenao Shinohara<sup>3</sup>; Tetsuya Kai<sup>3</sup>; Stefanus Harjo<sup>3</sup>; Masato Ohnuma<sup>1</sup>; Shigekazu Morito<sup>4</sup>; Takuya Ohba<sup>4</sup>; Akira Uritani<sup>2</sup>; Masakazu Ito<sup>6</sup>

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Japanese swords are very attractive not only as a work of art but also as a metallurgical point of view. There were five traditional styles (Gokaden) of Japanese sword making in the Koto (old sword) age; A.D. 987-1596. Since Japanese vintage swords became valuable, it is indispensable to establish non-destructive analysis method to identify some peculiar characteristics, related to the sword making processes. Neutron experiments are powerful tools to study metallic cultural heritages due to their high penetrating power and capability to give crystallographic information 1. Bragg-edge neutron transmission imaging gives real-space distributions of bulk information in a crystalline material. In addition, by analyzing position-dependent Bragg-edge transmission spectra measured at a pulsed neutron source, quantitative visualization of the crystallographic information of a sword can be achieved 2.

In this work, we investigated crystallographic information of a Japanese sword made by Noritsuna in Bizen (one of Gokaden) in the first quarter of the 15th century (1405). The experiments have been performed at the Energy-Resolved Neutron Imaging System, RADEN at J-PARC MLF BL22 3.

The Noritsuna sword was measured at three regions with a counting-type 2D detector, nGEM. The measured position-dependent Bragg-edge transmission spectra were analyzed by the RITS code [4] to obtain spatial distributions of the crystallite size, the texture variation, the d-spacing shift and its broadening. As a result, it was found that martensite phase exists near the cutting edge (Ha), small crystallites were distributed around the point of blade (Kissaki) region, and large crystallites were distributed at the tang (Nakago) region. The crystallographic characteristics of Noritsuna will be presented in detail.

This work partially includes the results of “Collaborative Important Researches” organized by JAEA, QST and The University of Tokyo.

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### Speaker Sessions and Seminars / 31

## A quadruple multi-camera neutron computed tomography system at MLZ

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Most neutron imaging systems can accommodate large samples of 15 -30 cm size, but recent interest is more focused on small cm-sized samples. With a small field of view for a camera-based detection system, the neutron flux per pixel decreases, and measurement time increases.

There were approaches to split a large field of view into smaller fields for individual CT measurements using a cogwheel-based adapter for the rotation stage [PSI] or using individual micro rotation stages [MLZ], but this leaves a smaller amount of camera pixels per tomography field, while many applications require the highest possible resolution even or especially for very small samples.

An alternative approach is followed at MLZ, using a multiple camera system with multiple rotation stages to make better use of the full size of the original neutron beam. With four cameras, only two rotation stages are required where samples are stacked in an aluminum tube with cutouts above each other. Cameras are stacked with two on top of each other, and two stacks beside each other.

A small, but high quality cooled CMOS camera is employed, each camera box contains lead shielding only in front and behind the camera for easy stacking, and sideways, joint shielding is built up with lead bricks and PE plates for the whole setup of four camera boxes.

The camera box and the mirror and scintillation screen holder are designed as separate parts so scintillation screen holders for variable size can be adapted.

The first prototype is already working, four more camera boxes are currently in production and will be completed by the time of the conference.

The talk will describe the system in detail.

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## Speaker Sessions and Seminars / 32

**Epithermal neutron radiography and tomography on large and strongly scattering samples**Burkhard Schillinger<sup>1</sup> ; Aaron Craft<sup>2</sup><sup>1</sup> *Heinz Maier-Leibniz-Institut (FRM II), TU München*<sup>2</sup> *Idaho National Laboratory***Corresponding Author(s):** burkhard.schillinger@frm2.tum.de, aaron.craft@inl.gov

While neutron imaging with thermal and cold neutrons has become a standard method at many neutron facilities world-wide, little research has been done on epithermal neutron imaging with electronic detectors. Indirect methods with dysprosium foils and film or imaging plates have been used for the examination of nuclear fuel at Idaho National Laboratory (INL) and other places, but a fully digital imaging system has rarely been employed beyond simple cadmium-filtered radiography. In a collaboration between INL in the USA and Heinz Maier-Leibniz Zentrum (MLZ) of Technische Universität München in Germany, several tests were conducted with a cadmium-filtered beam. At INL, the Neutron Radiography Reactor (NRAD) is optimized for high epithermal neutron output with a beam tube source position in close contact to the reactor core. At MLZ, the primarily cold and thermal energy spectrum of the ANTARES neutron imaging facility still contains sufficient epithermal neutrons that penetrate the undermoderated cold source to allow for reasonable measuring times with a cadmium-filtered beam.

Measurements include the effects of thermalizing epithermal neutrons in a heavily scattering sample, which can be removed by a second cadmium filter on the detector, and the first full epithermal neutron computed tomography on large technical samples in direct comparison to cold neutron tomography with the same setup without filters. Several examples of epithermal neutron imaging are included.

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**The ANTARES instrument control system for neutron imaging with NICOS/Taco at MLZ converted to a mobile system used at Idaho National Laboratory****Author(s):** Burkhard Schillinger<sup>1</sup>**Co-author(s):** Aaron Craft<sup>2</sup> ; Jens Krüger<sup>3</sup><sup>1</sup> *Heinz Maier-Leibniz-Institut (FRM II), TU München*<sup>2</sup> *Idaho National Laboratory*<sup>3</sup> *Heinz Maier-Leibniz Zentrum (MLZ), TU München***Corresponding Author(s):** burkhard.schillinger@frm2.tum.de

The Neutron Radiography Reactor (NRAD) at Idaho National Laboratory (INL) was designed for epithermal neutron radiography for examination of highly-radioactive irradiated nuclear fuel elements. Samples are lowered into the North Radiography Station from shielding flasks above, and a rail transfer system remotely positions radiography cassettes into the detector position for indirect radiography. Opening NRAD's two radiography stations on a regular basis for user operations was not foreseen when the facility was designed, but recent collaborative efforts seeking to introduce digital neutron imaging systems have demonstrated the need for simplified access to the radiography stations, which is an extraordinary challenge due to the high radiation environment and associated shielding requirements, and remote control of various imaging hardware inside the radiography stations. Two initial camera detector systems were built using a low-price, but high-quality scientific CMOS camera with massive shielding. For the first working tomography setup, it was a challenge to build a local standalone instrument control system without any existing electronic infrastructure.

In first tests in 2017, a Raspberry Pi computer with a stepper motor controller inside the East Bay of NRAD was interfaced with the standard windows camera software on a laptop outside, simulating mouse clicks to trigger image recording after each rotation step. In the high radiation field of the East Bay, the Raspberry Pi crashed after a few dozen of images even behind 5 cm of lead shielding. For a more sophisticated experiment in the North Bay in 2018, the instrument control system of the ANTARES imaging facility at Heinz Maier-Leibnitz Institut of Technische Universität München was scaled down to one laptop and three Raspberry Pi computers mounted outside the radiography bay, which was sufficient to control rotation and translation stages and to control and record tomography camera measurements.

The system is based on NICOS<sup>1</sup>, TANGO<sup>2</sup>, and LiMA<sup>3</sup>, all three free toolkits for building distributed control systems. TANGO and LiMA were originally developed and used at the European Synchrotron Radiation Facility (ESRF). TANGO uses little instrument servers for individual devices which can be controlled over the network, in this case by NICOS. NICOS, developed at MLZ, has a command/script executing daemon, controlled by a graphical user interface. The user may control devices by widgets, command line, or python scripts.

The INL NRAD tomography control system was the first standalone minimalist system CT implemented by MLZ, which can be extended in the future to control a multitude of devices.

1 <http://www.nicos-controls.org>

2 <http://www.tango-controls.org>

3 <http://lima.blissgarden.org/>

## Speaker Sessions and Seminars / 97

### Recent achievements and activities in neutron imaging at FRM II

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At the FRM II reactor in Garching, Germany the Heinz Maier-Leibnitz Zentrum operates the two neutron imaging facilities ANTARES and NECTAR. ANTARES provides a cold neutron spectrum which gives high sensitivity for even small changes of composition in a sample. Consequently ANTARES is used for neutron imaging with high spatial resolution as well as novel techniques such as imaging with polarized neutrons or neutron grating interferometry (nGI). The instrument NECTAR, in contrast, is a unique facility which provides a fast fission neutron spectrum which allows to investigate even very bulky samples and shows contrast complementary to X-rays or gammas.

In our contribution we will give an overview of recent achievements and activities of the MUnich Neutron Imaging Group (MUNIG). We have made several instrumental upgrades at both facilities. NECTAR is currently undergoing a complete redesign and an upgrade to additionally provide a thermal neutron spectrum which can be used when higher penetration than with cold neutrons is required in combination with high spatial resolution. Furthermore, at ANTARES we have designed and installed a dedicated <sup>3</sup>He cryostat for neutron imaging which allows to routinely reach temperatures as low as 500mK for imaging with polarized neutrons and nGI while allowing to keep the sample to detector position as short as 50mm. Additionally the nGI setup at ANTARES has undergone a major upgrade of the geometry and the employed gratings which allows us to achieve a visibility of 75% over the entire field-of-view. We will additionally show results of recently performed experiments at both beam lines which are of interest for the community.

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## High-resolution neutron depolarization microscopy of the ferromagnetic transitions in Ni<sub>3</sub>Al and HgCr<sub>2</sub>Se<sub>4</sub> by using Wolter mirrors

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Imaging with polarized neutrons has in recent years increasingly gathered interest due to its ability to visualize bulk magnetic properties and magnetic fields in 2D and 3D. Currently the spatial resolution of typical setups is limited to ~500µm by the space consumed by the polarization analyzer which needs to be placed between sample and detector. This increases the minimum sample to detector distance which is achievable and results in such mediocre spatial resolution.

To obtain higher spatial resolution, we employed a novel neutron microscope equipped with Wolter mirrors as a neutron image-forming lens and a focusing neutron guide as a neutron condenser lens at the instrument ANTARES at FRM II. The Wolter optic creates a magnified image of the sample at the detector position while at the same time removing the general requirement in neutron imaging to place the sample as close as possible to the detector. With the current prototype Wolter mirrors we could achieve a magnification factor of four and a spatial resolution of ~100µm was reached. The spatial resolution was in our case mainly limited by the surface quality of the employed neutron optical mirrors in the prototype optic and we see potential for the improvement by another order magnitude.

To demonstrate the potential of the technique we performed spatially resolved bulk imaging of ferromagnetic transitions in Ni<sub>3</sub>Al and HgCr<sub>2</sub>Se<sub>4</sub> crystals. These neutron depolarization measurements discovered magnetic inhomogeneities in the ferromagnetic transition temperature with spatial resolution of about 100 µm.

The images of Ni<sub>3</sub>Al show that the sample does not homogeneously go through the ferromagnetic transition. The improved resolution allowed us to identify a distribution of small grains with slightly off-stoichiometric composition. Additionally, neutron depolarization imaging experiments on the chrome spinel, HgCr<sub>2</sub>Se<sub>4</sub>, under high pressures up to 15 kbar highlight the advantages of the new technique especially for small samples or sample environments with restricted sample space. The improved spatial resolution enables to observe domain formation in the sample while decreasing the acquisition time despite having a bulky pressure cell in the beam.

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## Energy-selective Neutron Imaging of Radioactive Samples used for Neutron Capture Reaction Measurements

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Prompt Gamma-ray Analysis (PGA) and Neutron Resonance Capture Analysis (NRCA) are rapid and non-destructive method to analyze samples qualitatively and quantitatively. These promising tools

are used for the broad science field such as nuclear engineering, nuclear safeguards and materials science. To perform accurate analysis in their measurements, corrections of neutron self-absorption and multiple scattering effects in samples and of shielding effects for gamma-rays emitting from samples are important. In most cases, the correction factors are estimated assuming a uniform distribution of sample material. It is, however, not easy to confirm the assumption since most of Long lived fission products (LLFPs) samples are sealed to prevent the release of radioactive materials.

In this study, uniformity of sealed LLFPs samples of Pd-107, Tc-99, and I-129, being used for not only the developments of their analysis method but also neutron capture cross-section measurements, were investigated. Spatial distributions of gamma- and/or X-ray emitted from the Tc-99 and I-129 samples were visualized by putting them on an imaging plate which enables us to observe a crack and some non-uniformity in the I-129 sample. Neutron transmission images of those three samples were also obtained by a neutron imaging plate at the beam line No.4, "Accurate Neutron-Nucleus Reaction measurement Instrument" (ANNRI), installed at the J-PARC Materials and Life Science Experimental Facility (MLF). The similar non-uniformity was also recognized in the neutron transmission image of the I-129 sample while no significant non-uniformity was recognized in the Pd-107 and Tc-99 samples. The results of image plates brought about important information regarding the uniformity of the samples, though it was difficult to identify the isotopes of the sample.

In order to identify the isotopes which produced the non-uniformity of the sample, we utilized the time-of-flight (TOF) technique to perform energy-resolved two-dimensional neutron imaging measurements. The experiments were performed using a gas-electron multiplier (GEM) neutron detector (128 x 128 pixels) and the sealed radioactive isotopes (Pd-107, Tc-99, and I-129) at ANNRI. Thus, the non-uniform neutron transmission image of the I-129 sample was obtained in the corresponding resonance energy region of Na which was contained in a compound of "NaI" with I-129. The result was consistent with those obtained by the imaging plates mentioned above.

In this presentation, we verify the inside of the samples from the obtained images and discuss the effects of sample uniformity on the correction factors.

#### Speaker Sessions and Seminars / 89

### Current Status of The Energy-Resolved Neutron Imaging System, RADEN, at J-PARC MLF

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The world's first pulsed neutron imaging instrument dedicated to energy-resolved neutron imaging experiments, named RADEN, was constructed at beam port 22 in the Materials and Life Science Experimental Facility (MLF) of J-PARC 1. This instrument is designed to conduct state-of-the-art energy-resolved neutron imaging, such as Bragg-edge imaging, resonance absorption imaging, and pulsed polarized neutron imaging, together with conventional/energy-selective neutron radiography and tomography by fully utilizing the high intensity, short-pulsed neutron beam. The construction of RADEN was completed in 2014, and user operation was started from April 2015. By the end of April 2017, the number of conducted proposals reached 72 and about half of the total beam time was utilized by general users.

Besides the user program, the RADEN instrument group is continuing the technical development and improvement of the instrument so as to conduct more advanced energy-resolved and conventional neutron imaging experiments. To improve the detector performance, we exchanged the optical system of our camera-type detector for increased brightness to achieve fine spatial resolution, and upgraded an event-type detector, the  $\mu$ NID 2, for improved count rate, neutron detection efficiency, and spatial resolution. Also, the collimation system was upgraded and additional slits were introduced into the instrument for better beam shaping and efficient background reduction. The device



controlling software has been replaced with a newer version in order to make the interface more user friendly and to provide flexibility to easily include additional equipment under the control. Regarding the development of new imaging techniques, we have constructed a Talbot-Lau interferometer at the pulsed neutron source for the first time and applied the wavelength-dependent analysis to phase imaging [3]. Moreover, to analyze the spatial distribution of nano-scale structural information, a technique to extract the small-angle scattering contribution in the neutron transmission spectrum using orthogonally-arranged neutron Soller collimators has been developed [4].

In this presentation, we will report the current status of RADEN along with recent results of the technical development and application studies regarding energy-resolved neutron imaging techniques conducted at RADEN.

This work was partially supported by the Photon and Quantum Basic Research Coordinated Development Program from MEXT, Japan and the Momose Quantum Beam Phase Imaging Project, ERATO, JST (Grant No. JPMJER1403).

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## Observation of the magnetic field distribution leaked from an electric transformer using pulsed polarized neutron imaging

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Observation of magnetic fields emanating from electromagnetic devices gives important information on not only their structural and functional soundness but also on energy loss under operating conditions. A magnetic imaging technique using pulsed polarized neutrons would be suitable for direct observation of such magnetic fields because it enables us to visualize the field distributions in both massive objects and free space with a large field of view up to several square centimeters. We have developed such a pulsed polarized neutron imaging technique at BL22 RADEN at the Materials and Life Science Experimental Facility (MLF) of J-PARC and performed demonstration studies visualizing magnetic fields of a small electric motor and an electric transformer [1-2].

For the present study, the neutron imaging experiment was performed using the polarization analysis apparatus of BL22 RADEN 3. The object of study was a small electric transformer consisting of a step-lap rectangular core and 2 coils wound at both sides of the core (i.e., the primary and the secondary windings). A 50.25-Hz AC voltage was applied to the primary windings during the imaging experiment. The temporal change of the wavelength-resolved polarization distribution image was obtained by means of an AC field imaging technique developed in our previous study [4]. Leaked field from the core and the primary windings was observed as a deterioration of the polarization degree around the transformer in the polarization distribution image. The phase at maximum leaked field intensity was about  $0.7\pi$  radians, which was slightly delayed from the peak of the applied AC voltage at  $0.5\pi$  radians. This delay is thought to originate from the eddy current loss in the step-lap core. In order to investigate the leaked field distribution in more detail, we visualized the distribution of the field strength integrated along the neutron flight path by analyzing the wavelength dependence of the polarization degree point by point. As a result, the leaked field from the gap of the step-lap could be clearly observed. Moreover, the strength of the leaked field with an AC voltage applied to the primary windings was found to decrease compared with that observed when applying a DC voltage. The simulation study of the leaked field from the step-lap core clarified that the eddy current in the steel core acted to reduce the leaked field from the gap of the step-lap. Therefore, it was concluded that the observed reduction of the leaked field came mainly from the eddy current in

the core produced by the applied AC voltage.

In this presentation, we report detailed results of a leaked field study from the operating electric transformer and discuss the relationship between the eddy current loss in the step-lap core and the leaked field distribution.

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#### Speaker Sessions and Seminars / 25

## Flow visualization of heavy oil in packed bed reactor by neutron radiography

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The demand for petrochemical feedstock and middle distillate is increasing. Although utilization of heavy oils such as atmospheric or vacuum residue is also necessary, the heavy oils have not been used due to the high viscosity and low quality. Thus, desulfurization and upgrading processes are required to use the heavy oils effectively. A trickle bed reactor, in which a heavy oil and a gas are flowed concurrently through a packed bed of catalytic particles, is generally used as the upgrading process. In the reactor, channeling and consequent hot spots decrease the performance. Hence, the understanding of flow behavior in the reactor is significant.

Recently, the development of CFD simulator of hydrodynamics and reactions in the reactor has been advanced to clarify the flow behavior. On the other hand, the experimental works on flow visualization of the heavy oils have not been conducted. This is because the reactor was made of metal for operation at high pressure and high temperature, and consequently the visualization using visible light was not available. Therefore, the objective of this work is flow visualization of heavy oil in the packed bed reactor by neutron radiography.

In the experiment, the Kyoto university research reactor (KUR) was utilized as neutron source. KUR was operated at either 1 or 5 MW with a neutron flux of 1 or  $5 \times 10^7$  n/cm<sup>2</sup>·s, respectively. The heavy oil and N<sub>2</sub> gas were supplied concurrently to a packed bed reactor, i.e., a 1/2-inch stainless steel tube filled with Al<sub>2</sub>O<sub>3</sub> particles having the diameter of 1 or 3 mm. Atmospheric residue (AR) was used as the heavy oil sample. The reactor was heated to temperatures of 100°C and 250°C to change the viscosity of heavy oil. The flow rate of heavy oil was 2.5 mL/min and that of N<sub>2</sub> gas was set at 1 L/min at 25°C. Hence, the flow rates of N<sub>2</sub> gas in the reactor changed depending on the reactor temperatures. An image intensifier and a CCD camera at the framerate of 30 fps were used to obtain visualization images of the unsteady flow behavior. An image processing to reduce noises was performed for the obtained images.

The flow behavior of heavy oil in the reactor varied depending on the experimental conditions. Since the viscosity of heavy oil markedly varies with temperature, that is, the viscosity of heavy oil at 100°C is 10 times larger than that at 250°C, the head velocity of heavy oil flowing down at 100°C became approximately half that at 250°C for the particle diameter of 1 mm. In addition, the heavy oil at 100°C spread radially to the wall of the tube, whereas the heavy oil at 250°C did not spread. In the case of 3 mm particle diameter, the heavy oil did not spread at both 100°C and 250°C compared with the case of 1 mm particles, and the flow channeling occurred in the packed bed.

## Speaker Sessions and Seminars / 30

## Review and Prospect of hydraulic behavior research of rhizosphere, xylem and leaves using neutron imaging

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Neutron radiography which images interactions within the nucleus of atoms, rather than between electrons like X-ray (1), can identify the strongly interacting hydrogen in water molecules, and can be used to determine hydraulic behavior in soils and plants. The first of a series of neutron imaging (NI) is able to determine the water content and morphology of roots planted in pots embedded in the field. The results of a series of neutron imaging were used to diagnose root diseases in situ (2). Therefore, neutron imaging is the most appropriate method for studying the epidemiology of root-rot and rust because it can detect significant accumulations of inorganic elements of iron, aluminum, silicon, and magnesium ions and water of root in the soil, all of which interact with the fungi, mycorrhiza, and yeast inocula in the rhizosphere(3). The levels of water, phenolics, and inorganic elements in the roots are all indicators of root health.

The uptake of water and inorganic elements by roots is a crucial process for plant health. Dielectric cell pressure probes, magnetic resonance, and heat tracing can be used to map the fluid dynamics in the xylem sap and phloem, but they are destructive methods. By contrast, neutron dynamic imaging produces a 3D picture (4) of hydraulic movement in the vessels and sieve tubes, depending on solution ion concentration, pH, root pressure, osmotic pressure, capillarity, and nonpolar solvents during active metabolism and photosynthesis. Hydraulic movement from the root epidermis to the endodermis, apoplast, symplast, and transmembrane regions can be analyzed in vivo (5). Neutron imaging with the contrast agent, D<sub>2</sub>O, can be used to visualize in situ photomorphogenesis in the plant roots based on the sensitivity to different light wavelengths. These phenomena are largely uncharacterized at present. The application of neutron imaging shows us great promise for addressing many of the challenging questions related to plant hydraulics in the rhizosphere. In this paper, the related research will be reviewed and be looked in to the future of neutron imaging tools for an expanding agriculture and food field.

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## Speaker Sessions and Seminars / 16

## Development of energy-selective and element-sensitive imaging using a compact D-D fast neutron generator

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This work is focused on the development of energy-selective techniques using a compact Deuterium-Deuterium (D-D) fast neutron generator. This was done in the context of a custom D-D generator located at the Paul Scherrer Institute which was specifically developed to have a small emitting spot size for transmission imaging purposes <sup>1</sup>. The basis of this study lies in the physics of the D-D fusion reaction: the neutrons produced are quasi-monoenergetic with an energy dependent on emission angle from roughly 2.2 to 2.8 MeV, based on the acceleration voltage limitation of the device. Samples can therefore be imaged at different emission angles corresponding to different neutron energies. Since neutron cross-sections have energy dependence unique to each element (unlike X-rays), this combination of information from different angles can be used in principle to distinguish one element or chemical from another. The inverse can also be performed; instead of determining the content of an unknown sample, measurements of a known and uniform sample can be used to produce cross-section data.

The first steps of this investigation included a feasibility study of these techniques. Detailed angle-dependent source emission spectra models were created according to different target composition assumptions. These models were used to estimate attenuation vs. angle for several samples of known composition and thickness which have particularly prominent cross-section structures in the energy range of interest (e.g. alumina). Over the full range of emission angles, plastic scintillators were used to measure count rates with sample present, without sample, and with a shadow cone, in order to determine the sample attenuation. This was done with a custom, automated mechanical apparatus around the source. Scatter correction was also implemented based on detailed Monte Carlo simulations of the source and room geometry. The experimental attenuation data were compared with simulations and found to be in good agreement, demonstrating the fundamental feasibility of the approach.

Ongoing work aims to expand these measurements to include a range of materials which are of interest to industrial or homeland security applications. Furthermore, the next step of performing full tomographic reconstruction at multiple angles is being explored, both with simulations and measurements. The aim is to find the practical capabilities and limitations of determining the presence of materials of interest in samples of unknown composition. The latest results and progress towards this goal will be presented and discussed.

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## Speaker Sessions and Seminars / 41

### Investigation of ancient copper-alloy and ferrous artefacts from South-eastern Arabia

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Metal artefacts excavated from archaeological sites are often heterogeneous not only in their stylistic features but also in structure and composition. This is ultimately related to a variety of manufacturing processes developed within different socio-technological contexts. The current paper demonstrates how heterogeneous structures and underlying manufacturing techniques can be successfully

detected in ancient copper-alloy arrowheads from the Middle-Late Bronze Age site of Sharm in the United Arab Emirates. A non-invasive approach based on the combination of neutron tomography (NT), neutron diffraction stress analysis (NDS) and particle-induced X-ray emission analyses (PIXE) was exploited. Results suggest that the artefacts were made by casting an alloy of copper containing impurities of nickel and arsenic, and then subsequently subjected to different types of forging and heat treatment. The manufacturing process promoted specific types of elemental segregation and subsequent selective oxidation of the metal objects.

This paper also presents the results of NT applied to the investigation of totally corroded ancient ferrous artefacts from the early Iron Age site of Saruq al-Hadid, Dubai. Despite the severe state of degradation of the objects, NT allowed the detection of various features in the artefacts, including: 1) surface irregularities from plastic deformation by hammers and some other tools; 2) different corrosion products, and their specific distribution patterns, some of which can be associated with secondary recycling activities performed upon the objects; 3) various structural inhomogeneities such as mineralized pierced holes, incised patterns and ex-welding lines. Among the listed inhomogeneities, the ex-welding lines represent the major interest during NT investigation of corroded ferrous artefacts. These structural features can be found in almost every artefact, since corrosion preferentially evolves along these lines, and can be conveniently used for the comparison of different ferrous artefacts and their manufacturing techniques. The complementary invasive investigation of ferrous artefacts via analyses of remnant carburized areas using traditional optical microscopy techniques and analyses of slag inclusions by scanning electron microscopy coupled with energy dispersive spectroscopy (SEM-EDS) allowed a developed understanding of the socio-technological factors underlying the use of the identified iron welding techniques. These results provide a broader insight into the technologies and knowledge of the Iron Age societies of the Ancient Near East.

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## Imaging with $6\text{LiInSe}_2$ : Implications for Fast and Thermal Neutron Imaging

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In recent years,  $6\text{LiInSe}_2$  (LISE) was developed as both semiconductor and scintillator neutron detection material. In the present paper, LISe is evaluated as a neutron imaging media as a scintillator and semiconductor. An array of lithium indium diselenide (LISE) scintillators varying in thickness and surface roughness, were tested using both reflective and anti-reflective mounting to an aluminum window under thermal neutron irradiation. The spatial resolution of each LISe scintillator was calculated using the knife-edge test and a modulation transfer function analysis. It was found that the anti-reflective backing yielded a significantly higher spatial resolution over the reflective backing, and in most cases far exceeded the spatial resolution of a 50  $\mu\text{m}$ -thick  $\text{ZnS}(\text{Cu}):6\text{LiF}$  scintillation screen. Further, an apparent relationship between the observed increase in spatial resolution when using an anti-reflective backing and the quotient of the surface roughness to the thickness of the LISe scintillator was observed. Finally, the LISe scintillator array was used in neutron computational tomography to investigate the features of halyomorpha halys. Similar thermal neutron experiments with a  $9\times 7\times 0.5\text{ mm}^3$   $6\text{LiInSe}_2$  crystal coupled to a  $256\times 256$  channel Timepix ASIC with 55  $\mu\text{m}$  pixel pitch were performed. The imaging system performed a series of experiments resulting in a  $<200\text{ }\mu\text{m}$  resolution limit with the Paul Scherrer Institute (PSI) Siemens star mask and a feature resolution of 34  $\mu\text{m}$  with a knife-edge test. Furthermore, the system was able to resolve the University of Tennessee logo inscribed into a 3D printed 1  $\text{cm}^3$  plastic block. This technology marks the application of high resolution neutron imaging using a direct readout semiconductor. Finally, the semiconductor imager was exposed to a monoenergetic beam of 9 MeV neutrons from the  $d(d,n)$  reaction at Edwards Accelerator Laboratory. Using a 15 cm thick copper bar to create a knife edge across the sensor, a spatial resolution of 1.55 mm was determined from the 10-90% rise of the line spread function.

## Speaker Sessions and Seminars / 112

**Neutron Radiology Terminology in Imaging**Markus Strobl<sup>1</sup> ; Eberhard Lehmann<sup>2</sup> ; Ulf Garbe<sup>3</sup> ; Frikkie De Beer<sup>4</sup> ; Daniel S. Hussey<sup>5</sup> ; Thomas Bücherl<sup>6</sup><sup>1</sup> *Laboratory Neutron Scattering and Imaging, Paul Scherrer Institut*<sup>2</sup> *Paul Scherrer Institut*<sup>3</sup> *ANSTO*<sup>4</sup> *Necsa*<sup>5</sup> *National Institute of Standards and Technology*<sup>6</sup> *Technische Universität München, Radiochemie München***Corresponding Author(s):** markus.strobl@psi.ch

Neutron Imaging used to be a non-destructive testing (NDT) tool for decades and as such was listed with a few techniques in the ASTM standards. However, for the current state-of-the art in neutron imaging such NDT terminology, which mostly still refers to film detection, appears outdated in light of the digital imaging at advanced, large-scale neutron source facilities. We note that these digital imaging applications are the dominate activities of ISNR board members and comprise most of the WCNR presentations for more than a decade. Additionally, the rapidly increasing number of techniques and methods qualifying neutron imaging today combined with the outdated terminology have led to increasing confusion and lack of coherence in the use and creation of terms, which is required to clearly characterize measurement methods, developments, and applications.

Therefore, the board of the ISNR, at the last WCNR in 2014 in Grindelwald, Switzerland, established a working group to study issues of terminology. While corresponding higher order terms and definitions have been discussed and presented at the ITMNR in Beijing in 2016, an attempt for an extensive catalogue and system for a terminology in neutron imaging to be issued by the ISNR shall be presented and opened for discussion.

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Fig. 1 top: Context of Terminology for ISNR with respect to higher-level general umbrella terms like Radiology, Imaging, Radiography etc.; bottom: use of the term imaging over the last 210 years

## Speaker Sessions and Seminars / 47

**Conceptual design of a thermal neutron imaging facility at the Jordan Research and Training Reactor (JRTR) optimized by Monte Carlo neutron ray-tracing simulations**Mahmoud Suaifan<sup>1</sup> ; Jongyul Kim<sup>2</sup> ; Saed Almomani<sup>1</sup> ; Jongmin Lee<sup>2</sup> ; Baek-Seok Seong<sup>2</sup><sup>1</sup> *University of Science and Technology (UST), 217 Gajeong-ro, Yuseong-gu, Daejeon 34113, Republic of Korea, 2Korea Atomic Energy Research Institute (KAERI), 989-111 Daedeok-daero, Yuseong-gu, Daejeon 34057, Republic of Korea, 3Jordan Atomic Energy Commission (JAEC), JRTR Commission, 70 Shafa-badran, Amman 11934, Jordan*<sup>2</sup> *2Korea Atomic Energy Research Institute (KAERI), 989-111 Daedeok-daero, Yuseong-gu, Daejeon 34057, Republic of Korea***Corresponding Author(s):** mahma7@gmail.com**Abstract**

Recently, the Jordan Research and Training Reactor (JRTR) has officially got its operating license. The JRTR, 5 MWt, upgradable to 10 MWt, and neutron fluxes of orders of  $10^{14}$  n/cm<sup>2</sup>.sec, has started its activities to provide multi-purpose services according to the potential utilization plans. This paper discusses one of the most important and primary instruments in regards to the utilization

of the nuclear research reactors, and spallation sources as well, that is a thermal neutron imaging facility (NIF) to be installed at the sufficiently wide experimental hall of the JRTR site and be opened for local and international users of both sectors academia and industry. This paper focuses on the detailed works of the designing, optimizing, and verification stages of the conceptual design of the JRTR-NIF applying Monte Carlo simulations using McStas neutron ray-tracing packages. Initial simulation results show that the JRTR-NIF can provide competing flux values ranging between the orders of  $10^6 \sim 10^7$  n/cm<sup>2</sup>.sec at various sample positions, coupled with various L/D collimation selected ratios ranging between 80 ~ 1200, as well as good beam sizes, “effective” beam sizes up to 20 cm in diameter, with good resolutions compared to other pioneer facilities worldwide in order to cover a wide range of advanced applications required by various types of users.

**Keywords**

JRTR, Jordan, Neutron imaging, Neutron radiography, Neutron beam instrumentation, nuclear research reactor utilization, and Monte Carlo simulations.

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**Speaker Sessions and Seminars / 28****Recent Advances in Neutron Imaging using a High-Flux Accelerator-Based Neutron Generator**

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Neutron imaging systems have been designed and constructed by Phoenix LLC to investigate low density material attributes of composites and other materials where other Non-destructive evaluation, (NDE), methods do not suffice. The first-generation electronic neutron generator was commissioned in 2013 at a United States Army research facility to inspect munitions and other critical defense and aerospace components. A second-generation neutron imaging system has undergone extensive testing at the Phoenix laboratory with an increased total neutron output from an upgraded gaseous deuterium target of  $3 \times 10^{11}$  deuterium-deuterium (DD) neutrons/second. This system generates a higher neutron flux at the imaging plane, approximately  $1 \times 10^4$  n/cm<sup>2</sup>-sec, which reduces interrogation time, while maintaining high contrast and low geometric unsharpness. A further optimized system is currently under construction and promises yet even higher neutron output with increased image quality regarding signal to noise, contrast, and resolution. This system is expected to be installed at a production plant in 2018 and will be the first of its kind installed and used in a commercial setting. Phoenix’s technology offers high throughput and image quality for neutron radiographs, like images currently acquired at nuclear reactors, but with greater accessibility, an eased regulatory environment, at a much-reduced cost, and without great environmental or biological hazards. As neutron radiography becomes more accessible due to the increased neutron yield of accelerator-based systems, a wider range of inspection techniques will be possible including digital radiography and computed tomography, with shorter image acquisition times. A description of the Phoenix neutron generator and imaging system, including the beamline, target, moderator and collimator, various detector platforms, and post processing techniques including neutron interaction localization and computed tomography, are demonstrated in this presentation. Neutron radiographs captured with a Phoenix neutron radiography system will be presented for both analog and digital based formats.

Keywords:

Neutron, radiography, munitions, artillery, aerospace, imaging, direct radiography, computed radiography, computed tomography

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### Speaker Sessions and Seminars / 105

## Recent developments from NeXT-Grenoble, the Neutron and X-ray Tomograph in Grenoble

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NeXT-Grenoble is the Neutron and X-ray Tomograph born in 2016 from the joint effort of Universit  Grenoble Alpes (UGA) and the Institut Laue-Langevin (ILL), and takes advantage of its world-leading cold neutron flux. Specifically, the flux peaks at  $3 \times 10^8 n \text{ cm}^{-2} \text{ s}^{-1}$  for an L/D of 333 with an average wavelength above 3  .

The instrument relies on a suite of detectors ranging from fields of view above 170x[mm]x170[mm] to true resolutions below 10  m. They are constituted by a range of scintillators ranging from 200  m  $ZnS/6LiF$  : to 2.5 m  $^{157}Gd_2O_2S : Tb$  and a set of high aperture lenses.

Thanks to the uniquely powerful flux, the instrument can perform high speed tomographies (below 10 seconds) at large fields of view as well as acquire high resolution (below 10  m) tomographies in times comparable to those of microfocus x-ray setups.

A key feature of the instrument is the possibility to perform *simultaneous* x-ray and neutron tomography, in order to take advantage of the high complementarity of the attenuation coefficients of these two techniques.

The registration of the two volumes is made possible by recent mathematical developments which also provides phase identification, with much more ease than with either image individually.

A major upgrade of the instrument is foreseen in the forthcoming two years within the "Endurance 2" upgrade scheme of ILL to further improve its performances as well as to add further options (e.g. monochromation, polarised neutrons, grating interferometry).

This instrument is open for proposals through its dedicated website (<https://next-grenoble.fr/>).

This instrument is conceived with a wide range of both fundamental and engineering applications in mind and is capable of withstanding the weight of cells up to several hundred kilograms while remaining stable at high resolutions thanks to the granite exoskeleton. Correspondingly, the instrument allows for voluminous cells thanks to the movable detector and the abundant free space above (~ 1 m) and below the instrument (~ 1.5 m).

This, together with the aforementioned performances has already allowed a range of high pressure, high temperature and hydro-mechanical *in-situ* tests to be performed at high speeds.



## Speaker Sessions and Seminars / 95

**In-situ diagnostics of crystal growth by energy-resolved neutron imaging**

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There is usually a long delay between the discovery of novel single crystal materials and their use in practical applications. The new materials are often characterized with a small synthesized grain, while many applications require relatively large crystals (e.g. large enough to absorb gamma photons in case of gamma detectors). Introduction of new single crystal materials is often limited by the difficulties related to crystal growth. Optimization of single crystal growth techniques can benefit from the recent progress in high-resolution energy-resolved neutron imaging, which provides unique possibilities to perform in-situ measurements of process parameters, which currently can be obtained only indirectly.

This paper presents the results of recent experiments demonstrating the possibility to measure the elemental distribution, shape and location of liquid/solid interface and structural defects in several single crystal materials developed for gamma detection. The concentration of several elements is imaged with sub-mm spatial resolution during crystal growth, revealing the dynamics of elemental segregation across the boundaries between the solid and liquid phases as well within the liquid phases.

Our results indicate that the optimization of growth parameters can be performed through a feedback control as information on the growth process can be obtained in real time (minutes to hours in crystal growth terms). This should enable a quick path in the search for optimal growth parameters, thus greatly reducing timescale between the laboratory material discovery and upscaling to commercial/production.

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## Speaker Sessions and Seminars / 113

**'Neutron Microscope' instrument at PSI – recent upgrades and the first users experiments**

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The high resolution neutron imaging instrument ('Neutron Microscope') at the Paul Scherrer Institut (PSI) allows for neutron imaging down to 5 micrometres spatial resolution (Trtik & Lehmann, 2016). The transferrable nature of the instrument allows for its use at different beamlines of SINQ (namely at ICON, POLDI, BOA) and also at other neutron sources. The recent advances in both the spatial resolution and the available light output of the high-resolution scintillator screens based on highly isotopically enriched 157-gadolinium oxysulfide will be presented. On the top of the instrumental upgrades, the examples of the results of the recent user investigations will be presented. The authors list of this presentation will be amended accordingly with respect to the presented users' applications.

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## Neutron energy spectrum evaluation and radiography using metal foil activation and IP transfer method

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### Introduction

Neutron radiography is used as an effective nondestructive inspection method for hydrogen, hydrogen compounds or specific light elements such as lithium and boron, which are difficult to see by X-ray radiography. In recent high-intensity pulsed neutron sources, neutron imaging methods using resonances of specific isotopes are introduced with the progress of neutron time-of-flight (TOF) method and pulse synchronized two-dimensional neutron detectors. However, the TOF method cannot be applied to continuous neutron sources such as nuclear reactors or cyclotron accelerator neutron sources. Widely utilized gadolinium or dysprosium converter based neutron radiography is a method of thermal neutron imaging. Neutron energy distribution measurement of the irradiation field by means of Au foil and cadmium activation method to obtain Cd ratio can only determine the ratio of thermal neutron to epi-thermal neutron. Further improvements are required for precise neutron energy distribution measurement.

### Summary

This report describes the evaluation method of irradiation field neutron spectrum of continuous neutron sources by means of the resonance absorption and activation method of specific metallic nuclides. The arrangement of objects and foils of the multiple foil activation method and examples of transferred images. Neutrons, penetrated through step wedges of Dy, In, Au, Er and Manganin (Mn, Cu and Ni alloy) metals, activate foils of the same species at each resonant absorption energy. Activated metal foil images are separately transferred to IPs. A half portion of the step wedges is covered with Cd metal foil filter to cut-off the thermal neutron region, that leads to the improvement of S/N at the resonance region. The measured luminance of each metal foil step wedge was normalized by those in the area without objects, and the neutron flux corresponding to each resonance energy was obtained. The measured spectrum was in good agreement with the irradiation field spectrum calculated by PHITS analysis code. The developed method is effective for determining neutron spectral distribution up to 500 eV in continuous neutron sources, as well as imaging at specific energies.

## Magnetic field induced neutron phase contrast imaging with grating interferometry

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### Magnetic field induced neutron phase contrast imaging with grating interferometry

Magnetism has always been in the spotlight and neutrons have played an essential role in understanding this physical phenomena due to their intrinsic magnetic moment. Polarized neutron imaging and the grating interferometer (nGI) technique have been established as powerful means [1; 2] for investigating superconductors and domain wall of ferromagnetic materials 3.

Here we present an upgrade of the regular nGI setup, which allows to operate with polarized neutrons (p-nGI) in order to retrieve differential phase contrast images (DPCI) induced by the magnetic field and to visualize its spacial distribution. The DPCI yields quantitative information about the phase shift induced by the refraction of the polarized neutron beam on the phase object, due to the magnetic interaction between the sample and the neutron spin state.

The talk reports our experimental results achieved at the Beamline for neutron Optics and other Application (BOA) [4] at Paul Scherrer Institut (PSI).

A beryllium filter was used as energy selector in order to improve the sensitivity of the setup to the magnetic field strength.

Two different cases were taken into account for demonstrating the feasibility of this technique: a tailored sample, consisting of an homogeneous square-shaped magnetic field aligned parallel to the guide field, and a rectangular Neodymium permanent magnet as a general case. Hence, the magnetic phase shift image (PCI) of the experimental data was retrieved by integrating the DPCI, taking into account the energy spectrum of the beam and the visibility response function [5] of the p-nGI setup.

Subsequently, the experimental results were validated with the expected value calculated from the Hall probe measurements and finite element method (FEM).

We put particular emphasis on the understanding of the adiabatic and/or non-adiabatic nature of the process which define the condition for the accessible features.

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## Suitability of Gd<sub>2</sub>O<sub>2</sub>S:Tb/6LiF Scintillator for Industrial Film Neutron Radiography

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Commercial neutron radiography continues to be performed almost exclusively with gadolinium conversion screens and single emulsion x-ray film owing to several factors including the large image plane, high resolution, and the presence of standards. In recent years, the development of thin scintillation screens with improved resolution for digital neutron imaging has continued. This presents new opportunities for film imaging as well.

The imaging abilities of TriTec's 30 micron Gd<sub>2</sub>O<sub>2</sub>S:Tb/6LiF (80%/20% mixture) conversion screen with chemical vapour deposited gadolinium screens and a range of single emulsion films are compared based on image quality, and exposure requirements. The ASTM E545 image quality indicators and a line pair gauge are utilized to assess image quality in addition to test objects. Competitive quality was achieved, though practical limitations for use in industrial settings do exist.

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## Radiation Degradation of Silicon Crystal Used as Filter for Neutron Radiography

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Single crystals of some materials like silicon, sapphire or bismuth are often used as radiation filters for neutron radiography beams. These single crystals are relatively transparent for thermal and cold neutrons and can suppress fast neutrons and gamma radiation. Cross sections for thermal neutrons of these single crystals are 5 to 10 times lower compared with amorphous or polycrystalline samples of the same materials. When the single crystal is irradiated the crystal lattice is partly damaged and filter characteristics of the crystal can be deteriorated. This aspect of radiation degradation was studied for silicon single crystals.

During neutron irradiation of a silicon single crystal, defects in the crystal lattice are induced by two main ways: <sup>30</sup>Si nuclide is transmuted to <sup>31</sup>P by n-gamma reaction and Si nuclei are displaced by fast neutron scattering on the nuclei. The second type of induced defects can be partly removed by annealing of the crystal.

Experiments for silicon radiation degradation measurements were carried out in the LVR-15 research reactor, which is a multipurpose facility with maximal thermal power of 10 MW, situated in Řež near Prague. One horizontal channel is used for neutron radiography facility where cylindrical silicon single crystal with diameter of 78 mm and 1000 mm long is inserted into the channel as neutron filter. During reactor operation the crystal is permanently irradiated from reactor core. In the position of silicon cylinder base faced to reactor core, the average neutron fluence rate is estimated to about 1E17 cm<sup>-2</sup>/year.

Two experimental methods were used for estimation of radiation degradation of silicon crystals. First method was based on fluence rate measurement on the beam output by activation detectors repeatedly during four years when the filter was installed in the channel. In the second method, thermal neutron absorption of the silicon single crystal sample (cylinder  $\phi$ 78 mm x 100 mm) was measured before irradiation in the reactor core and after the irradiation. Neutron absorption was measured on LVR-15 neutron radiography beam by activation detectors. Silicon cylinder axis was parallel with the beam and the detectors were fixed on both cylinder bases. The results indicate that the radiation degradation of the filter in the LVR-15 neutron radiography facility is acceptable low for a few years of facility operation.

## Novel scintillation screen with significantly improved radiation hardness and very high light output

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Novel scintillation screen with significantly improved radiation hardness and very high light output

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That the bombardment with a high energy radiation has an effect on the material properties is well known since many years 1. Vacancies or disorder incorporated in the material can have a significant influence on the luminous intensity of scintillation material. In the development of scintillation material for high energy particle detection big efforts have been done within the last years 2. Actually the highly radiation resistant garnets seem to be the state of the art in that field 2.

It is known that neutrons affecting, especially the alpha and triton particle originating from the capture reaction, lead to a degradation of scintillator screens regarding the light output versus neutron fluence. Two different kind of scintillators are commonly used for neutron imaging applications: (i) 6LiF/ZnS scintillation screens with very high light output and reasonable resolution and (ii) Gd<sub>2</sub>O<sub>2</sub>S:Tb scintillation screens for very high resolution measurements but 10 times less light output.

Within a 2 years development project with PSI we wanted to understand for the 6LiF/ZnS scintillator type the effects which are responsible for the degradation mechanism and therewith be able to develop a more radiation hard neutron scintillator system. The goal was to not only improve radiation hardness but also to improve the light output in comparison to the traditional 6LiF/ZnS-scintillation screens.

In this talk the results of this development project, leading to a new type of scintillation screen with significantly improved radiation hardness, but still very high light output, are presented.

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## Dr

Rachel White<sup>1</sup> ; Filomena Salvemini<sup>1</sup> ; P.D. Callaghan<sup>1</sup> ; A Stopic<sup>1</sup> ; Paula Dredge<sup>2</sup>

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Title: First look neutron autoradiography of paintings on DINGO

Authors: R. White, F. Salvemini, P.D. Callaghan, A. Stopic, P. Dredge.

Abstract:

Neutron activation autoradiography has been used for decades to study the presence and distribution of activating pigment elements in paintings. The technique has been used to study the works of Georges de La Tour<sup>1</sup>, Jan Steen<sup>2</sup>, Rembrandt, Van Dyck and Vermeer<sup>3</sup>. These studies have examined the pigments used, the handling of the paint by the artist and subsequent conservation efforts.

The installation of the DINGO Radiography/Imaging/Tomography Station at the Australian Centre for Neutron Scattering has brought this capability to Australia. To prepare for the use of this technique on paintings in museum and art gallery collections a series of optimisation tests have been

carried out on prepared panels. The test panels were prepared in a range of artistic styles using pigments and paints that were used in the various eras and with the assistance of conservators at the Art Gallery of NSW. After irradiation the panels were exposed to image plates for set periods of time to detect the decay of pigment elements. Nuclear Activation Analysis was conducted on pigments and paints used to create the panels to assist in the optimisation of the windows of time for image plate exposures. This poster will show the results of the neutron autoradiography of a range of painting styles and across different eras of painting materials.

Figure 1 Left: Painting sample by Simon Ives using pigments traditional to Rembrandt's era. Right: manganese pigments visible in the autoradiograph (exposure for 5 minutes, taken 25 minutes after irradiation ended).

Figure 2 Abstract painting using modern paints by Celine de Courlon, showing copper containing turquoise paint as a block on the left and its variable thickness within the background colour. Autoradiograph from 4 days after irradiation of panel, image plate exposure of 4 days.

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## The 15th-18th terracotta doll investigation using a compact neutron tomography system at Thai research reactor

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It is well known that neutron imaging is a powerful non-destructive technique in archaeological studies, especially for visualization of organic contents or low-density parts inside antiques. In Thailand, the neutron imaging system has been developed to perform neutron tomography (NT) for archeological studies since 2015. A compact neutron tomography system, which composes of a CCD camera coupled with a LiF/ZnS fluorescence screen and an in-house developed rotation stage, was used to investigate the internal structure of an object. The experiment was set up at Thai Research Reactor (TRR-1/M1) of Thailand Institute of Nuclear Technology (Public Organization) with the power of 1.2 MWth. The neutron intensity at the radiographic position was about  $10^6 \text{ n.cm}^{-2}\text{s}^{-1}$ . The L to D ratio of the imaging system was approximately 50. In this work, an ancient sample of interest, namely, the 15th-18th terracotta doll was investigated to test the NT system. The resulting 2D neutron image showed a crack at the neck part and a small gravel inside the body. Then, the neutron tomographic images were reconstructed by means of the Octopus Image software. Even with the compact NT system, the 3D neutron image of the ancient doll was successfully reconstructed. The image revealed some hidden organic materials coated on the neck part of the doll. Moreover, the 3D neutron image of the ancient doll, which was obtained from the advanced neutron imaging facility at ANSTO, was used for comparison.

## Recent progress of neutron imaging facility and applications at China Advanced Research Reactor

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A thermal neutron imaging facility and a cold neutron imaging facility are under construction at China Advanced Research Reactor (CARR). At present, some main components, such as collimator, sample table, detection system, etal have been finished, and the others are under construction. The thermal neutron imaging facility will be operated in two modes: high intensity and high resolution depending on the distance between the sample and the aperture. The cold neutron imaging facility is more flexible, and sample can be placed at several positions, depending on the research demands. With the neutron imaging testing station built at the end of one neutron guide, many applications had been carried out, including testing Zr alloy nuclear fuel cladding, fuel cell, lithium battery, plants, rocks, fossils and so on. Both the neutron radiography and neutron tomography methods were studied.

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## A Study on Fusion Method of Neutron and X-ray CT Images

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Neutron and X-ray computed tomography produce different images of the same test workpiece. In order to overcome shortcomings of X-ray CT, we study method of using both neutron and X-ray CT to tomography and do data fusion of neutron CT and X-ray CT.

We designed and carried out experiments on the same model using X-ray and neutron respectively. After the reconstruction of tomographic images, and studied method based on Canny edge detection to detect the image edges, carried out Harris corner detection and used NCC algorithm to find matching feature points, then we did the RANSAC algorithm to eliminate the wrong pair of feature points then completed the image registration. We studied the fusion algorithm based on wavelet transform and average method, we got the final fusion image.

In this paper, the image fusion methods based on the Canny edge detection, Harris corner detection, NCC and RANSAC will be described, and the preliminary neutron and X-ray CT imaging and fusion experiments and the results will also be present and discussed.

We studied a generic way of automatic image registration based on algorithm of the Canny edge detection, Harris corner detection, NCC and RANSAC in our research. From the results that we got we can see clearly that the fusion image has a better result compared to the results based on either X-ray CT or neutron CT. It is clearly that data fusion is a way to make up the deficiencies of X-ray CT and data fusion will be more widely used in the future.

**Speaker Sessions and Seminars / 99**

## Neutron radiography of water imbibition in a smooth-walled fracture within sandstone

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Water spontaneous imbibition in unsaturated fractured rocks is a ubiquitous phenomenon in nature and engineering such as the enhanced oil recovery by water flooding, the storage of hazardous wastes underground and the development of geothermal et al. In the presented work, direct visualization of water imbibition in a vertical smooth-walled fracture with a width of ~114  $\mu\text{m}$  within a low permeability silty sandstone sample was achieved using neutron radiography at China Advanced Research Reactor (CRAA). The high-speed imaging mode i.e. 10 frames/second was employed to capture the rapid transport of water in the fracture at first 100 seconds of imbibition. Then the neutron image was captured every 2 seconds to improve the image quantity until the sample was saturated by water. Based on the neutron images, the wetting front was tracked on both vertical and horizontal directions to calculate the sorptivity. It was found six stages can be distinguished based on the varieties of sorptivity determined along the vertical smooth-walled fracture. The wetting front can travel at the height of ~17 mm along the smooth-walled fracture during the first 0.5 seconds of imbibition. Then the advance of wetting front along the vertical smooth-walled fracture slowed down due to the effect of gravity and water transport from fracture to matrix. Once the lower half of the sample was saturated by water, the infiltration of the wetting front along the vertical smooth-walled fracture accelerated again. The sorptivity determined along the horizontal direction varied in the range of 0.3073-0.3663 mm/s-0.5. Moreover, cumulative absorbed water volume in the investigated sandstone sample was determined at different imbibition time after the correction of neutron scattering and beam hardening effect. It was found the cumulative absorbed water volume increased linearly with the square root of time at two stages. Cumulative absorbed water volume in the sample grew much fast at the first 4 seconds of imbibition. At last, the time-lapse water content map of water imbibition in the investigated silty sandstone sample was presented. It seems the first report about the visualization of the full process of water imbibition in the smooth-walled fracture within a low permeability silty sandstone sample by neutron radiography.

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### Qualification and development of fast neutron imaging scintillator screens

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We have performed extensive testing and qualification of different commercial fast neutron scintillator screens in camera-based imaging detectors. These include BC400 organic scintillator from St. Gobain and ZnS(Cu) inorganic scintillator from RC Tritec AG. Furthermore, we have developed simple and inexpensive ZnS-based fast neutron imaging screens and their performance have been tested and compared to the aforementioned commercial ones. ZnS(Ag) and ZnS(Cu) powders have been mixed with optical epoxy, deaerated and casted into sheet form using an aluminum frame. Furthermore ZnS(Ag) was mixed with high viscosity glycerol to create suspension type imaging screen. The ZnS concentration and the screen thickness have been optimized using sample screen pieces. To initially test the performance of the screens, the fast tail of the flux in the thermal NEUTRA beam line at the SINQ spallation source of the Paul Scherrer Institute Switzerland has been utilized. Furthermore, extensive testing has been carried out at the RAD beamline of the 10 MW research reactor of the Budapest Neutron Centre (BNC), Hungary. The latter beamline is routinely utilized for thermal neutron imaging, however it has been adapted to enable fast neutron studies using in-beam filters against gamma and thermal neutrons. Our results indicated that the ZnS(Cu) commercial screen from the company Tritec AG had the best performance which could still be slightly improved according to our results. On the other hand, the BC400 screen performed the worst mainly due to



its low light output, which is detrimental in a camera-based imaging detector. The in-house ZnS-epoxy screens produced about 60% of light intensity of its commercial counterpart, which is mainly due to the lower hydrogen density of the optical epoxy compared to polypropylene. The glycerol suspension screen underperformed relative to expectations due mainly to an apparent separation of the scintillator powder and the glycerol. Some fast neutron radiographic images are shown to demonstrate the capabilities of the screens.

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### Development of kfps bright flash neutron imaging for rapid, transient processes

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The speed of thermal/cold neutron radiography is limited by the available flux even on the strongest spallation sources. The flux limitation can be alleviated using phase-lock, ensemble averaging techniques for periodic, repeating processes as it has been demonstrated on several samples like engines, pumps etc. However capturing rapid, transient, non-periodic processes by neutron imaging remains difficult. Recently we have demonstrated 800 fps cold neutron imaging 1 at the ICON beam line of the SINQ continuous spallation source utilizing the highest available flux of that beam line. Opposed to spallation sources, TRIGA reactors have the capability due to their special fuel composition to produce extremely bright neutron pulses for a short duration. This opens the possibility to image short, very rapid transient processes at very high rates. We develop bright flash thermal neutron radiography at the beam line of the 1 MW Penn State Breazeale research reactor. This TRIGA type reactor is able to produce pulses up to 1 GW with a FWHM of around 20 milliseconds. We have achieved bright flash radiography up to 4000 fps on a field of view (FOV) of around 15 square centimeters and at a spatial resolution of about 0.5 mm, however higher frame rates and FOV is feasible. The detector used is a CMOS camera based system featuring a 400 um thick LiF/ZnS converter screen. We demonstrate the method on air-water two-phase flow in a bubbler as simple, non-periodic dynamic process.

1 R. Zboray, P. Tritk, "800 fps neutron radiography of air-water two-phase flow", *MethodsX*, 5, pp. 96-102 (2018).

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### In-depth analysis of high-speed, cold neutron imaging of air-water two-phase flows

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We have recently published first results of very high frame rate cold neutron radiography of air-water two-phase flows in a bubbler 1. Gas-liquid two-phase flows play an important role in many scientific and engineering problems. These include the efficient and safe operation of chemical reactors, heat exchangers, boilers and nuclear reactors. Two-phase flows, in general, are very rapidly changing, non-periodic processes, requiring high temporal resolution instrumentation techniques to capture their dynamics. We have demonstrated imaging rates up to 800 frames per second by using a high-efficiency (relatively thick) scintillator screen in combination with the highest available flux

on the ICON beam line of the SINQ continuous spallation source and an imaging detector based on a high-speed sCMOS camera. This combination renders the spatial resolution to relatively modest value of about 0.5 mm, which is nevertheless sufficient for resolution of bubbles of the size down to about 1.0 mm in motion with rather high frame rates for neutron imaging of non-periodic processes. Here we present an in-depth analysis of the experiments taken at several air injection rates and at different imaging rates. The dynamically resolved gas fraction distribution is investigated together with the bubble size and the instantaneous gas interfacial velocity. The latter is evaluated using the optical flow (OF) method. The analysis can create a useful framework for analyzing dynamic neutron imaging sequences of the phasic velocities of a multi-component system in general.

1 R. Zboray, P. Trtik, "800 fps neutron radiography of air-water two-phase flow", *MethodsX*, 5, pp. 96-102 (2018).

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## Reviving and extending the neutron imaging capabilities at the Penn State Breazeale Reactor

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The Penn State Breazeale reactor, a 1 MW TRIGA type reactor has been utilized successfully for neutron imaging in the past. Presently a single beam line with a thermal spectrum is utilized for imaging, however the reactor is just about to undergo a major refurbishment involving the installation of a new core moderator assembly. This enables the establishment of several new neutron beam lines around the reactor including three with a cold spectrum. The new beam lines will feature improved flux and lower gamma background than the existing one. One of the new thermal beam line is solely dedicated to imaging. It will feature (re)moveable in-beam filters to enable multi spectrum imaging ranging from the standard thermal to epithermal and fast energy ranges. One of the new cold beam lines will have dual utilization including neutron imaging. This paper discusses in details the aforementioned new, imaging relevant beam infrastructure and its design. Some recent imaging results from the existing beam line utilizing new imaging detectors are discussed as well together with the future plans to implement a variety of different imaging methods and techniques. These will include bright flash imaging for rapid, transient processes, fast neutron imaging and multi-spectrum imaging.

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## Fast neutron imaging at a reactor beam line

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Fast neutron imaging is a promising nondestructive technique for testing dense and voluminous objects of practically any material composition. Only fast neutrons can provide images free of artifacts and with reasonable contrast for dense and voluminous samples containing mixed low-Z/high-Z materials, where photon-based techniques or even thermal neutron imaging would fail. Though fast neutron contribution in a thermal imaging beam line is typically considered as a burden, we have investigated its application for imaging. In many thermal beam lines the epithermal and fast

neutrons are intentionally suppressed by using typically sapphire filters, usually there is a significant flux of MeV neutron content from the fission spectrum tail in the beam. Of additional burden is the usually strong gamma background. We have utilized the RAD beamline of the 10 MW research reactor of the Budapest Neutron Centre (BNC), Hungary, and demonstrated the feasibility of fast neutron radiography and tomography. The beamline is routinely utilized for thermal neutron imaging having a thermal flux of around  $4e7 \text{ cm}^{-2}\text{s}^{-1}$ . The beam features further a relatively high gamma background of 8.5 Gy/h (non-attenuated) and a significant fast neutron contribution of  $2.7e7 \text{ cm}^{-2}\text{s}^{-1}$  ( $E > 2.5 \text{ MeV}$ ). Gamma and thermal neutron filtering has been applied and optimized on the beam line. We also optimized the imaging detector to improve the results. Fast neutron radiography and tomography have been performed using camera-based imaging detector in conjunction with different types of scintillator screens (organic and ZnS-based inorganic ones). A spatial resolution of around 1.6 mm has been achieved. Typically 10-15 minutes exposures were needed to obtain good quality radiographic images, whereas several hours of acquisition were needed to obtain a full tomographic set of images. High quality imaging results have been obtained on large (150 mm in diameter) and robust objects proving the potential of fast neutron imaging.

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## Plant Growth Promoting Rhizobacteria Enhance Plant Drought Tolerance by Changing Soil Physical and Hydraulic Properties

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Increasing global population results in unprecedented demand on agriculture for food. Two of the key challenges for sustainable agricultural development are water shortage and soil degradation. Therefore, it is critical to develop novel solutions for plant growth under restricted water availability. Plant-growth promoting rhizobacteria (PGPR) can benefit plants by increasing nutrient uptake, improving disease protection and drought tolerance, etc. Previous studies mostly focused on bacteria or the interaction between plant and bacteria, while little is known on how soil properties are modified by the presence of bacteria. Using UD1022, a *B. subtilis* strain, as a model bacteria, we demonstrated via soil water characteristic measurements using the HYPROP that UD1022-treated samples retained more water and had lower hydraulic conductivity than its controls. In addition, we investigated the effects of UD1022 treatment on soil water evaporation via combined neutron radiography, neutron tomography, and X-ray tomography imaging technique. Neutron radiography images showed greater water retention by UD1022-treated soil samples than their controls due to reduced water evaporation. Neutron and X-ray tomography 3D images revealed that water distribution in UD1022-treated soil samples during evaporation was less homogeneous, i.e., there were more disconnected water pockets compared with the controls where water was distributed more uniformly. Water from the disconnected water pockets transport to the evaporative surface through diffusion rather than capillarity, which reduces the soil water evaporation rate. Our study provides pore-scale mechanistic explanation for the reduced evaporation rate for UD1022-treated soil samples. Enhanced water retention in soil and reduced evaporation by UD1022 treatment is likely due to the production of extracellular polymeric substances (EPS) by UD1022, which was visualized by scanning electron microscopy (SEM).

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## Dynamic Lithium Diffusion in Lithium Batteries studied by Rapid Neutron Tomography

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Lithium batteries are considered one of the most transformative technologies of the 20th century. They are a reliable power source for portable devices which are used every day by billions of users, such as mobile phones, laptops, pacemakers and increasingly in electrical vehicles. Lithium batteries have high energy density and capacity, superior reliability and long shelf life of up to 20 years. This makes them the best choice for applications in extreme environments.

It is essential for the development of the next generation lithium batteries to have a deeper understanding of the macroscopic lithium diffusion processes insight the batteries during dynamic discharging to elucidate mechanisms which reduce the battery performance. To obtain such information three-dimensional imaging techniques, such as X-ray tomography, are state of the art. However, for direct imaging of lithium, X-ray techniques are often unsuitable due to the high transparency for low-atomic number elements like lithium. Neutrons offer a superior alternative with a high sensitivity for lithium, but neutron tomography suffers from insufficient spatial and temporal resolution. During the last decade, however, new high reflective neutron guides and high sensitive neutron camera systems have led to a significant reduction in the acquisition times.

We present time resolved in-operando neutron tomographies of the lithium diffusion process inside a commercial lithium – thionyl chloride battery (LS14250 from Saft) during discharging. The continuous three-dimensional imaging, with 10 minutes exposure time per tomogram, enables the visualisation of the lithium removal from the lithium-metal electrode and the lithium diffusion inside the thionyl chloride cathode. The experiment allows quantification of the removed lithium from the electrode as a function of time and correlate with electrochemical performance. Furthermore, the evolution of  $SO_2$  gas is detected which insulate regions on the anode and hinders the diffusion process in the cathode. Such processes can lead to a significant reduction in the capacity and performance of the battery. Our experiment demonstrated that neutron tomography is a powerful tool to image dynamic process in lithium batteries with a sufficient time resolution of the dynamic processes. Future work will focus on the application to a range of Li-ion chemistries and will seek to explore the degradation processes associated with long term operation and operation in extreme environments.

## Speaker Sessions and Seminars / 15

### Neutron imaging of Li-ion batteries with fission and thermal neutrons

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Neutron imaging provides outstanding sensitivity to light elements, e.g. high contrasts between hydrogen containing materials and metals. The neutron imaging facility NECTAR at MLZ regularly uses a fission neutron spectrum with a mean energy of 1.9MeV. These high energy neutrons allow insight in large objects of up to several ten centimeters with a high selective contrast for hydrogen. In contrast thermal neutrons with a mean energy at 28meV show lower penetration power but provide a

much better spatial resolution. A combination of these data will benefit from the even more selective contrast for hydrogen provided by fission neutrons, while thermal neutrons will serve to reach higher spatial resolution for structure materials surrounding the hydrogen containing materials. Therefore an upgrade of the instrument is currently ongoing to make both neutron energy ranges available at a single setup and benefit from their respective advantages to follow the electrolyte distribution inside lithium-ion batteries during operation.

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