

Imaging and Neutrons 2006

**Book of Abstracts
For Presentations and Posters**

**October 23—25, 2006
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
USA**

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Session MO1: Neutron Imaging Techniques and Challenges I

Chair: E. Lehmann, Paul Scherrer Institut

3D Imaging Techniques: A Comparison

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We review various imaging methods in 2D and 3D and try to evaluate the role of slow neutrons in the field.

Neutron Scattering as Imaging

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Because of flux limitations, relatively few of the applications of neutrons are in techniques that would be ‘conventionally’ considered as imaging, and these are restricted to length scales $> 10^{-5}$ m for the foreseeable future. On the $< 10^{-8}$ m scale there is an increasing demand for ‘conventional’ imaging using photons or electrons, where individual regions of a particular object are investigated. While the ability to do this is appealing, I would argue that the results can be scientifically inappropriate or even misleading. Individual small regions may not be representative of a larger system, and the brilliance of the probe required to investigate smaller and smaller regions inevitably leads to local damage and results which do not represent the equilibrium system. There is instead a strong argument in many cases for being able to visualize – ‘image’ – a valid average representation of a small region, as is typically done with diffraction techniques. The reason that these are not normally considered as ‘images’ is largely a question of tradition and presentation, but it could be noted that in the field of protein crystallography nowadays it is extremely rare to see diffraction data but very common to see real space ‘images’ of protein structures.

Experimental techniques are not the main limitation to exploiting this type of neutron ‘imaging’ for work on the $< 10^{-8}$ m scale. The limitation is almost entirely with the available software. There are many well established methods, for crystalline- and non-crystalline systems; these just need to be implemented (with suitably powerful computing resources) to directly produce real space images on the experimental time scale. Two major challenges can be identified, both experimental and in software but with major scientific potential. The first is to extend this to produce atomic resolution images up to 10^{-7} m scale systems, e.g., for proteins in solution. The second is to move into the time domain and produce moving images – ‘movies’ – on the atomic time and length scales, directly from experimental measurement.

The Neutron Imaging Facility ICON as Example for a State-of-the-Art Installation

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Coursed by the very positive experience in the operation of the first neutron imaging beam line at a spallation neutron source ever built – NEUTRA at the thermal beam port of SINQ- it was decided to construct a second facility at SINQ, using cold neutrons.

The design of the facility considered the NEUTRA principle (pinhole geometry without mirrored neutron guides) but takes into account new options and opportunities. These are phase-contrast enhancement, energy-selective studies, micro-tomography and the handling of much extended technical objects and arrangements.

In order to obtain optimal conditions for the beam properties extended studies with the Monte-Carlo code MACSTAS were performed, concluding in the option for a variable inlet aperture with six options for the opening (80 mm, 40 mm, 20 mm, 10 mm, 1 mm and 0.5 mm – all circular). In this way the neutron intensity, collimation and field-of-view can be tuned according to the demands of the application.

New features like variable beam limiters, fast shutters, variable flight tubes and sample manipulators together with comfortable space for experimental infrastructure inside the shielded and protected area enables a very flexible user program at ICON.

New devices were designed and implemented at ICON:

- a new CCD camera system for micro-tomography, based on advanced optical components and improved scintillator technology,
- the energy-selective imaging option with a selector-turbine based set-up in front of the beam line inside a separately shielded room,
- a scanning desk for samples of up to 500 kg and 150 cm extension in two directions,
- the grating interferometry setup for the direct experimental determination of the phase part of the transmission signal in imaging.

All these options enable an extension of the program at PSI for user support in neutron imaging in different directions, scientific and industrial based. Together with the options at the NEUTRA installation it can now comfortably be decided which conditions fit preferably to the particular problem in respect to transmission and contrast.

Absorption and Phase Contrast Neutron Imaging (overview)

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Within the last decade neutron tomography and radiography significantly gained importance. Especially its application in non-destructive testing for industrial components can be underlined. A good example is the automotive and aviation industry, where a high contrast for the used lubricants and adhesive materials is required. In contrast to X-rays, neutrons are able to penetrate thick layers of metals and provide on the other hand a high sensitivity to hydrogen containing materials. The charge-free neutron interacts with the core of the atom, while in contrast X-rays interact with the charge distribution of the electron shell. Therefore the X-ray attenuation coefficients increase with the atomic number of the elements, i.e. with the number of electrons. The interaction probability of neutrons with the atomic core depends on the coherent scattering length a_{coh} , which does not show a systematic dependence from the atomic number of the element. Summarizing the advantages of using neutrons for imaging one can state:

- neutrons are very sensitive to light elements like H, Li, B, where X-rays do not provide a good contrast (low and similar atomic numbers),
- the distribution of attenuation coefficients for neutrons is independent of the atomic number which helps to achieve contrast even for neighboring elements, for X-rays one finds an approximately exponential increase with the atomic number,
- neutrons easily penetrate thick layers of metals like Pb, Fe and Cu where standard X-ray imaging facilities with energies of several hundred keV fail, and
- neutrons can distinguish between isotopes (for instance ^1H and ^2H) which is not the case for X-rays.

The drawbacks of neutron imaging are related mainly to the coarse spatial resolution of few hundred micrometers due detection limits and the weak neutron sources (research reactors and spallation sources) in comparison with the synchrotron facilities.

Additional to the absorption contrast radiography a phase-contrast technique is available with thermal and cold neutrons. Phase-contrast radiography allows to visualize objects which provide very low contrast with the standard absorption radiography technique. Image formation in this case is based on a visualization of phase variations in a beam with a high lateral coherency by its transmission of the object. Due to the fact that neutrons could not be focused in a micrometer sized spot like X-rays, a small pinhole and a large distance between the pinhole and the sample is needed in order to achieve a good lateral coherence length of the neutron beam (propagation technique).

Session MO2: Neutron Imaging Techniques and Challenges II

Chair: B. Schillinger, Technische Universität München

Fast Neutron Radiography

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Due to the different dependences of the linear attenuation coefficients on the atomic numbers, radiography and tomography using fission neutrons offer complementary information compared to the well established techniques using X-rays, gamma-rays, thermal or cold neutrons. They can penetrate thick layers of high density materials with only low attenuation, while for light materials, especially hydrogen containing materials, the attenuation is rather high.

The Neutron Computer Tomography And Radiography NECTAR facility at the Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II) uses fission neutrons of a mean energy of about 1.7 MeV, created in two converter plates set-up of highly enriched uranium, for the non-destructive inspection of objects. The beam quality, i.e. the neutron divergence can be adapted to the object to be measured by using different collimators, resulting in L/D-values up to 300. The available neutron beam intensity at the measuring position is up to $1.6E+08 \text{ cm}^{-2}\cdot\text{s}^{-1}$ for a beam area of about 40 cm x 40 cm. For conventional imaging a two-dimensional detector system based on an electro-cooled CCD-camera (1024 x 1024 pixels) is used, other more specialized systems are available.

The actual setup offers an optimized environment for performing high quality neutron imaging, but still might be drastically be improved by increasing the efficiency of the neutron converting process. Up to now, the efficiency for this process is less than 1%. Together with the high gamma-background, caused by the fission process, this actually limits the usable dynamic range in imaging. Therefore the main challenge for the future in fast (here: fission) neutron imaging is to increase the detection efficiency without loss in spatial resolution (about 0.5 mm to 1 mm) on large detection areas (up to 40 cm x 40 cm).

As the actual results already indicate, the availability of such an enhanced detection system will establish fast neutron radiography and tomography as an additional, extremely valuable method for the non-destructive inspection of objects in many fields of application and research.

Tomography Technique Using Epithermal Neutrons

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Neutron-resonance absorption spectroscopy (N-RAS) is a new method for studying the dynamics of atoms by analyzing the Doppler broadening of their resonance peaks. The method enables us to investigate motions of a particular nuclide, because we can identify it with the resonance energies exist on the epithermal neutron region. The broadening of the resonance absorption peak is expressed by a formula including effective temperature, T_{eff} , as one of parameters. T_{eff} , which corresponds to the local temperature of the nuclide, has an asymptotic trend to the real temperature of the object above room temperature. Then we can obtain information about the kind and temperature of the nuclide.

In the current N-RAS the neutron absorption is recognized as the prompt gamma-ray emitted isotropic in space from the target nuclide. The neutron time-of-flight (TOF) technique is used to record the absorption spectrum. From this spectrum we can obtain information about the averaged nuclide motion inside the bulk object. In addition, the tomography calculations can be carried out using a set of the spectrum include the object spatial information of the beam slit position and the object rotation angle. We call the technique N-RAS/CT. Moreover, with carrying the tomography calculations for the each TOF channel we can reconstruct the N-RAS spectrum over the wide energy range at each position inside the object. Such technique, we call neutron resonance

imaging (NRI), gives us the analyzing ability of kinds of nuclide, nuclide distribution and temperature distribution over the object cross section with remote-sensing and non-destructive scan.

We made the N-RAS measurements for sample objects include ^{73}Ta with temperature gradient (from 310 K to 400 K) inside. With applying the computer tomography (CT) technique to the obtained set of T_{eff} we could reconstruct a cross sectional image of the nuclide and temperature distributions non-destructively. Then we developed the method as detailed analysis of TOF resonance absorption spectra. This improved way, NRI, was that the CT imaging are repeated at each TOF channel and pile up the reconstructed images in order to make the resonance spectrum. The number of calculations was increasing in such method but we could deduce more information from the reconstructed spectrum over the wide energy range. The absorption peak positions and shapes of the reconstructed spectrum had good agreement with the original TOF spectrum, and the nuclide and temperature distributions were reproduced original well.

In conclusion we think that the possibility of the technique was demonstrated in our study. But it still has the problem of the spatial resolution and neutron intensity. With the developments of a fine position sensitive neutron detector and an intense source, it can be applicable to many fields such as physics, chemistry, material science, nuclear engineering, mechanical engineering and biology.

Thermal Neutron Microscopy

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The results of thermal neutron microscopy experiments using biconcave, spherical compound refractive lenses (CRLs) and a wide-bandwidth thermal-neutron beam, are presented for the first time. The experiments were performed using a nuclear-reactor neutron source at the McClellan Nuclear Radiation Center (MNRC). We obtained micrographs of Cd slits with 2 to 5 X magnification. The resolution of CRL microscope was compared with that of a pinhole camera with the same aperture diameter as that of the CRL, as well as with a cylindrical tube of the same length and aperture diameter as that of the CRL. The modulation transfer function (MTF) of the CRL was calculated and compared with the measured modulation transfer factor at two spatial frequencies, showing good agreement.

Experimental and theoretical results demonstrated superior image quality of the CRL over that of the pinhole camera or a cylinder. Furthermore, simulations show the spherical lens CRL has better resolution than the parabolic lens CRL for bandwidths exceeding 2%. We show this is due to the mitigation of chromatic aberration by spherical aberration, and due to increased absorption of low energy neutrons relative to higher energy neutrons as the lens radius increases. Experimentally the spherical lens CRLs yielded better resolution than was expected with only the chromatic aberration considerations of a broadband thermal source.

The compound refractive neutron lens allows one to create a variety of neutron microscopes that are analogous to their light microscope counterparts. The penetrating ability of neutrons through most materials, and the contrast provided by certain elements such as hydrogen, boron, lithium, and gadolinium provide a basis for imaging with neutrons employing bright field, dark field, and phase contrast microscopy. Thermal neutron microscopy and cold neutron microscopy have a variety of imaging and diagnostic applications in the biological and physical sciences, as well as in medicine and industry.

The Important Role of Standardization in Neutron Imaging

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Neutron imaging is an important nondestructive testing and examination tool that is being utilized to provide information that addresses both industry and national security issues. For the first four years of neutron imaging, neutron radiography with single-coated film and metal conversion screens was considered the gold standard, providing images with excellent spatial resolution. A straightforward qualitative evaluation of the film images

could provide information that could not be obtained by other nondestructive testing techniques. Because the same technique was employed by almost all practitioners, there was good agreement on techniques and terminology. The last decade has seen rapid advances in imaging detectors and image processing hardware and software that have made it possible to rapidly and easily capture and process increasingly large digital neutron images. As a result, neutron imaging is now being used as the preferred technique in many new applications where the collected images are processed to yield quantitative measurements. The growth in applications and techniques in the absence of standards has created an environment in which the end user has no way of determining the beam or imaging system characteristics needed for the application. Further, the lack of uniform terminology and stated facility parameters make the selection of a test facility a hit or miss proposition.

It is important for those using neutron imaging in research, as well as those providing routine nondestructive inspections, to accurately and clearly communicate the capabilities and limitations of this imaging technique to the end users. This presentation presents examples where standardization of terminology, image quality indicators and beam parameters can be invaluable in avoiding misinterpretation of data and improving the user community's understanding of each neutron imaging facility's capabilities. As the neutron imaging community grows and new imaging beams and detectors are put into use, a consistent set of terms and characterization methods will be invaluable to the prospective users who are evaluating the possibilities of utilizing neutron imaging in their research or in a new application.

Session MO3: Challenges and Opportunities

Chair: R. McGreevy, Rutherford Appleton Laboratory

Development of New Neutron Imaging Techniques at ILL

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The high flux neutron imaging facility at ILL is dedicated to time resolved radiography and tomography and precise imaging with high statistics. Recently complementary techniques have been developed in addition:

Off-angular scattered neutron imaging visualizes the scattering cross section only. It complements attenuation images and could provide fundamental new insights. Coherently scattered neutron imaging and scattered neutron tomography are also possible. First millimeter-resolution radiographies and tomographies will be presented. Future improvements in terms of flux and spatial resolution will be discussed.

Neutron scanning microscopy offers superior spatial resolution with minimal background and excellent dynamics. It avoids the inherent resolution limit of scintillators and chip-based detectors and could broaden the range of neutron imaging to new length scales.

Phase and Microstructure Imaging at a Pulsed Neutron Source

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The 'natural' time structure of a pulsed neutron source can be exploited for energy discrimination in a range of imaging applications in engineering and archaeological sciences. Time-of-flight (TOF) applications for imaging phase contents and microstructural features are rather new, and typically of modest spatial resolution in the order of square millimetres. Crystallographic phase mapping utilizes the geometric aberration and corresponding TOF shifts of Bragg peaks [1]. Bragg edge transmission analysis is based on fluctuations of the total neutron cross sections of a material [2]. Bragg edges have their origin in structural properties, hence the recording and imaging of the Bragg edges is synonymous with mapping of phase content, microstructure, residual strain and texture of a material [3]. Recently, first preliminary and pioneering results of direct imaging of

microstructural features have been obtained with a light-intensified CCD camera system on a pulsed source [4]. Energy selection in neutron radiography provides a means for contrast enhancement and contrast variation with high spatial resolution [5]. Moreover, energy scans reveal microstructural variations that are not visible in conventional radiographic images. The potential of mapping the crystallographic texture of a material, by exploiting the Bragg edge transmission properties, is a promising prospect of energy-selective radiography at a pulsed source.

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Prospects for Neutron Probed Magnetic Resonance Imaging

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Magnetic resonance imaging provides a map of the spin lattice relaxation time (T_1) as a function of position in a sample. The traditional way to measure this map is to use Nuclear Magnetic Resonance with a small magnetic field gradient across the sample to provide the positional dependence. Polarized neutrons can also be used to probe nuclear polarization and thus be used to measure T_1 . Specifically the “brute force” method of polarizing the sample (with a large magnetic field or dynamical methods) will be discussed in light of recent high magnetic field technology. The new advances of pulsed fields up to ~ 60 T will allow T_1 measurements in samples at temperatures between 10 and 100 mK where the current limit is between 1 and 10 mK [1]. Alternatively the high magnetic fields of 40T and above would provide a sufficient Zeeman splitting to allow observation of inelastic scattering away from the incoherent line. The prospects of using this splitting in T_1 measurements will be discussed. These measurements provide T_1 at a specific spot on the sample and then an MRI image can be produced by rastering the sample through the beam. Advantages of using neutrons over RF will be discussed as well

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

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Detectors for Imaging at a Pulsed Source

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To take advantage of the Time-of-flight (TOF) technique available at pulsed sources a neutron detection system must record the neutron capture time. A modest time resolution on the order of a few μ s is needed, but the TOF region of interest may exceed 10 ms and current commercial technologies do not meet this requirement. CCD cameras and image plates produce high resolution images but they do not meet the time tagging requirements of a pulsed source. Image plates have no inherent time resolution and cameras cannot support the data transfer rates. Research is underway to develop new high resolution, real time detectors based on semiconductor and micro channel plate technologies and hopefully these will meet the need. In the short term it may be possible to use sampling cameras, like sampling oscilloscopes, to collect the neutron data. This technique would require several seconds to complete a frame scan, but multiple cameras could be used if required.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Neutron Phase Contrast Imaging

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Neutron and X-ray radiographic or tomographic imaging are invaluable tools in materials science and medical diagnostics. Today the majority of imaging applications is based on the attenuation of the radiation inside the object. For some specific applications, however, the imaging of the phase shift induced by the object can provide substantially new and otherwise not accessible information. In the case of x-rays, phase contrast imaging yields an increased contrast for biological samples, and thus is of potential interest for medical applications [1,2]. Neutron phase measurements, on the other hand, have a long and distinguished history in the exploration of the fundamental properties of quantum mechanics [3]. Thus, a combination of phase sensitive measurements with a neutron imaging approach has the potential of providing two- or even three-dimensionally resolved spatial information on the quantum mechanical interactions of neutrons with matter.

In this context, the aim of my presentation is mainly two-fold. Firstly, an overview over the current status, the basic principles, and the limitations of existing neutron phase imaging methods will be given. I will particularly focus on the recent development of grating based shearing interferometer and show how a setup consisting of three transmission gratings can yield quantitative differential neutron phase contrast images [4]. Several examples of applications with respect to neutron phase contrast radiography and neutron phase tomography will be given.

Secondly, I will identify some opportunities for exciting directions of new research particularly in the context of neutron phase imaging which could be addressed with a new imaging facility at the Spallation Neutron Source. Based on the previously shown results, the necessary experimental, technical, and theoretical developments to achieve the proposed goals will be discussed.

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Refraction and USANS – Radiography and Tomography

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Neutron refraction and (ultra) small angle neutron scattering (USANS) can be used as imaging signals for radiography and tomography. In both cases the position dependent changes of the scattering angles ($\sim \mu\text{rad}$, sec of arc), due to interior structures in a sample, are measured and transformed into image projections. The difference between refraction and USANS is based only on the size of the structure with respect to the lateral coherence width of the incident neutron wave. From these projections 2D or 3D images can be reconstructed. To measure these small angles a double crystal diffractometer with different analyzer crystals and detectors is used as a radiography and tomography set up. The method and some examples are given and compared with conventional radiographies and tomographies.

The presented method is one of the most sensitive techniques in radiography and tomography if nearly pure materials have to be investigated. All three interaction parameters – absorption, refraction and small angle scattering - are deduced from the same data set and can be used to reconstruct a complete 3D image. Small defects, clusters of app. hundred nanometer large particles in samples can be identified within a homogeneous matrix. This tomographic technique can be used as the “missing link” between scattering methods and direct imaging methods, between Fourier and real space. With improved spatial resolution ($< 100\mu\text{m}$) one can observe both, real images and Fourier space images depending on which data set is used. However, the limitation of this technique is given by the neutron flux. So it is one of the next tasks to improve the technique and to apply it to a large number of applications in basic and industrial research.

Nuclear Magnetism, Polarized Neutrons, and Imaging

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Dynamically polarized nuclei provide a powerful way for studying nuclear magnetism, molecular structure, as well as new prospects for imaging samples with neutrons. For adiabatic samples with very long nuclear relaxation time, dynamic polarization is the only practical way to obtain very high nuclear polarization of close to one hundred percent. The prospect of imaging samples with dynamically polarized nuclei will be discussed.

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Visualization of Combined Ultra-Small and Small Angle Neutron Scattering (USANS/SANS) data from Hierarchical Structures

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Over the past decade combined ultra-small-angle and small-angle neutron scattering became a powerful advanced tool of supra-molecular structural analysis, which has been successfully tested on many systems and found exclusively effective for studies of so-called hierarchical structures. This progress has been made mostly from the side of instrumentation after successful adoption of the X-ray Bonse-Hart ultra-small-angle technique for neutrons; however, the procedure of fitting experimental data to a theoretical prediction remains traditional so far. The method of mathematical modeling, which is usually applied on polydisperse systems, is significantly restricted by the number of possible analytical models. Besides, it allows calculation of structural parameters for a chosen mathematical model without giving impression about three-dimensional images of structures under study in real space.

An advanced approach in small-angle data processing based on the Monte Carlo modeling has recently been launched for monodisperse systems and three-dimensional real space images were calculated from diffraction data for complexes of biological macromolecules. Unfortunately this imaging technique cannot be directly applied for polydisperse hierarchical structures; however, there are some reasons to believe that real space visualization is possible.

The current presentation contains several attractive results of combined USANS/SANS studies obtained on different materials and should be considered as a proposal for the development of a new data processing technique. This advanced software will generate a computer image of the internal structure based mostly on the neutron diffraction measurements, however, the results of the other experiments and the database of the material under study also should be taken into consideration.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Spin Contrast Imaging

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The use of neutron spin precession in a magnetic field as a high-resolution "timer" has been applied in the neutron spin echo technique. The spin echo technique can measure a change in the energy or angle of the neutrons in neutron scattering experiments. In a similar fashion, we may apply the spin-echo technique to provide a contrast-imaging of an object. In this technique, a polarized neutron beam passes through the object and a magnetic field is applied perpendicular to the neutron spins to induce spin precession. Different materials in the object have different optical potential and result in slightly different flight time of the neutrons. The neutron spins therefore undergo different amount of precessions. By analyzing the neutron polarization, we may re-construct the optical potential and therefore the composition of the object. A previous measurement demonstrated that the main challenge comes from the difference in the optical potential of different materials are small. In this contribution, we will discuss the possibility of realizing the above spin contrast imaging technique using high magnetic field and long-wavelength neutrons.

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Session MO4: Cultural Heritage

Chair: H. Bilheux, Oak Ridge National Laboratory

Imaging Artifacts in the Louvre Museum

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The study of materials from the Cultural Heritage needs advanced techniques to shed new lights on ancient technologies and to help in their preservation. During the conference, I will illustrate current needs and potential contributions of imaging techniques, from the millimeter to the nanometer scales, using large scale facilities such as ion beam analysis at the Louvre, at synchrotron radiation and neutron facilities at Grenoble (France) as well as transmission electron microscopy.

The first example will deal with a study of an ancient hair dye formula. Reconstitution of the chemical process and observation of treated hair cross-sections by high resolution TEM allows us to highlight an early use of nanotechnology during the Greco-roman period. We find that the dye works by causing formation of nanocrystals of lead sulfide inside hair shafts without damaging the hair. The lead sulfide crystals look like the lead sulfide quantum dots synthesized recently using techniques from materials science (1).

The study of Egyptian makeup containers from the Louvre museum gives a second example of development of chemical technologies far more sophisticated than we had previously supposed (2). We have characterized the composition of large series of samples by Synchrotron radiation XRD and XRF and we verified the quality of the sampling and the homogeneity of powders preserved in the containers by neutron diffraction at the ILL (Grenoble, France). Egyptian cosmetic powders held by the Louvre contain ingredients which were so rare they could only have been synthesized to confer therapeutic properties to the eye make-up.

Another challenge is the imaging of paintings with different radiations to understand how the artists had worked. We will illustrate the current use of radiography and infrared imaging with the case of Leonardo's Mona Lisa. These observations and chemical analyses have given new information on the painter practices (3). How did Leonardo prepare his panel, mix his paints, and compose his picture?

(1) Ph. Walter, E. Welcomme, Ph. Hallégot, N. J. Zaluzec, C. Deeb, J. Castaing, P. Veyssière, R. Bréniaux, J.L. Lévêque, G. Tsoucaris (2006) - Evidence for early use of nanotechnology from an ancient hair dyeing formula. *Nano Letter*, Oct. 2006

(2) P. Walter, P. Martinetto, G. Tsoucaris, R. Bréniaux, M.A. Lefebvre, G. Richard, J. Talabot, E. Dooryhée (1999) - Making make-up in Ancient Egypt. *Nature* **397**, pp. 483-484.

(3) Mona Lisa: Inside the painting. Dir. J.P. Mohen, M. Menu, B. Mottin, HNA Books, Sept. 2006, 117p.

Session TU1: Cultural Heritage

Chair: C. Andreani, Università degli Studi di Roma Tor Vergata

Imaging with Epithermal Neutrons for Cultural Heritage Research

G. Gorini

Università degli Studi di Milano-Bicocca,
On behalf of the Ancient Charm collaboration

A large variety of chemical, physical and microstructural techniques are employed to characterize objects of cultural significance. Most of these methods are invasive and probes like X-rays and charged particles have limited penetration power in matter. Neutrons, on the other hand, can penetrate thick layers without substantial scattering or absorption depending on their energy. While the potential of neutron-based techniques is large, their development is recent in most cases. Especially, no attempt has been made to use the unique resonance absorption properties of epithermal neutrons for quantitative 3D imaging.

Many elements have neutron absorption resonances in the epithermal energy range. Neutron absorption is followed by the prompt emission of a gamma-ray cascade, and both the gamma emission and the neutron transmission can be measured. Spatially resolved information can in principle be obtained by a combination of tight neutron beam collimation, multiple positioning of the sample, and simultaneous measurement of neutron resonances with different strengths and therefore able to effectively probe depth.

Developing the “Neutron Resonant Capture Imaging” combined with “Neutron Resonance Transmission” (NRCI/NRT) as a non-invasive technique for 3D tomographic imaging and its use in cultural heritage research is the ultimate aim of the ANCIENT CHARM project. The idea of developing an imaging technique based on epithermal neutron absorption is totally new and presents a number of scientific and technical challenges which are best addressed by the joint development of two related 3D imaging methods: Prompt Gamma Activation Imaging combined with cold Neutron Tomography (PGAI/NT) and Neutron Diffraction Tomography (NDT). The three new imaging methods will provide a new, comprehensive neutron-based imaging approach, which will be applied for the 3D imaging of elemental and phase composition of objects selected as a result of a broad scope archaeological research.

Thermal Neutron Tomography: Archaeological And Geological Investigations

F. Andreoli, M. Palomba, R. Rosa

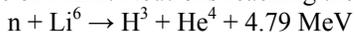
TRIGA Research Reactor, ENEA Casaccia

Via Anguillarese, 301 – 00060 S. Maria di Galeria – Rome – Italy

At the ENEA TRIGA research reactor (Casaccia Research Center, Rome), two equipments are available for neutron radiography applications, both including radiographic image digitalization systems based on a CCD camera and rotating units for tomography applications.

The equipments are operating at the two thermal neutron collimators existing at ground level of the reactor room: different outlet diameters, L/d and Cd ratios characterize the two positions.

After the interaction with the sample, neutrons are converted by a commercial LiF, ZnS (Ag) scintillator, with 96% as Li⁶ enrichment. The scintillating mixture, with a thickness of 0.4 mm, is layered on a aluminium plate of 1 mm. Neutrons reaching the scintillator react with the Li⁶ nuclei:



Tritons excite the luminescent levels on ZnS (Ag) emitting visible photons at 450 nm wavelength.

The image acquisition is performed with two different usual assemblies of mirror, lenses and CCD connected to personal computers located in the reactor room.

A Client-Server connection permits the effective acquisition of the tomographic images via Internet, allowing the complete control of the tomography by the authorized user. Museum and research institutions can have a “virtual” access to neutron tomography facilities, having their owned objects under investigation well protected in a “nuclear grade” patrolled secure location. Each institution will have the choice of using the available back-projection reconstruction software package (with output raw data sets allowing the use of commercial visualization software packages) or testing the algorithms of their personal reconstruction software.

Examples of 3-D reconstructions with virtual sections of archaeological and geological samples will be demonstrated.

The Study Of Roman Artifacts by Neutron and X-Ray Imaging Methods in Switzerland

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Many objects of archaeological relevance found in Switzerland are from Roman origin. Their uniqueness means, in most cases, any investigation of samples must be performed non-destructively.

Transmission experiments performed either with X-rays or neutrons, depending on the structure and size of the objects, can help to identify inner structures, composition, defects or the manufacture process. Furthermore, in some cases the treatment by conservators and restorers also becomes visible.

This report describes some examples from such investigations, pointing the archaeologist in the direction of which conclusions can be drawn from such experiments whilst highlighting the problems in interpretation which still exist. In the case of the neutron investigations, besides the transmission imaging as a radiograph the three-dimensional structure was observed by a tomographic technique. For of X-ray radiography, the images were obtained in the same digital format when a similar experimental method (imaging plates) was applied.

It becomes evident in the described examples that the combination and complementary use of both methods (neutrons and X-ray) brings insights in different aspects of the sample's properties and treatment. Whereas neutrons can penetrate objects of copper alloy much better than X-rays and organic traces made visible, the X-ray methods can favourably be applied to investigate larger iron and wooden objects.

The Swiss view can be extrapolated to other countries where these techniques are also simultaneously available in order to investigate other objects of relevance. The European network COST-G8 entitled "Non-destructive analysis and testing of museum objects" has been a very good network to support initiatives in this direction.

Session TU2: Medical and Biomedical

Chair: D. Myles, Oak Ridge National Laboratory

Neutron Stimulated Emission Computed Tomography

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Neutron spectroscopy is being used as a non-invasive technique to determine element concentration in biological tissue. Several disorders in humans are characterized by changes in trace element concentration in the affected tissue, e.g. breast cancer (Al, Br), prostate cancer (Ca, Cu), and liver iron overload (Fe). The preferred method of diagnosis for these disorders is invasive biopsy, which is an unpleasant procedure and has a 20% margin of error in determining element concentration. Neutron Stimulated Emission Computed Tomography (NSECT) uses a beam of fast neutrons to non-invasively determine the elemental composition of biological tissue in-vivo, and create two-dimensional tomographic images of element distribution through a single non-invasive tomographic scan. Principle: An incoming neutron scatters inelastically with a target atomic nucleus to excite it to a higher energy level that is often unstable. The excited nucleus then rapidly decays to its ground state, emitting a gamma photon whose energy is equal to the difference of the two nuclear energy levels. These energy level differences are unique to most elements and isotopes. The energy of the gamma photon is measured by an energy-sensitive detector, which allows identification of the emitting nucleus. NSECT experiments are currently performed using a Van-de-Graaf accelerator. This tunable neutron source is unable to provide narrow collimated beams with high neutron flux. While high flux wide beams are adequate for spectroscopy, narrow collimated beams are required for high-resolution tomographic imaging. The loss in flux translates to impossibly long scan-times that increase patient dose and reduce SNR by introducing more time-dependent background noise. A high flux collimated neutron source such as the SNS has the potential to overcome these limitations by providing narrow collimated neutron beams with high flux, allowing acquisition of high-resolution tomographic images in reasonable scan-times with better SNR and lower patient dose. In addition, a better-focused energy range would allow targeting specific elements and energy states. NSECT shows great potential in developing into a portable screening modality with the use of high-flux portable neutron sources and portable gamma detectors. Our preliminary data suggests that it may be possible to determine liver element concentration with less than 15% margin of error. Efforts are being made to improve the sensitivity of the system to allow trace element measurement through time-of-flight background reduction and attenuation correction. Our final goal is to implement a low-dose portable scanning system that allows a non-invasive diagnostic alternative for cancer and other disorders.

Microscopic Boron Imaging in Tissue Sections using High Resolution Quantitative Autoradiography

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Boron Neutron Capture Therapy (BNCT) is being investigated worldwide as a promising form of therapy for treating certain refractory types of cancer that presently have no cure. BNCT relies on selective delivery of the ¹⁰B isotope to tumor cells followed by neutron irradiation. The ensuing nuclear capture reactions release ⁴He and ⁷Li nuclei that deposit energy over a distance of approximately 10 μm. The short range of these reaction products means that BNCT can selectively sterilize tumor cells on a microscopic scale while sparing adjacent healthy tissue. Achieving efficacy with BNCT is critically dependent upon preferentially delivering sufficient amounts of boron to cells actively growing within the main tumor mass as well as those that may be infiltrating normal tissue or within poorly perfused parts of the lesion that are necrotic or hypoxic. Moreover, the neutron capture reaction products can be much more effective if boron is bound intracellularly, especially within the nucleus.

Developing and optimizing boron-containing agents to target tumors is now a major emphasis in BNCT research and these studies require analytical techniques for determining boron uptake in various biological samples. Tissue and tumor biopsies, however, inevitably contain a heterogeneous population of different cell types and a macroscopic analysis of these samples yields an average boron concentration that does not contain critically important information about the microscopic distribution. Consequently, these analyses often cannot be used to accurately represent biological effects from the short-range reaction products. High Resolution Quantitative Autoradiography (HRQAR) is a sophisticated technique that superimposes boron neutron capture reactions registered in a thin-film polymer track detector over stained tissue sections of the corresponding pathology. Boron containing tissue sections mounted on polymer-coated slides are packed in dry ice and irradiated in a thermal neutron beam port of the MIT Research Reactor to a total neutron fluence of approximately 10¹³ n cm⁻². Charged particle tracks are revealed using chemical etching and digital light microscopy followed by image analysis and Monte Carlo simulations to identify tracks and derive quantitative information, such as the boron concentration or microdosimetric quantities. These analyses can be carried out within certain cell populations to assess the uniformity and selectivity of compounds on a microscopic scale. HRQAR studies can be performed to evaluate boron compounds in advance of tumor therapy trials involving animal or humans that are generally costly and time-consuming. An overview of this technique is provided together with examples of results obtained from applied studies.

Multimodal Contrast Agents

Kenneth L Watkin

University of Illinois at Urbana, Champaign, Illinois

Recent advances in bioimaging contrast agents include the development of nano- particles for use as multimodal contrast agents that also have the potential to act as functionalized cancer treatment agents. In this presentation several new bioimaging contrast agents created for drug and gene delivery will be explored along with our recently reported dextran gadolinium oxide nanoparticle for MRI imaging and neutron capture therapy.

Imaging as Biomarker: Standards for Change Measurements in Therapy – Workshop Summary and Opportunities for Neutron Imaging

M. Vannier

University of Chicago, Chicago, Illinois

A workshop on imaging as a biomarker sponsored by NIST, NIH, and the FDA with participation by the pharmaceutical industry, professional imaging scientific societies, and academia was held on 14–15 September 2006 at the National Institute of Standards and Technology in Gaithersburg, Maryland.

Imaging as a biomarker of drug response is becoming an increasingly important field of research. The Food and Drug Administration (FDA), the National Cancer Institute (NCI), and the Centers for Medicare and Medicaid Services (CMS) have agreed to collaborate on improving the development of cancer therapies and outcomes for cancer patients through biomarker development and evaluation. A similar effort across the National Institutes of Health's (NIH's) Institutes and Centers (ICs) is being planned. Biomarkers are biological indicators of disease or therapeutic effects that can be measured by in vivo biomedical imaging and molecular imaging in particular, as well as other in vitro or laboratory methods. Recent work has shown that biomedical imaging can provide an early indication of drug response by use of X-ray, CT or PET-CT.

Neutron imaging has significant potential for biomedicine, especially as a biomarker in oncologic and vascular applications. In general, the technology for neutron imaging is less mature and many opportunities have not yet been explored.

Many sources of uncertainty exist in imaging as a biomarker. Biological variability, for example, is a factor that is both drug- and patient-dependent and thus difficult to characterize or model. However, other uncertainties are associated with the image data collection platform and the robustness of software tools required for reliable, quantitative measurement of change over time, such as tumor volume, radioactive tracer activity, or contrast agent dynamics. All these sources of uncertainty significantly affect the statistical power of clinical drug or therapy trials. Measurement of change over time with imaging for a variety of disease models will be presented.

The development of standards for image quality control, image data collection, and benchmarking of change analysis software tools, as well as image-specific statistical methods, could significantly reduce the size of clinical trials for drug response. The cost of drug development and submission to the FDA by the pharmaceutical industry may soon exceed \$1 billion. Standardized imaging methods may reduce these costs.

The scope of this workshop was focused on the need to standardize imaging methods for data collection and data analysis in the context of drug or radiation therapy trials. Topics for breakout discussions include:

- *Instrument quality control over the time sequence of a trial (modalities include: X-Ray, X-Ray CT, PET, PET CT, MRI, MRS, DCE and Diffusion MRI. Emerging modalities will also be discussed)

- *Harmonization of data collection across different commercial imaging platforms

- *Creation of standardized, objective performance metrics for image-analysis software using reference image databases or test beds

- *Standardized statistical methods for change measurement

- *Archival and access methods for image storage, related meta-data, and clinical outcome data

- *Innovative methodologies for the integration of image and other data for clinical decision making.

A summary of the workshop presentations and recommendations was formulated to help identify the areas of need and opportunity for application of neutron imaging technology as a biomarker in drug development.

State of the Art and Limitations in MRI and Optical Microscopy

W. Warren

Duke University

Modern magnetic resonance and optical imaging applications are undergoing a technological revolution due to recent developments in molecular and laser physics. In the MR case, new methods for creating "hyperpolarized" reagents (with 10,000 times thermal nuclear polarization), even in large molecules, are creating an entirely new class of contrast agents. These agents are particularly effective when probed by pulse sequences that exploit nonlinear evolution. In the optical case, femtosecond laser pulses can now be "shaped" in time (frequency and amplitude modulation) and in space, and can even have complex time-evolving polarization. While the initial goals of this work were largely to improve high-speed communication or control of chemical reactions, it is likely the most practical application will instead be to imaging, specifically of highly scattering

tissue. Our lab has shown that pulse shaping lets us extract useful optical signatures from a variety of molecular targets in tissue (for example, oxy- and deoxyhemoglobin, cytochrome c, and melanin). These signatures fall into two broad categories: nonlinear absorptions (which, for example, let us distinguish between eumelanin and pheomelanin) and nonlinear phase modulations (which highlight molecular structure while suppressing scattering). We will demonstrate applications of shaped pulses to image neurons fire, to explore deep tissue structure, and to characterize tissue composition.

Session TU3: Engineering

Chair: C. Hubbard, Oak Ridge National Laboratory

Imaging Single Grains in Polycrystalline Materials

John Root and Ron Rogge

National Research Council of Canada, Chalk River Laboratories

Because neutrons penetrate easily into most engineering materials, neutron diffraction contributes important knowledge in the domain of materials science, ranging from fundamental understanding to practical applications in material production or performance. While most other probes, such as X-ray diffraction, SEM, TEM, or laser interferometry reveal stresses and textures at the surfaces of materials, neutron diffraction provides truly representative sampling of bulk materials and can map stresses as a function of location inside bulk materials or components.

Typically, neutron diffraction measurements are made in situations where the sampling volume is much larger than the average crystallite size, so that tens of thousands of grains are sampled to give a reliable bulk-average result. "Images" of stress distributions throughout a specimen are often made by locating a small sampling volume at a selection of points. At each point, the sampling volume contains 10,000+ grains from which the "bulk-average", macroscopic stress is determined at that location.

The challenges begin when either (a) the typical grain dimension falls in the range such that the number of grains contributing to a diffraction peak is insufficient to assume uniform scattering intensity throughout the sampling volume or (b) it is desirable to understand the effects of one grain on another, or a second phase on a matrix. The way forward requires the development of instruments and methods that can deliver a sub-millimeter neutron beam with sufficient intensity to enable single-grain analysis of orientation and lattice parameter, then to scan this beam from one grain to the next. To add value to single-grain measurements at surfaces, which are already accessible by electron microscopy, the neutron microprobe must be able to capitalize on the penetrating capability of neutrons, beyond what may be possible with synchrotron X-rays, and reveal new insights about single grains in their three-dimensional environments.

In this presentation, we shall set the stage for discussing the feasibility of applying neutron beams to image single grains in polycrystalline materials, considering methods with monochromatic or full-spectrum beams, spatial resolution, and positioning in three dimensions.

Complex Multi-Phase Flow Visualization and Implications to Metal Casting using Neutrons

Dayakar Penumadu

The University of Tennessee, Knoxville, TN 37996

The application of dynamic neutron radiography for complex multi-phase flow visualization problems will be demonstrated for an application involving its use for lost foam casting (LFC) process. Lost Foam Casting (LFC), also known as Expandable Polystyrene Casting (EPC), is being adopted as a replacement process to the conventional metal casting techniques (sand and investment castings) based on its ability to produce metal castings with zero degree draft on locators, near-net-shape, reduced environmental waste, and simplified production techniques (little downstream machining and assembly). Many metal casting companies, such as General Motors, Mercury Marine, Saturn, and Robinson Foundry, are applying LFC for engine block and cylinder head castings with complex shapes to improve product quality with cost savings. Lost foam castings are

produced by pouring molten metal into an Expandable Polystyrene (EPS) foam pattern, which is surrounded by unbonded sand and compacted through vibration. In order to obtain controlled pyrolysis of EPS foam and smooth casting surface, the foam pattern is coated with refractory slurry. The EPS foam pattern degrades into liquid and gas products and escapes into the loose sand after the molten metal is introduced, and the metal then fills the foam pattern to yield the target casting. This is a relatively new technology and requires improved fundamental knowledge for large-scale implementation in foundry areas and provides an excellent opportunity for advanced basic research using neutron imaging techniques. One of the important aspects is to identify the molten metal and pyrolysis fronts during the LFC process, because the behavior of those fronts has determinate effect on the quality of castings. Results have shown that many casting defects are closely related to how well the decomposed polymer foam escapes, and the complex behavior of the pyrolysis front during the LFC process. Using digital image analysis and neutron radiographs, a fundamental multi-phase model and pyrolysis mechanism is being developed that seems to explain well the observations using metal pour for simple geometries of castings.

A significant advancement required for further research in this field is to improve the spatial resolution to sub-micron by still having a macroscopic view area with a diameter in the order of 10 to 15 centimeters, and time of exposure reduced to few milliseconds with acceptable signal to noise ratio. An exciting challenge in this field is to have energy selective imaging in three dimensions to visualize multi-phase flow with high spatial resolution for short exposure times.

Stroboscopic and Continuous Neutron Imaging at FRM II

B. Schillinger

Technische Universität München

The standard detection system employed at the ANTARES neutron imaging facility requires exposure times of a few seconds. For periodic processes, time resolution can be drastically improved by using a stroboscopic technique: A cooled CCD camera is coupled to a gate-able image intensifier that serves as a fast shutter. Identical time windows of the repetitive process are integrated on the CCD chip until a sufficient photon fluence is achieved, then the image is read out. By shifting the time window with a triggered delay generator, the whole period of the process can be recorded. This is demonstrated by several measurements of an electrically driven combustion engine, where the oil flux and oil cooling was made visible at rotation speed between 600 and 1000 rpm. The technique can also be employed to select energy windows in a time-of flight setup at a pulsed source.

A brief outlook is given about measurements with a high-speed camera capable of recording several thousand frames per second for continuous imaging.

Session TU4: Materials Research, Energy, Fuel Cells, and Aerospace

Chair: D. Penumadu, The University of Tennessee

The Experimental Program for Neutron Imaging at PSI (Switzerland)

Eberhard H. Lehmann, Peter Vontobel, Guido Kühne, Gabriel Frei and George Necola
Paul Scherrer Institut, CH-5232 Villigen PSI

PSI operates its spallation neutron source SINQ with success since 1997. With an averaged proton beam intensity of about 1 MW it is still world's strongest spallation source, corresponding to a research reactor of about 10 MW.

Neutron imaging has been performed at the facility NEUTRA just since the beginning of the beam supply in 1997. About 100 different experimental tasks with duration between few minutes and about one week take place per year for scientific and applied purposes. The users are from many countries round the world.

The overload of NEUTRA and the search for improved imaging conditions were the reason to design and to build a second facility – ICON. Its performance will be presented in a separate talk by G. Frei. Now it is possible to decide which conditions are preferably for a given problem in respect to the neutron spectrum (NEUTRA=thermal, ICON=cold).

Another feature at NEUTRA is the simultaneous use of X-rays (up to 320 keV) for referencing using a tube type source with the same divergence than the neutrons and the same detection system.

The progress in the imaging techniques with neutrons is the main reason why complete new approaches became reality. Only with the help of high-performance digital systems it was possible to realize neutron tomography, time-dependent investigations, phase-contrast enhancement and energy selective measurements. Further improvements at the beam line layout but also on the detector side will increase the application potential of neutron imaging options.

The interaction with industrial partners has been done mainly for non-destructive testing of components and for the intensive study of the performance of electric fuel cells. The talk will contain examples to show the performance in respect to the industrial needs. Further activities are concentrated onto the study of processes in time-dependency: running engines, infiltration processes, speedy tomography of sequences .

The experience in the collaboration with industrial partners from the last decade has shown, that a demonstration of the performance of the methods has to be done first before the customers will finally enter. Therefore, a dedicated beam line at SNS should exploit its special options at a pulsed source but also use the experiences from the most successful facilities.

Neutron Imaging for Fuel Cell Research

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The National Institute of Standard and Technology (NIST) has developed neutron imaging metrology, experimental facility, and research support infrastructure to address key technical barriers that are adversely affecting the rapid development of robust, efficient, and commercially viable low temperature hydrogen fuel cells. Lack of proper understanding of the *in situ* water transport inside an operating fuel cell is one such key technical barrier. Since Neutrons are highly sensitive to the presence light elements such as hydrogen, neutron imaging has emerged as one of the most effective tools to map and quantify water distribution in fuel cells. NIST has constructed and operates an advance thermal neutron imaging facility at the NIST Center for Neutron Research (NCNR) that is being utilized extensively by major automotive companies, fuel cell manufacturers, national laboratories, and researchers from academic institutions for fuel cell and hydrogen storage related research.

Neutron imaging technique (both thermal and cold) should not necessarily be limited to hydrogen economy related research but the technique has immense potential for broad and important uses in many other areas, from soft tissue to nuclear security related research. However, significant improvements in temporal and spatial resolution are needed before such potentials can be realized.

In this presentation, we will present an overview of the NIST facility, technical and scientific research efforts and challenges, and mode of operation for seamless integration of research efforts with industrial and academic partners.

Optics and Techniques for Diffraction and Contrast Imaging

Gene E. Ice

Oak Ridge National Laboratory*, Oak Ridge, Tennessee

Three-dimensional (3D) spatially-resolved diffraction imaging is an emerging research direction with the potential to revolutionize our understanding of the underlying basis for materials properties and dynamics. Although x-ray imaging methods on intense synchrotron sources can provide high-resolution 3D (tomographic) images of local crystal structure and defect distributions, analogous neutron techniques offer major advantages, particularly with spallation sources like the SNS. For example, neutron scattering can have far lower damage and good scattering cross sections for organic crystalline materials. This will make it possible to study the evolution of polymeric materials in-situ to understand and develop new processing techniques. Neutrons can also penetrate deeply into even high Z materials with good scattering at high momentum transfer from even low Z materials. This will open up the study of local stress and defect distributions deep within high Z large-grained materials and will allow for studies of low Z precipitate properties inside high Z samples. Because pulsed neutron diffraction provides neutron wavelength as well as scattering direction, data collected will include direct reciprocal space

maps with much better signal-to-noise than associated with Laue x-ray microbeam measurements. In addition, good neutron scattering at high momentum transfer will allow for ultra-precision measurements of elastic strain and local crystallographic rotations. These factors together with the large neutron magnetic scattering cross sections will allow for first-of-their kind studies of 3D magnetic domain evolution in materials.

In order to accelerate data collection and to achieve good strain *and simultaneously* good spatial resolution, new optics and approaches are needed. We discuss the technical challenges and scientific opportunities associated with three imaging approaches: (1) projection-magnified diffraction tomography; (2) objective-magnified diffraction tomography; and (3) differential aperture polychromatic micro-diffraction. Spatial resolution can be achieved in all three cases either by the use of small beams, by beam-defining apertures/virtual apertures, or by the use of high-resolution detectors. Some strategies to achieve small neutron beams and to improve the effective resolution of neutron area detectors in the context of imaging will be discussed. In addition to diffraction imaging, the techniques described above have possible applications to phase contrast imaging.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Session TU5: Homeland Security and Contraband Detection

Chair: J. D. White, Oak Ridge National Laboratory

Prototype Photonuclear Inspection Technology—An Integrated Systems Approach

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Active interrogation technologies are being pursued in order to address many of today's challenging inspection requirements related to both nuclear and non-nuclear material detection. The Idaho National Laboratory, along with the Los Alamos National Laboratory and the Idaho State University's Idaho Accelerator Center, continue to develop electron accelerator-based, photonuclear inspection technologies for the detection of shielded nuclear material within air-, rail-, and especially, maritime-cargo containers. This paper presents an overview and status of the prototype Pulsed Photonuclear Assessment (PPA) inspection system and its ability to detect shielded nuclear material by focusing on the integration of three major detection system components: delayed neutron measurement, delayed gamma-ray measurements, and a transmission, gray-scale mapping for shield material detection. Areas of future development and advancement within each detection component will be presented.

*Supported in part by the Department of Homeland Security under DOE-ID Contract Number DE-AC07-99ID13727.

Analysis Of Photon Interrogation Of Nuclear Materials For Nonproliferation Applications

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This work presents the results of ORNL simulations to reproduce time-of-flight data taken at the Idaho Accelerator Center (IAC) during photon interrogation of a depleted uranium target. The simulations were performed using the MCNPX/MCNP-PoliMi code system, which calculates the full statistics of the neutron/photon field generated from photonuclear reactions occurring during material interrogation with high-

energy photons. The results are in very good agreement with the measured data for interrogation of a depleted uranium target with 15 MeV endpoint bremsstrahlung photons for two different detector separation distances. Further simulations included a second stop-detector to enable correlation measurements. Correlation functions were obtained for both the depleted uranium target and a natural lead target, which has a photonuclear neutron production cross-section via a (γ , n) reaction that peaks at approximately the same energy as that for uranium. These reactions also have associated gamma-rays and thus can result in a similar detector response to interrogation of fissile material. However, it has been shown that the lack of multi-neutron producing reactions in lead leads to a dramatically different correlated-detector response when appropriate linear accelerator time structures are used. Our evaluation of the occurrence of accidental pairs of counts indicates that correlation measurements should be performed with an extremely low number of photons emitted per pulse. Preliminary results indicate that a large number of pulses are needed to obtain a meaningful ratio of real counts to accidental counts. This fact could lead to long measurement times. However, the measurement would become feasible with current and expected improvements in accelerator and detector technology. The response of benign materials to photon interrogation is of interest in the scanning of cargo containers or other shipping containers. This research has application in the areas of nuclear nonproliferation and homeland security.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Fast Neutron Resonance Radiography for Detection of Explosives and Contraband

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The central problem in the use of non-intrusive techniques for the detection of explosives or contraband is to devise a reliable method for determining both the composition and the shape of materials in a container. X-ray techniques based on either simple transmission measurements or on high-speed computerized tomography do not detect explosives; rather, they make it possible to distinguish volumes of different apparent densities and attempt to use this and shape to generate a detection signature. Unfortunately, while it is true that many explosives have e.g., a density of ~ 1.5 g/cc, it is not true that all materials with density ~ 1.5 are explosives. The result, both from the point of view of physics and practical experience in airports is that such systems are plagued by high false alarm rates.

We have developed a new method for detecting materials, based on Neutron Resonance Radiography (NRR). Element specific resonances in total neutron attenuation cross-sections, which are in the 1 to 8 MeV range, are exploited to enhance the contrast for imaging elements such as carbon, oxygen and nitrogen and others. This contrast enhancement mechanism is then used to produce elementally resolved images of objects under inspection and thus to identify the material composition of the object. Such an imaging technique has immediate application for homeland security but other applications in such areas as online analysis of minerals are also under investigation.

Fast neutron radiography requires the development of compact intense bright fast neutron sources in the energy range from 1 to 8 MeV. Currently small accelerators such as RFQs are used with high pressure gas targets but there are significant limitations on accelerator and target technology which limit performance. Further, the efficient detection and imaging of fast neutrons remains a significant problem, especially considering both spatial resolution and contamination due to gammas. For reliable imaging, a gamma-blind highly efficient fast neutron detector with \sim mm resolution will be required.

Flash Proton Radiography

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Los Alamos National Laboratory has used high energy protons as a probe in flash radiography for a decade. In this time the proton radiography project has used 800 MeV protons, provided by the LANSCE accelerator facility at LANL, to diagnose over two-hundred dynamic experiments in support of national and international weapons science and stockpile stewardship programs. In addition, 24 GeV protons, provided by the Alternating Gradient Synchrotron at Brookhaven National Laboratory, have been used to study the capability of proton radiography at higher beam energies. Through this effort significant experience has been gained in using

proton radiography as a technique to diagnose static and dynamic systems. The results of this experience will be presented by describing the spatial, temporal and density resolution capabilities and limitations of 800 MeV proton radiography.

Session WE1: Molecular and Cellular Biology

Chair: M. Vannier, University of Chicago

Whole Cell Imaging and Protein Localization in Biology and Biomedical Research

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Since the invention of the microscope, imaging has been a key technique in both biological and biomedical research. Over the years, a number of advances in technology have improved the spatial resolution at which cells can be imaged. However, considerable challenges still remain. This talk will discuss these challenges, and highlight them in the context of soft x-ray tomography, a relatively new technique for imaging sub-cellular architecture and the location of labeled protein in whole, hydrated cell. X-ray tomography produces images with unique information content, and provides us with insights that couldn't be obtained with the use of more conventional light and electron based microscopies. The current status of XM-2, the first x-ray microscope in the world to be designed and built specifically for biological imaging will be presented.

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X-ray Tomography Generates 3-D Reconstructions of the Yeast *Saccharomyces cerevisiae* at 60-nm Resolution: C. A. Larabell & M.A. Le Gros. *Molecular Biology of the Cell*. (2004) **15**, 957-962.

X-ray Imaging at the Advanced Photon Source and Applications in Biology

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X-ray imaging and microscopy is a major part of the user science program at the Advanced Photon Source (APS). Techniques that are supported by the X-ray Microscopy and Imaging Group at the APS include scanning x-ray microscopy, full-field imaging, 3D tomography, and coherent diffraction imaging. Several new beamlines have been proposed that include a bionanoprobe beamline for dedicated trace element imaging in biological cells and tissues, a high-throughput x-ray flow cytometry beamline, and dedicated beamline for coherent diffraction imaging. A variety of scientific problems are being investigated by beamline staff and users, ranging from nanoparticle transfection in biological cells with specific DNA targets to real-time phase contrast imaging of small animals for studies of internal physiology. This talk will provide an overview of examples of these bio-scientific studies with an outlook into the future based on developments of emerging and improved techniques that will enhance biological imaging at both subcellular/molecular and organism/medical levels.

This work was supported by the U.S. Department of Energy under contract No. W-31-109-ENG-38.

Prospective Applications of Neutron Holography in Biology

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Atomic resolution holography can be traced to Bragg's x-ray work and Gabor's electron interference microscope. Over the past decade, or so, there have been an increasing number of publications on atomic resolution holography using either electrons or hard x-rays. More recently, Sur et al.,¹ experimentally demonstrated the feasibility of atomic resolution thermal neutron holography via the inside source concept using a single crystal containing one S-wave incoherent scatterer (i.e., H atom, $\sigma_i = 80$ b) per unit cell. Subsequently, neutron holography using the inside detector concept was carried out by detecting neutron capture-gamma rays from lead nuclei in a Pb(Cd) single crystal.² Subsequently, Sur et al.,³ developed a kinematical formulation for the diffraction pattern of monochromatic plane waves scattering from a mixed incoherent and coherent scattering length distribution. The formulation demonstrates the conditions under which one can reconstruct thermal neutron holographic data from samples with either a single incoherent scatterer (e.g. H) per unit cell, or with numerous incoherent scatterers per unit cell, as is the case for biologically relevant and polymeric materials.

Biomimetic (e.g., lipids and proteins) and polymeric materials are typically rich in hydrogen and other low-Z atoms. Although membrane associated proteins are known to constitute approximately one third of all known proteins, fewer than 100 membrane protein structures have been deposited with the Research Collaboratory for Structural Bioinformatics protein data bank. The primary reason for this scarcity of membrane protein structures is the lack of high quality single crystals, a requirement for traditional scattering techniques. However, for atomic structure holography the only requirement is that the sample possess orientational order, translational order is not a necessary condition. Thus, commonly available "poor" quality protein crystals can potentially be solved, to atomic resolution, using holographic techniques.

Thermal neutron holograms obtained using a variety of samples and different (i.e., internal source and internal detector modes) holographic techniques will be presented. The seminar will also summarize the present status of neutron holography, and the key obstacles that need to be overcome in order to impact the biological sciences.

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Isotopic and Nuclear Spin Contrast

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The substitution of hydrogen ¹H by its heavier isotope, ²H (Deuterium) is most widely used in neutron scattering from hydrogenous materials. The applications in polymer science and structural biology are numerous. The polarization of the nuclear spin of the hydrogen spins opens a new dimension of polarized neutron scattering. The length b of coherent varies with nuclear polarization P

$$b(\text{H}) = (-0.374 \pm 1.456 P(\text{H})) 10^{-12} \text{ cm}$$

$$b(\text{D}) = (+0.66 \pm 0.28 P(\text{D})) 10^{-12} \text{ cm}$$

The minus sign applies, if the direction of neutron polarization is opposite to that of the nuclear spins. The neutron beam is assumed to be completely polarized.

The polarization of protons not only mimics deuteration. It also amplifies the range of contrast by a factor two, at least, with respect to deuteration. There is no incoherent scattering when the direction of the proton polarization coincides with the direction of the neutron spin polarization.

Massive deuteration, as it is required for neutron diffraction from protein crystals can be avoided by high proton polarization. The contrast from specifically deuterated regions of complex macromolecules or other subcellular systems is boosted considerably by proton polarization. Systematic errors are minimized, as proton

spin contrast variation is performed with one and the same sample. The in situ structure of a region which comprises less than 1 percent of a macromolecule (e.g., a mRNA fragment of 40 nucleotides, bound to the ribosome) can be retrieved.

A new field of applications comes from the method of dynamic nuclear spin polarization (DNP). Nuclear spins are polarized by microwave irradiation in the presence of paramagnetic centers, e.g. radicals. Polarized proton spin domains around radicals built up at the onset of DNP provide an additional scattering amplitude which is at least one order of magnitude stronger than that of magnetic neutron scattering. A radical density map is obtained by time-resolved polarized neutron scattering. The role of radicals is important in many fields of research from polymer chemistry to the biochemistry of cell death.

DNP is directional, at least in its first step of electron spin nuclear spin interaction. If the direction of the external magnetic field differs from that of the neutron flight direction, an asymmetry of neutron scattering from randomly oriented macromolecules will result. Neutron scattering meets NMR.

Session WE2: Physics, Chemistry, and Geology

Chair: L. Butler, Louisiana State University

Advances in Time-Resolved X-ray Detectors for Synchrotron Applications

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A novel photon-counting imager is described which is being developed for time-resolved experiments at synchrotron beamlines. By exploiting advances in microgap gas detector technology, together with an advanced ASIC-based readout, counting rates of up to 60 MCPS have been demonstrated with frame times as short as 1 microsecond and a spatial resolution of 50 microns. The design of the detector is described and test data are presented. The potential application of the technology to neutron detection is discussed.

Real Time Measurement and Imaging of Water and Elements in a Living Plant

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The University of Tokyo

Plants require only ions in water as nutrients to support their physiological activity. In the case of ions, imaging method has been drastically developed using various fluorescent probes. However, behavior of water itself has not been well known, for lacking tools for the research. To image water in a living plant, neutron beam can provide water distribution with high resolution. Neutrons enable to image roots imbedded in soil as well as fine structure of flowers or seeds nondestructively. Using a CCD camera and a sample rotating system, spatial water distribution can be visualized for analysis. However, neutron images provide only static distribution of water.

To know water movement in a plant sample, isotopes are preferable to use for nondestructive analysis. The application of positron emission tomography is one of the candidates. When water was irradiated with helium beam, ¹⁸F, which half-life is 110 min. is produced from ¹⁶O in water. To some extent, ¹⁸F, a positron emitter, can trace water movement in a plant. Generally, half-lives of positron emitters are very short, after a while, most of the radioactivity decays out and enables to repeat the same experiment using the same sample, which is a great advantage to eliminate errors in analysis due to using the different samples. However, in the case of thin tissue, such as leaves, positrons are easy to escape, therefore, we could obtain mainly stem images rather than those of leaves in a plant sample. The resolution of the image by positron emitters is in mm order.

To analyze water movement quantitatively, we targeted a soybean stem, 1cm in length, and measured the uptake amount of ¹⁵O water, which was produced by ¹⁴N(d, n)¹⁵O reaction. We found that tremendous amount of ¹⁵O water was constantly leaking out from xylem vessels, which were regarded as mere pipes to transport water, and re-entered to the vessels. Since the half-life of ¹⁵O is only 2 min., we could perform the measurement for

about 20 min. Through simulation, half of the water existed in the stem was renewed by freshly absorbed water in about 20 min.

Since beta-emitting isotopes provide higher resolution of the image than those of positron emitters, we constructed real-time imaging system for conventional isotopes, such as ^{14}C , ^{45}Ca or ^{32}P . The sensitivity of this imaging method was more than 10 times higher than that of an imaging plate for ^{45}Ca , with similar resolution.

Fingerprinting Marbles by Combining Neutron Diffraction and Imaging

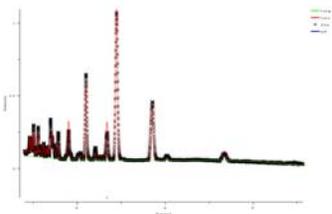
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The use of neutron tomography (NT) for archaeometric purposes is quite recent, with the first report dating back to 1996. NT is now a fast growing technique, due to the high penetrability of neutrons, to the fast development in digital recording and processing and to the development of new detectors and related electronics which are constantly improving space and time resolution. Clearly the possibility of getting information without destruction or damage of valuable archeological and historical objects is of primary importance. The ability of thermal neutrons to penetrate thick layers of metals (up to 3 cm of steel) makes NT a unique tool to investigate metal samples like historical weapons even when they are covered with thick calcareous concretions like in the case of finds from sunken ships. On the other hand, the ability of neutrons to detect even small amounts of hydrogenous materials due to the high attenuation coefficient of H (82 barn) makes them useful e.g. when the structure of fossils is characterized. Of course, NT must not be considered *the technique*, but rather *a technique* able to complement information obtained with other techniques. Finally the fact that, when neutrons are scattered by multi-component solid natural materials, most of the scattering comes from the interface between the voids and the material, the analysis of the data in terms of models is greatly simplified. This is certainly the case of stones and other geological materials, for which often a two-component modeling is fully adequate, despite the intrinsic complexity of these materials. Therefore, it is not surprising that by combining a variety of neutron techniques like Neutron Diffraction (ND), Small Angle Neutron Scattering (SANS), Ultra Small Angle Neutron Scattering (USANS) and Neutron Tomography, or more in general Neutron Imaging, is possible to cover a range of structures from the micro-structure (nm or fractions of nm) to meso-structure, to macro-structure (mm or fractions of mm) able to fully characterize the nature and the origin of geological materials. Information on a few selected cases will show that the possibility of finger printing marbles and other stones of archaeological interest is indeed possible. As an example, Neutron diffraction data (Fig. 1) and Neutron Tomography (Fig. 2) are shown for one sample of white calcitic marble recovered from the Villa Adriana (Tivoli, Italy) shown in Fig. 3.



Posters

Understanding Fuel Cell Operation Through Neutron Imaging

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Neutron imaging has recently been identified as a useful tool for in-situ, non-destructive analysis of hydrogen fuel cells. Hydrogen has a large radiographic cross-section for neutrons, making this technique suitable to observe in-situ formation and transport of water in the channels of an operating fuel cell, without disturbing its operation. This is a valuable capability that could be used for fuel cell development and optimization because water is possibly the most important component in the operation of proton-exchange membrane fuel cells (PEMFC). A fully equipped fuel cell has been set up at Georgia Tech to study the effects of operating parameters on fuel cell performance. Experimental observations have shown a strong dependence on humidity, pressure difference across the proton-exchange membrane (PEM), and electrical resistance. In parallel, a molecular dynamics simulation study of hydrated Nafion (PEM membrane) at water contents ranging from 5 to 20 wt% was performed at The University of Tennessee to examine the structure and dynamics of the hydrated polyelectrolyte system. The simulations show that the system forms segregated hydrophobic regions consisting primarily of the polymer backbone, and hydrophilic regions with an inhomogeneous water distribution. We find that the water clustering strongly depends on the water content. At low water content, only isolated small water clusters are formed. As the water content increases, it becomes increasingly possible that a predominant majority of water molecules form a single cluster, suggesting that the hydrophilic regions become connected. The goal of the present work is to couple neutron imaging with molecular modeling results, as well as traditional and novel electrochemical analysis techniques, to provide a better understanding of water formation, accumulation, and transport in proton-exchange membranes and their effects on the fuel cell performance.

Science Program at ILLs Neutron Imaging Facility

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The high flux neutron imaging facility at ILL is dedicated to time resolved radiography and tomography and precise imaging with high statistics. Areas of application include materials research and engineering, hydrogen technology, geology and others, such as friction stir welding and transient liquid phase bonding, fuel cells, heat exchangers and car engines, water transport and nondestructive testing.

The Neutron Imaging Facility at ILL

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The high flux neutron imaging facility at ILL is dedicated to time resolved radiography and tomography and precise imaging with high statistics. It delivers a thermal flux of $3 \cdot 10^9 \text{ cm}^{-2} \text{ s}^{-1}$ to a sample area of 18cm by 18cm. Spatial resolution is typically 100 microns, 14-bit images can be acquired in 50ms. Different cameras and optics, fully integrated instrument control and data acquisition, and additional sample environment allow for a wide range of experimental setups.

Identification of Hidden Object of Archaeological Interest

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When objects of artistic or archaeological interest are studied, the fragility, the historic and even the economic value of samples analyzed must be taken into account. Neutron tomography in conjunction with other methods (i.e. neutron diffraction), seem to be an useful technique for testing objects of artistic or archaeological interest. Neutron tomography is a non invasive technique and thermal neutrons are able to penetrate thick layers of sample. Therefore, samples of greater mass than the ones allowed with other probes can be investigated. Recently at the University of Palermo (Italy) we have started a research program with the aim to obtain tomographic 3D reconstructions on a selection of ancient artillery and other objects recovered from ship wrecks in several locations close to Sicily shoreline (Sicily channel and Tyrrhenian sea). In particular some of these ship wrecks have been found near the south coast (approximately 1 mile from the shoreline) where there is, still nowadays, a very dangerous cliff (Scoglio della Bottazza). Most of the objects found belong to ships operating in the Mediterranean sea between the 4th century A.D. and the 17th century A.D.. Indeed near Scoglio della Bottazza, in shallow waters, it has been possible to spot, during a preliminary investigation, part of the artillery weapons and other arms used for self defense. The artillery parts recovered are clearly indicating that we are in presence of bulky metallic objects embedded in a thick calcareous concretion matrix (sea shells in many cases are clearly visible). Typical objects are swivel guns, long range cannons, various ammunitions and connection parts very well conserved, swords and simple parts.

We have performed measurements on a large number of small finds and on the handle of a sword for which little information was available because of the thick calcareous concretion surrounding the arm. The sword is made of three pieces (the handle and two additional pieces which should make the entire arm). Surprisingly clear images of the handle have been obtained, despite the difficulty of the experiment given the strong adsorption of the find which would have prevented the use of other probes. Even in such difficult experimental conditions Neutron Tomography (NT) has shown to be of extreme importance for obtaining the digital 3D reconstruction of the objects before the restoration work. In figure 1 the reconstructed image is the result of stacking four images. The original handle is also shown. More data are being analysed, and more experiments are needed on the remaining pieces. Still there are many other objects that need to be recovered from the sea and the NT technique might help in building a database for identifying the correct historical frame period.

So far the experimental part of the research program has been carried out at the German BENS facility in Berlin.



A High Spatial Resolution Neutron Radiography System by Using Micro-channel Plate Neutron Detector and CCD camera

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A neutron sensitive Micro-channel Plate (MCP) detector with pore pitch of 11.4 μm is combined with a high-resolution cooled CCD camera to provide a high spatial resolution neutron radiography system. The optical path includes a high reflection front surface mirror for keeping the camera out of neutron beam and a macro lens for achieving maximum magnification that could be achieved. All components are assembled into an aluminum light tight box with heavy shielding to protect camera and to provide a dark working condition. A remote controlled step motor is also integrated into the system to provide on-line focusing ability. The system resolution is determined through the modulation transfer function (MTF) that is measured by the edge method. The edge profile is provided by imaging a Gadolinium foil with a sharp edge. An angling technique is used to overcome aliasing due to undersampling. The scintillator screen and other CCD camera and CID camera can also be integrated into the system and their performances are compared in terms of MTF. The best resolution achieved by the system at the TRIGA reactor at the University of Texas at Austin is 16.2 lp/mm, which is equivalent to a minimum resolvable spacing of 30 μm .

Simulation and Analysis of Correlated Neutron and Gamma-Ray Detection from Photofission

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We describe Monte Carlo simulations of the photon interrogation of uranium and plutonium metal samples of varying mass and composition. The simulations are performed using a Monte Carlo package consisting of modified versions of the codes MCNPX and MCNP-PoliMi. The cross-correlation functions simulated consist of correlated detection of these neutrons and gamma-rays. Firstly, these physical mechanisms are described, and the ability of the resulting correlation signatures to characterize shielded special nuclear material is discussed. Secondly, a methodology based on Monte Carlo simulations and artificial neural networks that is aimed at determining the characteristics of the fissile material. In the simulations, we modeled a simple geometry consisting of fissile material shielded with Celotex, placed between two plastic scintillation detectors. We show that the methodology can reliably differentiate between highly enriched uranium and plutonium. Furthermore, the mass of the material is determined with a relative error of about 7%.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Momentum Distribution of Atoms in Condensed Helium

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Deep Inelastic Neutron scattering measurements of light atoms is the focus of our current research. Such measurements reveal the dynamics of single particles. In this poster, we present a survey of our measurements of the shape of the momentum distribution of atoms $n(\mathbf{k})$ in liquid and solid ^4He , and in ^3He - ^4He mixtures. The measurements were performed on the MARI time-of-flight spectrometer at the ISIS spallation neutron source, UK. Data from the early 1990's onwards are presented. These measurements show unequivocally that the $n(\mathbf{k})$ in normal liquid ^4He , superfluid ^4He and solid ^4He , are not Maxwellian, in agreement with calculations and as expected for cold quantum systems. The kurtosis δ which provides the magnitude of the leading deviation from a Gaussian $n(\mathbf{k})$ is determined from the data. In bulk liquid ^4He , the second leading deviation from a Gaussian is

also determined. In $^3\text{He}^4\text{He}$ mixtures, we find a condensate fraction, n_0 , which increases with increasing ^3He concentration. We also find that the ^3He $n(\mathbf{k})$ in mixtures has a small step height at the Fermi surface and a substantial high-momentum tail characteristic of a strongly interacting Fermi liquid.

Achievements in Neutron Radiography at INR Pitesti, Romania

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There are presented the two neutron radiography facilities, the underwater neutron radiography facility and the dry neutron radiography facility from INR Pitesti. It is shown the design, involved methods and results of their operation. The underwater facility it is intended for nuclear fuel investigations based on transfer method with indium and dysprosium foils and track-etch method. The second newer facility is located at the tangential beam tube of the TRIGA ACPR reactor. This new facility enlarges the applications of neutron radiography, diversifies the applied methods and makes the neutron radiography easier, compared with older underwater facility. Beside the methods of the underwater facility it is used a detector with scintillator, image intensifier and a high resolution CCD camera proper for tomographic investigations. Soon will be in practice the direct method based on gadolinium.

Synthesization And Characterization of High Quality of Single Crystal CeCoIn₅

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Novel electronic materials play an ever-increasing role in technological applications. They cover a rather broad spectrum of materials that exhibit exotic transport and magnetic properties. These include superconductors, narrow band gap semiconductors, electronically active polymers, colossal magneto resistive systems, nanoscale electronic materials, and Fullerenes, to name a few. Understanding the interplay between strong electronic correlations and structural properties of these materials is important. In this communication, we report the growth and preliminary characterization of single crystal Cerium based intermetallic compounds that are known to show superconductivity at $T_C = 2.3\text{K}$.

Synchrotron X-ray Tomography (Absorption and Phase Contrast Enhanced) of Low Contrast Materials: Biological Tissues and Polymer Blends

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Synchrotron X-ray tomography ranks as one of the leading high-resolution 3D imaging methods for materials, polymer blends, microfabricated devices, biological samples, and environmental samples. The tunability of synchrotron X-ray energy enables better contrast for images as well as 3D elemental concentration mapping. But imaging low contrast materials by x-ray tomography, such as biological tissues, has been a challenge. Here we are presenting tomographic images on cat claws and a fiberglass-reinforced polymer blend with a new generation flame retardant. Compared to other composite materials or objects, both samples are difficult to image due to low contrast between tissues in cat claw and between fiberglass and the polymer blend. To improve the image contrast between tissues, several different assessments were done, such as phase contrast imaging and the infusion of different contrast agent into the biological tissues. To investigate the chemical composition of this polymer blend, new procedures and algorithms were developed to produce, segment and analyze a chemical concentration distribution that assesses the flame retardant distribution throughout the blend, especially around the fiberglass reinforcement.

Visualization of the Internal Structure of Responsive Polymer Films *via* Neutron Reflectivity – Modeling *vs.* Direct Inversion

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Polyelectrolyte multilayers (PEMs) present a new class of nanostructured materials obtained through alternating self-assembly of water-soluble polymers at solid-liquid interfaces. The use of pH-responsive polyelectrolytes for multilayer construction offers the advantage of engineering pH-responsive coatings of controllable film structure and thickness. A variety of potential applications in, for example, drug delivery and tissue engineering urge understanding of the fundamentals of the self-assembly and post-assembly response, specifically, information on structural changes within polyelectrolyte multilayer films upon exposure to environmental stimuli.

We are using neutron reflectivity as the principal tool to probe layer distribution and the degree of layer interpenetration at assembly and post-assembly steps. We studied two related classes of responsive PEMs, where interactions between adjacent layers are controlled either by electrostatic or by hydrogen-bonding forces and found hydrogen-bonded layers to be highly interdiffused. The degree of interpenetration was controlled by the strength of interlayer adhesion and by polymer nature. We also addressed a question of pH-induced structural change in electrostatically bound multilayers and observed dramatic randomization of layered film structure resulting from selective release of one of the film components.

Neutron reflectometry is a powerful technique to unravel the structures of layered materials. Intrinsically, the interpretation of reflectometry data via modeling is a challenge due to the loss of phase information in the diffraction process. Most interpretation is done through tedious trial and error, which yields non-unique structural solutions. Direct inversion using multiple measurements with reference layers has been experimentally demonstrated, but the method involves elaborate preparation and long data collection times and so is not routinely done.

Using conventionally measured PEM data, we have shown that the Patterson function can be adapted to assist in determining the number of layers and their thicknesses. We have also evaluated the “sufficiency” of the data for direct inversion, and the ambiguity of the solutions. These measurements show the Patterson function to be very helpful in depth profile determination in many practical situations. In the worst case, it can provide a sanity check for data fitting results. For conventionally collected reflectivity data, this represents a small, but significant step toward real-space imaging of buried layers.

*Managed by U.T. Battelle, Inc., for the U.S. Department of Energy under contract DE-AC05-00OR22725

Development of Novel Neutron Optics for Imaging at Future Neutron Optics Test Station at MIT Nuclear Reactor Lab

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The Spallation Neutron Source (SNS) will catalyze a new generation of neutron instrument development, a new generation of neutron scientists, and therefore, a new generation of scientific research with neutrons. Neutron imaging, in particular, has a great potential for applications in science and engineering thanks to unique features of the neutron, such as large penetration depth into most materials, considerable variations in neutron scattering cross-section among chemical elements and isotopes, and magnetic interactions between the neutrons and magnetic materials.

The challenges of pushing the resolution and contrast of neutron imaging to the levels necessary for such applications as biomedical imaging or hydrogen storage cannot be met with today’s neutron imaging optics. Most of today’s neutron radiography instruments utilize neutron absorption phenomenon and use minimal image formation optics. Novel imaging methods, such as phase contrast and differential phase contrast, which utilize the wave nature of the neutrons, promise significant improvements upon traditional radiography. However, significant advances in neutron optics are needed to overcome low spatial and temporal resolution of the neutron imaging. The combination of the novel neutron optics and great improvement of the neutron flux will enable

construction at SNS of fundamentally new instruments, such as neutron microscope in absorption and phase contrast, which was not practical before.

The Neutron Optics Test Station, currently under construction at MIT Nuclear Reactor Lab, is aimed directly at developing novel instrumentation and educating the next generation of neutron scientists. Our own research will emphasize developing novel focusing and imaging methodologies. In particular, we are interested in developing Wolter-type curved mirrors, which will be optimized for use with diffuse neutron sources. In addition, we are planning to support a user program, when a portion of the beam time at this new instrument will be provided to outside users for testing new optical elements and detectors.

Spectroscopic Neutron Imaging Experiments Performed at Pulsed Neutron Sources in Japan

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Neutron radiography using a pulsed source is a useful tool for non-destructive tests. By using a pulsed source, we can obtain energy dependent images at once by using time-of-flight (TOF) method for energy analysis. As one of applications we performed experiments to obtain the energy dependent images of the object including thin plastic and thick one covered with Cd. From the results we could recognize the thin plastic in a low energy image and thick one in the image with energy over the Cd cut-off. This indicated that the energy dependent imaging was very useful to catch the total image of the objects.

The radiography using the pulsed source means in some sense the measurement of the total cross-section of the sample material depending on the position, so it gives spectroscopic images. A characteristic feature of the total cross-section is Bragg edges at a low energy region, which appear for materials scattering the neutron coherently. By analyzing the structure of the total cross section, we will be able to get information on crystal structure, crystalline size and so on. Therefore, we performed experiments to apply this feature of transmission spectra to the material distinction and also to the material identification. By fitting the simulated TOF spectra to the experimental ones it was suggested that the material identification was possible.

Further information of the object is also obtained by analyzing the total cross section. Total cross section will depend on the micro-crystalline size since effect of extinction appears more clearly if the crystalline size becomes larger. We measured a heat treatment stainless steel and a surface treatment one. The cross section around the Bragg edges was smaller than that of the surface treatment one. This may be attributed to the large microcrystalline size of the heat treatment sample. In this transmission data we also found the sub Bragg edges on the tail in shorter wavelength side, which may be due to the microcrystalline. Similar characteristic feature was also found in the data of the other sample artificially stressed.

Therefore, these results indicated that the transmission spectroscopy image would be a new method for analyzing the material structure of the objects depending on the position.

The Role of Neutron Imaging in Hydrogen Research

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Current attention of many researchers in materials science, chemical engineering, and physical sciences, including me, is focused on changing hydrocarbon based energy source to hydrogen. However, to meet this ultimate goal, we have to build successful key technologies for both hydrogen fuel cell and hydrogen storage (including safely storing and delivering hydrogen in a fuel cell vehicle).

Among all the possible fuel cells, proton exchange membrane fuel cell (PEMFC) is a leading candidate with applications as replacements for rechargeable batteries, to remote power for houses and automobiles. (The key components of PEMFC are: the membrane electrode assembly (MEA), the anode gas diffusion medium (GDM), the cathode GDM, and the respective gas flow channels.) However, an important issue to its successful deployment is water management: a delicate balance between properly hydrating the MEA and keeping the GDM pores free of water to allow efficient gas flow to the MEA. Neutron Imaging (NI), which may provide two dimensional (2-D) radiograph of liquid water in sample, is capable to unambiguously distinguish the key

components of PEMFC, to analyze the mass flow of water through the GDL and out of the fuel cell through the flow channels, and to acquire tomographic (3-dimensional) images of an operating PEMFC.

On the other hand, a fuel cell vehicle may function efficiently only if it has an efficient means to store and deliver hydrogen. Neutrons are generally viewed to play an important role in the development of the engineered systems used to provide hydrogen storage onboard vehicles because of their unusual sensitivity to hydrogen.

The limitation of current imaging techniques, from my point of view, is the resolution or length scale of imaging. Thus R&D is needed to better the resolution through ways such as changing neutron-light conversion scintillator into neutron-electron conversion converter at neutron camera part. An increase of 10-50 times in sensitivities would be very valuable to my research interest in both fuel cell and hydrogen storage.

Application of Neutron Radiography to the Lost Foam Casting Process: Visualization and Characterization of the Pyrolysis Front

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Lost Foam Casting (LFC) has started to be widely used as a replacement to the traditional casting due to its unique advantages: no size limit, casting products obtained as near net shape, environmentally friendly, energy saving. The LFC process and products are largely depended upon the complex behavior of pyrolysis front during the interaction with liquid metal. Therefore, visualization of the LFC process helps to develop a fundamental understanding of key issues governing the science behind LFC process. However, it cannot be achieved by traditional approach such as X-ray radiography, but by neutron radiography due to the unique neutron cross-section of the materials involved. This study aims to utilize advanced and innovative technology provided by neutron radiography to identify and visualize the pyrolysis front during the LFC process. Experiments are conducted by using refractory coated Expanded Polystyrene (EPS) foam patterns having simple General Motors test plate geometry with different gating systems, and aluminum alloy samples are cast at 1400 F. Real-time neutron videos are recorded for the entire LFC process that includes phase transitions. Still images are extracted from real-time neutron videos, then further processed and rendered by image processing.

Based on the results from neutron radiography combined with the image processing and analysis, the pyrolysis front can be clearly identified and isolated from the molten metal as well as the undecomposed polymer foam. For the side-gating sample with a thickness of 24 mm, the range of pixel intensity representing the pyrolysis front is between 105 and 130, while the range representing the undecomposed polymer pattern is between 80 and 105, and the range representing the molten metal flow is between 132 and 178. There is remarkable difference in the filling characteristics for the casting processes in which different thickness of the foam patterns and different gating are used. The irregular volatilized pyrolysis flow patterns composed of gaseous styrene monomer are observed more pronouncedly when thicker EPS foam patterns interact with liquid metal during the lost foam casting process. The proposed approach in this study will prove to be a powerful in-situ monitoring and visualization system for both quality control and improvement for metal casting techniques such as the LFC.

We are grateful to the guidance, experimental collaborations, and interpretations over the past few years from our collaborators from General Motors Powertrain (GMPT) group and those who were affiliated with Casting and Development Validation Center (CDVC).

Atomic Resolution Neutron Holography - Basic Principles and Field of Applications

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The basic principles of atomic resolution holography are proposed by Ábrahám Szöke. Later on, László Cser et al. discovered how to apply Szöke's ideas for neutron holography. Atomic resolution neutron holography occurred to be an extremely useful tool for three dimensional imaging with atomic resolution of those

orientationally ordered samples which are out of scope of the electron and X-ray approaches. In this talk we present the scheme of the two basic approaches of the holographic measurements, i.e. the internal source and the internal detector concepts. The way of their experimental realizations is discussed as well. This technique provides answering on the questions like what is the position of the hydrogen atom in crystal lattices including metal hydrides organic crystals and potentially biological samples as well, what kind of local deformation is caused by an impurity atom, the effect of external electromagnetic field, or clarify the mechanical lattice deformation (e.g. residual stress) on picometer-scale range. The ability of this method is illustrated on the holographic investigation of PbCd alloy.

Methodical aspects of the atomic resolution neutron holography

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Atomic resolution neutron holography is a unique tool for three dimensional imaging on atomic resolution scale the atomic structure of ordered (and even of orientationally ordered) samples. This method in some sense is complementary to the X-ray holography, but some times it is the only approach to carry out holographic study of many samples. However, several methodical problems have to be considered for successful performing of a holographic experiment. In the present talk we discuss some aspects of these problems like the choice of the size of the domain of observation, the instrumental effects and the statistical noise. The conditions of the optimization of the holographic measurement are described as well. The feasibility of TOF experiment for neutron holographic measurement is also demonstrated. Till now only Cd nucleus was used for internal detector, but there is a considerable number of further suitable elements/isotopes which can be expected to be of use in neutron holography. Possible candidates for both techniques will be presented and peculiar properties of some of them briefly discussed. This aspect is especially promising for such high intensity pulsed neutron sources as SNS. The benefit of a dedicated holographic instrument is shown in comparison with a standard diffractometer. Such a facility installed at the 10 MW Budapest Research Reactor is described.

Present Status of Radiography Research Activities in JAEA

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The available neutron flux over 10^8 n/cm²s and high collimator ratio larger than 100 at the JRR-3M thermal neutron radiography facility (TNRF) has expanded the application field to dynamic but clear imaging of moving objects and fluid phenomena. JRR-3M TNRF is facilitated with three major imaging systems using a fluorescent converter, being characterized by spatial and/or temporal resolutions: Static neutron radiography (SNR) system using a cooled charge coupled device (CCD) camera, Real-time neutron radiography (RNR) system using a silicon intensifier target tube camera or an electron bombard CCD camera with 30 frames/s and High-frame-rate neutron radiography system using the combination of an image intensifier and a high-frame-rate digital video camera with more than 1,000 frames/s. SNR has been used for three-dimensional visualization of air-water two-phase flows in a simulated reactor core for new type nuclear reactors. Also, three-dimensional computed tomography clearly illustrated hydrogen concentration distribution in alloys and tanks. RNR has been applied to quantitative visualization of void distribution in gas-solid fluidized beds and also applied to generated water distribution measurement in a fuel cell. HFRNR has been found to be a powerful tool to visualize rapid transient phenomena.

Although JRR-3M TNRF supplies advanced imaging systems to users, it is also utilized as an testing facility for neutron imaging devices such as a neutron image intensifier. Excellent characteristics of the neutron image intensifier make fast neutron computed tomography possible.

Preparation and Characterization of Nanocrystalline Aluminum Hydroxide Powders

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It has been proposed that as particles become smaller in size they may take on different morphologies that may alter their surface chemistry and adsorption properties in addition to increasing their surface areas. The preparation and characterization of aluminum hydroxide powders in basic medium are described. Aluminum hydroxides were prepared from supersaturated solutions of sodium aluminate, obtained by mixing aluminum metal with NaOH. The two key parameters were the concentration of the basic solution and the ratio $R=[\text{NaOH}]/[\text{Al}]$. Identification was made using the matching of the x-ray diffraction peak positions and intensities between standards and precipitates. Raman, infrared, and Scanning Electron Microscopy techniques have also shown to be good methods for identifying and distinguishing between phases. Nanocrystalline bayerite and gibbsite phases were obtained.

3-D Deep Penetration Neutron Imaging of Thick Absorbing and Diffusive Objects Using Transport Theory

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The goal of the proposed project is to radically improve the computational tools used for non-destructive investigation and 3-D imaging of thick absorbing and diffusive heterogeneous objects using neutron transport theory. Such imaging capabilities are of enormous interest in a wide range of fields, from the detection of Special Nuclear Material at ports of entry to the United States, to the characterization of spent nuclear fuel, and to biomedical imaging. For these applications, neutrons are particularly useful since they penetrate deeper than X-rays and are also able to distinguish fissile materials.

In the past, such imaging problems were most often used by resorting to the two extreme cases of neutron transport that are described by either straight ray or the diffusion approximation. The resulting equations are solved using the inverse Radon transform, or optical tomography techniques, respectively. However, in many applications, the medium to be imaged does not satisfy the conditions under which these approximations hold. In particular, this is the case for imaging containers at ports of entry: The object to be imaged contains voids (or air) and dense diffusive and absorbing parts at the same time.

Accurate imaging techniques therefore require the use of the full neutron transport equation, rather than approximations. On the other hand, the use of the more complex full transport model requires the development of new techniques beyond what is routinely used in nuclear engineering, to ensure efficient and accurate solutions of the imaging problem. In particular, this includes the following areas: (i) accurate and efficient solution of 3-D energy-resolved neutron transport problems, (ii) accurate computation of the gradient of objective functionals to minimize the mismatch between predicted and measured neutron fluxes, (iii) efficient Newton-type optimization algorithms, (iv) methods that are able to distinguish between fissile and non-fissile materials, and (v) adaptive mesh refinement.

We propose to develop these techniques within the scope of this project. We will base our work on prior experience gained from similar applications such as reflector homogenization and in particular in biomedical imaging where we could show that the use of techniques such as those above can lead to very accurate and efficient 3-D imaging algorithms that not only yield more than 10 times the resolution compared to previous approaches, but also at a fraction of the numerical cost.

If successful, this project will lead to the development, verification, and validation of reliable, accurate, and efficient 3-D imaging (tomography) algorithms and software that may be used in a variety of applications both inside and outside the Department of Energy. In addition, the results and techniques of this proposal will be incorporated into the classes taught by the investigators, and thereby be used in the education of the next generation of nuclear engineers. The proposal also calls for the involvement of one graduate student. The

participation of two departments will lead to an increased exchange of knowledge between different disciplines not only between researchers, but also between the involved students. Finally, we will provide undergraduate students with research experience through this project.

Development of a Near-Field High-Energy Gamma Camera for Use with Neutron Stimulated Emission Computed Tomography (NSECT)

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Certain elements are vital to the body and an elemental imbalance can be either a symptom or cause of various diseases. Several studies have found elemental composition differences between malignant and benign tissue in the breast, prostate and brain. Additionally, Hemochromatosis and Wilson's disease are identified by iron and copper overload respectively, in the liver. Current techniques for identification of these diseases typically involve needle biopsy, which is invasive and painful. We are developing a new technique, Neutron Stimulated Emission Computed Tomography, that can measure and image elements *in vivo* in a non-invasive, non-destructive manner. The region of interest (ROI) is interrogated via a beam of high-energy neutrons (MeVs) that excite elemental nuclei through inelastic scatter. These excited nuclei then relax by emitting characteristic gamma radiation. Acquiring the gamma energy spectrum in a tomographic geometry allows reconstruction of elemental concentration images. Our current tomographic method relies on the first generation CT technique of: scan a thin line, translate, and repeat. This is time consuming and limits resolution to the thickness of the beam, and it is difficult to maintain adequate neutron flux as the beam narrows. A better solution would be to interrogate the entire ROI at once and then image with a position sensitive gamma camera. Unfortunately, current gamma cameras operate in too low an energy range for use in NSECT spectroscopy. Their spatial and spectral resolution is limited by crystal size and collimation and is usually only practical for energies less than 511 keV. To obtain adequate spectral resolution we use High Purity Germanium (HPGe) but their large diameter (~7cm) provides no spatial information. The challenge is to obtain both spectral and spatial information. We propose to adapt spectral imaging technology that is currently being applied to space-based applications. A rotating modulation collimator (RMC) consisting of two parallel slat collimators offset from one another (5cm-50cm) is placed in front of the HPGe. Counting the number of incident gamma rays at each collimator rotation angle allows for reconstruction of images. The challenge is to identify a RMC geometry that allows this method to work in a near-field environment, which has far fewer assumptions than the infinite focus of space imaging. To that end, we have designed, built and tested a prototype RMC. This allowed for the creation and validation of a Monte Carlo simulation environment that can be used to iterate on and identify a successful RMC design.

Neutron Tomography Based on Neutron Transport Problem

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The goal of our neutron tomography project is to reconstruct the material distribution in an unknown object undergoing a nondestructive detection with a neutron beam. We assume that we are given an incident beam (or beams) of particles and also given signals from detectors around the periphery of the object. We focus on problems in which significant scattering may occur inside the unknown object. We are particularly interested here in applications in which radiation can undergo significant scattering (and/or fission and/or production of other types of radiation) within the object. We also take the count of the cases in which radiation is actively introduced through the object boundary as well as cases in which radiation is spontaneously emitted by sources within the object.

We assume that if we knew everything about the object (material and source distributions) and any incident radiation, then we could use a "forward model" (i.e., solve the transport equation) to obtain reasonably accurate

calculations of the measurements obtained by external detectors. Given this ability, one family of approaches performs a sequence of “forward” calculations, each with some (hopefully intelligent) guess of material and source distribution. The goal of such “optimization” methods is to determine distributions that minimize some measure of the difference between predicted and measured results.

We believe that our approach to this problem is new and advantageous in many respects. One key feature is that we treat the material (a discrete quantity) as the unknown, as opposed to a set of cross sections (continuous variables). This changes the mathematical nature of the problem, and in fact greatly reduces the dimension of the search space. Another key feature is we take a hierarchical approach to reduce computational costs of a stochastic optimization procedure. By the approach, we reduce the dimensionality of the search space (number of spatial cells in which we are searching) and the number of possible values in each dimension (number of candidate materials in each cell). We begins at learning some characteristics of the object from relatively crude (inexpensive) calculations, and then use knowledge from such calculations to guide successively more sophisticated calculations.

Prompt Gamma-Ray Imaging

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A new imaging modality called prompt gamma-ray imaging (PGI) has been identified and investigated primarily by Monte Carlo simulation. Currently it is suggested for uses on small animals or neutron capture therapy, where the tolerated dose could be relatively high. The approach consists of irradiating objects in the thermal or epithermal neutron beam of a nuclear reactor. Then prompt gamma rays are emitted from the elements in the sample by the radiative capture (n, γ) reaction. These prompt gamma rays are produced in energies that are characteristic of each element, and they are also produced in characteristic coincident chains. After measuring these prompt gamma rays by a surrounding spectrometry array of detectors, the distribution of each element of interest in the sample is reconstructed from the mapping of each detected signature gamma ray by either electronic or physical collimation. In addition, the transmitted neutrons from the beam can be simultaneously used for structure imaging, which provides the registration for the elemental distributions obtained from PGI. Geant4-based Monte Carlo simulation codes have been developed for this purpose. Preliminary results indicate that this approach will be successful for either elements of relatively high neutron absorption cross section like Gd, Cd, Hg, and B or elements of high abundance in objects like H. This approach has great potential to be used in neutron cancer therapy to monitor neutron distribution and neutron-capture agent distribution.