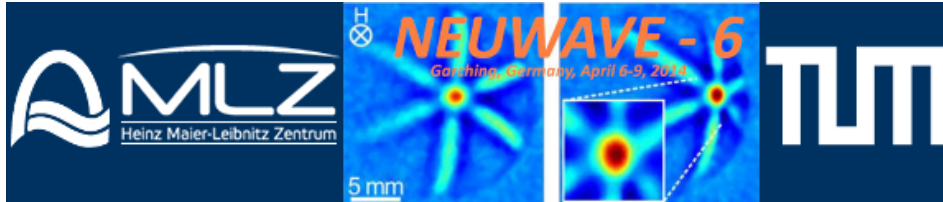


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Book of Abstracts

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ToF Instrument projects I / 21

Present status of Pulsed Neutron Imagin in Japan**Author:** Yoshiaki Kiyonagi¹¹ Nagoya University**Corresponding Author:** kiyonagi@qe.eng.hokudai.ac.jp

We are developing the pulsed neutron imaging method and expanding application such as cultural heritage. Here, we introduce present status of the pulsed neutron imaging in Japan.

We have improved the RITS code to analyze martensitic phase. We introduced a Gaussian distribution of lattice spacing to express realistic behavior in the martensite. From this analysis it is found the width increased toward the periphery, which indicating increase of martensitic phase in this region. The trend was very similar to that of hardness of the iron along radial direction.

As cultural heritage applications, Japanese swords were measured and in the transmission spectrum we found similar gradient Bragg edges to the martensite at edge area of the sword. We thought it was also martensitic phase. By analyzing this area by the RITS code, we succeeded in indicating the martensitic characteristics. Coins were measured and it was indicated that texture changed depending on its produced age.

Hydrogen is one of important elements for NRG, and hydrogen storage material research is popular. We studied TiCrMo alloy, and at high content of hydrogen it showed a hump in neutron total cross section and no hump for low hydrogen content one. This indicated that metal hydride was formed at high content and probably relatively free hydrogen existed at low content. This suggests possibility to study hydrogen bound state depending on position.

With the use of resonance transmission we can obtain the elemental information. However, it is not easy to evaluate quantitatively. For this purpose we developed synthetic pulse function of the J-PARC neutron source and implanted it into the REFIT code. After then we succeeded in obtaining quantitative values.

Detectors are very important part of the pulsed neutron imaging. A camera type detector combined with a high speed camera and a high-resolution digital camera has been developed. A spatial resolution of several 10 μm was attained by the high-resolution camera and time-of-flight spectrum with 100 μsec -channel width was obtained. A counting type detector of 8-pic has been developed. DAQ system was improved to be compact and high speed data transfer, and it was proved to have a spatial resolution of about 100 μm .

We have been performing pulsed neutron imaging under the project of JAEA, and collaborative work among Hokkaido University, JAEA, Ibaraki University, KEK and Nagoya University is on going. In the presentation we present outline of major activity and topics in obtained results.

The author sincerely thanks members of the project group. This work was partially supported by JSPS KAKENHI Grant Number 23226018.

ToF Instrument projects I / 0

Status of ODIN imaging project at ESS**Author:** Markus Strobl¹¹ ESS AB**Corresponding Author:** markus.strobl@ess.se

After the concept for the ODIN imaging instrument has been endorsed by the ESS Scientific Advisory Committee (SAC) just before NeuWave-5 held at ESS in Lund, Sweden, ODIN was also approved by the ESS Steering Committee (STC) and consequently the project was moved into the construction phase 1 according to ESS policy. ODIN is a collaborative project involving various partners which are

foreseen to partly also significantly contribute to the construction. Apart from that the project also involves various cooperations concerning method development and scientific projects. These are meant to build partnerships with universities, future users and institutions with valuable expertise, which is key for the future of the project and instrument. An overview of these activities as well as a snapshot of the current state of the project, the ESS as a whole and the instrument ODIN, will be provided together with an outlook on planned progress and perspectives.

Instrumentation I and Applications 0 / 19

Energy-selective imaging at CONRAD-2

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The neutron imaging instrument CONRAD at HZB was upgraded recently. The neutron guide system was replaced by a new one based on super mirrors. This has reflected in higher neutron flux and larger beam at the sample position. The capabilities of the instrument in the field of energy-selective imaging were strengthened by construction of new double crystal monochromator and installation of velocity selector. The parameters of the devices were studied and procedure for their calibration was developed. Effects like wavelength and intensity inhomogeneities were observed and analyzed. New method for energy selective imaging using the wavelength gradient over the image plane due to the mosaic of the monochromator crystals was developed. The parameters of the energy-selective capabilities of the CONRAD-2 instrument will be presented and recent will be discussed.

Instrumentation I and Applications 0 / 16

Distinction of Liquid Water and Ice using Dual Spectrum Neutron Imaging

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The use of fuel cells in automotive as well as in autonomous power supply applications requires them to be able to start and operate at sub freezing temperatures. At those temperatures, the water produced by the electrochemical reaction can freeze and block the access of gaseous reactants, leading to a failure of the cell. Previous experiments [1] indicated that liquid water in supercooled state plays an important role in the cell operation at sub-freezing conditions. In this context, a direct method from the distinction of liquid water and ice is highly desired. The measurement is based on the differences in microscopic cross-section between liquid and frozen water [2] –related to a different inelastic scattering behavior of the neutrons. To avoid time consuming energy-selective

measurements, a “dual spectrum” method was developed and implemented at the ICON neutron imaging beamline (Paul Scherrer Institut, Switzerland). In this method, a Beryllium filter is used to remove the high energy part of the spectrum and focus on an energy range (< 5 meV) where the differences of cross section are visible. Using the comparison of the sample attenuation with and without filter, a “liquid fraction parameter” calibrated to be 0 for ice and 1 for liquid water can be extracted [3]. As seen in Figure 1, the liquid fraction parameter for a test water column shows a clear step at $t = 4$ h, where the state changes from ice to liquid water. At $t = 8$ h, no transition is visible, as the water stays liquid in the supercooled state. Further experiments were performed using small scale fuel cells. Although the absolute detection of the aggregate state of water was not possible, the phase transitions between liquid water and ice (and inversely) were clearly identified.

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Summary:

Dual Spectrum Neutron Imaging was established as a method for distinguishing liquid water from ice. Although an absolute detection remains challenging, the identification of phase transitions in time series was demonstrated both in a test water column and in small scale fuel cells.

Applications I / 1

Wavelength Dependent In-Situ Neutron Radiography Investigations of the Phase Change of Zirconium Oxide and Zirconium Nitride during Air Oxidation

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Oxygen stabilized metallic zirconium (Zr(O)) reacts with N₂ at temperatures between 700 and 1400°C. Post-test examinations at room temperature, however, show a mixed structure consisting of ZrO₂ and ZrN. No information is available about which phases are formed during the reaction at these high temperatures. It is possible that both phases are produced directly or that a Zr(O,N)_x mixing phase is produced which decomposes into ZrN and ZrO₂ during cooling down to room temperature. Wavelength dependent neutron radiography was used to perform in-situ investigations of the phase composition of the zirconium at different temperatures. The aim was to check the presence of Zr(N,O)_x and at which temperature the two-phase mixture ZrO₂ / ZrN and the phase change in the structure of ZrO₂ from tetragonal to monoclinic occur. In this presentation we discuss the background noise observed in the radiographs due to a change in furnace temperature when performing wavelength dependent in-situ neutron radiography experiments.

Applications I / 2

Analyzing crystalline structures by means of neutron imaging at pulsed and continuous neutron sources

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When the grains in polycrystalline materials (crystallites) are large enough (compared to the spatial resolution), diffraction of neutrons out of the direct beam depends on whether or not a crystallite in the beam path is oriented as such fulfilling Bragg's law. Spatial variation in transmission contrast appears across the sample between those grains that do and those that do not. Rotating the sample changes these conditions and makes tomographic extension non-trivial as a grain does not contribute equally to all projections.

The energy-dependent coherent elastic scattering cross-section exhibits distinct peaks rather than the well known Bragg edges in this case. The ensemble of peaks holds information on the crystallite's phase, orientation and shape. High energy-resolution is required to accurately resolve these peaks which have a typical width of $< 0.05\text{\AA}$. Hence, preference is given to the time-of-flight option, stressing the importance of neutron imaging at pulsed sources. Based on energy-scans of coarse grained iron and nickel-based superalloy samples made at ISIS (RAL, UK), we present the feasibility of grain mapping and an outlook towards tomography.

At continuous sources, convolution of the sample cross-section with the monochromator wavelength spread, compromises this method. However, one can think of the crystallites as monochromators themselves, diffracting a particular wavelength out of the polychromatic direct beam based on their orientation. Capturing these diffracted neutrons yields a projection of that grain, with the position on the detector indicative of the orientation. These projections can in turn be used for algebraic reconstruction, which yields a grain volume as well. An example of neutron diffraction contrast tomography (nDCT) performed on an aluminium strain sample made at SINQ (PSI, CH) will be shown.

The methods for imaging with the transmitted or diffracted neutrons for coarse grained material will be presented, examples given, pros and cons assessed and necessary hardware discussed.

Applications I / 14

Neutron Depolarisation Imaging Study of the Weak Itinerant Ferromagnet Ni₃Al

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Quantum phase transitions (QPT) are phase transitions that are driven by quantum fluctuations instead of thermal fluctuations. In practice this implies that QPTs even occur at zero temperature as a function of non-thermal control parameters such as hydrostatic pressure, magnetic field, uniaxial stress or chemical composition.

The weak itinerant ferromagnet Ni₃Al is a stoichiometric system, which crystallizes in the fcc Cu₃Au-structure. At ambient pressure, a Curie Temperature of $T_C = 42\text{K}$ is found [1]. A small amount $x = 0.4$ of excess Al in Ni(75-x)Al(25+x) can reduce the magnetic ordering temperature to zero [2],

which indicates that Ni₃Al is located close to a magnetic instability. This renders Ni₃Al an ideal candidate for the investigation of a ferromagnetic QPT as a function of chemical composition. At the re-designed neutron imaging beam line ANTARES at FRM II, a new and advanced setup for polarized neutron imaging is available, which includes a neutron velocity selector for monochromatization, a polarizer, an analyzer and a manipulation stage which allows to handle a closed cycle cryostat containing the sample and an electromagnet. With this new setup, neutron depolarization imaging studies were performed on two differently treated polycrystalline samples of Ni₃Al to investigate the influence of tempering on the spatial distribution of TC. Furthermore, an evaluation procedure was developed to obtain information about the homogeneity of the sample in beam direction from the temperature dependence of the neutron depolarization without performing time-consuming tomographic methods. The information on the variation of TC over the sample can then be used to verify measurements of other physical properties, such as magnetic susceptibility or the magnetic neutron scattering cross section, which both strongly depend on the transition temperature of the sample.

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Methods + Software / 10

BENEFITS AND CHALLENGES OF USING ENERGY SELECTIVE NEUTRON IMAGING AT TIME OF FLIGHT AND REACTOR SOURCES

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The presentation will outline highlights and challenges based on our personal experiences in using energy selective neutron imaging at reactor sources and spallation sources. Rather than “just” showing exciting results possible from energy selective neutron imaging and scattering for identifying phase, texture, strain, and microstructure, we will focus on an active discussion regarding:

- Experimental setup
- Data collection
- Data analysis and reconstruction

From an engineering and materials science perspective, we will outline which applications appear to be useful to be implemented at reactor sources vs. spallation sources. While analysis from data collected at reactor sources is mostly straight-forward, the data from spallation sources at the present time offers unique challenges and will be addressed during our talk.

The research results to be discussed at the workshop has been performed in collaboration with the Helmholtz Zentrum Berlin (N. Kardjlov, A. Hilger, I. Manke, M. Boin), the University of Berkely (A. Tremsin), Rutherford Appleton Laboratory (J. Kelleher, W. Kockelmann), the Los Alamos Neutron Science Center (B. Clausen, S. Vogel), NIST (D. Hussey, D. Jacobson), the ESRF (W. Ludiwig, P. Reischig), and the Oak Ridge National Laboratory (A. Pyzant, C. Hubbard).

Methods + Software / 11

Visualization of bulk magnetic properties by Neutron Grating Interferometry

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see attached

Summary:

The neutron Grating Interferometer (nGI) is a standard user instrument at the cold neutron imaging beamline ICON [1] at the neutron source SINQ at Paul Scherrer Institute (PSI), Switzerland. The setup is able to deliver simultaneously information about the attenuation, phase shift (DPC) [2] and scattering properties in the so-called dark-field image (DFI) [3] of a sample. Due to the interaction of neutrons with the nucleus only, they are able to penetrate deeper into matter, in particular heavier materials, than X-rays do. A further advantage of neutrons compared to X-rays is the interaction of the neutrons' magnetic moment with magnetic structures that allows for the bulk investigation of magnetic domain structures using the nGI technique [4].

In the contribution, the nGI-setup and its technique for imaging with cold neutrons will be presented. The main focus will further be on magnetic investigations of electrical steel laminations. Both, grain-oriented (GO) and non-oriented (NO) laminations will be presented in two parts. GO-laminations are widely used in industrial transformer applications, while NO-sheets are common in electrical machines. For grain-oriented sheet, domain walls were visualized as well as their dynamic displacement process in external applied magnetic fields. In figure 1a) a dark-field image of a grain oriented transformer sheet is shown in the absence of an external applied magnetic field. In well-oriented Goss grains single domain walls are visible (horizontal dark lines), the corresponding domains have a size of several millimeters. Furthermore, dark areas are observed. These are misoriented grains where wide basic domains are superimposed by supplementary domains [compare: Magnetic Domains, A. Hubert, R. Schäfer (1998)] that destroy the dark field image by multiple scattering. By applying a magnetic field, favorably magnetized domains grow and less domain walls become visible. In a magnetic field of 6000 A/m (see figure 1b)), the well-oriented grains are saturated and only the misoriented grains show a remaining contrast. This contrast indicates that supplementary domains are still present in these grains. By further increasing the external magnetic field, also these areas become transparent and the whole sample is in the saturated state.

The resolution of the nGI is limited in terms of resolving domain walls individually in non-oriented sheets. Here a relative density distribution of domain walls is rather imaged. The darker the DFI-signal, the more domain walls are present at this particular point. For non-oriented sheet the influence of treatment and cutting processes was investigated. On the one hand mechanical cutting was compared with laser cutting techniques. On the other hand magnetic flux properties were investigated and the ability to influence the flux propagation by laser treatment was verified. With the possibility of decreasing the magnetic flux, together with the capability of visualizing the magnetic flux density itself in these laminations spatially resolved, new insights in the magnetic behavior of bulk samples become possible.

Methods + Software / 8

Determination of the spatial resolution with the (magnetic) modulation transfer function

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One of the most important parameters in imaging (neutron, X-ray, etc.) is the determination of spatial resolution of the used system. Various methods may yield different results sometimes, mainly due to lack of agreement in standards of measurement. A simple and direct measurement can be achieved with the “Siemens star” [1]. Another, very commonly accepted method is the determination of the modulation transfer function (MTF) of an imaging system [2]. The MTF can be directly measured, as the image contrast as a function of spatial frequency of absorbing grids having increasing numbers of line pairs/unit length. Usually such absorbing grids are not easily available for thermal or cold neutrons. Moreover, they have different disadvantages, such as finite transparency or absorption and also they have to be adapted to the system under investigation, i.e. they do not cover the frequency range continuously.

Using polarized neutrons one can produce a fringe pattern due to path length-dependent depolarization of a neutron spin by traversing a magnetic field of a coil. Changing the current in a coil one can tune, i.e. increase or decrease the number of line pairs/unit length and thus measure the “magnetic” MTF (MMTF) of the imaging system [3]. We compared the MMTF - results of coils with different shapes (circle, quadratic, triangle, rhomboid) with the MTF yielded with straight edge measurements and with those, where a constant fringe pattern was moved vertically in steps of 50µm and the corresponding change of gray values in a pixel line was measured. Further, the spatial resolution was measured for different distances of a coil (110mm, 150mm and 170mm) from the detector. Thus, one gets realistic values for the expected spatial resolution of a sample, measured at these distances.

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ToF Instrument projects II / 23

Recent advancements in energy-dispersive neutron imaging at LANSCE

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We recently applied novel detector technology for the first time to perform element-specific neutron imaging at LANSCE. Utilizing neutron absorption resonances, we were able to image the tungsten

and uranium concentrations in mock-up nuclear fuel pellets enclosed in a steel cladding. Several urania pellets were sintered in contact with tungsten metal plates, leading to diffusion of the tungsten into the urania matrix. With energy-dispersive neutron radiography we were able to collect data that allowed to reconstruct the tungsten distributions in the pellets. Similarly, we examined fragments of the Chelyabinsk meteorite and were able to non-destructively visualize iron grains by detecting resonances of cobalt impurities in the iron. This contribution will provide an overview of our recent work. Furthermore, we will discuss the application of Bragg-edges for energy-dispersive neutron imaging.

ToF Instrument projects II / 4

Progress of Constructing Energy-Resolved Neutron Imaging System at J-PARC MLF

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Construction of the Energy-Resolved Neutron Imaging System (ERNIS) has started in 2012 at the Material and Life science experimental Facility (MLF) of J-PARC. Beam line shields, a new beam shutter, in-shield devices except a T0 chopper, sample stages and detector stages have been installed in the fiscal year ended March 2013 as shown in the attached photograph. The first beam will be delivered in November 2014. After on-beam commissioning use programs are planned to start in 2015. In this presentation progress of the construction is introduced, and then characteristics of ERNIS are discussed.

The new beam shutter having three different size of holes and two rotary collimators provide various neutron beams: the maximum thermal neutron intensity of 3×10^7 n/s/cm²/MW with 100 mm square in beam size, the maximum beam size of 300 mm square, and the highest L/D value of 7500. Two sample positions of upstream (L=18 m) and downstream (L=23 m) are equipped in the shield having a large spare with high ceiling of 3.8 m from the floor. Samples up to 1-ton in weight can be controlled in X-Y-Z and theta coordinate by a large sample stage at the downstream position. A middle one at the upstream position and a portable small one are also available. Large samples and accompanying equipment are brought about through the 1.2 by 1.5 m hatch above the upstream position or through the 1.5 by 3.0 m one above the downstream position using cranes in MLF.

A well-established neutron imaging apparatus consisting of a cooled-CCD camera viewing a flat neutron scintillator was assembled for conventional neutron radiography. Aiming at the highest spatial resolution 4- and 9-inch "neutron color image intensifier" systems are developed. A new Image Signal Accumulation Sensor (ISAS) [1] is also developed aiming at high spatial and time-of-flight resolution simultaneously. As proven neutron counting detectors two detectors are prepared: the micro-pixel chamber (μ -PIC) -based neutron imaging detector [2] utilizing He-3 gas for spatial resolution of about 0.1 mm, and the B-10 coated Gas Electron Multiplier (GEM) detector [3] for higher counting rate. These counting detectors having 100 mm square of field-of-view will be mainly used for neutron Bragg-edge, resonance and magnetic imaging.

Software for device control, data analysis and computed tomography are developed. In addition to the Filtered Back Projection (FBP) software the Simultaneous Algebraic Reconstruction Technique (SART) one is designed to be calculated by a dedicated GPGPU parallel computer system for ERNIS. We fully appreciate all proposals, requests, comments, etc. from potential users, the most important requirement to make this ERNIS project a major success.

ToF Instrument projects II / 15

Recent progress of the IMAT project at ISIS

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The materials science instrument IMAT is currently under construction at the second target station of the ISIS pulsed neutron source [1]. IMAT will offer neutron radiography, tomography and energy selective imaging measurements. In the long run, diffraction detectors on IMAT will offer spatially-resolved crystallographic phase, residual strain and texture analyses of regions of interest in a sample. IMAT will exploit the energy dependences of total cross sections of materials for contrast variation and, specifically, for mapping residual strains and texture in metals and alloys. To do this, energy discrimination is required which is achieved by making use of time of flight techniques on the pulsed 10Hz neutron source. A flexible selection of relatively narrow bandwidths (down to 0.5 Å) or large bandwidths (up to 15 Å) is achieved by operating two double disk choppers at different frequencies and opening times. Energy selection with much higher resolution as required for Bragg edge analysis is achieved with time-of-flight measurements by synchronizing the imaging camera with the ISIS source trigger. The wavelength resolution is ultimately limited by the neutron pulse width given by the slowing-down process of neutrons in the moderator. Monte Carlo simulations of the instrument and experimental data from the IMAT moderator indicate that the energy resolution for imaging will be better than 0.8% [2]. The flexible selection of neutron wavelengths using choppers and time-of-flight capable cameras is achieved for a field of view up to 200 mm.

A straight neutron guide has been installed to transport the neutrons to an aperture selector which will define L/D values up to 2000 at a distance of 10 m from the aperture. Current activities include development and construction of two double-disk choppers and a T0 chopper, a pinhole selector, a heavy-duty sample positioning system, and a camera positioning system. The commissioning programme will start with two neutron imaging cameras: a gated CCD imaging system [3] developed by CNR Messina, Italy, and a time-of-flight capable high-resolution imaging detector based on a neutron sensitive MCP [4] developed at Berkeley, USA. Two relatively small diffraction detectors at scattering angles of 90 degrees will be commissioned as well. It is envisaged that IMAT will have first neutrons in summer 2015.

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Neutron transmission of monocrystalline solids: perfect crystals, mosaic crystals and texture components

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After passing through a single crystal, a polychromatic neutron beam presents a series of dips in intensity at specific wavelengths, due to neutrons removed from the beam as a result of Bragg reflection on the crystal planes. These reductions in intensity can be exploited as a contrast agent on wavelength-dependent neutron imaging of single crystal components, or objects having microstructures close to that of a single crystal.

The position, width and depth of those dips depend on the material, the crystal orientation and the degree of perfection of the single crystal. Real monocrystalline solids of macroscopic size present always some degree of imperfection, with slightly misaligned regions ranging from seconds of arc for perfect crystals to tens of minutes for mosaic crystals.

Here we will discuss details of the wavelength dependent transmission of monocrystalline objects, presenting some analytic expressions for the total cross section. Typical widths and depths of the signal for different materials are considered. Possible information to be extracted from such signal (crystal orientation, elastic strain, misorientation) is exemplified through experiments performed in Cu mosaic crystals [1].

Finally, we show recent theoretical advances towards describing the transmission of textured polycrystalline materials as a sum of contributions from many individual single crystallites.

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Summary:

The wavelength dependence of the neutron transmission of monocrystalline solids is discussed, for different degrees of crystal perfection. Analytical expressions for the total cross section are compared to time-of-flight transmission experiments performed on the ENGIN-X beamline, at Isis, UK.

Applications II / 3

Fast-Neutron Energy Selective Imaging for Container Screening - and beyond

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This contribution presents a summary of the recently finished “ACCIS” project where the technique of Fast Neutron Resonance Radiography was investigated on its potential as possible candidate for a screening technique of small and medium sized cargo containers.

This contribution presents a summary of the recently finished “ACCIS” project where the technique of Fast Neutron Resonance Radiography (FNRR) was investigated on its potential as candidate for a screening technique of small and medium sized cargo containers.

The talk will briefly describe the method, the different detector techniques developed and tested in the project and the still ongoing work on producing intense pulsed fast-neutron beams. Furthermore practical aspects of implementation will be addressed, like acceptance of neutron based techniques, legal aspects as well as activation and radiation protection issues.

The talk will finish with conclusions of future applicability of this techniques in civil security and present an outlook on ideas and first experiments and results to alternate applications FNRR and FN-R/-CT for industry, energy and material research.

Methods + Software II / 22

Refraction based edge enhancements at structural materials –improved studies with highest special resolution and by MacStas simulations

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Edge enhancement features can be observed for several structural materials (Fe, Al, Zr, Pb, Cu, Ti, ...) under certain conditions. In several dedicated experimental runs, preferential at the cold imaging beam line ICON, systematic studies were performed to understand this phenomenon from the experimental side.

On the other hand, a MacStas tool was developed for the simulation by ray-tracing of either refraction, reflection or transmission using the well-known data of material dependent refraction indices. For the first time, a setup with 6.5 μm pixel size and a 14 mm FOV was used. The neutron imaging data were obtained from a 10 μm GadoX screen with cold neutrons of the white ICON spectrum. Beside some common test samples, we focused on two studies: (a) how the edge effect develops when a 20 μm steel film rotates around the axis close to the beam direction, (b) what happens if objects with different refraction behavior get close together.

Most of the measurements fit to the simulations qualitatively. However, there are several open questions to be studied in detail as the relation between the attenuated and refraction beam parts quantitatively. Furthermore, the role of beam divergence and individual spectral components needs to be discussed.

Gaps between material layers and their optimized visibility and the interference of materials with refraction of opposite sign need to be studied in more detail by means of the current setup of highest spatial resolution.

Methods + Software II / 5

ARTEFACTS IN NEUTRON CT –THEIR EFFECTS AND HOW TO REDUCE THEM

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Neutron Computed Tomography has some boundary conditions that are not found in X-ray CT. The intensity of recorded projections is influenced by the build-up of afterglow of the neutron scintillation screen, the projections of the sample itself often contain additional intensity next to edges which is caused by refraction in the sample. The additional intensity acts like 'negative absorption', i.e. emission close to the sample, which conflicts with the assumptions made for tomography. In the reconstructed CT image, it leads to a halo around the sample which may become so strong that the useable data range for real sample details is strongly reduced if the reconstruction software uses automatic scaling for the use of integer variables.

The talk will present several examples of artefacts and their effects in reconstruction, and show how these can be reduced by simple additional preprocessing of projection data.

Methods + Software II / 17

TOWARDS A REALLY QUANTITATIVE PHASE ANALYSIS THROUGH NEUTRON TOMOGRAPHY: OPEN QUESTIONS STILL TO BE SOLVED.

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Neutron imaging techniques have a huge potential for the characterization of morphological, compositional and microstructural properties of materials. The possibility to apply Energy Selective Neutron imaging is an added value opening enormous possibilities in the field of metallurgy both for the characterization of industrial products and to gain information on historical artifacts. In spite of an excellent development of the technique, some problems related to data treatment and to neutron monochromatic beam generation still remain.

Concerning white beam imaging they are:

- the normalization of the tomographic data at the end of the processing performed using commercial software that is not clearly described and is scaled in an undetermined way with respect to the expected attenuation coefficient;
- the correct determination of the beam hardening parameters;
- the blurring effect in the voxels during the laminographic reconstructions.

Concerning energy selective imaging the main problem is related to the determination of the energy distribution and resolution into the field of view and to the methods to obtain a reliable map of it and how to deal with it during the tomographic reconstruction.

This abstract aims to present an overview of this open question with the goal to rise discussion hoping in the creation of a working group devoted to deal with these topics.

Detectors / 12

Introducing the GP2 detector; Event-mode neutron imaging using the 'PImMS' sensor with gadolinium converter

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The demand for energy resolved neutron imaging has generated several recent advances in detector instrumentation and techniques[1], particularly with the use of borated MCP's[2] or fast, gated, CCD technology[3]. This presentation reports on the development of a new type of detector, the Gadolinium-PImMS-2 detector. GP2 utilizes a PImMS-2 CMOS sensor[4], so named as it was developed for Particle Imaging Mass Spectrometry[5], modified to record event-mode data from a pulsed

neutron source[6]. The CMOS sensor has been made neutron sensitive by using gadolinium; the sensor directly detects the conversion electrons generated from neutron capture.

The active area of the current version of GP2 is 22.6mm x 22.6mm, with a single pixel size of 70 μ m. This gives a total of 104976 pixels. Each pixel has four 12-bit SRAM registers for storing timing information, allowing each pixel to record up to four independent hits per frame. The PImMS architecture records a hit after signal shaping and discrimination, meaning no dark-field correction is required. The discrimination level can be independently adjusted for each pixel, which improves the uniformity of the sensitivity, compensating for gain variations. A range of gadolinium thicknesses were measured to determine the optimum detector efficiency, which is ~10% for neutron wavelengths above 1.8Å, using a single layer of $\text{^{225}Gd}$.

Summary:

The new GP2 neutron imaging camera will be introduced, reviewing results from the last two years of R&D. A full detector specification will be given, discussing neutron efficiency, gamma sensitivity, spatial resolution and temporal resolution. Measurements from standard samples demonstrate the detector's capability to measure Bragg-edges, radiographs and tomograms.

Detectors / 20

High Resolution Energy Resolved Neutron Imaging and Resonance Transmission Analysis of engineering samples, geological objects and gamma ray scintillators

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The recent progress in the development of high resolution neutron counting detectors with microchannel plate and Timepix readout enables simultaneous detection of a wide range of neutron energies. That allows for simultaneous acquisition of transmission spectra in each pixel of the detector for epithermal, thermal and cold neutrons. The presence of resonances at energies below 1 keV allow unambiguous elemental and isotope mapping within the samples, while Bragg edge imaging can be used to study the crystallographic properties of the samples. We present the recent improvements of detector characteristics and demonstrate new results of recent experiments conducted at pulsed and reactor-based sources. The 320 μ s readout time of our electronics enables measurements of a wide range of energies with small readout deadtime. The results of our recent experiments with single crystal engineering samples, scintillator crystals and geological single crystal samples will be discussed. The possible studies of advanced nuclear fuel elements at pulsed sources are demonstrated by our experiments. The first attempts on the resonance tomography will also be presented, where certain elements can be unambiguously imaged independent of other materials present in the object. The studies of the structure of various welds were performed by the Bragg edge and resonance absorption imaging. The future improvements of the neutron counting MCP/Timepix detection technology will be discussed.

Applications III / 6

Observation of temperature dependent magnetic flux trapping in superconductor of type I

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The understanding of underlying physics of Meissner effect and flux trapping in type I superconductors is of great importance, both from fundamental and industrial research point of view [1]. In ideal type I superconductors, such as Lead (Pb) as investigated in the present study, is expected to exhibit complete expulsion of externally applied magnetic field. This behavior is independent of whether the sample is in single crystal or poly-crystalline form. In the present work, we investigate both single crystal with $\langle 100 \rangle$ orientation and poly-crystalline Pb samples of high purity (99.999%) using polarized neutron tomography measurements, with varying temperature. Contrastively, we observe that magnetic field expulsion as well as the flux trapping depends not only on the sample, but also on the crystallographic orientation of sample with respect to an external magnetic field and temperature variation. Both $\langle 100 \rangle$ Pb single crystal orientation and poly-crystalline samples exhibit partially expelled and trapped magnetic field in the field cooled (FC) data. Similar images patterns are observed for both the samples. Above TC ($\approx 7.19\text{K}$), no fringe pattern is evidenced, while below TC evolution of fringe pattern is seen around the sample. This indicates the expulsion of magnetic field in superconducting state. Also, further investigations show that after the sample is field cooled below TC and Bext is switched off, signatures of flux trapping (figure shown below) are observed. Although, poly-crystalline Pb sample showed larger flux trapping than $\langle 100 \rangle$ Pb single crystal. This indicates that both Pb single crystal and poly-crystalline sample did not show the ideal superconducting behavior. It is interesting to note that this behavior, particularly in comparison to $\langle 100 \rangle$ Pb single crystal, is in contrast to the previously reported results $\langle 110 \rangle$ Pb single crystal [2]. Wherein, $\langle 110 \rangle$ Pb sample apparently showed an ideal Meissner effect, i.e. no flux trapping has been evidenced. In conclusion, the present results indicate that the fundamental complex physics has not been fully understood, and therefore call for further theoretical studies.

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Applications III / 9

Bragg edge neutron imaging applied to reduction induced strains in anodes and anode supports for Solid Oxide Fuel Cells.

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The Solid Oxide Fuel Cells (SOFCs) are used for a direct conversion of chemical energy into electrical energy and are composed of three layers: a porous anode, a solid electrolyte and a porous cathode.

The greatest market entry barrier of the SOFCs is their durability, and it has been shown [1] that their degradation is caused mainly by processes taking place in anode. A common anode material is Ni –YSZ (yttria stabilized zirconia) cermet. During the initial operation of the SOFC, nickel oxide particles are reduced to pure nickel, which determine the porous microstructure of anode and anode support. This microstructure determine the mechanical and electrochemical properties of the fuel cell [1], [2], which have direct influence on the performance and durability of the SOFC.

Simultaneous exposure to reducing atmosphere and external stress leads to accelerated creep, which changes the stress field in the SOFC [3]. This phenomenon is not fully understood, and the best way to explain it is simultaneous in-situ observation of phase transition and stress field development in NiO-YSZ layers. The best method for such measurements is energy, i. e. wavelength resolved radiography.

We present the results of Bragg edge neutron imaging experiments on SOFC anode supports performed ex-situ at different facilities (ISIS in UK, HZB in Germany, J-PARC in Japan). The anode supports were reduced under different temperatures in different times and under different values of stress, which was applied in direction causing bending of the sample (thus one side of the sample was under compression, and the opposite side was under tensile stress). We observed different content of Ni and NiO phases in different areas of the samples. In particular, we could resolve areas of thickness 200 μm in layers of thickness 1 mm, which allow us to see the gradient of reduced phase amount within one layer. With such resolution we were able to compare phase amount at different sides, which was crucial for the samples reduced under nonsymmetrical stress. We have shown that compressive stress accelerates the reduction rate, since we observed higher amount of reduced phase on the compressed side than on the opposite side. Moreover, it was possible to resolve the edges characteristic for NiO and Ni already even after 2 min of exposure, while the reduction process takes about 1 hour. Therefore, we can conclude that the achievable spatial and time resolution is sufficient to conduct in-situ measurements, which will be the next step in this research project.

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Applications III / 13

Microstructural characterization and phase-mapping distribution of Indian sword blades

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India was famed in literary and history accounts since Greek and Roman time for the traditional crucible steel. According to Will Durant, the technology passed to the Persians and from them to Arabs who spread it through the Middle East. In the 16th century, the Dutch carried the technology from South India to Europe, where it gave rise to steel mass production [1].

In this process, small pieces of iron can be separated from the bloom and then heated in a close crucible together with charcoal (which is almost entirely composed by carbon), until a partial or

total melting takes place. Rapid absorption of carbon can lead to the formation of cast steel (“crucible” steel), with a very high (1.2-1.6 wt% C) carbon content, which needs further little hardening. Controlled cooling and forging can then develop a pattern, resembling watered silk, on the surface of the blade (wootz steel, misnamed Damascus steel). This was the procedure used in Iran, Central Asia and India, where it remained in operation until 19th century, with products that were high in quality but small in production scale [2-4].

However, several unresolved issues still remain about the forging methods of the so-called wootz steel, that is a hypereutectoid steel characterized by an ordered pattern of cementite grains in a pearlite matrix. How to obtain such a peculiar microstructure is still unresolved.

In order to shed light on these open questions, preliminary metallography analyses have only been possible upon some broken blades, which were very kindly supplied by the Nizam’s Armoury of Hyderabad [5]. The obtained results have been consistent with the neutron diffraction data obtained from selected Indian blades from Hyderabad and other private collections [6]. Thanks to neutron diffraction, “crucible” steels have been discriminated by the cementite content and the subclass of wootz steel has been identified in a non-destructive way.

In the presented work, four Indian blades of princely quality have been selected and investigated by means of white beam and energy-selective neutron tomography on ANTARES at FRMII in Garching (DE). The samples have been made available by the Wallace Collection (London).

A preliminary white beam tomography has been done to characterize the non-metallic components (slag inclusions, mineralised and/or oxidized parts) and determine the presence of hidden cavities (cracks) into the blades. Suddenly, the energy-resolved tomography has been performed selecting neutron wavelength intervals immediately above and below the 110 ferrite Bragg edge. Since ferrite is only present into pearlite (a lamellar structure made of alternate layers of ferrite and cementite) in hypereutectoid steel, the contrast enhancement has been exploited to determine the space location of cementite inside the pearlite matrix.

Enhancing the contrast factor of the selected phase, the cementite has been mapped at a microscopic scale, complementing the available information on historical Indian blades made of wootz steel. These data allowed us to shed light on the wootz manufacturing techniques.

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Summary:

In the presented work, four wootz steel Indian blades from the Wallace Collection (London) have been investigated. Enhancing the contrast factor of a selected phase by means of energy-selective neutron tomography, the phase distribution has been mapped at a microscopic level, complementing the available information on the ancient manufacturing techniques of wootz steel.

Instrumentation II and wrapup / 7

A Neutron grating interferometer for the ANTARES Beamline and possible applications on domain imaging in superconductors.

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Neutron grating interferometry (nGI) is an advanced neutron radiography method based on the Talbot-Lau effect which allows a simultaneous recording of differential phase contrast (DPC) as well as dark field images (DFI) in addition to the neutron transmission image (TI) [1] [2]. The setup consists of three different neutron phase and absorption gratings installed at a conventional neutron imaging beamline. DFI, DPC and TI are calculated out of a sequence of detector images taken during a stepping scan of one of the gratings. The applications range from phase contrast and dark-field radiography or tomography as a tool for material differentiation to the observation of domain walls in ferromagnetic materials like transformer sheets or even more bulky samples [3].

Because of the one-dimensionality of the grating array, the method is only sensitive to scattering and phase shift components perpendicular to the grating lines. To extract the complete scattering information a rotation of the sample around the beam axis is therefore necessary which has recently been shown in x-ray grating interferometry. [4]

Unfortunately, a rotation of the sample is not always possible in case of oversized samples or a complex sample environment connected to the sample position. To circumvent these problems an nGI setup was constructed, which allows a simultaneous rotation of all three gratings without changing the Talbot distance or the alignment of the phase to the analyzer grating and therefore avoids a readjustment of the interferometer after rotation.

In the first part of our contribution we will discuss some of the properties and design characteristics of the new setup. In addition, we will show results of a new approach for the fabrication of the analyzer grating using Gd sputtering instead of Gd evaporation. This nGI setup will be implemented at the ANTARES beamline as user instrument.

In the second part we will explain possible applications and advantages of this method on the example of neutron dark field imaging of domain structures in superconducting material. For such experiments a combination of the nGI method with a cryostat and a magnet is necessary. Investigations of this kind will highly benefit from the new setup and the extensive sample environment at FRM II.

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