Abstracts of Scientific Presentations

1. Introductory session

The Bonse-Hart Technique for Neutrons

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We present an overview of the most significant experimental and theoretical studies made in the past two decades, which resulted in adoption of the Bonse-Hart USAXS technique for neutrons. Having over four orders of magnitude smaller absorption neutrons penetrate much deeper inside condensed matter revealing “internal” dynamical diffraction effects “invisible” for X-rays. These effects have not been studied before more or less systematically, thus a serious experimental and theoretical research has been done to make performance of the USANS instrument comparable to its X-ray analog.

Application of the Bonse-Hart USANS instruments clearly demonstrated a real breakthrough to the μm-scale range of the neutron diffraction structural analysis. The new instrument extended a total dynamical Q-range of the neutron scattering by two orders of magnitude.

A Brief History of the Wing Problem

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The Bonse-Hart double-crystal (double-crystal diffractometer, DCD) ultra-small-angle scattering (USAS) instrument takes advantage of the very sharp angular resolution of the double-crystal rocking curve to access information about scattering at very low momentum transfers corresponding to length scales larger than can be probed by any other neutron or x-ray scattering technique. It was recognized as early as the inception of this technique nearly forty years ago that its application is limited primarily by the background intensity in the wings of the DCD rocking curve. Thus, DCD instruments for USAS used multiple reflections in channel-cut monochromator and analyzer crystals to sharpen the rocking curve. This greatly improved performance, but the residual scattering in the wings remained orders of magnitude greater than that predicted by straightforward application of scattering theory. Surface defects, parasitic reflections in the channel cut crystals, thermal diffuse scattering, and parasitic scattering from beam-defining slits have all been suggested as sources of this residual instrument background. In this talk I will briefly summarize the efforts to characterize and control for these effects that have resulted in DCD USAS changing from a curiosity for specialists working with special samples to a generally applicable technique for user facilities.

The Effects of Multiple Scattering on the Analysis of USANS Data

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Small-angle neutron scattering (SANS) and ultra-small-angle neutron scattering (USANS) are widely used to investigate the micro- and nano-structure of condensed matter. However, measurements on materials such as cements, clays etc. are often affected by multiple scattering.

If a substantial number of neutrons experience more than one scattering event within a sample, the result is broadening of the angular distribution profile. Often samples cannot be made thin enough to guarantee single scattering and even in cases where it is possible to make thin samples, surface effects can sometimes lead to results that are not representative of the bulk material. In addition, for many SANS measurements there is no reliable method for determining whether or not a sample is thin enough to produce mainly single scattering. Even SANS measurements from samples with different thicknesses may not reveal the presence or otherwise of multiple scattering.

We will show that the problems presented by multiple scattering can often be overcome by using USANS measurements. The ability to measure scattering intensities at very small angles using USANS allows us to quantify the effects from multiple scattering. Rather than trying to eliminate multiple scattering from SANS and USANS experiments, it is more advantageous to use multiple scattering to extract information from these measurements that would otherwise be impossible, or at least more difficult, to obtain.

SASProFit - Program for USANS Data Evaluation

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The program SASProFit has been developed to address problems specific to double crystal SANS diffractometers. Particularly, it permits to

- treat slit-smeared scattering cross-sections directly, without preliminary conversion to pin-hole geometry,
- take multiple scattering in diffraction regime into account,
- fit simultaneously data taken at different *Q*-ranges or even different instruments, including 2-dimensional SANS spectra measured in pin-hole geometry
- evaluate scattering from anisotropic systems.

On the lowest level, the scattering model is defined in real space as the auto-correlation function of scattering medium projected on the plane perpendicular to the incident beam. Scattering functions in infinite-slit geometry can be then calculated easily for any sample rotation in the scattering plane. The program offers basic set of particle types, either monodisperse or with free size distribution. Optionally, hard-sphere structure factor in local monodisperse approximation can be applied. Data treatment with SASProFit is demonstrated on both simulated data and experimental data from cavities in superplastically deformed ceramics measured at different instruments.

SASProFit is freeware available at http://omega.ujf.cas.cz/~saroun/SAS.

2. Design and Application of the Existing USANS Instruments

First Neutron Experiments with “Bonse-Hart” Channel-Cut Crystals. Applications of the USANS Instrument at the FRJ-2 in Jülich

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We will give a review of the first experiments on “Bonse-Hart” channel cut crystals and will demonstrate -on basis of curves of resolution- the improvements of this technique made up to date at the “Jülich” USANS instrument. Experiments on a model system and some simple applications on material science will conclude this part.

The second part of this talk will deal with a pinhole SANS instrument, which allows a minimum Q of the order of 10^{-4} Å^{-1} after implementation of a focusing mirror optical element. Such instruments obey the criteria of neutron USANS technique. First results from the worldwide first focusing mirror instrument, which was built at the Jülich research center, will be discussed.

The Neutron Bonse-Hart Diffractometer for USANS at NIST

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The Perfect Crystal Diffractometer at NIST's Center for Neutron Research is now in routine operation. The performance of the instrument will be presented. Design measures taken to optimize its performance will also be discussed. The instrument is located on a dedicated thermal neutron beam port and utilizes a vertically focussing pyrolytic graphite pre-monochromator. The channel-cut monochromator and analyzer crystals for the instrument are large Si(220), triple-bounce, crystals to provide a beam, of fixed wavelength 2.4 ± 0.1 Å, with a cross section at the sample position of up to 5 cm x 5 cm. The Q-range of instrument is 4×10^{-5} Å^{-1} $< Q <$ 0.02 Å^{-1}. The peak-to-background is 2×10^{6}. The maximum beam current is 60,000 n/sec. The instrument is part of the NIST/NSF Center for High Resolution Neutron Scattering (CHRNS) with up to 2/3rd of the available beam time to be allocated to outside-user experiments.

Design and Applications of the S18-USANS Instrument at ILL

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The S18 USANS Instrument has been successful in operation for about 5 years. We present the principal design features and recent experiments from different users. The instrument is equipped with two triple-bounce channel-cut perfect silicon crystals in 220 orientation. Wavelength selection between 1.6 and 2.9 Å is provided, but the instrument is routinely operated at 1.9 Å. The thermal neutron supermirror guide H25 offers excellent flux conditions with up to 6000 n/cm²s. This allows also for time resolved measurements and very small sample sizes and even in-situ experiments of material formation processes. A representative overview of selected experiments performed will be given. They range from neutron optical investigations of artificial lattices to industrial relevant materials like tire-components and cellulose fibers. S18 is a CRG-C instrument at the ILL, where 100% of the beam time is allocated to the Atominstitut. External users are encouraged to join the experimental program.

Optimization of the USANS Instruments at ILL, PSI and in Vienna

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Bonse-Hart double-crystal diffractometers (DCDs) with multi-bounce channel-cut crystals show rocking curves that depart dramatically from dynamical diffraction theory in their wings. The intrinsic background is many orders of magnitude higher than the predictions of dynamical diffraction theory. Therefore different ways were tested. In the first step the contamination was eliminated by cutting a groove in the middle of the back plate of the channel-cut crystals and inserting a cadmium absorber in this groove. With this modification an additional suppression of the wings of the rocking curve of about one order of magnitude was achieved. After this, we developed a new design for a DCD. The concept for this new crystal design was to avoid the back reflection and the thermal diffuse scattering. The different steps on the way to produce these crystals are presented in a detailed way. The crystal preparation and the different instruments where these crystals have been tested are also described.

3. New Projects of USANS and TOF-USANS Instruments

The Time-of-Flight Ultra-Small-Angle Neutron Scattering (TOF-USANS) Instrument for SNS

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We present the results of our latest calculations of the TOF-USANS instrument for SNS, which lead to the following conclusions.

- the flux gain factor related to the multi-wavelength performance makes the TOF-USANS flux at a sample position comparable with that for the best reactor-based USANS instruments;
- the TOF-USANS extends the value of Q_{min}, by additional order of magnitude, from $2 \cdot 10^{-5}$ Å^{-1} to ~ $2 \cdot 10^{-6}$ Å^{-1}; this allows measurements of enormously large? agglomerates in condensed materials with dimensions up to ~ 200 µm;
- parallel measurements at different wavelengths will be very helpful for diagnosing of multiple scattering in USANS experiments.

5. Alternative (none Bonse-Hart) Ultra- and Very-Small-Angle Neutron Scattering Techniques

Equipping a SANS Instrument with an USANS Option

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We present a 2D-USANS option aimed at extending the q-range of long baseline SANS instruments to $q \approx 10^{-5}$ Å^{-1} at $\lambda = 10$Å. This option is based on a 2D-multi hole aperture at the begin of the collimator, a 2D-multi lens array near the sample and a neutron camera with about 0.2 mm spatial resolution and ca. 10×10 cm^2 area. In this way, several hundred individual images of typically 0.6-mm width (FWHM) are superposed and the stringent relation between intensity and resolution in standard SANS does not apply. To trade q-range against intensity, multi-hole apertures and multi lens arrays of different lattice constants will be applied. Due to their large F-number of $\approx10^3$, the lenses can be made in a simple economic way. The USANS-option requires gravitational correction by prisms near the multi lens array.

The system may also be applied at pulsed sources: In this case, gravitational corrections can be done by image processing, i.e. without prisms in the beam. No proper achromatic lens system seems to exist at present, and the multi lens array must be replaced by a multi hole array (a pin hole camera system), with sacrifice in intensity. The USANS option should be rapidly (typ. 1 min) interchangeable with standard SANS and can be built and implemented at low costs. A test version is near completion at TU-München. The planning of such an USANS option for D11 at ILL-Grenoble is underway. Optimum intensity would request a large area incoming neutron beam (typ. 10×10 cm^2), well above the present beam area at D11.

Phase Transitions in Colloidal Crystals Studied with the Spin-Echo SANS

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The structure of hard sphere colloidal suspensions is measured at different concentrations using recently developed Spin-Echo Small-Angle Neutron Scattering (SESANS) technique. It is shown that SESANS measures real space correlations ranging from the size of a single particle for a dilute suspension to several particle diameters for a concentrated suspension, glass and crystalline state. Experimental correlation functions are compared with theoretical ones. SESANS is demonstrated to be a powerful tool to study colloidal structures of sub micron.

6. USANS Application in Materials & Polymer Science

USANS Study of Bends of Linear and Branched Polyethylene

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Polyethylene (PE) is produced in many forms, each of which has different properties. High density PE (HDPE) is the most crystalline form, because the chains contain very little branching. Typical low density PEs (LDPE) contain both short chain branches (1-3 per 100 backbone carbon atoms), as well as long chain branches (0.1 - 0.3 br./100° C). Linear low density PE (LLDPE) is produced by co-polymerizing ethylene with an alpha-olefin such a hexene and can have a wide range of branch contents. The properties of the individual species can be altered by mixing the components and blends of HDPE, LDPE and LLDPE are widely used commercially. However, understanding of the mechanical and melt flow properties of such blends is handicapped by the absence of a consensus concerning the melt miscibility of the components. For example, electron microscopy and thermal measurements on rapidly quenched (solid) samples have been interpreted as reflecting liquid-liquid phase separation in the melt, whereas pinhole small-angle neutron scattering (SANS) experiments, which probe the melt directly, indicate that HDPE/LDPE blends are homogenous. Similarly, SANS indicates that mixtures of HDPE and short-chain branched polyethylenes, such as LLDPE are homogenous in the melt when the branch content is low, but phase separate, when the branch content is higher (> 10 br./100C).

However, it has been claimed that these experiments do not provide unambiguous evidence for a 1-phase (homogenous) melt for HDPE/LDPE blends, and that the data might also be interpreted as arising from a bi-phasic melt with very large (micron-sized) particles. The previous experiments were performed with a maximum spatial resolution ~ 103Å, so if the domains had micron-size dimensions, much of the scattering from the different phases would be undetectable. We have addressed this hypothesis via a new ultra-high resolution (USANS) instrument9, which increases the spatial resolution to ~ 30µm. The experimental results confirm that HDPE/LDPE blends are homogenous in the melt on length scales probed by pinhole SANS and also by USANS; we have also studied a blend of linear (HDPE) and short-chain branched polyethylenes. It has been shown that USANS can directly resolve both the size of dispersed phase (~ 4µm), and the forward cross section, which is six orders of magnitude higher than for homogenous blends, thus confirming the conclusions drawn from previous SANS studies.

USANS and SANS Study of the Structure of Vycor Glass

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The extensive application of porous solids in separation and catalysis technology requires a thorough determination of their pore structure. The commercially available porous Vycor glass (PVC) has been widely used in the above-mentioned applications as well as in fundamental studies of the influence of pores on the behavior of confined fluids. PVC is obtained from a borosilicate glass-forming melt which has undergone a phase separation, one of the two phases being removed by leaching. As a result, a porous material with nominal pore sizes ~ 40 Å and the pore volume ~ 30% is formed.

The structure of PVC has been widely investigated before by neutron and X-ray small angle scattering in the range of scattering vectors $0.001\ \text{Å}^{-1} \leq Q \leq 0.2\ \text{Å}^{-1}$. Previous investigations were mostly concerned with studying the „Vycor correlation peak“ at $Q \sim 0.25\ \text{Å}^{-1}$ as well as pore surface properties in the high-Q region, and generally neglected an „upturn“ in the scattering at $Q < 0.006\ \text{Å}^{-1}$, which may be indicative of the availability of larger pores. Here we report the results of combined USANS and SANS studies of the structure of Vycor glass in the Q range $\sim 4\text{x}10^{-6} < Q < 2\text{x}10^{-1}\ \text{Å}^{-1}$ and demonstrate the existence of pores with dimensions of the order 8 – 10 μm in this porous matrix. We tentatively interpret the existence of the micrometer pores in Vycor as due to the late stages of spinodal decomposition during the manufacturing process. However, more systematic USANS studies are needed in order to determine the nature of such large pores in Vycor, and such investigations are currently in progress.

Multilevel Structure of Reinforcing Silica and Carbon

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Multiphase materials, phase separated at the nanometer scale, are widely recognized as the materials of choice for advanced applications. These materials, however, are often complex in that they show structural features on length scales extending from angstroms to hundreds of microns. Structural complexity presents a formidable challenge to the morphologist seeking to elucidate the relationship between synthetic protocol, structure and properties. Complexity, however, also opens new opportunities for materials synthesis. By assigning different tasks to different length scales, it is possible to achieve properties exceeding those of simple single-phase analogues. In a sense, physical strategies replace chemical strategies in these new synthetic schemes.

The paucity of instrumentation capable of resolving structures in the 1000 Å range has severely limited our ability to design morphology into complex materials. Numerous small-angle x-ray instruments work well for structure below 200 Å and light scattering often covers the region above 1000 Å. No-man's land, however, lurks between these limits. The development of the ultra low-q neutron instruments opens new opportunities to investigate the forbidden regime around 1000 Å. Neutrons are particularly desirable because of their large penetration depth and susceptibility to contrast matching.

This talk will show examples from the field of nano composites where complex hierarchical structures are often found. Rubber, for example, is typically reinforced with colloidal silica or carbon black. In this case, three levels of structure emerge, primary particles, aggregates and agglomerates, each of which plays a role in materials performance. Nanophase carbon is another example where in reality morphological features are found over 5 decades in lengths scale. Here again, the presence of large-scale structures has evaded adequate characterization.

Nano- and Microporous Hydrogels Constructed via Peptide Self-Assembly

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We are exploring new methods of materials construction via aqueous molecular self-assembly, specifically peptidic molecule self-assembly. By using peptidic molecules in the self-assembly design process, one can take advantage of inherent biomolecular attributes, namely secondary structure and intramolecular folding events, in addition to more traditional self-assembling molecular attributes such as amphiphilicty, to define hierarchical material structure and consequent properties. The self-assembled nature of the resultant material imparts beneficial rheological properties (e.g. shear thinning, self-healing) for ease of processing. The utility in material design with block copolypeptides will be discussed. Hydrogels, with unique nano- and microstructure, vs. membrane suspensions can be built with block copolypeptide amphiphiles relative to the polyelectrolyte character of the hydrophilic block (ionic = gel, nonionic = membrane). USANS has been combined with laser scanning confocal microscopy and bulk oscillatory rheology to provide an interesting structure-property relationship in the gels. Specifically, when gels are formed at polypeptide concentrations high enough for significant rigidity (G' > 1 kPa) the gel exhibits a clear microporous structure that seems to scatter clearly as a surface fractal with well-defined, smooth interfaces (where D = 2 and log (I) vs. log (q) = -4) between the gel matrix and water pores and channels. When the gels are formed at lower concentrations where rigidity is lost, the microporosity is much less defined, and the low q scattering slope drops significantly indicated a roughening/broadening of the gel matrix/water matrix interface.

7. X-ray Session

Application of the Darwin Theory for Structural Study of Surface Layers

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First we review recent developments of Darwin's dynamical theory of diffraction. In his original work, only the diffraction in the symmetric Bragg geometry was discussed and the well-known Darwin curve was obtained. We have developed the theory to be applicable for any incidence condition including the grazing incidence geometry. We have further extended the theory to the multi-beam case.

In the Darwin theory, a crystal is divided into atomic planes parallel to the surface, and the scattering amplitudes by an atomic plane are calculated. Then the effect of multiple scattering among atomic planes is taken into calculation. Therefore the theory is suited for the treatment of crystals whose surface layers are distorted or different from those in bulk crystals. We show the results of recent studies using the modulation of the crystal-truncation-rod scattering under the exciting condition of a Bragg reflection.

USAXS on a Rotating Anode Source

Thomas Rieker

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A Bonse-Hart USAXS instrument on a rotating anode source provides a Q-range of ~ $3\cdot10^{-4}$? $^{-1}$ < Q < 3 $\cdot10^{-2}$? $^{-1}$ and spans about 8 orders in intensity. This Q-range overlaps that of pinhole instruments, making it possible to study materials structure over nearly 4 decades in length scale using a single technique, by combining data from both types of instruments.

We study a wide range of materials at the New Mexico / Sandia National Labs / University of Missouri Small-Angle Scattering Lab. The lab is well known for the work on mass- and pore-fractal materials, enabled by the Bonse-Hart instrument. I will briefly describe our Bonse-Hart instrument, its performance, and recent work on fractal structures in materials.

The UNICAT Ultra-Small-Angle X-ray Double-Crystal Diffractometer at the Advanced Photon Source

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Ultra-small-angle x-ray scattering (USAXS) methods at a third generation synchrotron source are opening up new areas of microstructure characterization in materials science. However, high brilliance and angular resolution, and small beam size, must be coupled with a robust but flexible instrument design. Continuous development by NIST of a USAXS double-crystal diffractometer at the UNICAT sector of the Advanced Photon Source (APS) has resulted in a world-class facility now available for advanced materials research. The emphasis is on absolute-calibrated microstructure characterization, applied contiguously from the nanoscale through the mesoscale range.

The facility consists of an advanced Bonse-Hart camera, fully-automated instrument control, data reduction, and evaluation software packages, and dedicated staff to support users. Current instrument performance parameters include: undulator X-ray source delivering ~10^{13} photons/sec at 10keV on the sample, 7 keV - 19 keV energy range, a Q range from 0.0001 $Å^{-1}$ to 1 $Å^{-1}$, 9 decades of intensity range, primary (standardless) absolute intensity calibration, fluorescence rejection in the scattered beam, a range of beam size from 1.2 mm^2 down to 0.04 mm^2 with a 1-D spatial resolution down to 40 ? m. Instrument capabilities encompass: semi-automated data reduction, analysis using state-of-the-art structure factors and models, anomalous SAXS and USAXS-imaging capabilities. Both 1-D collimated (slit smeared) and 2-D collimated configurations are routinely available.

Examples of application include studies of sintering-resistance in advanced thermal coatings, gradient microstructures in fuel cell layers, nano-particle agglomeration and assembly, hierarchical polymer structures, nano-composites, carbon nano-tube fillers, cement hydration, soil structure, metal precipitation, polymer deformation, creep phenomena in silicon nitride, dislocation structures in aluminum, diesel soot formation, and in-situ nano-particle formation within flames.

Development of this facility continues (e.g., current exploration of high-energy operation) in an active partnership between the instrument scientists and the user community.

USAXS studies at the ESRF

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This presentation will give an overview of the ultra small-angle X-ray scattering (USAXS) studies at the ESRF. The beamline ID2 features both pinhole based and Bonse-Hart USAXS cameras that can be used in a complementary manner. The high flux and low divergence of the undulator beam, together with a high dynamic range detector permit time-resolved measurements in the second range using the Bonse-Hart setup. Recent USAXS studies of structure and dynamics of interacting colloidal systems will be presented. The future possibilities for high brilliance USAXS will be discussed.

8. Application of USANS in Petrology, Archaeometry, Natural & Industrial Sciences

USANS and SANS Study of Sedimentary Rocks

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Sedimentary rocks are complex and somewhat ill-defined "dirty" systems. Following the emergence of fractal geometry in early 1980's and the subsequent significant research using tools as diverse as SANS and SAXS, SEM, optical microscopy, molecular adsorption and mercury injection porosimetry, the microstructure of sedimentary rocks has been deciphered and described in terms of the mix of fractal and Euclidean geometry in the length scale range 0.5 nm to 100 µm. At the same time important theoretical inroads have been made, both into the formal description of the pore space microstructure and understanding the principles of its formation.

Building on these foundations, since mid-1990's we have been conducting research into the petroleum-related aspects of the microstructure of sedimentary rocks: hydrocarbon generation and expulsion in clastic source rocks and coals and, recently, the microstructure of sandstones. SANS, and USANS have been at the heart of these investigations, providing a versatile, non-invasive probe into the rock micro-architecture, generation and migration of fluids and the thermal evolution of the organic rock matrix (for coals). This work has led to the assessment of the generative potential of hydrocarbon source rocks in commercial oil and gas wells, adding SANS and USANS to the routine toolbox of organic geochemistry.

Application of the USANS Technique in Natural Sciences and Archaeometry

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Examples of applications of combined SANS-USANS technique in Archaeological and Natural Sciences will be presented. Among the natural composites of archaeological interest, there is a wide variety of stones used in buildings, monuments, statues and other objects of archaeological or cultural heritage interest. Marble is one of the most common stone used for these purposes; in the Mediterranean basin there are about ten main marble supply districts of antiquity together with a few tens less important. The provenance of stone objects is of key importance to archaeology in so far as artistic, technological or commercial exchange patterns may be studied and correlated to historical events and social contacts between cultures. Authentication of works of art in museums is also of great concern, particularly as a number of rather expensive fakes have been acquired by museums from dubious sources. We have performed a series of USANS and SANS measurements on a large number of marble samples coming from different Mediterranean locations. Preliminary analysis of experimental data has shown that there are essentially two classes of textures. The first one can be seen as a network of fractal clusters formed by solid primary particles while the second one consists of a network of non fractal aggregates built of solid primary particles. Data analysis for the fractal aggregates has been performed using a hierarchical model that essentially describes two regions of the scattering curve. For small values of the momentum transfer the slope of the scattering curve in a log-log plot indicates the fractal dimension of the cluster which is essentially a measurement of the branching of the aggregate itself. For larger values of the momentum transfer it is possible to derive the radius as well as the surface structure of the solid primary particles. Another example of application of combined SANS-USANS techniques is the study of biogenic platforms resulting from a massive overgrowth, at the tide level, of cylindrical shells of vermetid gastropods. In the Mediterranean area, the vermetid gastropod *Dendropoma petraeum* is the dominant reef building species along the lower midlittoral fringe. Preliminary observations on macroscopic samples suggested that vermetid growth follow a fractal pattern. In order to reveal the fractal nature of the vermetid formations at mesoscopic and microscopic level, and to study how the different natural conditions may influence their growth, Small and Ultra Small Angle Neutron Scattering (SANS and USANS) seem to be the most appropriate techniques. Preliminary investigation by means of X rays diffraction and scanning electron microscopy have shown that vermetid shells are mainly constituted by calcium carbonate in the form of aragonite. Experimental data concerning both solid sample and powders have shown a power law dependence, indicating a porous structure. Samples are probably constituted by fractal aggregates at different length scales or, in other words, a network of fractal clusters formed by solid primary particles with a rough surface. Experiments have been also performed doing contrast matching of the solid matrix (calcium carbonate) with a mixture of D_2O and H_2O in order to obtain structural information on the porosity of wet sample. One contrast matching condition has been measured and a network of fractal clusters has been observed.

Results from Time Resolved Measurements on Hydrating Cement Pastes

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It has been known for some years that that substituting D_2O for H_2O in hydrating cement slows down the hydration process by a factor of about three. Measurements of the physical properties of cement pastes samples hydrated in H_2O and in D_2O have failed to reveal noticeable differences between those samples. On the other hand, SANS and USANS measurements of cement samples hydrated in H2O and D2O have shown significant differences. To investigate the effect of D_2O for H_2O substitution further we have carried out time resolved SANS measurements on cement samples hydrating in H_2O and D_2O at the USANS facilities at the Hahn-Meitner Institute and the Nuclear Physics Institute at Rez near Prague. To account for multiple scattering, measurements were taken from a number of samples of different thickness. Totally unexpected results were obtained from the D2O hydration measurements. These results which are difficult to interpret probably reflect the complicated nature of the initial stages of the cement hydration process.

9. “Non-Conventional” Application of the Bonse-Hart DCDs

USANS Studies of Artificial Structures

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Various artificial structures have been investigated by means of ultra-small angle neutron scattering at the USANS set-ups at the TRIGA reactor in Vienna and at the high flux reactor of the ILL in Grenoble. Structures with dimensions in the order of µm show a distinct diffraction pattern in the µrad range which can be resolved by the USANS technique based on a Bonse-Hart camera with channel-cut crystals and a related tail suppression system. Diffraction peaks up to the 50th order have been observed from a two dimensional hole-structured silicon plate. For this sample characteristic diffraction maps in reciprocal space have been obtained. Various artificial lattice structures and filament structures have also been investigated. How the measured results relate to the quality of such structures will be discussed. In this connection the investigation of pores in various materials become a challenging topic. The intensity of the diffraction pattern strongly depends on the phase shift differences of the neutron wave between the different parts of the sample. By means of phase matching additional information can be obtained.

A Tunable Bonse-Hart Camera

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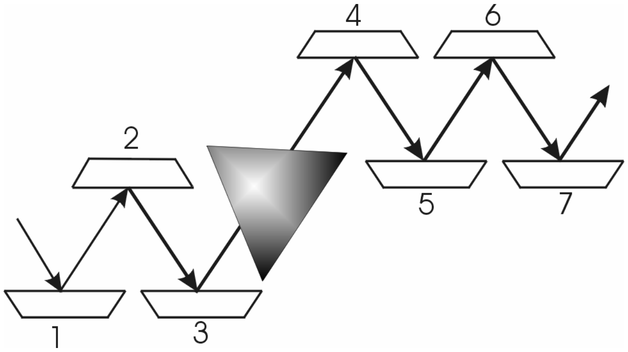
The full width at half maximum of a channel cut crystal (CCC) can be tuned, if the reflection pattern from one crystal slab is shifted by a certain angle relative to the reflection pattern of the following one. One can reduce the resulting Darwin width of such a CCC down to a fraction of the “natural” width and use the narrow reflection curve of such a monochromator and analyser of a Bonse-Hart camera for high angular resolved experiments. To maintain the properties of a CCC a special sevenfold channel cut crystal was developed and successfully tested.



A wedge shifts the reflection pattern relative to the following one and only the common intensity product passes the CCC. So the Darwin width of such a CCC can remarkably be reduced down to a fraction of the “natural” Darwin width.

Tomographic Imaging with a Bonse-Hart Camera

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We used a special Double Crystal Diffractometer (DCD) as a tomography apparatus and investigated refraction and small-angle scattering as imaging signals. The recorded data from this DCD include much more information about the object than one gets from common absorption tomography. This is due to the additional, simultaneous registration of absorption, refraction and small-angle scattering. Refraction data were used to reconstruct the image of samples that showed weak or even no attenuation (absorption) contrast. It was possible to reconstruct images of samples using refraction data only, even in cases where common techniques failed. Small angle data were used to image different concentrations of 150nm particles carotine in D_2O. This new imaging signal offers a number of new possibilities in high resolution non-destructive testing.

Residual Stress and Reflectivity Measurements on Ni/Ti Multi-Layer Films

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It has been shown in our previous studies that the back-face rocking curve (co-called "neutron camel") is extremely sensitive to the ultra-small deformation strain. The residual stress in a thick Si crystal coated with a 2000 Å Ni film can be easily calculated from asymmetry of the back-face rocking curve using the Stoney formula. Thus, these experiments clearly demonstrated a possibility of the residual stress and reflectivity measurements on the same sample of a neutron supermirror or a multi-layer monochromator coated on a thick Si substrate.

We calculated and prepared three samples of the Ni/Ti multi-layer monochromators coated on 8 mm thick Si(220) substrates in order to detect a possible correlation between the reflectivity and the residual stress. The Si(220) substrates were cut and polished at the ANL Advanced Photon Source Crystal Optics Shop; the substrates were coated with Ni/Ti milti-layer films at the S-DH Company, Germany. The reflectivity and residual stress measurements will be done at the National Institute of Standards and Technology, USA.