Poster Session A

A-1 A Prototype of a Multichannel Collimator installed in a SANS Instrument: Test Results.

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Both analytical [1] and computer simulation work shows that the performance of typical small angle neutron scattering instruments can be improved by using a properly designed converging multichannel collimator. A prototype of such a collimator has been designed. The prototype will be installed at a small angle neutron scattering facility for test work. The present paper will report on and discuss test results and introduce guidelines for the implementation of multichannel collimation. [1] Margaça, F.M.A., Falcão, A.N., Salgado, J.F. and Carvalho, F.G., Physica B 276-278 (2000) 189

A-2 Renewal of the D11 collimation

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The collimation of the incident beam is carried out at D11 by a system of moveable glass guides. All mechanical components are under normal operating conditions heavily used and degradation is particularly critical for the original parts of the instrument ($\sim$ 30 years old). Therefore the D11 collimation system will now be completely renovated within the ILL Millenium programme. The project includes a replacement of all neutron guides by new glass guides of cross section 30x50 mm over the whole lengths of 38 m. Two new intersections will be introduced: (i) with a new collimation distance of 27 m away from the sample a detector position intermediate between 20 m and 36.7 m can be used with optimized flux conditions. (ii) the refurbishment of the sample area permits installation of a another new collimation distance at 1.5 m away from the sample which will lead to a considerable flux gain at the shortest detector distance (L=1.1 m). This flux gain is particularly important in view of the new generation of the fast 2 MHz SANS detectors for D11 & D22, which are also developed within the ILL Millenium Programme.

A-3 Development of a neutron material lens and prism based on neutron refractive optics

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We have developed compound refractive lenses for cold neutrons. To prevent an increase in neutron absorption with beam size increasing, we have developed Fresnel lenses using the electrolytic in-process dressing (ELID) grinding technique. The lens characteristics were carefully investigated with experimental and numerical simulation studies. The lenses functioned as neutron focusing lens, and the focal length of 14 m was obtained with a 44-element series of the Fresnel lenses for 1 nm neutrons. On the other hand, we have developed a neutron prism. This development will lay the important groundwork for the next characterization of the two-dimensional compound refractive lenses; moreover, the neutron prism will bring us a new application for energy analysis in the Time-of-flight neutron scattering instruments.

A-4 Performance of the PRISMA straight supermirror guide system

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The primary spectrometer of the PRISMA instrument at the UK ISIS Facility has been rebuilt to incorporate a straight, converging supermirror neutron guide system. The impressive measured flux gain factors of up to 10 for incident energies 2-30 meV are consistent with results of Monte Carlo modelling, and the low background and good resolution of the instrument have been retained. Additionally, the incorporation of adjustable beam optics such as a variable aperture disc chopper, interchangeable collimation and variable size beam apertures have resulted in an instrument with considerable flexibility. The success of the upgrade demonstrates that with careful design, short, straight guide systems are a viable option for spallation source instrumentation.

A-5 Engineering design of the PRISMA supermirror guide system

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The converging, straight, supermirror neutron guide system recently installed on the PRISMA spectrometer at ISIS was required to maintain a low background signal in the instrument whilst enhancing the overall incident beam flux. The straight guide system converges to a focus point at the sample position 9m from the moderator. 2mm thick Ni/Ti supermirror glass components are clipped onto 2m rectangular and cylindrical precision machined steel support structures to enable simple installation in the shutter and target station wall. Frame overlap filters, a T0 nimonic chopper, variable aperture disc chopper, 1m diverging guide, beam monitors, collimators and beam defining jaws have all been squeezed into the remaining 3m. The collimators and jaws are controlled by linear motor systems capable of operating in the high stray fields produced by a cryomagnet at the sample position. We provide a breakdown of the whole system to demonstrate the features included to achieve the project specification.

A-6 Thermal neutron optical experiments with a high resolution double crystal diffractometer

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A double crystal diffractometer (DCD) using perfect crystals as monochromator and analyser usually does not enable experiments on the field of neutron optics which is the domain of neutron interferometry or cold neutrons. However, due to the development of special 7 bounce channel cut crystal (7-CCC) it was possible to investigate the effects of slit and edge diffraction in ultra small angle neutron scattering (USANS) due to a PNR (peak-noise-ratio) of better than 10^5 at the twice FWHM position of the rocking curve. Furthermore with this new 7-CCC the Darwin range

of 100% reflectivity can be tuned down to a fraction of its natural width without a change of the beam geometry which was not possible before. Applications of this new equipped DCD will be given.

A-7 Accuracy Evaluation of Hexapole Electromagnets

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Hexapole magnets can be used as lenses to shape neutron beams. The influences on the neutron optical performance of a hexapole magnet pair in focus to focus configuration in the presence of a bipolar field component due to magnet asymmetry or misalignment of otherwise ideal hexapole magnets with respect to the optical axis are determined by numerical simulation based on ray tracing of individual neutron paths. Radial and axial components of the magnetic flux density have been measured inside the vacuum tube of 10 mm diameter of two hexapole magnets, using a fluxmeter with tangential Hall probe and a holder for positioning the probe with resolutions of 0.5 mm in radial and 5° in azimuthal direction. The results are used for parameter identification of the magnetic field analytical expression in the magnet airgap. The process allows the elimination of errors due to probe holder misalignment and uncertainty of sensitive volume radial position.

A-8 Observation of neutron standing wave by the use of neutron reflectometry

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Permalloy/Ti/Gd layered structure deposited on Si substrate sample was studied by the use of neutron reflecetometer installed at the Budapest Research Reactor. The sample was embedded in gaseous proportional chamber. The radiation products (gamma-rays and conversion electrons)emitted after neutron capture by Gd were registered simultaneously with the reflected neutron beam. Resonances characterizing the generated standing waves were observed. The data are interpreted in terms of the layer properties, i.e. the scattering length density profile.

A-9 A very cold neutron bottle for precise spin interferometry and optics

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We propose a new very cold neutron(VCN) bottle for precise spin interferometry and optics. There are a lot of sequential garland reflections of VCNs on the inside surface of a cylinder. These VCNs are storaged using the gravity effect. The momentum and reflection angles of VCNs in the bottle are approximately defined. This characteristics is different from conventional UCN bottle method. This means that we could control the interactions of neutron spin with magnetic and electric fields by using echo conditions in spin interferometry. In this paper the principle and performances are described and some preliminary performance test results will be discussed.

A-10 Neutron beam control using a magnetic doublet

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The sextupole magnetic field functions as a focusing or defocusing lens for neutrons depending on neutron spin polarity. The focusing effect of a prototype sextupole magnet was experimentally studied and a neutron intensity gain of about 36.5 was obtained. Combining two functions of the sextupole magnet such as focusing and defocusing functions, we can control neutron beam shape and divergence more flexibly. Adiabatic and non-adiabatic field connections make it possible to realize the magnetic doublet system. We realized the magnetic connection for the magnetic doublet and investigated the magnetic doublet experimentally and numerically. In this paper, the details of experimental and calculational results of the magnetic doublet will be discussed.

A-11 Diffraction of UCN on a moving grating and phase modulation of a neutron wave

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The results of the recent experiment for the observation of the ultracold neutron diffraction by a moving grating are reported. The essence of the observed phenomenon is that the moving of the grating with period $2a$ across the neutron beam results in modulation of the transmitted wave with the frequency $\Omega = \pi V/a$ in each point of a beam (V is grating velocity) Then the resulting state is a superposition of coherent waves with a discrete spectrum. Experiment was performed with the Gravity UCN Spectrometer with Interference Filters, that are the neutron analogous of the optical Fabry-Perot interferometers.

A-12 Neutron Spectra at Resonant Tunnelling through Interference Filter

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We investigated the spectra of UCNs tunnelled through the Neutron Interference Filters in a resonance of quasi - bound state. Fore a number of samples we detected the effective shift of the spectra when filter moved parallel to its surface. It was recognized later, that spectrum of the tunnelled neutrons is not defined by the solution of the one-dimensional quantum problem, but distort remarkably by neutron scattering at optical imperfections. Due to the resonant character of the neutron interaction with filter, scattering cross-section increases dramatically, on some order of magnitude. Recently obtained experimental results as well as theoretical analysis are strong ground for believing that phenomena of the line shift at filter moving, caused by resonant scattering of neutrons during the tunnelling.

A-13 Enhancement of reflectivity small d-spacing multilayer mirrors by ion polishing in combination with ion beam sputtering

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One of the most important problems in producing the small d-spacing multilayers is the reduction of the interface roughness that becomes larger with the number of bilayers deposited. We have applied and succeeded in ion polishing in combination with ion beam sputtering deposition for fabricating very small d-spacing multilayers. Ni/Ti and Ni/Mn multilayers were deposited and ion-polished using an ion beam sputtering system. The ion beam polishing was applied immediately after the deposition of each layer to smoothen the surface of layer. The dependencies of ion polishing time, ion acceleration energy and incidence angle on the interface roughness were firstly studied to optimize the conditions of Ar+ ion polishing by using X-ray and neutron reflectometry. TEM observation was conducted on these multilayers. It was observed that the reflectivities and the evaluated interface roughnesses of Ni/Ti and Ni/Mn multilayers with d-spacings of 100Åand 10 pairs were obviously improved by using ion polishing. In the case of ion-polished Ni layers, the evaluated interface roughness reaches a minimum value of 3.5Åat an ion polishing time of 69 sec, ion acceleration energy of 100eV and incidence angle of 10 degree. While an evaluated interface roughness is 7Årms in the case without ion polishing. Smaller d-spacing multilayers were investigated. Ni/Ti and Ni/Mn multilayers with d = 20 - 30 Åand N = 50 - 300 pairs were deposited and ion-polished. The evaluated interface roughness of the multilayer with d = 31.8Åand N = 50 pairs decreases to a minimum value of 4.4Årms treated by ion polishing. Furthermore, the interface roughness is kept to 4.3Årms in the case of d = 29.4Åand N = 300 pairs. On the other hand, the interface roughness decreases to 8.2År.m.s in the case of d = 20Å. It may be concluded that the intermixing of interface takes place by argon ion bombardment when the layer thickness is 20Åand the critical boundary of layer thickness at which intermixing takes place may exist between the layer thickness of 20Åand 26Å. Finally we would like to discuss about its application and the possibility of multilayer mirrors with d-spacing of less than 20Åwhich may realize a normal incidence reflection devices for cold neutrons.

A-14 Diffraction on commercial ruled and holographic optical gratings. Application to the energy analysis of a cold neutron beam.

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We present diffraction measurements on ruled and holographic gratings measured in reflectivity conditions at grazing incidence. The periods of these gratings range between 200 nm up to $50\mu m$. We show that it is possible to obtain large diffraction efficiencies (up to 10%) over a rather large lambda band (1 nm). The diffraction efficiency and lambda band of these gratings can be enhanced by coatings adapted to neutron reflection. We discuss the optimal choices for the periods and materials. These optical gratings are readily available over large surfaces (at the moment up to 50x100mm^2). We discuss the use of such gratings in neutron optics for the energy analysis of a cold neutron beam in specular time of flight reflectivity measurements. These optical devices could greatly improve the efficiency of TOF spectrometers on steady neutron reactors. These measurements have been performed on the TOF reflectometer EROS at the LLB.

A-15 Gain factors with the new supermirror guide system at the Budapest Neutron Centre

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In parallel with the installation of a cold neutron source (CNS) at the Budapest Research Reactor, the neutron guide system has been redesigned and replaced by modern neutron optical elements. MC calculations have been used to determine the optimal conditions for the guide parameters, taking into account the geometrical constraints of the existing infrastructure as well as the cost-effectiveness of the planned replacement. For the 3 cold neutron beams nearly 80 m of new guides were installed, a great part is made of supermirrors. The new in-pile guide system and the individual shutters enable minimal losses at the starting sections. The out-of pile part was optimized for the experimental stations; four instruments are in use so far. The neutron flux measurements were compared with the simulated values. The combined effect of the CNS and the guide system yields a gain factor in the flux as high as 30-60.

A-16 Neutron physical properties of a multiblade velocity selector

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A novel neutron velocity selector has been designed and constructed using the basics concept of the widely used multidisc rotor devices [1]. In this new design the disks are replaced by ”wheels” with a large number of thin blades on the peripheries. These blades coated with neutron absorbing material form channels for the path of neutrons with the proper velocity. The distance between the 10 mm wide wheels is calculated so, that the number of wheels should be minimal on one hand, and the trajectories for the non-desired neutrons to be eliminated, on the other hand. This third generation of KFKI-type selectors offers enhanced performance for all the major parameters: transmission up to 90%, wavelength resolution down to 5% and range in-between 0.2 to 5 nm. Neutron test experiments will be presented. 1. L. Rosta, Physica B, 156&157 (1989) 615

A-17 A large angle neutron bender using sequential garland reflections

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We discuss a basic structure and performance of a new cold neutron bender using sequential garland reflections, which can bend a neutron beam with large divergence by large angle. Using this bender at a pulsed neutron source we can distribute cold neutrons (polarized cold neutrons if necessary) to plural spectrometers without the problem of frame overlap.

A-18 Development of Neutron Optical Components at ILL

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The neutron optics laboratory at ILL carries out an innovative research program for the development of neutron optical components.The directions include monochromators:controlled mosaic crystals,improved Heusler crystals and gradient crystals;multilayers:large critical angle mirrors,high efficiency polarisers and neutron waveguides;and polarised Helium 3 gas. Additional programs for the development of refractive lens systems,micro-collimators and simulation techniques are also actively followed. An overview of recent highlights will be given.

A-19 Concept and Realization of a fully configurable and programable Data Acquisition System for the Neutron Scattering Instruments at SINQ

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We present the basic concept and the realization of our fully configurable data acquisition HW for the neutron scattering instruments at SINQ. This system allows for the collection of the different data entities and event related data generated by the various detection units. Further, it offers several synchronization options including a time measuring mode for time of flight determinations. Based on configurable logic (FPGA, CPLD) events at rates up to the MHz range can be preprocessed and transmitted into a programmable on line data reduction system (Histogramming Memory). It is implemented by means of a commercially available VME Power PC module running under a real time operating system (VxWorks).

A-20 Experimental corrections in Deep Inelastic Neutron Scattering experiments for light nuclei

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Deep Inelastic Neutron scattering technique aims at the obtainment of the momentum distribution of each atomic species present in the sample. However a series of nontrivial steps must be followed in order to obtain such a goal. In this work we show that the data processing is especially critical in the case of light nuclei, because the peak-shape critically depends on the incident spectrum, the total cross section of the resonant filter and the detectors efficiency. An algorithm to calculate Multiple scattering and attenuation effects in the sample is presented. Experimental data on a variety of systems are presented and the present corrections are applied on them. Finally a general prescription on data treatment in such kind of experiments is proposed.

A-21 Method of analysis of multiphonon and multiple scattering effects in inelastic neutron scattering experiments

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We present experimental results of inelastic neutron scattering experiments on a Ti-Zr alloy and on polyethylene samples of different shapes, together with total cross section measurements. A method of analysis of inelastic neutron scattering experiments is proposed, aimed at obtaining a correctly normalized density of phonon states. This method makes use of total cross section data as a necessary normalization condition. Furthermore it is shown that with the concurrent use of both techiques, reliable values of the mean-square displacement of the atoms are obtained. This method is particularly useful in the case of incoherent scatterers like the present, where diffraction methods fail to provide such information. The need to ellaborate different strategies for data processing according to the employed spectrometer is emphasized.

A-22 Remote Access and Display of Neutron Data

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With the proliferation of high intensity neutron sources and involvement of scientists from diverse areas, it is important to provide methods for remote access and display of data. We have developed an Integrated Spectral Analysis Workbench (ISAW) that can read, merge or combine, operate on, and visualize large arrays of data. ISAW is written in Java to allow it to run on common user workstations and to facilitate network communications. Recently, we added the ability to remotely access live data made available through a data server running on the control computer. This data server receives UDP data packets from a data sender running as part of the Data Acquisition System and sends TCP data packets requested by ISAW clients running on user desktop systems.

A-23 The determination of the instrumental corrections in experiments with polarized neutrons

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For scattering experiments which use polarized incident neutrons and analyse the polarization of the scattered neutrons, a two-by-two matrix method is introduced for the determination of the instrumental correction factors. The parameters which describe the efficiencies of the polarizer, analyser, and spin flippers, are defined slightly unconventionally so as to result in a system of equations which is easily solvable even in the general case. In the present method the intensity losses are also considered. In real experiments, a calibration measurement in which the sample is absent or replaced by a dummy that does not influence the spin state of the neutron beam is necessary for the determination of the correction factors. The present method, due to its straightforwardness, is also useful in experiments with time-dependent efficiencies, e.g. those with a polarized ^{3}He gas spin filter.

A-24 Sub-Nanosecond Multi-Channel Time-to-Digital Converter for the Area Detectors at TriCS and AMOR

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We report the development of an ultra-fast multi-channel time-to-digital converter (TDC). The TDC is currently being installed as the front-end readout electronics of the (delay line based) area detectors at the SINQ instruments TriCS and AMOR. The high temporal resolution of the TDC (120 ps) allows the conversion of the neutron-induced detector signals into accurate 2-D scattering coordinates. Preliminary tests suggest that the neutron impact positions can be resolved with an accuracy of about 1 mm.

A-25 Ceramic neutron image plates

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To improve the optical properties of neutron image plates the organic binder used in commercial plates has to be avoided. A new way to synthezise ceramic NIPs containing only the storage phosphor and a neutron converter is presented. Based on a mixture of BaFBr:Eu as storage phosphor and Gadolinium or Lithium as neutron converter the parameters for synthezising NIPs were optimized and the resulting optical properties, such as DQE, spatial resolution and photostimulated luminescence are compared. Gadolinium thereby is used as material with a high neutron capture cross section and Lithium to reduce the g-sensitivity of the phosphor.

A-26 Neutron image plates with low gamma-sensitivity

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Commercially available image plates (IP) for two-dimensional, position-sensitive detection of neutrons comprise a mixture of the neutron converter Gd_2O_3 and the storage phosphor BaFBr:Eu^{2+} dispersed in an organic binder. Gd is used due to the large capture cross section for neutrons; however, owing to its high atomic number it is also a strong gamma-absorber. While this is nonrelevant for high neutron doses, in experiments with low neutron dose at sites with a high gamma-background Li is more applicable as a converter material. In this work we compare image plates containing Li or Gd and show how their properties can be tailored by parameters such as composition, thickness and preparation to obtain a gamma-insensitive, low resolution IP with a high sensitivity to thermal neutrons.

A-27 Development of large-area 2D neutron detector

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In the last decades a new type of detector based on photostimulable storage phosphor was developed. A wide dynamic range with a linear response, a high spatial resolution and a large size of the Image Plate available characterizes the performance of such detector. We present the experimental results obtained for a Neutron Image Plate, commercially available from FUJI, and discuss capabilities of this Image Plate for the purposes of measurements of weak reflexes and diffuse scattering at a thermal neutron diffractometer. The problems of gamma-ray background, image fading effect and an inherent noise are discussed. We discuss our plans for developing of a Neutron Image Plate Detector with smaller inherent noise and reduced sensitivity to gamma-ray background. The latter can be achieved by use of ^{6}LiF as neutron converter instead of Gd_2O_3 (as in commercially available BAS-IP ND, FUJI). It is also important to optimize phosphor to be quite adequate for our needs.

A-28 Large Area Thermal Neutron Imaging Datector with ^{6}Li-foil converter

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We report on the development and first beam test of a thermal neutron imaging detector with a metallic ^{6}Li neutron converter foil and wire chamber based charged particle read out [1]. The advantages of this detector are parallax free imaging, low gamma background, good space- and excellent time resolution as well as high counting rate capability. For the first time, a large area (300 cm^2) metallic ^{6}Li-converter foil with optimised thickness (0.135 mm) for maximum detection efficiency and good mechanical and chemical stability was produced. The wavelength dependent detection efficiency e was measured by TOF (e = 24% at 0.18 nm) and is in good agreement with the calculations. The position resolution is 0,6 mm (fwhm), the time resolution is better than 100 ns. Applications for time resolved neutron radiography and diffraction are discussed. [1] V. Dangendorf et.al., Nucl. Instr. Methods A350, (1994), 503.

A-29 Concept and Realization of 2D and 1D Detector Readout Systems with new Adjustement and Calibration Features

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We present our concept and the realization of the new 2D Readout- System for the CERCA XY 128x128-7.5 neutron detection chamber running on the SINQ SANS Instrument. The adjustment and calibration of the detector response is performed entirely under SW control. This new design includes a time of flight option, too. Based on this successful design we are developing now the 1D TOF readout-system for the POLDI detection unit.

A-30 Calibration of Neutron Linear Position Sensitive Detector

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Linear Position Sensitive Detectors (PSDs) are attractive candidates to become standard neutron scattering detection systems. Modern electronics have provided good stability and spatial resolution for these detectors. Incorporating a bank(s) of PSDs, as an example in a neutron diffractometer, a wide range of angles can be covered inexpensively and at the same time the detectors bank can move close to or away from the sample giving high flexibility to the experiments, i.e. high resolution versus high counting rate for a dynamic experiment. However, these detectors suffer from a disadvantage which manifests itself when spectra collected from different detectors or detector positions do not overlay exactly. This is due to the fact that the usual transformation of detector channels to spatial positions does not take into account the imperfect analog electronic system. A method to transform the detector channels to scattering angles based on both measurements and a theoretical development, which corrects of the electronics imperfections, is presented. The overlay of the same Bragg peak measured from different detectors and/or different angles even at the tails of the peak is better than a few minutes of the arc -much better than the overall resolution of the instrument.

A-31 A New Detector System for the Structure Powder Diffractometer (SPODI) at the FRM-II in Garching

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This article describes the concept of one of the new technical features of SPODI (supported by BMBF under KFZ03-FU5FRM), the Position Sensitive Detector (PSD) array including the read-out electronics. 80 single ^{3}He detectors each with an active length of 300 mm will be arranged vertically in a detector bank. The position resolution enables to evaluate larger parts of the Debye-Scherrer cones and to improve the determination of the 2-Θ positions of Bragg peaks. The set-up of this detector system which is known as Individual Counter Array (ICA) will be discussed. Test measurements with the prototype of the read-out electronics have already been performed. A software package will store the signals in a 2D matrix and allow various evaluation procedures of the data.

A-32 Energy Resonance Detectors for Neutron Scattering in the 1-100 eV Region

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The Resonance Detector Spectrometer (RDS) is a promising instrument for spectroscopy of neutrons with energies above 1 eV at spallation neutron sources. A conceptual design of a RDS instrument is presented here based on the use of (n,gamma) converter foils with a high yield of low energy gammas, and a gamma spectrometer that can be easily incorporated in a detector array. Different choices of converter foils and gamma spectrometers will be compared and discussed especially in terms of their efficiency and background insensitivity.

A-33 Development of 2-dimensional imaging detector based on neutron scintillator with wavelength shifting fibers.

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Neutron detectors with large sensitive area and 2-dimensional imaging capability with good spacial and time resolution play a key role in various experiments with slow neutron beam. We have been developing the imaging detectors for the purpose to evaluate neutron optical devices such as a lens and prism. The detectors consist of neutron scintillator plates (ZnS(Ag)+^{6}LiF, ^{6}Li glass) with the size of 50×50mm, and wavelength shifting (WLS) fibers (Kuraray Y11, B2) with the cross section of 0.5×0.5mm. Two arrays composed of 100 fibers were optically coupled onto both sides of the plate in orthogonal directions to each other. Each end of the fiber array was in contact with the photocathode of a multianode photomultiplier tube, where one end was bundled every ten fibers and the other was gathered every tenth fiber to reduce terminals of light output. In this paper, we report on the development of the detectors and their performance measured with cold neutron beam at the C3-2 beam line of the JRR-3M research reactor of JAERI.

A-34 Two-Dimensional Position-Sensitive Gaseous Detectors for High Resolution Neutron and X-Ray Diffraction

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Two dimensional position-sensitive gaseous detectors have been developed at the Geesthacht Neutron Facility (GeNF) for high-resolution neutron and X-ray diffractometers. They are multi-wire proportional counters with delay line readout filled with Ar/CO_2, ^{3}He/CF_4 or Xe/CO_2 for detecting x-rays, neutrons and hard x-rays, respectively. The detectors have a sensitive area of 300 × 300 mm^2 or 500 × 500 mm^2. The performance of the

detectors are demonstrated for the case of one neutron detector being used at the ARES diffractometer at GKSS which is dedicated to the analysis of residual stresses. Further detectors were used for analyzing textures and residual stresses at the hard X-ray beamline PETRA-2 at HASYLAB. Finally, the design of a novel detector for the neutron reflectometer REFSANS at the research reactor FRM-II in Munich/Germany is introduced.

A-35 Enhancement of TOF diffractometer Sirius with 90° detector bank

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A TOF powder diffractometer, $Sirius$, installed at Neutron Science Laboratory (KENS) of High Energy Accelerator Research Organization (KEK), has realized both the high resolution and the high intensity with its large backward detecter bank and supermirror guide. Sirius also has a large 90° detecter bank (0.54 str. covered by 288 Position Sensitive Detecters), which is used for diffraction study under special environment, such as high-pressure, high-temperature, and so on. Recent works using high-temperature furnace with gas control device (modeled on the Miller Furnace at IPNS), high-pressure cell (Paris-Edinburgh cell) will be presented.

A-36 Formation of glassy storage phosphors

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Commercially available neutron image plates contain a storage phosphor and a neutron converter both in form of crystalline powders. In this study we report on new storage phosphors based on fluoroglasses with the potential advantage of enhanced spatial resolution due to reduced light scattering of the photo-stimulating laser beam. Fluoroglasses are investigated as promising candidates for neutron detection due to their ability for high rare-earth doping and formation of electron and hole traps. First results on scintillation as well as storage properties are presented.

A-37 A novel image plate readout technique for a fast and large area neutron detector

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A new type of image plate scanner is being developed at Agfa medical systems. Instead of using a rotating polygon mirror and a single laser, the new system employs a whole line of laser diodes for stimulation and a CCD line sensor for data readout, very similar to a fax machine. This technique allows for a 300 x 300 mm^2 area to be read in under five seconds with 0.1 mm resolution, 0.05 mm pixel resolution is possible on an area up to 420 x 420 mm^2. Aim of the design is a very compact scanner with a fixed image plate in a cassette that can replace a standard X-ray film cassette in medical systems. This detector appears ideally suited for neutron detection, and TU München and Siemens are starting to develop neutron sensitive image plates for this particular machine. The new Agfa system at least equals the flat panel amorphous silicon detector arrays concerning resolution, speed and dynamic range, will be lower in cost and may be the future standard detector for X-ray and neutron radiography and computed tomography.

A-38 An investigation into modular position sensitive neutron detector systems

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The neutron diffraction facility at the NECSA SAFARI-1 research reactor has been using a commercial linear (10 cm active length, RC encoding) position sensitive detector (PSD) for several years. A recent increase in the number of diffraction stations, as well as the construction of a future Small Angle Neutron Scattering facility, has precipitated a need to implement several additional PSDs. A performance evaluation of the commercial unit over the past several years is presented. An outline of its performance factors (e.g. position resolution, signal stability, signal to noise ratio) and technical problems encountered are discussed. Recent results on the in-house development of a similar type of detector (20 cm active length), as an alternative to the commercial unit, are presented. Implementation of these new detectors in a modular star-like configuration for 2-D operation will be discussed.

A-39 Instrumentation Development at the ILL

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The ILL is currently involved in an important effort of modernization of its instruments ; this require the development of detectors with larger area, faster and more accurate. After a long period of R&D, MicroStrip Gas Chambers can now be considered as a reliable technique; two large area 1D MSGC are currently giving excellent results on 2 ILL instruments and 2D medium and large size MSGC, due to their very good position resolution and counting rate, offer new perspective in neutron instrumentation. Nevertheless, Multi Wire Proportional Counters have not been neglected and at the present time a considerable effort is made to improve their performances, with the recent realization of a 64×64 detector with $3 \times 3mm^2$ cells and individual readout and a project to build a 128×128 detector. A new Position Sensitive counter, 1 m long with an outer diameter of 8 mm has been fabricated to sensibly improve the limit in position resolution of those detectors. There is a growing interest in non-gas detectors, which is represented by the development of a detector based on Lithium scintillators coupled to multi-anode photomultipliers with wavelength shifting optical fibers. In order to respond to new experimental conditions the whole acquisition chain has been redesigned, including a new circuit for the charge amplifier and the discriminator, with reduced sensitivity to EM noise, and a simplified method for center of gravity localisation.

A-40 The high flux backscattering spectrometer (RSSM) for the FRM-II reactor in Munich

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A lot of slow processes in matter, i.e., relaxation processes in polymers, diffusion processes in liquids or tunnelling spectroscopy within molecular crystals require a very good energy resolution while studying which should be in the order of μeV. The new backscattering spectrometer for the

FRM-II (RSSM) reactor uses all possibilities to optimize this type of spectrometer. The good energy resolution is achieved by using a Bragg angle of 90^o not only at the Doppler monochromator (Si111) in the primary but also at the analyser system within the secondary spectrometer (Si111). The large solid angle covered by the analyser system compensates the low intensity which is a result of the good energy resolution. The most advanced components are 1st) a so called Phase space transfomation chopper (PST) and 2nd) a fast Doppler monochromator. The PST incereases the neutron intensity at the elastic energy of 2.08 meV by a factor of $\approx$4 by using the reflection of neutron at a moving crystal (PG002). The Doppler monochromator modulates the energy of the incoming neutrons and gives access to a dynamical range of $\pm 38\mu$eV. We present the latest technical solutions used for the PST chopper and the Doppler monochromator and give an overview about the basic principles and the status of the project.

A-41 Status of the new Structure Powder Diffractometer (SPODI) at the FRM-II in Garching

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The new Structure POwder DIffractometer (SPODI), a project of the Technische Universität Darmstadt and the Ludwig-Maximilians Universität München supported by BMBF under KFZ03-FU5FRM, is currently built up at the new FRM-II neutron source in Garching near München. The design is finished based on an optimisation of the components by Monte Carlo simulations. First devices are ready and tested. This article will give an overview of the whole concept and the status of the main components including the special and new features, selected for the set-up. The realisation of this concept aims at improved resolution, higher intensity and better profile shape.

A-42 Neutron optical considerations for the Materials Science Reflectometer at the FRM-II

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The instrument will use a monochromatic beam of cold neutrons with variable energy in the FRM-II guide hall. Experimental options include evanescent Bragg scattering, beam polarization, polarization analysis and in-situ X-ray analysis. Horizontal and vertical sample arrangements are possible to allow large momentum transfer and accomodate samples with a free liquid surface. We will describe the quantitative neutron optical evaluation of primary beam extraction, " focusing" and collimation based on Monte Carlo simulations (McStas). The performance of the instrument will be demonstrated using standard reflectivity profiles simulated with McStas for different instrument configurations.

A-43 The Novel Reflectometer REFSANS for Analyses of Liquid and Soft Surfaces at the New Research Reactor FRM-II in Munich/Germany

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A novel reflectometer is being built at the high flux reactor FRM-II in Munich. It is dedicated to the analysis of interfaces, phase boundaries and surfaces at the air-water interface of liquid/soft samples. REFSANS will go into operation in 2001 and will be open for national as well as international users. The design of REFSANS as well as new perspectives for biological applications are outlined. The latter result especially from novel neutron optics of REFSANS which will offer new possibilities for analysing diffuse surface scattering and, thus, lateral heterogeneities of surfaces.

A-44 Optimization of a partially non magnetic primary radiation shielding for the triple-axis spectrometer PANDA

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At the new high flux research reaktor FRM-II in Garching the cold triple-axis spectrometer PANDA is under construction. Monte Carlo Simulations have been used to optimize the two sections of the primary radiation shielding of PANDA. The first section contains a neutron guide, a collimator exchanger and a horizontal diaphragm which serves as virtual neutron source. The second section is the monochromator shielding. Special attention has been paid to build a compact and highly efficient shielding, partially non-magnetic, with a total biological radiation dose of clearly less then 10 μSV/h on the outside of the shielding. Specially considered was the determination of the composition of an albedo reducer which is important to minimize the background in the experiment outside the shielding. By using the Monte Carlo program MCNP-4B the destiny of the total spectrum of incoming neutrons and gammas from the beam tube SR-2 have been determined during the 3-dimensional diffusion process in different types of heavy concrete doped with Boron, borated Polyethylen, ^{6}LiF and lead.

A-45 The thermal three-axis spectrometer PUMA at the new neutron-source FRM-II

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Here we present the main features of the thermal three-axis spectrometer PUMA, who's installation on the beam tube SR7 of the new neutron-source FRM-II is well underway. The instrument has been optimised to provide a competitive neutron flux, keeping the configuration as flexible as possible. The variable horizontal slit in the beam-shutter, double-bent monochromators and analyser, a velocity selector as higher-order filter inside the monochromator shielding - these are some of the technical highlights. A versatile sample-table provides the base for all kind of sample environment. The future options include polarisation analysis using ^{3}He spin-filter technique, time-resolved experiments and a multi-analyser-detector unit.

A-46 The Time-of-Flight Spectrometer with Cold Neutrons at the FRM-II

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The progress in design, optimization and construction of the new cold time of flight spectrometer at the FRM-II is presented. The final geometry of the primary and secondary spectrometer is given. The main design criteria were high intensity at the sample position, variable energy resolution (including high resolution), well-defined resolution function and low background. We show in which way these criteria are implemented. Special attention is given to the final prototype of the carbon fibre composite chopper disks. This new material was chosen to obtain the high chopper speeds necessary for high resolution measurements.

A-47 NECTAR - The fast neutron tomography facility at the FRM-II

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At the new research reactor FRM-II a **NE**utron **C**omputer **T**omography **A**nd **R**adiography (**NECTAR**) facility using fast neutrons for transmission measurements is under construction. It will be used for the non-destructive characterisation of industrial and scientific objects having maximum dimensions of 80 cm × 80 cm × 80 cm and mass of 500 kg. The fast neutrons are created by fission in a special converter facility made out of highly enriched uranium. Via beam tube and optimised collimator systems the neutrons are guided to the measuring position. Several filters may be used for manipulating the spectrum. For counting and imaging of the transmitted neutrons different detector systems will be available. An actual status report of the NECTAR-facility will be given.

A-48 The new material science diffractometer at the neutron source FRM II

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By using neutron diffraction the three - dimensional bulk residual stress state and the texture of a material can be determined. Neutron residual stress and texture analyses thus have been establised e.g. as non-destructive testing techniques for large industrial components. Since most material manufacturing and forming processes as well as welding techniques lead to the formation of both texture and residual stresses, a material science diffractometer, which is optimised for both texture and residual stress investigations, is built at the new neutron source FRM II. This new diffractometer is presented and examples for its future application to materials science studies are given.

A-49 HEiDi, Single Crystal Diffractometer at the Hot Source of the FRM II

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HEiDi ist the name of the single crystal diffractometer at the hot source of the new FRM-II neutron source. It will be established at beam line SR-9. The short wave neutron radiation from the hot source is suitable especially for diffraction experiments concerning the following matters: Measurement of Bragg reflections up to very high hkl values gives a significant improvement of accuracy of structural parameters, e. g. anisotropic mean square displacements. This allows the investigation of phase transitions with disorder or anharmonical effects. Further applications are the localization of light elements, e. g. hydrogens, in neighbourhood to heavy elements and compounds with isotopes (e.g. Sm, Gd) with extreme absorption at larger wavelengths, magnetical structures and wavelength dependend extinction effects. On ICNS 2001 advanced details of HEiDi will be presented.

A-50 Reseda, the new NRSE-Spectrometer at the FRM-II

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We present the new resonance-spinecho-spectrometer Reseda at the FRM-II that is near completion. It will cover a time range from some ps to 30 ns and work with two independent analysing arms.As a typical application, an experiment on the itinerant antiferrromagnet chromium is discussed.We demonstrate that the spinecho-technique allows a determination of the space and time correlations near the spin-flop transition at 121 K. The measurements indicate that there is no gap in the magnetic excitation spectrum.

A-51 D4c: a very high precision diffractometer for disordered materials

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The disordered materials diffractometer D4 at the Institut Laue-Langevin has been thoroughly upgraded through improvements in detectors, collimation and shielding to become the D4c instrument. A larger solid angle of detection has increased the total count rate by a factor of 5, thereby reducing random error in the diffraction measurement, and a corresponding factor of 5 improvement in detector stability has reduced the principle cause of systematic error. The overall precision of the instrument has therefore been increased by a factor of 5 as compared to its previous version, D4b. We present an overview of the D4c instrument's design, as well as some results of the successful very high precision experiments performed at D4c since its commissioning in May/June 2000.

A-52 The High Intensity Neutron Diffractometer D20 at ILL : new and future improvements and examples of experiments

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D20 is a variable-resolution 2-axis powder diffractometer at ILL, equipped with a stationary curved linear position sensitive detector (PSD) covering a wide 2Θ range of $153.6°$. Two vertically focussing monochromators, 5 take-off angles and optional Soller collimators provide a large choice in Q-space, resolution, wavelength and flux. The future Germanium monochromator (available by the end of year 2001) will make high-resolution powder diffraction possible, thanks to take-off angles of up to $122°$. The actual copper monochromator will be replaced by two new ones of higher quality and optimised vertical focussing. These monochromators will be used at low take-off angles ($28 \pm 2°$) and to ensure maximum flux on the sample at $44 \pm 2°$ take-off angle. The very high flux available makes D20 an ideal tool for in-situ diffraction studies with time constants even below a second. D20 is adapted to various levels of crystallographic complexity and to kinetic measurements, as will be shown through examples of recent experiments performed on D20.

A-53 IN20B - the high flux polarised neutron TAS.

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As part of the ILL Millenium Programme the primary spectrometer of the IN20 polarised TAS has been entirely renewed. A new beam tube, providing a neutron source diameter increased to 170 mm, has been installed together with an adjustable heavy input slit in front of the monochromator. The increased beam cross-section and divergence is matched by a new doubly focusing Heusler monochromator of unprecedented size 230 x 150 mm ^{2}. The polarised flux gain factor of 5-10 is expected in the commissioning tests in late April. In combination with the already implemented horizontally focusing analyser, the data collection rate will be boosted by almost two orders of magnitude as compared to the original IN20, representing the state-of-theart in the 90's.

A-54 The small-angle neutron scattering instrument D22 at the ILL

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Since its commissioning in 1995, the small-angle neutron scattering (SANS) instrument D22, with a detector of nearly 1 square meter and 128x128 pixels, has been in permanent use for five years. The D22 SANS facility provides the highest constant flux at the sample in a wavelength range of 0.45 to 4 nm. With such intense flux it is feasible to perform experiments on extremely weakly scattering samples (e.g. thin-film magnetism), or time-resolved studies (¡100 ms) using stopped-flow or flash-light devices. A project in the context of the ILL Millennium Program, and already far in advance, aims to install a new detector capable of counting at least 2 MHz with dead-time losses of not more than 10% (cf. 50 kHz for the current detector) while leaving the other parameters unchanged.

A-55 Development of a very cold neutron spin interferometer at the ILL

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We are developing a very cold neutron (VCN) spin interferometer for investigations in neutron optics and spectroscopy. Using polarizing neutron mirrors, we can separate in space and recombine the two spin states. Such a set-up allow performance tests of special polarizing neutron mirrors dedicated for neutron spin interferometry and a new neutron spin echo spectrometer. As a first step towards such a powerful spin interferometer, two mirrors and a precise slit system have been installed at the PF2/VCN beam position at the ILL. In this paper first VCN spin interference patterns will be presented and prospects for future applications will be discussed.

A-56 ILL's renewed thermal three-axis spectrometer IN8C: a progress report

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The IN8C project - the renewal of ILL's thermal three-axis spectrometer IN8 - aims to increase the monochromatic flux at the sample position and at the same time to reduce the background level of the instrument. This is possible using large double focusing monochromators, short distances as well as a wider beamtube, and adopting a beam geometry with a horizontal and vertical virtual source. Also the instrument's flexibility will be improved by enlarging the range of accessible scattering angles at the monochromator and sample position [1]. ILL has undertaken the IN8C project in collaboration with its Spanish scientific partners. The monochromator protection and focusing mechanics have been manufactured and successfully tested in Spain. The instrument is now being installed on site, implying significant building work in the H10 experimental zone. Instrument commissioning should start before the end of this year. Besides the scientific case, the expected experimental performance and the technical concepts we will present the current status of the project focusing on the most recent achievements during factory trials and installation on site. We will also discuss several additional instrumental options aiming for a further increase in experimental flexibility. [1] for details see: A. Hiess et al., Physica B276-278 (2000) 91-92

A-57 The new ILL strain imager

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The neutron strain imaging technique covers a wide range of applications in basic, applied and industrial research and materials testing. Accordingly an instrument for the determination of mechanical stresses must be extremely flexible. The ILL is constructing such a strain imager in collaboration with the University of Manchester and partially funded by the EPSRC. Special features of the instrument will be: a hexapod for the precise and most flexible positioning of specimens up to 1000 kg and 2 m length; beam optics that inhibits the surface error; a big wavelength range of 1.3Åto

4.5Å; a set of monochromators including a double focusing one; and a super mirror neutron guide. The lateral resolution will be suitable as well for measurements at interfaces and surfaces. The paper describes the design and the basics of the individual components of the instrument.

A-58 New perspectives on the IN5 time of flight spectrometer

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The cold source time of flight (ToF) spectrometer IN5 at the ILL is being upgraded since January 2001. IN5 stayed nearly unchanged since the seventies. In the meantime, similar instruments were developped on less powerful sources making the modernization of IN5 necessary. As a first step the primary spectrometer is concerned: the neutron guide and the chopper system. The challenge was to take advantage of the 20cm high beam coming out of the reactor and to focus it to a $\sim 2 \times 5cm^2$ at the sample position in order to gain in flux, while keeping the high versatility which was part of IN5 success. We use a converging guide in both directions. The mirrors are either natural Ni or supermirror up to M=3 coated. The chopper system is composed of 3 pairs of counter rotating disks: 750mm diameter for the first pair and 690mm for the others, turning at a 17000 rpm speed. The expected gain in flux is between 4 and 5 for the guide alone and 7 and 10 at equivalent resolution.

A-59 A neutron diffractometer with an adjustable in-pile fan collimator for focusing in reciprocal and real space

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In the past a diffractometer was developed at HMI for focusing in real and reciprocal space by illuminating a wide horizontally curved monochromator by neutrons transmitting an in-pile slit and reflecting them to the sample. Focusing in the scattering angle 2Θ is achieved by adjusting the distance between monochromator and sample thus establishing an appropriate correlation of the neutrons directions with their wavelengths. We present a modification of the focusing design where the neutron slit is replaced by an adjustable fan collimator which allows to control the correlation with the fan opening. The improved performance with respect to resolution and intensity in an experiment with fan geometry will be compared to the slit geometry and the conventional diffractometer. First measurements show that enhancements of the intensity up to a factor of 5 are feasible for small samples compared to a conventional diffractometer with Soller type collimators without losing 2Θ resolution.

A-60 Refurbishing a thermal three axes spectrometer at BENSC

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The thermal spectrometer E1 of the Berlin Neutron Scattering Center (BENSC) at the Hahn-Meitner-Institut Berlin is presently equipped with 3 beam channels at fixed monochromator scattering angles only. A continuous variation of the incident wavelength is so far not possible. To convert E1 into a state-of-the-art three-axes spectrometer a new monochromator shielding is planned. Presently, a feasibility study is under way for to find the best design for a new shielding which will allow one to change the monochromator scattering angles between 20 and 90 degrees continuously. Shielding properties and neutron will be optimised by Monte Carlo simulations. The use of a velocity selector for higher order suppression and of a fan collimator will be discussed.

A-61 New evanescent wave surface diffractometer at the LLB

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A new surface diffraction spectrometer has been mounted on the reflectometer EROS at the Laboratoire Léon Brillouin. The originality of the design lies in the fact that we are working in a Laue type configuration. The sample is mounted horizontally on a goniometric table. A Position Sensitive Detector (200x100 mm^2) can be swept around the sample in the horizontal plane. The sample is illuminated by a white beam hence no flux is lost and the wavelength spectrum is selected by the sample itself and determined by the sample mosaicity. Time of flight measurements are also performed to check the wavelength accuracy. Preliminary measurements on 1 cm^2 samples show that the intensity in the surface diffraction peaks are about 1-2 counts per second. Measurements have been performed on non-magnetic crystals and on magnetic epitaxial thin films.

A-62 Chopper time-of-flight powder diffractometer

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In order to demonstrate the performance of the Time-of-Flight Monochromator principle which has been introduced for the instrumentation at Long-pulse spallation sources, a prototype instrument has been set up for powder diffraction at the KFKI reactor in Budapest. Results obtained at a reactor source can readily be scaled to apply for a long-pulse spallation source. A double chopper produces pulses of 10 s FWHM with precise triangular lineshape, or 0 to 200μs FWHM (triangular or trapezoidal). The resolution is wavelength dependent, e.g. near backscattering, Δd/d= 0.001 for d= 0.1 nm and pulses of 10μs. Measurements of a sample of sintered alumina with high resolution show, that a TOF powder diffractometer performs far better than a conventional high resolution diffractometer with monochromator and collimators. Since in high resolution powder diffraction the peak width is often broadened by sample properties (e.g. strain, grain size or inhomogeneities), a considerable gain in intensity can be achieved by relaxing the resolution of the instrument, which is done for instance by changing the chopper phases. Single crystal reflections can also be investigated easily with high resolution. As an example the reflection (002) of pyrolytic graphite was recorded. It shows a lorentzian line shape with a width Δd/d= 0.0032.

A-63 Small-Angle Scattering Studies of Polymeric Membranes

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Polymeric membranes are used in industrial and analytical separation techniques. Our aim is to study the dynamic coagulation/aggregation processes which take place in the initial state of membrane formation and which have basic influence on structure and properties of the membranes. Small-angle scattering with neutrons (SANS) and synchrotron radiation (SAXS) proved to be suitable for the characterization of this kind of structures in the nanometer size range. As model system we chose the spinning of a hollow fiber membrane, enabling us to analyze the dynamic processes of membrane formation by measuring at increasing distances of the spinning nozzle. In preliminary small-angle scattering investigations diverse polymeric membranes were characterized by size distributions calculated from the resulting scattering curves.

A-64 Modulated Structure Formation in Microphase Separating Binary Paraffin Mixtures

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On cooling from the molten to solid state, binary n-alkane mixtures can spontaneously form stable solid solutions at one extreme and, at the other, fractionated solids in which the components form two pure (macrophase-separated) phases. In between, some binary mixtures of n-alkanes undergo demixing in the solid solution, with the molecules slipping along their long axes to separate into lamellar components, to form intermediate (microphase) structures. We have investigated the phase behaviour of binary C_nH_{2n+2}:$C_{36}D_{74}$ systems for n equal 20 to 34 using small-angle X-ray and neutron scattering (SAS) and showed that microphase formation is more general, with the rate of demixing being determined by chain-length mismatch. Recently, we have focussed on the $C_{28}H_{58}$:$C_{36}D_{74}$ system since it not only displays the fastest rate of microphase formation but also possesses the largest mismatch for which a microphase is formed without significant precipitation of the longer-chain component. We present here the SAS from recent experiments in which these mixtures were studied as a function of composition and provide evidence for the formation of incommensurate modulated structures.

A-65 SANS studies of critical phenomena in ternary mixtures

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Quasi-binary liquid mixture of 3-methyl pyridine with heavy water was investigated by small-angle neutron scattering. Addition of moderate amount of different salts change the phase behavior of the system, causing expansion or shrinkage of the closed immiscibility loop. The crossover region between the Ising-type and the mean field critical behavior was studied, preliminary results will be presented.

A-66 SANS study of micellar aggregation of multi-headed surfactants

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This work deals with the study of aggregation properties of recently synthesized novel single-chain surfactants bearing one, two and three head groups. Small-angle neutron scattering (SANS) studies in aqueous solutions of these surfactants show that micelles become dramatically smaller in size with the increase in the number of head groups. The hydrocarbon chains in these micelles no longer remain in extended conformation to accommodate the increase in the number of head groups. More interestingly, unlike single-headed surfactant where the micelles grow on addition of salts e.g. KBr and sodium salicylate, it is seen that sizes of micelles of multi-headed group surfactants are independent of these additives. These studies thus demonstrate that the micellar properties of surfactants could be remarkably influenced by manipulation of the charge densities at the head group.

A-67 The Length Scale Dependence of Strain in Networks by SANS

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We present a SANS study on the length scale dependence of chain deformation by means of a suitable labeling in dense, crosslinked elastomers of the HDH-type. This lenght scale is controlled by the size of the label as well as the crosslink density. The results are compared to long homopolymers. The data are analyzed by means of the tube model of topology in rubber elasticity in combination with the Random Phase Approximation (RPA) to account for interchain correlations. Chain degradation during crosslinking is treated by the standard RPA-approach for polydisperse multicomponent systems and confirmed in an in-situ study by both SANS and shear-rheology. Current models of rubber elasticity could be distinguished naturally, solely basing on the gradual importance of chain interactions. The transition from locally freely-fluctuating to tube-constrained segmental motion was investigated without sacrificing the mechanical quality of the rubber.

A-68 SANS study of micellar behaviour of pluronics in aqueous salt solutions

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Small-angle neutron scattering (SANS)measurements have been carried out from the aqueous solutions of pluronics F88 and P84 in presence of salt KCl. Pluronics are PEO-PPO-PEO tri-block copolymers and the amount of PEO in F88 is much more than that in P84. The measurements were done for the fixed concentration (5 wt%)of the pluronics and with varying concentration (0 to 300 mM)of KCl. The results are compared with the

effect of increasing temperature (25 to 70°C)on the pluronic solutions. It is found that the effect of addition of salt is similar to that of increasing temperature. The pluronics are as unimers at low temperatures and their micellization takes place when the salt is added or the temperature is increased. The structure of micelle depends on the salt concentration and the temperature of the solution, and the structural changes in the micelles are different for F88 and P84. The measurements have also been carried out to compare the effect of different salts of the lyotropic series on the above pluronic systems.

A-69 SANS ANALYSIS OF PERFLUOROPOLYETHER WATER IN OIL MICROEMULSIONS BY HARD SPHERES AND ADHESIVE HARD SPHERES POTENTIALS

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Ternary water-in-oil microemulsions composed by an ammonium carboxylate PFPE surfactant of molecular weight 710 and a PFPE oil of molecular weight 900 leading to microemulsions that show a dynamic percolation phenomenon (Phys. Rev. vol. 56, 4356 (1997)) were investigated by SANS to characterize the microstructure. Hard sphere and adhesive hard sphere potentials were used to model the droplet - droplet interactions supposing the droplets polydispersed spheres, with a Schultz distribution of sizes. The validity of the model is discussed in relation to the previously characterized percolation phenomenon.

A-70 Interfacial Characteristics of Tapered Block Copolymers Studied by Neutron Reflectivity Measurements

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Microphase-separated interfaces of tapered block copolymers were investigated in comparison with that of regular block copolymer by neutron reflectivity measurements. Samples are anionically-polymerized two styrene-d8-isoprene tapered block copolymers with two different tapered region and a regular diblock copolymer all with total molecular weight of around 40k and polystyrene content of 50 vol.%. Sample films with about 80nm thickness were spin coated on silicon wafers from dilute solutions of samples in toluene at 2000rpm. Reflectivity apparatus used is a new pulsed-neutron reflectometer (ARISA) with a vertical scattering-plane geometry installed at one of the thermal neutron port of KENS. It has been found that the interfacial thicknesses of the microphase-separated tapered block copolymers are considerably thicker than that of a regular block copolymer.

A-71 Relaxation of entangled model H-shaped polymers: a SANS investigation

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This study is related to the understanding of rheology of long chain branched polymers. A model compound for long chain branching (H-shaped) was investigated in elongational flow as a function of time after a step strain to λ=2. The experiments were performed in a strain rig with temperature and strain rate control. The structure factor was measured after specific relaxation times intimately connected to the microscopic hierarchy of the polymer structure. A description of the correlation hole effect and quenched disordered in the scattering in the RPA approximation was adopted from the similarity with permanent rubberelastic networks and modified to permit the observation of strain locally along the faster relaxing arms. The data are consistent with the time scale of linear shear rheology from which shift factors were derived. They confirm the fact that backbone and arm relaxations can be treated in a decoupled, hierarchical way in time.

A-72 Segment-segment interactions of poly(*N*-isopropylacrylamide) in aqueous methanol solutions by using small-angle scattering

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SANS and SAXS from semi-dilute solutions of poly(*N*-isopropylacrylamide) (PNIPAM) in aqueous methanol solutions have been measured in poor solvent regime and determined the binary and ternary cluster integrals, B_1 and B_2. The systems for SANS measurement were PNIPAM in D_2O and deuterated methanol in D_2O mixtures just below LCST. SAXS measurements were made in methanol and 70% methanol at 30 and 40°C. SANS and SAXS measurements were performed with KENS-SAN spectrometer, and with an Anton Paar Compact camera. In SANS measurement, the values B_1 and B_2 were negative for each system. We evaluated the contribution of segment-segment interaction of the entropy S_{int} and the enthalpy H_{int} . In the measured temperature range, S_{int} and H_{int} were positive and those values decreased with increasing methanol content. This means that the special interactions such as hydrogen bonding polymer between segment and solvent molecule and hydrophobic hydration exist in the solution. The cononsolvency occurs due to the decreasing of H_{int}. Temperature dependence of B_1 and B_2 obtained from SAXS measurement was not observed, and both values were positive. Therefore S_{int} and H_{int} became negative. In this condition, hydrogen bonding between segment and solvent molecule and hydrophobic hydration were negligible.

A-73 Polymer Boosting Effect in the Droplet Phase Studied by Small Angle Neutron Scattering

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Microemulsions consisting of water, oil, and non-ionic CiEj-tenside are well established model systems. On the microscopic level, the tenside forms a film between the otherwise non-miscible water and oil. We investigated the one phase region, where oil droplets are dissolved in water. The addition of amphiphilic block copolymers increased the miscibility of oil in water (boosting effect). Different poly(ethylene-propylen)-poly(ethylene-oxide) diblock copolymers were investigated, such that one polymer was attached to one droplet. We used the contrast variation method to obtain the 6 partial scattering functions. One polymer showed a weak boosting effect. Due to the depletion interaction, the polymer

avoided the vicinity to the droplets, and was homogenously distributed beyond a certain radius. Another polymer lead to a substantial increase of the droplet phase. First SANS studies showed differences to the weakly efficiency-boosting polymers.

A-74 SANS Study of Structural Evolution in NIPA/SA Gel on Dehydration

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Gels which have hydrophobic and hydrophilic bases in a network show interesting behaviors, such as the volume phase transition (Tc). N-isopropylacrylamide/sodium acrylate (NIPA/SA) gel belongs to this kind, in which NIPA-base is hydrophobic and SA-base is hydrophilic above Tc .Recently, occurrence of a microphase separation in the dehydrated NIPA/SA gel was observed by a small-angle x-ray scattering (SAXS) experiment. To understand the mechanism of the microphase separation, it is necessary to investigate evolution of water distribution in the NIPA/SA gel during the dehydration process. Therefore, the authors conducted a small-angle neutron scattering experiment with decreasing water content of the gel, in which the SANS profile evoluted with three stages: in the beginning the intensity around q=0Å^{-1} increased, then a broad peak around q=0.02Å^{-1} emerged and became intense, however, the trend switched to decrease at the last stage. This result indicates inhomogeneous water evaporation through the different hydrophilicity between NIPA- and SA-bases.

A-75 SANS Aanalysis of Aqueous Ionic Perfluorpolyether Micelles

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The behaviour of aqueous ionic perfluoropolyether micelles was investigated by SANS. The surfactant molecule is composed of a fluorinated tail of chemical formula Cl-$(C_3F_6O)_n$-$(C_2F_4O)_m$-$(CF_2O)_q$-CF_2- (where n>>m and q close to zero) and a carboxylate head group with ammonium or potassium counterion. The role of concentration and temperature are studied. SANS data were analysed assuming a two-shell model for the micellar form factor. The structure factor is the result of a screened Coulomb repulsion in addition to hard - sphere repulsion.

A-76 Conformation of Cyclic and Linear Poly(Dimethylsiloxane) in the Melt: a Small Angle Neutron Scattering Study

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Despite the wide range of experimental data available for cyclic polymers in dilute solution, the static and dynamic properties of ring polymers in the melt remain largely unexplored. To date, no experimental study of the radius of gyration of rings in bulk has been reported. Ring polymers differ considerably from linear chains, as they cannot interpenetrate as their linear counterpart. It has been suggested that due to the presence of topological constraints, rings in the melt may be more compact than Gaussian chains. We report SANS measurements of cyclic and linear PDMS in the melt. We show that the cyclic chains are partially collapsed and do not follow Gaussian statistics. We find that $Rg \propto N^{2/5}$, a result recently confirmed by computer simulations.

A-77 Small-Angle Neutron Scattering Study on the Aqueous Mixtures of Short-Arm Fullerene-based Star Ionomers and Sodium Dodecyl Sulfate

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The synthesized water soluble fullerene-derivatives $C_{60}[(CH_2)_4SO_3Na]_6$ (FC4S) and $C_{60}[CO(CH_2)_5O(CH_2)_4SO_3Na]_6$ (FC10S) have potential biomedical applications such as free radical scavenging or antioxidant-action. In our previous studies, using SANS and SAXS, we found that FC4S formed globular aggregates in aqueous solutions and FC10S form rod-like aggregates [1]. These aggregates have porous structures that might be able to adsorb other molecules. By using SANS and SAXS, the FC10S and SDS complex aggregates were found to have a cylinder-like shape. It is found that there is a saturation limit of the number of SDS molecules that can be adsorbed into the aggregates of FC10S and the ratio of adsorbed SDS to FC10S is about 0.7 to 1. In this paper we will report our small-angle neutron scattering studies on the mixtures of FC4S and SDS. Our preliminary analysis showed that the addition of SDS has little effect on the structure of FC4S and some of the added SDS were adsorbed into the FC4S aggregate. [1] U. Jeng, T.-L. Lin, C.-S. Tsao, C.-H. Lee, L.Y. Wang, L.Y. Chiang, C.C. Han, J. Phys. Chem. B 103 (1999) 1059.

A-78 SANS Structural Characterization of Fullerenol-Derived Star Polymers in Solutions

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We have studied the conformation of fullerenol-derived star polymers in two organic solutions using small angle neutron scattering (SANS). The SANS results indicate that the six poly(urethane-ether) (PU) arms chemically bonded on the fullerenol ($C_{60}(OH)_{12}$) of the star polymer have a Gaussian chain behavior in Toluene, whereas these arms exhibit an exclude volume effect in dimethylformamide (DMF) solutions. Concentration (0.04 - 4 wt%) and temperature effects (10-70°C) on the morphology of the star polymer are also studied. We use a scattering model to extract the radius of gyration Rg values and the persistent length of the star polymers from the SANS data. The temperature-sensitive Rg values obtained are compared to the fully stretched length of the PU arms, ∼200 Å , and the linear dimension of the PU arms, ∼120 Å , in fullerenol cross-linked PU films.

A-79 Composites Reinforcement by Rods. A SAS Study.

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The mechanical properties of composites are governed by size, shape and dispersion degree of these so called reinforcing particles. Among them polymeric fillers based on thermodynamically driven microphase separation of blocks of segments offer the opportunity to study a model system for the case of rodlike filler particles. A PBPSPB blockcopolymer was chosen and SAS measurements carried out both with x-rays and neutrons, in order to characterize separately the hard phase and the crosslinked PB matrix. The latter has been modeled through the Heinrich-Straube approach. The properties of the material will depend strongly on the way how stress is carried and transferred between the soft matrix and hard fibers. The failure of the strain amplification concept and the change of topological contributions to the free energy and scattering factor have to be addressed. For this the composite shows similarity to a two-network system, i.e. interpenetrating rubber and rodlike filler network.

A-80 Silica Filled Elastomers: Polymer Chain and filler Characteriztion by a SANS-SAXS Approach.

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We studied composites made up of commercially intersting fillers based on Silica particles and a blend of protonated and deuterated polyisoprene (PI).We varied the filler volume fraction and the applied strain.For the first time the extraction of the SANS scattering function of the polymeric phase in both the undeformed and deformed state has been performed, for a three component system in absence of composition matching.A parallel SAXS study provided information, not accessible to neutrons, on the filler structure.The Koberstein approach was used and modified for the structural fitting of the unlabeled analogue with Beaucage functions.The silica filled rubber shows a response to mechanical strain in the sense of the commonly accepted laws of the hydrodynamic reinforcement.Differently, the microscopical characterization of the single chain under deformation, in the frame of the Heinrich-Straube model, suggests a substantial independence of the reinforcing factor on the volume fraction.

A-81 A Structural Study of Lamellar Phases Formed by Nucleoside Functionalized Lipids

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Bilayer stacks formed of di-palmitoyl-phosphatydil-Adenosine (DPP-Adenosine), DPP-Uridine and their 1:1 mixture were investigated after equilibration in a 98% relative humidity atmosphere, with an external contrast variation (1:0, 1:1, 0:1 H_20:D_2O atmosphere), and of the temperature. A surprisingly different behavior has been found for the Adenosine and the Uridine derivative at 40°C, despite the apparent very similar chemical composition. While for DPP-Adenosine the spectrum can be accounted (in analogy to DPPC) for by a lamellar phase with a smectic period of about 60 Å, DPP-Uridine displays a not so straightforward behavior, that we have tentatively ascribed to coexisting hexagonal and lamellar phases. In the 1:1 mixture the lamellar mesophase of DPP-Adenosine is retained, while the contribution of the supposed hexagonal phase of DPP-Uridine appears not relevant. It should be stressed that this behavior is not ideal, and can be considered as an indication of specific interactions taking place between polar heads, during the recognition process.

A-82 SANS study of three-layer micellar particles

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Three-layer nanoparticles prepared by polymerization of methyl methacrylate monomer in micellar aqueous solutions of polystyrene-block-poly(methacrylic acid) and poly(methyl methacrylate)- block-poly(methacrylic acid) were studied using small-angle neutron scattering. The resulting polymer forms a layer on the core surface of the original micelles. SANS curves were fitted using a model of coated cores of spherical or ellipsoidal shape. The particle size (for the presented examples, 20 - 50 nm in diameter) can be finely tuned by variation of monomer concentration. Time-resolved SANS experiments were carried out to describe growth of the particles during polymerization.

A-83 Polarized neutron study of the magnetic mesostructure in $(Pd_{1-x}Fe_x)_{1-y}Mn_y$.

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The low temperature magnetic behaviour of dilute alloys $(Pd_{1-x}Fe_x)_{1-y}Mn_y$ (y=0; 0.05) was studied by 3-dimensional neutron depolarization analysis (neutron wave length $\lambda = 0.15$ and 0.23 nm) and small angle neutron scattering $\lambda = 0.9$ nm. Alloys with different Fe-concentration (x=0.0035, 0.016) were investigated in order to study the influence of flustration on the magnetization behaviour in the temperature range between 2K and 50K and in magnetic fields up to 40 A/cm. The temperature dependence of the mean magnetization differs from that of both a simple ferromagnet and a re-entrant spin glass. A very complicated behaviour of depolarization was observedin alond the magnetizing/demagnetizing loops. This indecates drastic changes in the magnetic structure on a mesoscopic length scale and a breakdown of FM order with temperature.

A-84 Magnetic Studies in Mesoscopic Length Scale

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Neutron depolarization technique, a powerful probe for carrying out magnetic studies in the mesoscopic length scale, has been used extensively by us at Trombay [1] on a variety of magnetic systems including ferrites, intermetallics and CMR-perovskites. Our findings have shown the necessity and usefulness of such a mesoscopic probe in order to bridge the gap (and hence to resolve the discrepancies) between the results in macroscopic and microscopic length scales [2]. [1] S.M. Yusuf et al.,Pramana-J.Phys. **47**(1996)171; Neutron News **8**(1997)12. [2] S.M.Yusuf et al.,J.Phys.:Condens. Matt. **7**(1995)5891; S.M. Yusuf et al.,Phys. Rev.B **53**(1996) 28; S.M.Yusuf et al.,Solid State Commun. **101**(1997)145; S.M. Yusuf et al., J. Magn. Magn. Mater.**166**(1997)349; S. M. Yusuf et al., Phys. Rev. B **62**(2000)1118.

A-85 Local Susceptibility and Anisotropic Magnetization Parameters in Polarized Neutron Diffraction

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Polarized neutron diffraction provides information about the magnetization density of each individual crystallographic site. In the present paper the role of local susceptibility tensor accounting for the magnetic response of an atom in the external magnetic field will be discussed. The symmetry of this tensor is very similar to those of the tensor Uij describing the thermal motion of atoms. By analogy with the atomic displacement parameters (ADP) the anisotropic magnetization parameters (AMPs) can be introduced. The six independent anisotropic magnetization parameters can be refined from the flipping ratios and visualized in three dimensions as "magnetic ellipsoids" in a way similar to "thermal ellipsoids". In the case of small local anisotropy the "magnetic ellipsoids" can be described as spheres with the diameter proportional to the induced magnetization. In other cases "anomalous" (elongated or flattened) ellipsoids will occur. Different examples of "anomalous" magnetic ellipsoids will be presented.

A-86 Neutron Polarization Analysis Studies of Magnetic And Nuclear Short Range Order In β-Mn Alloys

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While the magnetic ground state of β-Mn is a quantum spin-liquid, μSR measurements demonstrate that β-($Mn_{1-x}Al_x$) exhibits spin-glass order at low temperatures for x>0.09, suggesting that the substitution of Al into β-Mn damps the spin fluctuations in the system, and localizes the Mn moments. Magnetisation data taken on β-$Mn_{1-x}Co_x$ and β-$Mn_{1-x}In_x$ also show evidence of moment-localization. Using full XYZ neutron polarization analysis, we have isolated the nuclear and magnetic diffuse scattering unambiguously, enabling the study of the interplay between the magnetic and atomic disorder in the lattice. For Al and In doped alloys, we find that the magnetic correlations are of much shorter range in the spin-glass region than in the spin-liquid region, indicating that the relief of magnetic geometrical frustration is less important in the determination of the magnetic properties of these alloys than the introduction of chemical disorder in the lattice.

A-87 Neutron depolarization in magnetic fluids during magnetizing/demagnetizing cycles

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The magnetization behaviour of ferrofluids (FF) along a magnetizing/demagnetizing loop was studied by 3-dimensional polarization analysis of the neutron beam transmitted through the sample. The experiment aimed to study the FF structure which the nanoparticles formed under influence of a small magnetic field (significantly smaller than the saturation field). Althrough a hysteresis of the mean magnetization is not observed, a hysteresis in the depolarization occurs and it gets stronger as the nanoparticle concentration is increased. When the applied field is changed stepwise, the depolarization behaviour depends strongly on the step value. A model for the FF magnetic structure is proposed and discussed.

A-88 High Pressure Magnetic Phase Diagram of CeP Studied by Neutron Diffraction

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The reported magnetic phase diagram of the low carrier system, CeP under pressures up to 5.6 GPa shows interesting features of this system. However, there are still several unknown regions in the reported phase diagram, which demand further study. The present work is devoted to fill out this gap along with the investigation of magnetic phase up to the highest ordering pressure. Sapphire-anvil cell was used for generating the pressure with a very small sample ($\sim$ 0.017 mm^3, CeP#2). The experiments were carried out at TAS-1 of JRR-3M reactor in JAERI, Japan. Under the pressure of 2.05 GPa a magnetic structure consisting of ↑,↑,↓,↓ stacking of ferromagnetic (001) planes was found below 43 K. At 2.57 GPa, an anti-ferromagnetic phase (k=1/3) was observed to exist between 31.7 and 45 K, and a ferromagnetic phase was also found below 31.7 K. Experiment with 3.2 GPa shows ferromagnetic phase below 51 K. Further experiment with higher pressure will be performed soon.

A-89 Magnetic P-T Phase Diagram and Magnetic Structures of CeSb

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We have performed neutron diffraction experiments on CeSb up to 4.6 GPa in order to investigate the magnetic order above about 2 GPa where the pressure-induced huge peak of the electrical resistivity appears below about 60 K. The results clearly show that the enormous enhancement of the resistivity, shifting to higher temperature with increasing pressure, is accompanied by the type-I antiferromagnetic order of Γ_8 state Ce ions. Furthermore, the development of type-IA antiferromagnetic order below about 33 K leads to the rapid decrease of the resistivity. We also present the new P-T phase diagram of CeSb, which solves the discrepancy between the reported phase diagram by previous neutron diffraction experiments and that by the resistivity measurements.

A-90 Neutron diffraction study of the $U(Pd_{1-x}Fe_x)_2Ge_2$ magnetic structure under high pressure

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Magnetic structure of $U(Pd_{1-x}Fe_x)_2Ge_2$ at low temperature is strongly depends on Fe doping level x. At x<0.015 low temperature structure is longitudinal spin-density wawe (LSDW), but at x>0.015 the structure changes to simple AF. Theoretical predictions maintains that this transition is connected with a changing of unit cell volume due to Fe doping. This work presents results of low temperature magnetic transitions studies under high pressure at different fixed Fe doping level.

A-91 Neutron diffraction studies of the antiferromagnetic $ZnCr_2Se_4$ and $Cu_{0.5}In_{0.5}Cr_2S_4$ under pressure

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The neutron diffraction studies under pressure of the spinels $ZnCr_2Se_4$(up to 56kbar) and $Cu_{0.5}In_{0.5}Cr_2S_4$(up to 10kbar) at temperatures 1.5-300K have been conducted. In the $ZnCr_2Se_4$ under the pressure of up to 56kbar, the simple spiral magnetic structure is preserved but the period of the spiral changes from Ls= 22Å(P=0kbar) to 15Å(56kbar). The T_N rises and changes with the velocity DN/ DP 1grade/kbar. The magnetic moment of Cr^{3+} decreases under pressure and this value changes from 2.87(0.27) at P=0kbar to 2.26(0.29)μ_B at P=15kbar. The extrapolation Ls=f(P) shows that the period may be equal to the lattice parameter of ≈10Å, and k=[00m] of the magnetic spiral structure may be [001] at the pressure 75 kbar (T=1.5K). The investigation of the $Cu_{0.5}In_{0.5}Cr_2S_4$ (Sp. gr. F43m) under pressure up to 10kbar showed that T_N increases from 39K(P=0) to 49K (P=10kbar). The magnetic structure remains collinear antiferromagnetic (k= [001]) and does not change under pressure.

A-92 Complex magnetic phase diagram of $TmCu_2$

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The orthorhombic intermetallic compound $TmCu_2$ orders antiferromagnetically (AF) below $T_N = 6.5$ K. Bulk studies indicate complex magnetic phase diagram (MPG) in fields along b-axis. Neutron diffraction on single crystal of $TmCu_2$ confirmed the complexity of MPG. Four different AF phases were observed below T_N in zero magnetic field, the ground-state AF1 corresponds to the squared-up AF structure with propagation vector τ_1 = (5/8 0 0) and mg. unit cell $8a \times b \times c$. Four ferrimagnetic phases were found in $B < 0.5$ T parallel to the b-axis and field-induced ferromagnetic phase exists in higher fields. The individual phases were identified and MPG along the b-axis is presented.

A-93 Pressure-induced change of magnetic order in $Tb_{1-x}Y_xNiAl$ and $TbNi_{1-x}Cu_xAl$

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We have studied the influence of hydrostatic pressure on a transition from antiferromagnetic (afm.) to ferromagnetic (fm.) order that occurs in the intermetallic series $Tb_{1-x}Y_xNiAl$ and $TbNi_{1-x}Cu_xAl$ between $x = 0$ and $x = 0.1$, using neutron powder diffraction. The Curie temperature is $T_C = 47$ K. The ternary compound TbNiAl shows afm. order. A critical value of x exists in both series where both fm. and afm. types of domains coexist in parallel. We have already established earlier that applying hydostatic pressure shifts the balance between the two types of domains to favour afm. ones. New results will be presented which suggest that the transition is actually more complicated than previously assumed: Increasing the pressure stepwise in the compound $Tb_{0.97}Y_{0.03}NiAl$, first fm. domains grow, but above $p = 2$ kbar magnetic order gradually changes to afm. At $p = 7$ kbar the transition is completed: no fm. domains are detected at this pressure.

A-94 Field induced phase transition in a type III fcc - antiferromagnet

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In the fcc symmetry the type III structure is stable only when both the JN and JNN interaction are antiferromagnetic. This situation is realised in K2IrCl6 which orders at 3.1 K and is very close to the nearest neighbour Heisenberg case with S=1/2. We have investigated the field dependent ordering and find in B=5T a phase transition from the type III ordering to a multiple q - structure. The neutron polarisation analysis data on the field induced structure suggest that higher order interactions are the reason for the observed phase transition. The relevance of such a contribution

is justified by quantum fluctuations expected due to the frustration of the fcc lattice for antiferromagnetic order and weak next nearest neighbour interaction .

A-95 Magnetic order in the fcc - symmetry: Phase diagram and structure of ReB12

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The rare earth borides (Re=Tm, Er, Ho,...) with the chemical formula ReB12 are metals which crystallise in the fcc symmetry. The electron mediated RKKY interaction can be described by the free electron approximation. The strength of the dipolar interaction is comparable to the RKKY interaction, but the ratio of both interactions varies over the series of Re=Tm, Er, Ho. Our investigation shows that the compounds TmB12 and ErB12 have a simple phase diagram. In HoB12 the dipolar interaction is less relevant compared to the RKKY interaction. There, a more complex phase diagram is found in applied field and neutron diffraction reveals unconventional antiferromagnetic order at q=(2/3, 1/3, 1/3) and (0, 2/3, 2/3). The results are comparable to the nuclear ordering of Cu and Ag and the first principle theories developed for magnetic order in the frustrated fcc symmetry.

A-96 Structure genesis and magnetic ordering in compounds of the $ThCr_2Si_2$ type

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It is shown in a phenomenological approach that the symmetry space group $I4mmm$ of the paramagnetic phase in compounds of the $ThCr_2Si_2$ type arises as a result of a structural phase transition from a close-packed paraphase with space group $Im3m$. It is found that the real magnetic ordering in compounds of the $ThCr_2Si_2$ type is described by transition parameters belonging on a a single direction , along the line joining the points of maximum symmetry in the Brillouin zone of the $I4/mmm$ group. It is shown that the variation of the modulus of the wave vector are a consequence of a change in the dopant concentration. The spatial dependence of the order parameter in the incommensurate phases is obtained for the corresponding universality classes.

A-97 A two phase magnetic structure of $DySi_2$

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Neutron diffraction performed on the polycrystalline binary $DySi_2$ had shown that, at room temperature the crystal structure can be equally described by adopting either the orthorhombic Imma system with the Dy occupying the 4e site and Si the 8h site, or the lower symmetry Pnc2 system with both rare earth and the silicon on the 4c site. At 2K the antiferromagnetic nature of the compound is confirmed. Two distinct magnetic phases are assumed in interpreting the magnetic structure with propagation vectors: the commensurate and the with ordered magnetic moments along the c-axis and with and, respectively. The spin configurations are discussed in terms of magnetic exchange interactions and crystal field effects. [1] K. Sekizawa K. Yasukochi, J.Phys. Soc. Japan 21, 274 (1966) [2] K. Sekizawa , J.Phys. Soc. Japan 21, 1137 (1966) [3] J. Pierre E. Saiud D. Frachon J. Less Common Metals vol.139, no2 p321-9

A-98 Pulsed-neutron diffraction studies of two-dimensional magnetic ordering in $DyRu_2Si_2$

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We have investigated magnetic orderings of $DyRu_2Si_2$ by pulsed-neutron diffraction. Below T_N=29.3 K the magnetic ordering is confirmed to be one-dimensional with Q=(2/9 0 0). However, below T_t=3.5 K a number of magnetic satellites have been observed not only on high-symmetry lines, but also on low-symmetry lines with the fundamental modulation of Q=(2/9 0 0) and its odd harmonics in the $a^\star$-$b^\star$ reciprocal plane. These magnetic satellites show that in this region the magnetic structure is not one-dimensional as previously reported, but described by a model of two-dimensional magnetic ordering with a 18a×18a×c magnetic unit cell, which contains two non-magnetic Dy ions.

A-99 Magnetic structures of some strongly correlated electron Ce-Ni-Ge systems

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Magnetic measurements (MM) and neutron powder diffraction (NPD) have been used on intermetallics in the Ce-Ni-Ge system, to establish correlations between chemical composition, crystal structure [1], magnetic properties and cerium valence state. MM [2, 3] show that $Ce_xNi_yGe_z$ richer in Ge are antiferromagnetic. From NPD $Ce_3Ni_2Ge_7$ [4] and $Ce_2Ni_3Ge_5$ magnetic structures are collinear, with ordered M_{Ce} magnetic moments at 1.4K equal to $1.98(2)\mu_B$ and $0.4(1)\mu_B$ respectively. $CeNiGe_3$ magnetic structure has both commensurate and incommensurate magnetic components, with $M_{Ce} \approx 1\mu_B$. These magnetic structures will be analysed in connection with crystal structures and Ce-Ni, Ce-Ge hybridizations. [1] P Salamakha et al, JAC 236 (1996) 206-211; [2] B Chevalier et al, JMMM 196-197 (1999) 880-882; J Mater Chem 9 (1999) 1789-1792; [3] Z Hossain et al, Phys Rev B 62 (2000) 8950-8953; [4] L Durivault et al, JMMM (2001).

A-100 Magnetic structures of RPd_2Ge

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Crystal and magnetic structures of RPd_2Ge (R=Tb-Er) compounds are determined on the basis of neutron diffraction data. All the compounds are antiferromagnets. The Neel temperatures are 19K, 12.5K, 4.4K and 3K for the compounds with Tb, Dy, Ho and Er respectively. $TbPd_2Ge$ and $DyPd_2Ge$ undergo a further phase transition in the ordered state and at 1.5K they exhibit a collinear magnetic structure described by the propagation vector $\mathbf{k}$=(0,0,1/2). With increasing temperature the structure changes into a sine-wave modulated one (at 10K and 5K for R=Tb and Dy, respectively). $HoPd_2Ge$ has a sine-wave modulated structure and $ErPd_2Ge$ is a collinear antiferromagnet at 1.5K. In both cases the magnetic moments are parallel to the c-axis.

A-101 Magnetic Structures of HoAuGe and ErAuGe

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The magnetic structures of HoAuGe and ErAuGe, both crystallizing in the hexagonal LiGeGa-type structure, have been investigated by high resolution neutron diffractometry in the temperature range between 1.5 and 7.7 K. For ErAuGe additional low temperature diffraction experiment have been performed between 40 mK and 1.4 K. Both compounds order antiferromagnetically at low temperatures. The magnetic structure of HoAuGe is described by the propagation vector k=[1/2,0,0]. The low temperature diffraction pattern of ErAuGe is similar to that obtained for HoAuGe, however, some magnetic reflections of ErAuGe show a significant broadening. This broadening is due to the magnetic domain size effect. Close to the Neel temperatures incommensurate magnetic structures appear. These structures can be described by the propagation vector k=[0.4461(5),0,0] at 4.5 K for HoAuGe and k=[0.4185(40),0,0] at 3.0 K for ErAuGe.

A-102 The structure of ferrofluids in the vicinity of the silicon-ferrofluid interface

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Specular reflectivity and off-specular scattering experiments have been performed on the interface of a flat silicon single crystal and colloidal solutions of ferromagnetic nanoparticles (ferrofluid, FF) at several applied magnetic fields with polarized and unpolarized neutrons. The data provide us with information over the nuclear and magnetic structure of the FF in the vicinity of the interface, i.e. the depth dependence of the scattering length density as well as the lateral correlations, and may reveal the previously assumed magnetic-field induced reorganization of the FF particles.

A-103 Magnetic Phases in $La_{1-x}Y_xMn_2Si_2$ - High Resolution Diffraction

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$La_{1-x}Y_xMn_2Si_2$ with $x \sim 0.2$ presents a critical concentration between ferromagnetic $LaMn_2Si_2$ and antiferromagnetic YMn_2Si_2, exhibiting magnetic instabilities with temperature and composition that are dependent on the ferromagnetic and antiferromagnetic contributions. We have recently shown that the mixed magnetic region corresponds to a two-phase structure [1], however accurate determination of the crystallographic distinction between these phases was restricted by the medium resolution diffractometer. Here we present a high resolution neutron powder diffraction study (SIRIUS diffractometer, KEK) in which these structural differences are resolved for the first time. [1] M Hofmann, S J Campbell and S J Kennedy, J Phys: Condens Matter, 12 (2000) 3241

A-104 Antiferromagnetic ordering and magnetic structure of the layered cerium carbide halides, $Ce_2C_2X_2$ (X=Br,I)

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The compounds $Ce_2C_2X_2$ (X=Br,I) crystallize with a layered structure consisting of a close-packed arrangement of X-Ce-Ce-X slabs. C_2 units are located within Ce_6 octahedral interstices. Depending on the stacking sequence there exist two phases (1s- and 3s- stacking variants). Powder magnetic susceptibility measurements show antiferromagnetic ordering below 13 K for $Ce_2C_2Br_2$ and 15 K for $Ce_2C_2I_2$. We have performed neutron powder diffraction experiments on the two samples, $Ce_2C_2X_2$ (X=Br,I). From the Rietveld refinement of the low temperature diffractograms we derive the magnetic structures of $Ce_2C_2X_2$ which can be indexed by a propagation vector $k = (0, 0, 1/2)$.

A-105 The magnetic structure of the neodymium carbide halides, $Nd_2C_2X_2$ (X=Br,I)

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$Nd_2C_2Br_2$ and $Nd_2C_2I_2$ are both congeners of a group of isotypic compounds, $RE_2C_2X_2$ (RE = rare-earth ; X = halogen). These compounds crystallize as layered structures consisting of close-packed arrangements of X-RE-RE-X slabs, with C_2 dimers centering the distorted RE_6 octahedra. Magnetic susceptibility meaurements indicate antiferromagnetic ordering below 12 K for the bromide and below 30 K for the iodide. Based on the Rietveld refinement of the low temperature powder pattern of $Nd_2C_2I_2$, the magnetic reflections can be indexed assuming identical crystallographic and magnetic unit cells (k = [0,0,0]). The final Rietveld refinements reveal that the Nd magnetic moments lie entirely within the *ac*-plane with a magnitude of 3.4(1) μ_B, and are arranged parallel across the C-C dimers. The detailed magnetic structure and the magnetic properties of these new Nd compounds will be discussed.

A-106 Crystal and magnetic structure of the two-dimensional coordination polymers CoCl2(bpy-d8) and NiCl2(bpy-d8) (bpy-d8 = 4,4' -bipyridine-d8)

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Neutron diffraction studies of deuterated polycrystalline samples of the two-dimensional coordination polymers CoCl2(bpy-d8) and NiCl2(bpy-d8) (bpy-d8 = 4,4' -bipyridine-d8) have been carried out at various temperatures in order to determine their crystal and magnetically ordered structures. For both compounds, we observe an orthorhombic structure, space group Pban. In this structure, infinite chains of (-CoCl2-) or (-NiCl2-) units are cross-linked by the bipyridine ligands. The two pyridine rings of the bpy exhibit a relative tilt of 30°, which was not observed in the previous single crystal x-ray study. Previous magnetic susceptibility studies of CoCl2(bpy) and NiCl2(bpy) revealed long range antiferromagnetic ordering below 5.0 K and 8.5 K, respectively. The magnetically ordered structures were determined from low-temperature neutron diffraction data. A simple antiferromagnetic arrangement was observed in which the metal ions are ferromagnetically coupled within the metal-chloride chains and nearest-neighboring chains couple antiferromagnetically. Next-nearest-neighboring chains linked by the bpy ligands are aligned ferromagnetically. The ordered moments at 1.5 K are 2.8(2) μ_B and 2.0(2) μ_B, respectively, with a moment orientation along the bpy bridges.

A-107 Neutron diffraction study of the magnetic and structural phase transitions in the deuterated molecular ferromagnet Fe(dtc)2Cl

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Magnetic susceptibility curves versus temperature measured in single crystals of Fe(dtc)2Cl show a ferromagnetic phase transition at 2.45 K with the easy axis of magnetisation given by [101]. In addition, there has been observed an anomaly in the specific heat which has been ascribed to an structural phase transition occurring at around 150 K. This structural transition is at the origin of the difficulties found to refine from the single crystal data the nuclear structure at low temperature as well as the magnetic structure. We have determined the nuclear and magnetic structures at several temperatures between 1.5 K and RT by powder neutron diffraction in a deuterated sample. Systematic thermodiffractograms reveal that the structural transition extends over the temperature region 175 to 140 K. In this structural transition the ethyl groups which at RT are disordered between two non equivalent positions become frozen in only one position. A canting angle of around 20 degrees has been estimated from the analysis of the magnetic diffraction data at low temperature. We have also determined the evolution with temperature of the magnetic (magnetic moments, canting angle, critical exponent) and structural parameters.

A-108 Dynamics of coupled zigzag S= $\frac{1}{2}$ chains: Cluster expansion approach

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We have studied dimerized spin systems by realizing the cluster expansion to high order. Using this tool we have investigated the incommensurate region of zigzag chains with isotropic exchange coupling constants near the disorder-line where the dispersion curve exhibits a minimum at a finite wavevector. Our approach clearly shows the gradual transition between the minimum of the dispersion at wavevector 0 and wavevector π within this region. We have extended the dimer expansion to cover weakly interacting chains for a quantitative description of three dimensional materials like $KCuCl_3$ and $Cu_2(C_5H_{12}N_2)_2Cl_4$. By comparison with recent data from neutron scattering experiments we are able to determine the strength of interaction between individual spins.

A-109 Magnetic properties of the coupled edge-sharing CuO_2 chains in $Ca_{2+x}Y_{2-x}Cu_5O_{10}$

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Neutron scattering experiments were performed on the quasi-one-dimensional magnet $Ca_{2+x}Y_{2-x}Cu_5O_{10}$, which consists of the ferromagnetic edge-sharing CuO_2 chains. In undoped $Ca_2Y_2Cu_5O_{10}$, the magnetic excitation peak width in energy becomes broader with increasing Q along the chain although sharp excitations are observed around the zone center and perpendicular to the chain. We revealed that the anomalous magnetic excitation spectra are caused mainly by the antiferromagnetic interchain interactions. In slightly hole-doped $Ca_2Y_2Cu_5O_{10}$, exchange interactions change mostly in the chain. The broadening of the magnetic excitations at high Q's and energies is still observed.

A-110 Spin-Gap and Antiferromagnetic Correlations in Low-Dimensional $PbNi_{2-x}A_xV_2O_8$ (A=Mg,Co) Compounds

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Our experiments explore important issues in quantum magnetism that contribute to our knowledge of phase transitions and critical phenomena. We focus on low-dimensional magnetic model compounds embedded in a three-dimensional lattice to probe how the singlet ground state and the energy gap in the magnetic excitation spectrum are suppressed and give their place to magnetic long-range order. For this purpose we employ high-intensity neutron powder diffraction complemented by bulk magnetic susceptibility on selected quasi-1D compounds. The composition phase-space of the $PbNi_{2-x}A_xV_2O_8$ system doped at the S=1 Ni sites by non-magnetic Mg (S=0) or magnetic Co (S=3/2) ions, presents a series of isostructural solids with sharp contrast in the nature of the magnetic exchange interactions and the onset of the accompanied spin-freezing transitions. We show that spin vacancy induced antiferromagnetic order is in strong competition with the disordered spin-gap state.

A-111 Neutron diffraction studies of two-dimensional magnetic ordering in $TbRu_2Si_2$

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We have investigated magnetic orderings of $TbRu_2Si_2$ by neutron diffraction. Below T_N=57 K the magnetic ordering is confirmed to be one-dimensional with Q=(3/13 0 0). However, below 5 K a number of magnetic satellites have been observed not only on high-symmetry lines, but also on low-symmetry lines with the fundamental modulation of Q=(3/13 0 0) and its odd harmonics in the $a^\star$-$b^\star$ reciprocal plane. These magnetic satellites show that in this region the magnetic structure is not one-dimensional as previously reported, but described by a model of two-dimensional magnetic ordering with a 26a×26a×c magnetic unit cell, which contains two non-magnetic Tb sites. At approximately 1.8 K this magnetic order changes to another two-dimensional one.

A-112 Antiferromagnetic quantum spin chains in high magnetic fields

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One-dimensional antiferromagnetic spin systems are known for macroscopic quantum ground states similar to the superconducting ground state. The characteristic properties of the ground state wave function are reflected in the magnetic excitation spectrum which cannot be understood in terms of precessing spins. The quantum character of ground state and excitations becomes most evident at high applied magnetic fields. Quasi-one dimensional antiferromagnetic spin systems are realized by transition metal compounds with strong superexchange along one crystal axis, and negligible along the other directions. We discuss elastic and inelastic neutron scattering results on antiferromagnetic Heisenberg chains with spin 1/2 and 1 in high magnetic fields.

A-113 Lattice dynamics of the spin-Peierls inorganic compound $CuGeO_3$

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Inorganic compound $CuGeO_3$ is a spin-Peierls system with transition temperature T_{sp}=14 K. Phonon softening related to the lattice dimerization at T_{sp} has not been observed yet. Longitudinal optical phonon propagating along $b^\star$-axis ($b^\star$-LO) observed with transfer energy 1 meV at zone center by inelastic neutron scattering. The temperature dependence of this $b^\star$-LO phonon peak is measured, and as a result of the measurement phonon energy and peak width are independent on temperature, but phonon intensity deviates suddenly from Bose factor curve below 60 K. In conclusion structural phase transition at T_{sp} is not displacive type but order-disorder type and short range order of low temperature phase progresses from 60 K.

A-114 Ordering in two dimensional antiferromagnets at finite temperatures.

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We present a theoretical study of the thermodynamic and critical properties of quantum Heisenberg antiferromagnets on the square lattice,in both the isotropic and the easy-axis anisotropic case, and for different spin values S. These models describe the magnetic behaviour of many real compounds, at different values of spin. such as $Sr_2CuO_2Cl_2$, La_2CuO_4, and CFTD (S=1/2), La_2NiO_4 and K_2NiF_4 (S=1), Rb_2MnF_4 and K_2MnF_4 (S=5/2). The method we use is the pure-quantum self-consistent harmonic approximation (PQSCHA), which allows us to determine the temperature and spin dependence of fundamental quantities such as the correlation length, the staggered susceptibility, magnetization and the specific heat. Being the method parameter-free, the comparison between our theoretical results and the available experimental data does not require best-fit procedures, and it hence stands as an excellent tool to interprete and understand experimental data. In this work we have mainly focused out attention on the temperature and spin dependence of the correlation length and staggered susceptibility. We show that, in accordance with neutron scattering data [1-2], and at variance with predictions of the quantum non-linear sigma-model field theory, no crossover towards a quantum critical regime is observed for S>1; indications of such absence be persisting in the S=1/2 case, are also given. As for the anisotropic model, the agreement found between our results and the neutron scattering experimental data allows us to conclude that the finite-temperature divergence of thecorrelation length and staggered susceptibility observed in most real compounds, is due, no matter how small the easy-axis anisotropy, to the onset of two-dimensional easy-axis long-range order, i.e. to the occurrence of a two-dimensional Ising-like phase transition [3]. [1] Y.S.Lee, M.Greven, B.O.Wells, R.J.Birgeneau, G.Shirane, Eur.Phys. J.B5, 15, 1998. [2] K.Takeda, M.Mito, K.Nakajima, K.Kakurai, K.Yamagata, Phys.Rev B63, 2001, in press. [3] A.Cuccoli, T.Roscilde, V.Tognetti, R. Vaia, Eur.Phys. J B 2001, in press.

A-115 Magnetic Ordering and Spin Excitations in Quasi-1D $Mn(dca)_2(pyz)$ [dca = $N(CN)_2^-$; pyz = pyrazine]

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Although molecular magnets comprised of dicyanamide (dca) bridging ligands have received much recent interest, we have little knowledge of the magnetic excitations. In particular, $Mn(dca)_2(pyz)$ features a novel interwoven ReO_3-like network structure which AFM orders below T_N = 2.53 (2) K. The Mn^{2+} moments align parallel to the Mn-pyz-Mn chain axis in zero-field. Due to exchange and single-ion anisotropies, a spin flop (SF) transition is observed at 0.43 T while a SF to PM transition occurs at an unusually low field of 2.83 T. Inelastic neutron scattering studies of the spin excitations in zero- and applied-field environments provide a better understanding of the link between electronic structure and magnetism.

A-116 Neutron Scattering Study of the Field-Induced Phase Transition in the Spin Gap System $TlCuCl_3$

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$TlCuCl_3$ is the strongly coupled spin dimer system with the singlet ground state. Neutron elastic scattering experiments have been performed in magnetic fields parallel to the b-axis. The magnetic Bragg peaks which indicate the field-induced magnetic ordering were observed for magnetic field higher than the gap field $H_g = \Delta/g\mu_B = 5.5$ T at $Q = (h, 0, l)$ with odd l in the $a^\star - c^\star$ plane. The spin structure in the ordered phase was determined. The temperature and field dependence of the sublattice magnetization and the phase boundary obtained, together with previous magnetization and specific heat data, are discussed in connection with a recent theory which describes the field-induced magnetic ordering as a Bose-Einstein condensation of magnons.

A-117 Spin Structure of $CsCu_{1-x}Co_xCl_3$ in the Magnetic Field

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The elastic neutron scattering have been performed in order to determine the magnetic structure of the antiferromagnetic triangular system $CsCu_{1-x}Co_xCl_3$ ($x \approx 0.03$) in the magnetic field. $CsCu_{0.97}Co_{0.03}Cl_3$ undergoes two phase transitions at $T_{N1} = 10.4$ K and $T_{N2} = 7.4$ K at zero field. The low temperature phase is the new ordered phase produced by the doping effect of Co^{2+} ion. It is found that the spin structure in the new ordered phase is the oblique triangular antiferromagnetic phase in which spins form the triangular structure in a plane tilted from the basal plane. The phase transition field at which the new phase becomes unstable is minimum for $H \parallel c$ and maximum for $H \perp c$. The field variations of the tilting angle ϕ depend on the field direction. With increasing field ϕ decreases gradually for $H \perp c$. On the other hand, for $H \parallel c$, ϕ is almost constant up to $H \approx 3$ T and becomes zero abruptly. On the presentation, we will present the detailed magnetic structure and discuss the origin of the oblique triangular structure.

A-118 Magnetic excitations in the triangular-lattice antiferromagnet $CuFeO_2$

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We report inelastic neutron scattering measurements on a single crystal of $CuFeO_2$. The spin wave dispersion in both the low-temperature commensurate and the intermediate temperature incommensurate phases along the [110] and the [001] directions were observed. For the low-temperature phase, four spin-wave branches were found. In the incommensurate phase, the magnetic excitation spectrum is strongly softened. The $CuFeO_2$ structure consists of triangular layers of magnetic Fe ions separated by nonmagnetic ionic layers of Cu and O, stacked along the c-axis. It has previously been presumed therefore, that the magnetic interactions are 2D in character. The main result of this experiment however, is that there is a significant dispersion of magnetic excitations propagating in both the basal plane and along the hexagonal axis. This observation suggests that the magnetic interactions in $CuFeO_2$ are 3D in nature and undermines the validity of a simple 2D Ising model proposed for $CuFeO_2$.

A-119 Contrasting Antiferromagnetic Order Between $FePS_3$ and $MnPS_3$

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Transition metal thiophosphates, MPS_3 (M=Fe,Mn etc.), make up a class of antiferromagnetic materials with quasi 2-dimensional magnetic behaviour. The metal atoms occupy a honeycomb lattice in which, for $MnPS_3$, the first neighbour interaction is 400 times that between the planes with little anisotropy so that the antiferromagnetic order is not well established perpendicular to the planes. We present the first true powder neutron diffraction patterns of $FePS_3$ which show by the absence of trailing edges on the magnetic Bragg peaks, seen at low temperatures for $MnPS_3$, that they are point like rather than rod shaped in reciprocal space. This indicates that the order is truly 3-dimensional.

A-120 Instability of a quasi-magnetic structure on a layer compound, $Cs_{0.5}Rb_{0.5}VF_4$

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Magnetic properties of $Cs_{0.5}Rb_{0.5}VF_4$ are studied by a neutron diffraction and a neutron diffraction camera. The layer compound shows an antiferromagnetic phase transition at about 35K and the magnetic unit cell is $2ap \times 2bp \times 2cp$, where $ap \times ap \times cp$ is an ideal unit cell of $TlAlF_4$-type. However, the magnetic cell is transformed to $2ap \times 2ap \times cp$ under a field cooling of 2T. The quasi-magnetic structure shows unusual magnetic properties depending on the applied field and specimen temperature.

A-121 Magnetic ordering of Fe in NCH_5-intercalated iron phosphate $Fe(OH)PO_4$

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The magnetic properties of a novel organic templated layered material $FeOHPO_4(NCH_5)$ have been studied via ac magnetic susceptibility, dc magnetization, and neutron diffraction measurements. Temperature dependencies of both the in-phase and out-of-phase components of the ac

magnetic susceptibility display a sharp peak at 30 K, signifying the antiferromagnetic ordering of the Fe spins. Weak but definitive magnetic hysterisis were seen at temperatures below 30 K, suggesting the existence of a weak ferromagnetic component. Neutron magnetic diffraction measurements reveal a spins structure consists of ferromagnetic sheets coupled in a canted antiferromagnetic arrangement, with an ordering temperature of 30 K and a saturated moment of 3.35(5) Bohr magtron for the Fe ions.

A-122 Magnetic Excitations and Exchange Interactions in the Spin Gap System $TlCuCl_3$

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The magnetic excitations in the spin gap system $TlCuCl_3$ were investigated by neutron inelastic scattering experiments. The constant-Q energy scan profiles were collected in the $a^\star - c^\star$ plane at $T = 1.5$ K. Well-defined one magnetic excitation mode was observed. The dispersion relations along four different directions were determined. The lowest excitation occurs at $Q = (h, 0, l)$ with odd l as observed in $KCuCl_3$. The experimental results are supplemented by a theoretical analysis based on dispersion curves calculated from a dimer series expansion, which allows the determination of exchange parameters between individual spins.

A-123 Neutron Scattering Studies of the S = 2 Antiferromagnetic Chain $MnCl_3(C_5D_{10}N_8)$

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Quasielastic and inelastic neutron scattering studies of the quasi one dimensional S = 2 antiferromagnet $MnCl_3(C_5D_{10}N_8)$ are reported. The quasielastic measurements exhibit a broad peak at Q≈0.68 $Å^{-1}$ which is consistent with short range antiferromagnetic coupling between neighboring Mn^{3+} ions. Inelastic experiments, at $T = 30$ mK and $Q = 0.68Å^{-1}$, reveal decreased magnetic scattering at energies less than 0.2 meV when compared to similar studies at $T = 20$ K. These results provide microscopic evidence for the presence of a Haldane gap and are consistent with the bulk magnetization measurements of Granroth *et al.*, Phys. Rev. Lett. **77**, 1616 (1996).

A-124 Dimensional crossover in the strongly fluctuating antiferromagnet $KCuF_3$

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We present neutron scattering data on the quasi-1D spin-1/2 Heisenberg antiferromagnet $KCuF_3$. Measurements of the dynamical susceptibility were made using the MAPS spectrometer at ISIS and HB1 spectrometer at HFIR both below and above the Neel ordering temperature. $KCuF_3$ lies in a dimensional crossover region and shows contrasting behaviors over different temperature and energy ranges. At energies from 25-110 meV the two-spinon continuum characteristic of a 1D spin-1/2 antiferromagnetic chain is observed at all temperatures. While below 25meV in the ordered phase the Goldstone spinwave modes characteristic of a 3D magnet are observed. An additional mode appears at low temperatures between the spinwave branches at 17meV. Polarized neutron measurements show that this is longitudinal in nature. We discuss our results in the context of dimensional crossover in a system near quantum criticality.

A-125 Nanostructured intermetallic alloys with GMR behaviour

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We present a polarised SANS investigation of nanostructured Au-Fe alloys and Fe/FeO "core shell" compounds. The samples were prepared by melt-spinning and inert gas condensation. The SANS experiment performed with an external magnetic field (2.5 T) allowed us to separate the nuclear and magnetic contributions to the total signal, and gave us information on particle size distribution, particle density and mean interparticle distance as function of the synthesis parameters. The FeAu alloys show the presence of thin flat Fe precipitates with no magnetic contribution. In the core-shell samples, the polarisation analysis shows the existence of correlations between nuclear and magnetic form factors. The data can be interpreted in terms of a distribution of composite particles with a magnetic core (∼1 nm), and a non-magnetic outer shell with average thickness ∼0.5 nm.

A-126 Quantum fluctuations in the model 2D, S=1/2 Heisenberg antiferromagnet CFTD.

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Quantum fluctuations in many-body systems act to suppress the classical order parameter. In some cases it may be quenched completely so that new ground states with no classical analogues emerge. The nature and excitation spectra of such quantum ground states attract considerable attention, but the same fundamental considerations apply to the quantum fluctuative part of the ground states of systems where the reduction of classical order is not complete. Here we present recent neutron scattering measurements on $Cu(DCOO)_2 \cdot 4D_2O$, a model 2D quantum Heisenberg antiferromagnet on a square lattice, where quantum fluctuations reduce the ground state staggered magnetisation to 60% of its classical value. The

experiments were performed on MAPS at ISIS, and illustrate the potential of this instrument for exploring excitation spectra over large volumes of reciprocal space, while maintaining good Q resolution. Contrary to the predictions of spin wave theory, we observe a dispersion of the spin wave energy along the zone boundary. By combining our data with numerical calculations and existing theoretical work, we find indications that the ground state overlaps with quantum states of a nature that is qualitatively different from the Neel state. In addition we present the first experimental evidence for a multimagnon continuum in this model.

A-127 Magnetic and elastic fluctuations in the 2D S=1 XY antiferromagnet $BaNi(PO_4)_2$

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As part of an effort to understand the inevitable dynamic couping between magnetic excitations and fluctuations of the underlying crystal lattice, we have performed a polarised neutron scattering investigation of $BaNi(PO_4)_2$, which represents the 2D S=1 XY antiferromagnet on a hexagonal lattice. The spin wave excitations are found to remain well defined well above T_N=23 K, albeit damped and softened. The damping of propagating excitations in the short range ordered system is in itself of fundamental interest to our understandng of the 2D XY model, and the results are reated to theoretical expectations. To our initial surprise, we observed an oscillatory behaviour of the final neutron polarisation as a function of wave-vector along the spin wave dispersion. By mapping the low energy phonon dispersion at high wave-vector and temperature, we demonstrate that the maxima of the apparent depolarisation coincides with where the spin wave and phonon dispersions cross. Hence, at low temperatures and wave-vector, where it is impossible to observe the phonon scattering directly, it reveales itself in the polarisation of the spin wave branch. This observation encourraging for subsequent studies of systems, where quasiparticles of coupled magnetic and elastic fluctuations are expected.

A-128 Structure and excitations in the high-field soliton phase of $CuGeO_3$

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$CuGeO_3$ is a good physical reaisation of a spin–Peierls system, where coupling of the S=1/2 chains to the 3D lattice creates a dimerised singlet quantum ground state. Above a critical applied magnetic field of 12.5 T, the elementary excitations condense into the ground state forming an incommensurate lattice of solitons. Applying neutron scattering techniques in magnetic fields up to 14.5 T, we have obtained a complete characterisation of the structure and excitations in this soliton phase of $CuGeO_3$. The observed soliton structure is well accounted for by existing theories, while much less has been predicted concerning the excitation spectrum. We observe three well defined excitations, all gapped, which may be interpreted as two commensurate transverse excitations and an incommensurate low-energy phason mode.

A-129 Magnetic Excitations in Zigzag Charge Ordered NaV_2O_5

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We report on an inelastic neutron scattering study performed in the quarter-filled spin ladder-system NaV_2O_5 both above and below the charge ordering temperature T_c = 35 K. We observed a distinct dispersion relation along $b^\star$-axis with the zone boundary energy of about 90 meV at 6 K below T_c. It becomes slightly vague at 60 K above T_c. These characteristic features, including the $c^\star$-dependent excitations, are discussed in connection with the zigzag-type charge ordering.

A-130 Field dependence of the magnetic ordering in the XY quantum magnet Cs_2CoCl_4

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Low-dimensional quantum magnets are ideal test beds for investigating non-linear effects in strongly fluctuating systems. Cs_2CoCl_4 has been proposed as an experimental realization of a spin-1/2 XY antiferromagnetic chain [1], predicted to show unusual quantum spin liquid behaviour. To determine the Hamiltonian and the magnetic phase diagram we have made neutron diffraction measurements on a single crystal as a function of temperature down to 80 mK and magnetic field up to 6.5 T. In zero field, long-range antiferromagnetic order was observed below T_N=217 mK and models for the magnetic structure will be discussed. The long-range antiferromagnetic order disappears when the field applied perpendicular to the chain axis reaches 2.1 T.par [1] H.Yoshizawa et al, Phys. Rev. B28, 3904 (1983).

A-131 Spindependent diffuse scattering from polarizing supermirrors

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We present measurements of the spindependent diffuse scattering under grazing incidence from polarizing supermirrors (reflection edge 2.5 $\theta_{c,Ni}$ for spin $\uparrow$) measured with polarized neutrons and polarization analysis. The mirrors have been investigated in different magnetic states between remanence (mirror is magnetized opposite to the field direction and reflects spin $\downarrow$ neutrons) and saturation at 30 mT, as well as in high magnetic fields. The diffuse scattering of these mirrors is strongly spin polarized. At scattering angles below the specular reflection angle, a non-negligible amount of diffuse scattering with spin $\downarrow$ has been observed. Additionally, we present an analysis of the diffuse scattering in the framework of the DWBA, which yields information about roughness and magnetic domains (orientation and size) within the layered structure. The measurements have been performed on the HADAS reflectometer in Jülich and on the PRISM reflectometer in Saclay.

A-132 Magnetic configuration at the interface of an exchange bias system studied by polarised neutron reflectometry

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A study of the magnetic configuration at the interface of $Gd_{40}Fe_{60}/Tb_xFe_{1-x}$ bilayers by Polarised Neutron Reflectometry (PNR) is presented. GdFe is soft ferrimagnetic layer and TbFe is hard magnetic one. Depending on the composition of the TbFe alloy the exchange bias phenomenon observed is either negative or/and positive. PNR measurements perform on GdFe/TbFe showed that a magnetic domain wall (DW) is created at the interface as the GdFe magnetisation rotates. The DW thickness could be tuned by increasing or decreasing the applied magnetic field. The DW size and shape could be deduced by fitting PNR measurements. The sample was also rotated in the field which permits to create DW of different shapes (60° DW, 90°). Magnetic profiles obtain from PNR measurements are found to be in very good agreement with micromagnetic calculations.

A-133 Small-angle Neutron and X-ray Scattering Study on Super-paramagnetic Co-Al-O Granular Films

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Metal-nonmetal Co-Al-O granular films composed of nanoscale ferromagnetic particles ($\approx$ 3 nm) embedded in an amorphous oxide matrix, exhibit interesting magnetic properties depending on their microstructures. Films with high Al-O contents show super-paramagnetism, while films with low Al-O contents exhibit soft magnetic properties. In order to clarify the microstructural difference, transmission electron microscope (TEM) observation and small-angle X-ray scattering (SAXS) have been performed. In addition to these technique small-angle neutron scattering (SANS) has been applied to this study for clarifying the local magnetization distribution. Though TEM and SAXS for super-paramagnetic films show that the particles are well isolated, SANS profiles imply that some fraction of these particles are magnetically coupled. The difference in the fraction of the particles which are in contact with each other, is dominant factor for magnetic properties and are discussed in detail.

A-134 Single-crystal DHCP and FCC phases in Ce/Pr Superlattices

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Cerium usually comprises a mixture of polycrystalline FCC and DHCP allotropes. Single-crystal Ce has been stabilised in Ce/Pr superlattices grown using molecular beam epitaxy. It is found that FCC or DHCP phases can be obtained depending on superlattice composition and growth conditions. Low-temperature neutron scattering was performed on Ce/Pr samples using the triple-axis spectrometer D10 at the ILL. These measurements showed that one sample, [Ce20Pr20]60, is a single crystal with a DHCP unit cell; while another, [Ce30Pr10]56, is a mixture of FCC and DHCP phases. Antiferromagnetic ordering is observed in the DHCP sample (Neel Temperature=11.1K) with a magnetic structure similar to that of beta-Ce. Surprisingly, the magnetism is confined solely to the Ce blocks. Further, it is found that at low temperatures, the lattice contraction found in bulk cerium is supressed in FCC Ce/Pr superlattices.

A-135 NSE for Self-Diffusion in Intermetallic Alloys

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Backscattering spectrometers like IN16 at ILL allow to study diffusion in intermetallic alloys with CsCl structure [1]. Diffusion in these systems is comparably slow, so that temperatures close to the melting point have to be applied in order to achieve quasielastic effects which, however, are still at the limit of what even the best backscattering spectrometers can resolve. NSE is expected to extend the dynamical range towards longer times and to give a more direct picture of the different time scales that are involved in the diffusion process. We present first results of an experiment performed at the spin-echo spectrometer SPAN at the BENSC/HMI. [1] M. Kaisermayr, J. Combet, B. Sepiol, H. Ipser, H. Schicketanz and G. Vogl, Phys. Rev. 61, 12038 (2000); Phys. Rev. B 63, 054303 (2001).

A-136 Neutron structural investigations of para- and antiferroelastic phase of potassium dysprosium double tungstate

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The potassium dysprosium double tungstate $KDy(WO_4)_2$ (KDyW) belongs to the class of magneto-elastics with interrelation between magnetic and elastic ordering. The structural phase transition (SPT) of cooperative Jahn-Teller (CJT) type from para- to antiferroelastic state was observed at 6.38K with the magnetic phase transition taking place at 0.60K. The neutron powder diffraction experiment was directed to determine the atomic structure of the antiferroelastic state of the KDyW. Anomalies in the atomic structure were observed near the SPT temperature. Additionally we find an increase of the width of Bragg reflections suggesting increasing strain in very good agreement with the CJT character of the SPT (J-T distorsions).

A-137 Inelastic and critical neutron scattering in the ergodic phase of relaxor ferroelectric PbMg1/3Nb2/3O3

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Results of study of low energy excitations and critical scattering in the field induced ergodic ferroelectric phase of PMN relaxor ferroelectric are presented. It is shown that transition to the ferroelectric phase does not result in the appearance of clearly defined soft mode. Observed quasielastic scattering demonstrate dependence similar to that of dielectric constant and is strongly anisotropic. Q-dependence of the scattering intensity follows $q^{-\alpha}$ law, with a changing from a$\sim$1.6 (170K) to a$\sim$2.9 (300K). The most important conclusion is that heating of PMN, cooled in electric field, above depolarization temperature $\sim$210K does not restore the scattering pattern observed in "virgin" sample. Work was supported by the RFBR (grant 99-02-18074) and Russian program "Neutron researches of Solids"

A-138 Neutron powder diffraction study of structural phase transitions in some ferroelectric complex metal oxides

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The unusual physical properties of ferroelectrics are dependent on the adopted atomic structure and an understanding of structural chemistry of these materials is crucial for developing novel compositions with anomalous dielectric properties. With the aim to achieve insight into the structural mechanism of ferroic ordering in complex metal oxides, this neutron powder diffraction (NPD) study was initiated. NPD patterns have been collected at the Swedish Research Reactor R2 (Studsvik) in the temperature range 10-1000K for the following oxides: the perovskites $APbO_3$ (A=Ba,Sr), the ferroics with stibiotantalite-type structure $ANbO_4$ (A=Sb,Bi), the aluminates with tridymite derivative structures MAl_2O_4 (M=Ba,Sr). From sequental Rietveld refinements the correct symmetry of ferroic phases and polar atomic displacements were studied with temperature. The structural peculiarities of different phases are discussed and possible structural models of phase transitions are presented. The crystal and magnetic structures of $Pb(Fe_{0.5}B_{0.5})O_3$ (B=Nb,Ta) and $Sr(Fe_{0.67}B_{0.33})O_3$ (B=W,Te) have been investigated at different temperatures. Possible Fe^{3+} magnetic moment orderings in these systems are analysed. The correlation between ferroic distortions and the magnetic ordering is also briefly discussed.

A-139 Ferroelectric phase transitions in Nb-doped $KTiOPO_4$

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Crystals of $KTiOPO_4$ (KTP) have a unique combination of physical properties (non-linear optic(NLO), ferroelectric(FE), superionic). In an attempt to further understand the role of Nb doping in KTP on FE and NLO properties, the NPD and SHG studies were initiated. KTNP crystals with 2, 3 and 10% Nb were grown using the flux method. The powder specimens were prepared from simgle crystals by grinding. NPD patterns have been collected at the Swedish Research Reactor R2 (Studsvik) at the different temperatures below and above Tc. All the structures are found to be orthorhombic (s.g.Pna21 at T$<$Tc and s.g.Pnan at T$>$Tc) and are isostructural with KTP. The substitution of Nb for Ti leads to expansions along a and b axis and contraction along c axis. Nb atoms strongly favor the Ti(1) sites reducing the octahedral distortion. The charge balance occurred by creation of vacancies preferentially in K(1) site. The major atomic displacements associated with FE transitions were studied and the magnitude of Ps was estimated. Polar distortion at phase transition is accompanied by relatively large shifts of K cations and this sublattice may be responsible for appearance of spontaneous polarization Ps. Above Tc partial disorder of K cations was also found. The possible type of phase transition (displacive or order-disorder) is briefly discussed.

A-140 Metal foam evolution and decay studied by neutron radioscopy

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The formation process of metal foams was investigated with neutron radioscopy. Two kinds of experiments were carried out: in the first lead foams were generated in a furnace from foamable precursor material containing metal and blowing agent. During foam expansion neutron absorption profiles were recorded. The vertically oriented foam columns were scanned with a beam of thermal neutrons from which the time-dependent local density was derived. The resulting density profiles reflect the kinetics of drainage in metallic foams. In the second experiment pre-prepared lead foams were melted by heating them in a furnace. During melting and after neutron absorption profiles were obtained from which drainage kinetics were derived. Results from both types of experiment are presented and compared with drainage curves of aqueous foams.

A-141 USANS investigation of early stages of metal foam formation

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Metallic foams are on the verge of being used in industrial applications. However, the mechanism of foam creation, especially the early stages, are still unexplored. Ultra small-angle neutron scattering (USANS), performed with the double crystal diffractometer (DCD) at the Geesthacht Neutron Facility (GeNF), is a promising method for obtaining a three-dimensional average of a pore size distribution in a wide size range from about 100 nm to about 20 microns. Zinc foams were prepared by expanding compacts containing powders of Zn and ZrH_2, the latter acting as a blowing agent. Foaming was interrupted at an early stage by quenching the expanding samples, thus allowing to prepare foams with porosities ranging from 0.5 to 20%. Analysis of the neutron scattering curves yielded pore size distributions which conformed with the results obtaining by microscopy.

A-142 Multiple Small Angle Neutron Scattering Studies of Anisotropic Materials

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Various authors have recognized the power of multiple small-angle neutron scattering (MSANS) analysis in providing information on coarse, concentrated microstructures involving micrometer length-scales larger than those accessible in conventional SANS studies. Following previous work that considered the case of spherical scatterers [1,2] and, later, randomly-oriented spheroidal scatterers [3], we now describe a MSANS analysis for non-randomly-oriented spheroids and discuss this with reference to studies of the multi-component void morphologies encountered in plasma-sprayed ceramic thermal barrier coatings of technological interest [4]. [1] N.F. Berk and K.A. Hardman-Rhyne, J. Appl. Cryst., 18, 467 (1985) and 21, 645 (1988). [2] G.G. Long et al., J. Neutron Res., 7, 195 (1999). [3] A.J. Allen et al., J. Appl. Cryst., 27, 878 (1994). [4] A.J. Allen et al., Acta Mater., in press (2001).

A-143 Crystal-field spectrum of $R_{1-x}Ca_xBa_2Cu_30_{\sim 7}$ (R = Ho, Er) high-T_c superconductors in the overdoped regime

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Recent inelastic neutron scattering measurements revealed the crystal-field (CF) spectra of the overdoped $R_{1-x}Ca_xBa_2Cu_3O_{\sim 7}$ (R = Ho, Er, $0 < x < 0.2$) high-T_c superconductors to consist of the two spectral components associated with the optimally doped and overdoped domains, respectively. Increase of the Ca concentration does not affect the local charge density of the optimally doped domains, but increases the spectral weight of the overdoped component of the CF spectrum. In the sense of this "two-phase" picture, which was established earlier for the underdoped region, there is a smooth crossover between the under- and over-doped parts of the phase diagram. In spite of this, we argue that these two regions are characterized by qualitatively different type of the in-plane charge distribution.

A-144 Additional low-frequency modes in zirconium hydrides

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Vibrational modes in $ZrH_{0.1}$ were studied by neutron spectroscopy at room temperature (α-Zr and γ-hydride) and around 528K, the FCO γ-phase to FCC δ-phase conversion temperature given by the literature. The optical modes analysis in terms of H vibrations specific to each hydride agrees with recent neutron diffraction and specific heat measurements indicating a lower temperature for this phase transition. Using different experimental resolutions the inelastic scattering was well-separated by the quasielastic one. Additional low-frequency modes (around 5 meV) revealed by spectra measured at all temperatures are discussed in terms of strong overdamped phonons confined to an interstitial H and its surrounding nearest neighbours and characteristic of resonant vibrations.

A-145 Calculation of restricted rotational states in the methyl group

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The methyl group attached to the molecule in solid phase has a certain amount of hindrance in rotational motion. The rotational potential usually can be expressed by the 3rd order and the 6th order of periodic functions. In the case of the larger potential, the collected parts of energy levels approach to the so-called tunneling levels. The lowest part consists of two levels (A, E) with 3-fold symmetry or four levels (2A, 2E) with 6-fold one. In the intermediate region with respect to not merely the field strength but the degree of mixing of two components, much variety appears in the structure of rotational energy levels. The energy values correspond to the various molecular surroundings. The matrix elements are also computed which gives the intensity of inelastic neutron scattering spectra.

A-146 Long Range Atomic Order in Irradiated $Mn_{20}Cu_{80}$

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Short range order in CuMn alloys is observed as a diffuse (1 $\frac{1}{2}$ 0) peak in neutron scattering spectra. There has been speculation for many years on the nature of the corresponding long range order. A sample of $Mn_{20}Cu_{80}$, irradiated in a nuclear reactor to produce a large number of crystal defects at a low temperature, has developed long range order. This order is not seen in an identical non-irradiated sample.

A-147 Neutron diffraction study up to 1900K of 3:2-mullite

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Neutron- and supplementary x-ray diffraction studies were carried out on pure and Cr-doped 3:2-mullite ($Al_{4+2x}Si_{2-2x}O_{10-x}$, with x=0.25) up to the decomposition temperature. Main aim of the work was a detailed investigation of the thermal expansion and structural re-ordering in the high temperature regime of this technically important material. Generally we observe two regimes which are tentatively analysed by linear expansion coefficients. The expansion along b (orthorhombic s.g.) is largest. Cr-mullite shows lower expansion. There are only minor changes in the structural parameters, e.g. a decrease of the occupation of the Al(2)-site above 1300K, which is more significant in Cr-mullite. The idea of a relation between an anisotropic lattice expansion and an anisotropic expansion of the basic building units, i.e. AlO6-octrahedra, will be discussed. The results gained from the different neutron diffractometers will be compared.

A-149 Temperature dependence of ordering in the γ-phase of Ni-based superalloys

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Short range order (SRO) in model $gamma$-phases of several Ni-based superalloys is investigated by elastic diffuse neutron scattering as a function of temperature. The composition of the single-crystalline samples has been modified by small amounts of additional elements like Mo, Re, Ru and W in order to study their influence on SRO which is clearly dominated by peaks of the (1 1/2 0) type. The intensity of SRO scattering remains nearly unchanged up to about 700°C, above this temperature SRO decreases gradually and finally vanishes around 1000°C. Correlation lengths do not change significantly with temperature.

A-150 Direct comparison of SANS data with SEM image analysis

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Ultra small-angle neutron scattering (USANS) technique permits to investigate large-scale microstructure in the size range overlapping with the resolution of SEM micrographs. In principle, image analysis of sections parallel to the scattering plane permits to calculate directly the Fourier image of the slit-smeared scattering functions. Results of both methods can be then compared directly using this Fourier image as the common characteristics describing the microstructure. We have tested this technique on data simulated for various 2-phase systems in order to assess the influence of image resolution and statistical errors due to the limited image area on the precision of such direct comparisons. Apart of connecting SEM and USANS data, this technique can be employed to calculate the total scattering cross-sections, which is important parameter for correct evaluation of data affected by multiple scattering. It is particularly useful for the investigation of strongly scattering porous materials.

A-151 SANS Characterisation of Pillared Layered Catalysts

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Pillared inter-layered clays (PILCs) are materials in which aluminosilicate layers are intercalated with nano-oxide pillars. Such porous solids have potential for many industrial applications, due to their special sorption/catalytic properties. Despite several studies, there is no detailed sorption mechanism available, the major reason being their heterogeneous nature. In order to study the structure of PILCS, SANS measurements were performed on a series of parent clays as well as their alumina-pillared homologues. The spectra of the pillared samples are characterized by strong peaks, which are absent from the scattering curves of the parent clays. Additionally, surface roughness changes are observed after pillaring. Based on contrast variation, discrimination of the different entities is possible and structural information can be obtained.

A-152 Small-angle Scattering Studies of Carbon Sorbents Porous Structure

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The research of a porous structure of the carbon sorbents (CS) were performed by the method of the small angle neutrons scattering(SANS) The measurements were carried out on the industrial sorbents, prepared by traditional methods. All investigated samples have similar the SANS curves in an interval of scattering vectors q=0.006-1Å^{-1}. For an explanation of the experimental SANS curves a model is suggested that the CS consists

of two groups of carbon particles of the different sizes. The small particles have a size about 10A. They make a half of a volume part of scattering particles and have a strongly rough surface with fractal dimension Ds=2.5. The other group of particles has a power distribution on the sizes N(R)$\sim$ R^{-n}, where the R changes between R_{min}=25Åand R_{max}=300Å. The experimental value of the n is 3.2. This group of particles has a volume part less than 1%. Using our model we could satisfactorily explain the experimental SANS data for the different CS. The work is executed at support RFBR (grant 01-02-96412).

A-153 Structure Evolution and Formation of Pre-melted State in $NaNO_2$ Confined within Porous Glass

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The results of neutron diffraction study of $NaNO_2$ embedded in porous glass are presented. It is shown, that $NaNO_2$ forms complex dendrite clusters inside glass. The details of the structure including the anisotropic thermal parameters are determined both below and above ferroelectric phase transition temperature Tc. It is demonstrated that growth of dielectric permittivity could be attributed to the giant increase of thermal displacements and possible formation of "pre-melted state". On cooling ferroelectric phase transition suppresses this "looseness" and below Tc normal ferroelectric phase exists with structure nearly identical to the bulk $NaNO_2$. The work was supported by the RFBR (grants 01-02-17739), the Russian Program "Neutron Researches of Solids".

A-154 SANS investigation on pore surface roughening in rocks

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Pore-matrix interface and pore morphology in some metamorphosed, sandstones and igneous rocks have been investigated in length scales of 20-1000 nm by small-angle neutron scattering (SANS) to reveal the fractal nature of the interface. Multiple scattering effects in these specimens has also been looked into. Fractal dimension of sandstones and metamorphosed rocks has been estimated to be 2.8 but that for igneous rocks has been found to be 2.3. Attempt has been made to explain the fractal nature of the former rocks with a computer simulation model based on their formation mechanisms. SANS data indicate the existence of the upper cut-off of fractal for igneous rocks and sandstone, but no unambiguous cut-off has been observed metamorphosed rocks in the accessible length scale.

A-155 Small angle neutron scattering investigation of microporosity in marbles

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Small-angle neutron scattering (SANS) measurements have been carried out on different marble and limestone samples, of relevance to cultural heritage safeguard, to investigate the micropore distribution in the size range between 1 and 100 nm. Different varieties of marble have been studied using a $CaCO_3$ single crystal, pore-free, as reference. A series of red scaglia limestone samples has also been studied obtaining for this material well reproductible results. Namely identical SANS cross-section are found for speciemens issued from a modern quarry and for samples issued from an historical building. Furthermore, the micropore volume distribution functions, obtained by transformation of the SANS data, are in accordance with the porosimetry result for pore sizes larger than 50 nm approximately. SANS measurements carried out to try and distinguish close and open microporosity are also reported.

A-156 Layered structural determination of ternary systems based on clays: precursors in the synthesis of mesoporous solids

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A neutron diffraction study was carried out in D16 Station at ILL, with the a twofold purpose: (a) to evaluate the quality of two different macroscopic vermiculites to be employed as reference materials in the basic study of the arrangement of organic molecules in their interlayer space and (b) to analyse the level of contrast incorporated in the target systems (composed of the silicate, a surfactant component and a swelling agent). For the (a) purpose we have chosen two well-characterised macroscopic vermiculites, Eucatex and Santa Olalla, and they have been intercalated with fully deuterated dodecyltrimethylammonium. The neutron diffraction patterns of both systems show that there is a large number of (00l) diffraction basal reflexions. However, a more ordered organic/silicate mesophase is formed with vermiculite from Santa Olalla, a more precise neutron scattering profile along the c-axis being obtained. Regarding the (b) objective, the study of the influence of the organic swelling agent has been carried out in the most ordered organic/silica system. The effect of absorbing three different alkanes with length chain from C8 to C12 can be summarized as following: They act as swelling agents and the basal spacing increased up to ca. 40 Å. The basal space remains almost constant and the series of basal reflections exhibit similar profiles for all the systems.

A-157 Small Angle Neutron Scattering (SANS) observation of channels in nuclear membranes

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Nuclear membranes are thin polymer foils (typically 10m thick) irradiated by heavy ions which create defects located along the ions trajectory, the track. It is possible to open rather monodisperse channels by chemical etching (0.25N NaOH at 80*C for PolyEthyleneTerephtalate). SANS experiments have been performed in LLB (PAXE) and ILL (D11) on PET membranes prepared in the FLNR (JINR, Dubna), and later in GANIL. Previously only a Gaussian approximation was used [1,2] for the data treatment. A new experimental protocol allows to obtain more fully interpretable spectra. For the biggest channels we could observe the oscillations of the Bessel function (radial part of a cylinder form factor). The rotation of the sample around a vertical axis provides 3 dimensional information about the channel shape. [1] R. Spohr, Nuclear Instrument and methods, 173 (1980) 229-236 [2] D. Albrecht thesis, (1983), GSI report 83-13, Darmstadt, Germany

A-158 Small Angle Neutron Scattering (SANS) observation of channels porous silicon

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When electrochemically etched in HF, silicon wafers develop a porous layer. The geometry of this layer is strongly dependent upon the initial doping of the silicon. There exist a critical doping when the porosity changes from an amorphous to an organised state with parallel channels.While electron microscopy can give only a local picture of the channel shape, SANS provides overall information about channels shape.Early experiments at the Budapest reactor allowed to determine average channel diameters and showed that the diameter dispersion follows a Schultz-Zimm law [1,2]. The channels exhibit a very large aspect ratio which makes their study difficult; for a recent experiment performed on D11, at the ILL, we used a new specific experimental protocol, including the rotation of the sample around an axis orthogonal to the neutron beam, which allows to obtain 3 dimensional information about the channel shape. Previous experiments had shown that the doping range where the porosity change takes place is very narrow. We shall present a new model for the channel diameter, cross section and overall 3D shape. [1] G. Kadar, G. Kali, Cs. Ducso, E. Vazsonyi: Small angle neutron scattering in porous silicon, Physica B, 1997, Vols. 234-236, pp. 1014-1015 [2] G. Kadar, E. Vazsonyi, S. Borbely, G. Kali: Small angle neutron scattering in P+ doped porous silicon, J. Porous Materials, 2000, Vol. 7, pp. 331-334

A-159 SANS investigation of nitrogen sorption in mesoporous silica

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The mechanisms of nitrogen sorption in mesoporous silica with pore sizes between 5.5 and 10 nm were investigated by small-angle neutron scattering (SANS). Using contrast matching conditions for silica and condensed nitrogen, SANS curves were recorded at 77 K at various relative pressures. The experiment offers the unique opportunity to study pore structures and sorption mechanisms in one single experiment. Using novel suitable evaluation methods, the changes in the SANS patterns were quantitatively related to different sorption mechanisms, which were found to be micropore (2nm) filling, nitrogen layer formation and mesopore condensation. In particular, an unambiguous proof and a quantification of this additional microporosity was obtained for the first time.

A-160 Optical data storage material $Sr_{0.61}Ba_{0.39}Nb_2O_6$: A single crystal neutron diffraction study.

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$Sr_xBa_{1-x}Nb_2O_6$, SBN, $0 < x < 1$, is a very attractive material for technological applications and basic research, because of its outstanding electrooptic and photorefractive properties. The space group of SBN in the ferroelectric phase is P4bm with a positional modulation of the NbO_6 - octahedra and an occupational modulation of the Sr/Ba atoms. The ideal structure $(A1)_2(A2)_4(C)_4(B1)_2(B2)_8O_{30}$ is not fully occupied. Five Ba and Sr atoms are distributed over six sites, whereby the $(A1)_2$ position is only occupied by Sr, on $(A2)_4$ Sr and Ba are present, the $(C)_4$ position is empty and the $(B1)_2$, $(B2)_8$ positions are occupied by the Nb atoms. We performed neutron diffraction experiments on TriCS at SINQ in order to explain the influence of the positional modulation of the oxygen-octahedra on the modulation in the Sr/Ba occupation in the congruent melting composition $Sr_{0.61}Ba_{0.39}Nb_2O_6$ by detecting the main and satellite reflexes of the first order.

A-161 Neutron and X-ray powder diffraction investigated of the new compound $Bi_{2.53}Li_{0.29}Nb_2O_9$

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New compound was synthesized with a purpose of search of materials with high ionic conductivity. The crystal structure is defined using both x-ray and neutron diffraction data. The structure of $Bi_{2.53}Li_{0.29}Nb_2O_9$ is orthorhombic with sp. gr. Cmc21 and the parameters of an elementary cell a=24,849 Å, b=5.453 Å, c=5.462 Å(x-ray data were indexed in tetragonal sp. gr. I4/mmm, a=b=3,857 Å, c=24,849 Å) at T=290 K. Compound was investigated in a wide temperature range from 10 up to 870 K. Structural phase transitions in this area of temperatures is not founded. The Ritveld refinement was carried out at 10 and 290 K. Under two temperatures coordinates of atoms and thermal factor in isotropic approach were determined. The analysis of the received data with use bond valence calculation is carried out.

A-162 Neutron Diffraction Study of Structural Transformations in Ternary Mercury Chalcogenides at High Pressure

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Structure of ternary mercury chalcogenides $HgSe_{1-x}S_x$ (x=0.3, 0.5, 0.7) and $HgTe_{1-x}S_x$ (x=0.15) has been studied by means of neutron diffraction at pressures up to 4 GPa using sapphire anvil high pressure cell equipment. A phase transition from the cubic zinc blende phase to the hexagonal cinnabar phase was observed in both systems at P∼0.6 GPa and 1.5 GPa, respectively. Lattice parameters and positional parameters of Hg and Se/Te/S atoms as functions of pressure were determined. The differences between cinnabar phases of $HgSe_{1-x}S_x$ and $HgTe_{1-x}S_x$ are discussed. The work was supported by RFBR, grant 00-02-17199.

A-163 The Structure of 6-line Ferrihydrite

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A sample of 6-line nanocrystalline ferrihydrite (bulk formula: 5,$Fe_2O_3 \times H_2O$) obtained by 12 minutes hydrolysis of Fe(III) nitrate at a temperature of 75,°C was investigated by neutron diffraction at 5,K, 293,K and 343,K. In comparison to the typical 6-line X-ray diffractogram neutron

diffraction shows a much more detailed pattern. Basing on earlier discussed models the structure of ferrihydrite can now be given as a sum of a defect-free phase and a defective phase. 6-line ferrihydrite is antiferromagnetic already at ambient temperatures with a spin sequence of $+--,+$ at the iron sites and a moment of 3.2,μ_B/,Fe at 5,K.

A-164 Neutron powder diffraction study on the thermal expansion of cuprite

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The temperature behaviour of cuprite, Cu_2O, was investigated from 8 K to 530 K by the analysis of about 100 neutron diffraction patterns collected in steps of about 5 K. Cu_2O crystallizes in a rare cubic structure (SG Pn3m) representing a framework of corner sharing OCu_4 tetrahedra. According to Rietveld refinements the unit cell volume reveals a minimum around 270 K and increases towards both low and high temperatures. The linear thermal expansion coefficients from 8 K to 270 K and 270 K to 530 K are approximately -1.7×10^{-6} K^{-1} and 5.5×10^{-6} K^{-1}, respectively. Observations of significant amounts of non-Bragg scattering on Cu-reflections indicate the existence of transversal vibrational modes which are assumed to cause the negative thermal expansion. The isotropic thermal displacement parameters of both Cu and O increase continuously from 8 K to 530 K. The neutron results are discussed in the context with previous findings by other experimental techniques [1]. [1] M. Ivanda et al., J. Raman Spectrosc. 28 (1997) 487.

A-165 Neutron Diffraction Study of $Bi_{12}MO_{20}$ Single Crystals with Sillenite Structure, (M = Si, $Si_{0.995}Mn_{0.005}$, $Bi_{0.53}Mn_{0.47}$)

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Series of $Bi_{12}MO_{20}$ single crystals were synthesized by the Chochralski and TSSG methods. The isomorphism of all studied compositions was established by neutron diffraction: the compounds crystallize with the cubic sillenite type structure (space group $I23$, Z=2), a=10.104Å, 10.109Åand 10.153(1)Åfor M = Si, $Si_{0.995}Mn_{0.005}$ and $Bi_{0.53}Mn_{0.47}$, respectively. The BiO_n polyhedron can be described as a distorted octahedron with one axial position occupied by the free $6s^2$ electron pair of Bi^{3+}. In Si-containing crystals the tetrahedral position (2a) is fully populated by Si^{4+}. In the synthesized new compound $Bi_{12}(Mn_{1-x}Bi_x)O_{20}$, Bi and Mn atoms statistically occupy (2a) sites. The Mn-containing samples were found to be oxygen deficient.

A-166 Crystallization Behaviour of Bulk Metallic Alloys by IN-SITU Neutron Diffraction

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We have performed in-situ neutron diffraction crystallization experiments on ZrTiCuNiAl, ZrTiCuNiBe, PdNiCuP alloys and we have looked into details to the first phases appearing at crystallization. We have compared the observed transition temperatures to the first DSC crystallization peaks. For several compounds the first phase appearing is an icosahedral phase, meaning that in the glassy state the short range ordering must be of icosahedral type. In the system ZrTiCuNiAl, the composition range for which the icosahedral order appears is smaller than the composition range for which a bulk glassy alloy is produced, other first crystallization phases, like Zr_2Ni, are also formed. In the system ZrTiCuNiBe, an icosahedral phase is formed for the Vitr4 composition, but is not obvious for the Vitr1 composition. The experiments on the PdNiCuP system does not show any intermediate crystallization.

A-167 Neutron Scattering Studies of Functional Carbon Materials

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High-resolution neutron powder diffraction and small angle neutron scattering (SANS) experiments were carried out on three different carbons by TOF method using pulsed spallation neutron source at KENS, KEK, Japan. Materials selected on the basis of their performances as anode in secondary lithium ion batteries were Swedish natural graphite, meso carbon micro beads (MCMB) and carbon fiber. SANS results confirmed the existence of nanopores in carbon fiber, which is the origin of high capacity of hard carbons. MCMB showed difference in the low Q region of the scattering curve that may arise from the turbostratic disorder. The hexagonal (AB) and rhombohedral (ABC) phase information in graphitic structures were also revealed from neutron diffraction results. All these results are discussed to explain their electrochemical performances.

A-168 Neutron and X-Ray Study of stoichiometric and doped $LiNbO_3$:$Zn_{0.08}$

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$LiNbO_3$ (LN) crystals possess useful optical properties, which are strongly dependent on both the crystal stoichiometry and content of dopants such as Mg^{2+}, Zn^{2+}, In^{3+}, Sc^{3+}. Such elements drastically reduce photorefraction at a sufficiently high "threshold" concentration. This value is for Zn in the range of 6-7 at.% and was supposed to be connected with the change of the dopant atom localization in the lattice. We report the results of a single crystal neutron (D9, ILL) and X-ray (Moscow) study of stoichiometric and doped (with 8.2 at.% Zn) $LiNbO_3$ at T= 78 K and 300 K and also a "multi-pattern" powder neutron (D2B, ILL) and X-ray (STOE, Univ. Muenchen) Rietveld refinement of the crystal structure and electron density. LN single crystals are very perfect and extinction problems are discussed.

A-169 Structural study of the $LaNi_{4.6}Ge_{0.4}$-D_2 system using neutron diffraction

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Intermetallic compounds that reversibly store hydrogen gas at room temperature and pressure are used in numerous devices including hydrogen gas storage units, hydrogen purification and heat-pump systems. $LaNi_{4.6}M_x$ (M=Sn, Ge), substitutional derivatives of $LaNi_5$, are being used in sorption cryocoolers for applications in space [1]. This study deals with the structural characterisation of $LaNi_{4.6}Ge_{0.4}$ and of its hydride (deuteride) including the hydrogen insertion sites in the metallic matrix and their occupancies. Results are compared to properties of Sn substituted phases. [1] L.A. Wade, et al., Adv. Cryogenic Engineering, 45 (2000) 553.

A-170 Crystal Structure and Spiral Magnetic Ordering of $BiFeO_3$ Doped with Manganese

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$BiFeO_3$ belongs to the class of the so-called ferroelectric-magnets exhibiting both electric - dipole (T_c = 1118 K) and spiral magnetic ordering (T_N = 653 K)[1]. We report on changes on the crystal and magnetic structure of $BiFeO_3$ with Mn doping for samples $BiMn_xFe_{1-x}O_3$ (x =0, 0.1, 0.2) as analysed from constant wavelength and time-of-flight neutron powder diffraction. With increasing Mn concentration the periodicity of the magnetic spiral increases whereas the average magnetic moment and the ordering temperature decrease. The Mn doping leads to a non-Brillouin-type behaviour of magnetisation. [1]. Sosnowska I., Peterlin-Neumaier T., Steichele E., J. Phys C, 115 (1982) 4835.

A-171 Neutron powder diffraction study of ilvaite with very high Mn content.

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Ilvaite, $Ca(Fe^{2+},Fe^{3+})_2Fe^{2+}(Si_2O_7)O(OH)$, is a mixed valence iron silicate. Mn is a common component of natural ilvaites substituting mainly for Fe. In all ilvaites studied up to now the MnO content is less than 10 wt%. Recently, ilvaites with 13.5–13.8 wt% MnO were established in the Madan ore district, Bulgaria. Powder neutron diffraction study on the TOF diffractometer ROTAX at ISIS was performed to determine the Fe/Mn distribution. The structure refinements in space group P 2_1/a (a = 13.0246(8) Å, b = 8.8511(5) Å, c = 5.8485(3) Å, β = 90.15(1) degree) show that Mn substitutes for Fe only in the M2 octahedral site (occupancy= 0.510(3)). Orthorhombic symmetry of the unit cell can be ruled out. The Mn occupancy of the Ca site is 0.105(3). The order parameter Q, determined from the refined bond lengths, is equal 0.66(34). The relationship between Q and β is discussed.

A-172 Neutron Powder Diffraction Study of $LaONO_3$

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The crystal structure of lanthanum oxynitrate, $LaONO_3$, has been reported as tetragonal with C_{4v} as the lanthanum site symmetry [1]. Analyses of the luminescence spectra of $LaONO_3$:Eu^{3+} and $GdONO_3$:Eu^{3+} [2] have proved that the Eu^{3+} site symmetry can be no higher than C_{2v} possibly due to different orientations of nitrate groups. The neutron powder diffraction study was carried out to give more detailed structural information. The results suggest that the structure solution from X-ray diffraction data is only an approximation of the actual one. [1] Gobichon, A.-E., Auffredic, J.-P., and Louër, D., Solid State Ionics 93 (1997) 51. [2] Hölsä, J., Kestilä, E., and Karppinen M., Z. Phys. Chem. 187 (1994) 61.

A-173 Vacancy ordering in nanosized maghemite from neutron and X-ray powder diffraction

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Maghemite (γ-Fe_2O_3) is a deficient inverse spinel of current interest for magnetic recording media preparation. Common view is with a long range ordering of the vacancies on the octahedral sites, which lowers the symmetry from $Fd\bar{3}m$ down to $P4_332$. We studied a nanocrystalline sample with needle shaped grains focusing our interest on the distribution of vacancies and shape effects. Both neutron and X-ray diffraction measurements showed the presence of superstructure peaks, the whole pattern was indexed in $P4_12_12$. The structural parameters were determined from Rietveld refinement, while the examination of the diffuse scattering yielded details of the local structure and short range ordering.

A-174 Neutron diffraction study of the structure $TiC_xO_yN_z$

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The crystal structure of $TiC_xO_yN_z$ (x+y+z = 0.67-0.68) have been investigated by high resolution neutron diffraction method. Initial samples had NaCl-type (sp.gr.Fm3m) crystal structure. The lattice parameter decreased with increase of the nitrogen content. The shape of reflections of 00l type did not changed after annealing at 1000K-890K (120 hours). Splitting and distortion of the structure and superstructure peaks decreased with increase of the nitrogen content. It is shown that the trigonal ordered structure (space group R3m; $P3_121$) of Ti_2C type was formed after annealing.

Cubic ordered structure appropriate to Fd3m space group was observed at $TiC_{0.31}O_{0.08}N_{0.29}$. Anneal at 1100 K has resulted in disordering of structure (sp.gr.Fm3m).

A-175 Neutron Scattering Investigation of Lead-Potassium Eutectic Alloy

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Binary Pb-K system is a matter of multiple microscopic investigations (see e.g. [1]). A singular interest to this system results from Zintl clusters which exist in liquid Pb-K (> 25 at.%) alloy as a form of quasi-molecular groups, $(Pb_4)^{-4}$ (K^+4), with the tetrahedral packing of atoms. However, there are no data on that for Pb-K alloys with potassium concentration lower than 25 at.%. We have investigated liquid lead and Pb-K (9 at.%) eutectic alloy by neutron diffraction and inelastic neutron scattering at 630 and 870 K to obtain information of its atomic structure and microdynamics. It is found that the Zintl pre-peak in static structure factor is absent for the Pb-K (9 at.%) eutectic alloy, that is the clusterisation of this system is absent. [1] H.T.J. Reijers, W. van der Lugt, C. van Dijk, M-L. Saboungi, J. Phys.: Condens. Matter 1 (1989) 5229.

A-176 Phase stability of layered manganates $Ca_{3-x}La_xMn_2O_7$

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Neutron diffraction studies have been carried out to study the solubility of Lanthanum in the parent compound $Ca_3Mn_2O_7$ and the stability of the layered $Sr_3Ti_2O_7$ structure in samples of nominal composition $Ca_{3-x}La_xMn_2O_7$, prepared by solid-state reaction route. The results of these studies show that single phase layered compounds form only in the concentration range $0 \leq x \leq 0.4$. The $(La,Ca)MnO_3$ phase starts appearing at x=0.4. At x=1.2, they become majority phases with CaO and the layered phase being minority phases. Our neutron diffraction results clearly disprove earlier reports that the layered phase is formed over the entire concentration range, x and confirm our earlier XRD results. Our studies also demonstrate that neutron diffraction technique is necessary for an unambiguous structural characterization of the layered manganates.

A-177 Temperature Dependence of Lattice Parameters in n and p Type CdTe by Neutron Scattering

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Single crystal neutron scattering studies of n and p type CdTe have been performed using the time of flight high-resolution diffractometer OSIRIS. The integrated intensities and lattice parameters of four (hhh) Bragg reflections were determined as a function of temperature between 2 and 250K and analysed with a Debye model. Distinctive features are found according to the acceptor or donor character of the crystal.

A-178 *ex situ* Structure Changes in Lithium Manganese Spinel after High Temperature Storage

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Ex situ neutron powder diffraction method has been applied systematically to study the change of the crystal structure in lithium manganese spinels in electrochemical cells. $Li_{1+x}Mn_{2-x}O_{4-\delta}$ (x = 0.0 - 0.1) after several storage temperatures were investigated by the Rietveld analysis of high resolution neutron powder diffraction data. With increasing temperature of storage in an electrolyte solution, oxygen site was found to be more deficient. In chemically Li-deintercalated $Li_{1.03}Mn_{1.97}O_4$, which was used as a model system for studying the stability of charged states with different charge depths, phase separation due to variation of Li occupancy in the structure proceeded in slightly de-intercalated spinels after the storage at 80°C.

A-179 Mechanochemical Treatment of α-Fe_2O_3 - Microstructural Analysis

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The effects of milling haematite, α-Fe_2O_3, under a variety of environments and milling conditions have been investigated by several groups recently. Of particular interest is the transformation of α-Fe_2O_3 to defect magnetite, Fe_3-vO_4, on wet-milling haematite in vacuum under low energy conditions. Our aim is to clarify the extent to which the intermediate phase maghemite, γ-Fe_2O_3, influences the transformation and the resultant milled products. We have milled α-Fe_2O_3 for 72 h and 144 h and investigated the products by in situ neutron diffraction measurements up to $\sim$ 700°C. The fractions of Fe_3-vO_4 and other oxide phases have been determined by Rietveld refinement. Our findings agree with chemical analysis for the Fe^{2+} present in the milled products.

A-180 Neutron Diffraction Study of the Crystal Structure and Dissolved Oxygen Content of Yttria-Sintered Aluminum Nitride

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Oxygen impurity and its related defects are thought to be the most influential factors in controlling the thermal conductivity of AlN. It is thought that the defect population within the crystal structure of this material is influenced by oxygen incorporation into the lattice and as a result, the phonon conductivity, which dictates the heat transfer properties of AlN, is affected. Neutron diffractometry was used to determine the dissolved oxygen content and crystal structure changes of AlN. The diffraction data were analyzed through Rietveld refinement method. The lattice parameters a

and c, Debye-Waller factors, relative microstrain and, most importantly, accommodation sites and atomic percentage of oxygen impurity atoms in the lattice were determined. The range of oxygen determined through this method decreased from 0.59(5)at.% for the as-received material to as low as 0.20(3) at.% for the sintered specimens. The oxygen was accommodated both substitutionally with a saturation limit of 0.39(4)at.%.; and interstitially. The resulting Rietveld models were used to explain the relationships between the crystal structure changes (such as unit cell volume, c/a ratio and Debye-Waller factors variation) and the removal of oxygen from AlN structure.

A-181 Neutron diffraction investigation of texture and elastic anisotropy of the olivine xenolithes

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To interpret the seismic anisotropy and to study deformation processes in the Earth's upper mantle texture investigations of rocks are necessary. Since olivine is the most widespread rock forming mineral, olivine xenolithes from various Europe regions were collected. Olivine textures were measured by the SKAT neutron texture diffractometer at the reactor IBR-2 (Dubna, Russia). Both the experimental and texture-derived spatial P-wave distributions were compared. The preferred orientation of olivine grains mainly controls the elastic properties at high confining pressures. The reconstruction of the rock deformation tensor was carried out. The p-T conditions of texture forming process were estimated.

A-182 Relating damage in an A359/SiC composite to creep strain and temperature using neutron diffraction

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Time of flight and fixed energy neutron diffraction is used to measure lattice strain in the matrix and reinforcement phases of an aluminium A359/SiC composite during creep and during subsequent room temperature mechanical tests. The stress exponent for creep rate is about 3 when determined from the stress in the Al matrix phase measured by neutron diffraction. This is opposed to about 8 when determined from the macroscopic applied stress. This discrepancy is related to the evolution of damage during creep. Room temperature mechanical tests are used to quantify damage by relating the elastic strain in each phase to macroscopic modulus measurements. The degree of damage is related to the creep strain, load and temperature of prior creep. A micromechanical analysis of composite elasticity and the kinetics of creep is introduced and found to give good agreement with experiment. Macroscopic creep data and modulus measurements are found to be consistent with neutron diffraction results.

A-183 Neutron texture study of natural gneiss mylonites affected by two phases of deformation

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Three samples of ductilely deformed gneiss from the Penninic nappes (Adula-Tambo, Swiss Alps) were studied using the texture diffractometer SV7-b at the Forschungszentrum Jülich. Pole figure data processing yielded complete textures for several constituent mineral phases (quartz, chlorite, mica). In combination with optical crystallographic orientation anlysis, image analysis of grain boundaries, and geological field work, this allowed to separate two stages of deformation of the samples which occurred under different temperature conditions and in a different kinematic framework, giving important information on deep-seated tectonic processes during the formation of the Alps.

A-184 Measurement of the crystallographic texture and internal strain in two phase Cu-Nb wire using neutron diffraction

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Two phase Cu-Nb wire produced by co-deformation techniques has exceptionally high tensile strength and electrical conductivity. The high strength results from the rigid niobium phase which is present in the wire as long axially aligned fibres. We report here the results of measurements of the crystallographic texture and the residual elastic strain in two phase Cu 18 wt% Nb wire using neutron diffraction. Samples of the as-fabricated wire were analyzed at room temperature using monochromatic neutron diffraction to determine the angular position of the niobium (220) and the copper (222) diffraction peaks. The niobium and the copper phases were both strongly textured with the niobium (220) and the copper (222) aligned in the axial direction of the wire. The niobium (220) inter-planar spacing was compared with the spacing in strain free niobium. The results indicated that the niobium fibres in the as-received wire are under a residual tensile strain of approximately 1.82%. This measured internal strain is significantly more precise than previously reported values which were inferred from measurements of the curvature of extracted niobium fibres.

A-185 Neutron diffraction method for investigations of strains in single-crystal structured materials

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Using high resolution neutron diffraction strains in a specimen can be evaluated from angular difference in position of diffracted peaks with respect to those measured by strain-free standards. But in the case of monocrystals this position is very sensitive to the orientation of the sample with respect to the incident beam. To overcome this problem, a set of diffraction patterns was recorded during sample rotation in vicinity of the Bragg reflection. The sum of all measured patterns then can be regarded as a simulation of the measurement of a polycrystalline sample. This method has been applied to strain investigations in a two-phase $Al_2O_3/Y_3Al_5O_{12}$ (YAG) single-crystal and thermal cycled Ni based superalloys.

A-186 Mineral preferred orientation and magnetic properties as indicators of varying strain conditions in naturally deformed iron ore

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A folded layer of iron ore was taken as a structural domain to investigate the degree of interdependence of mineral preferred orientation, magnetic properties and varying strain conditions. Hematite and quartz pole figures measured by means of neutron diffraction were recalculated by reducing the ODF into a set of texture components. The degree of preferred orientation increases considerably from the limbs towards the core of the fold. Corresponding changes of magnetic susceptibility, its anisotropy and hysteresis parameters (e.g. saturation magnetisation, remanence, coercivity) reveal information about the domain structure of hematite and texture modifying deformational processes.

A-187 Stress Induced Transformation and Textures in Pseudoelastic CuAlMnZn Shape Memory Alloy Deformed in Tension

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Martensitic transformation processes in shape memory alloys are characterized by reversible texture transformations. The commercially available SMA products as wires are typically fiber textured. Due to the lattice correspondence between austenite and martensite phases, this drawing texture is partially inherited upon transformation and possibly modified by preferential formation of martensite variants if the transformation to the martensite takes place under applied stress. In this work, in-situ neutron diffraction texture measurements of CuAlMnZn alloy bar specimen upon tensile and compressive loading were carried out. The results (ODFs) are combined with the conventional structural analysis using TOF spectra (GSAS Rietveld refinement) collected in another experiment in axial and radial orientation with respect to the load axis.

A-188 Determination by neutron difraction of residual stresses in PM 6061Al-15%volSiCw composites with different whisker orientation

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The determination of residual stresses, RS, in 6061Al-15vol%SiCw composites was carried out to correlate them with whisker orientation, distribution and aspect ratio. The composites were obtained by a powder metallurgical, PM, route and consolidated by extrusion at four different temperatures, Text (300°, 359°, 498°, and 534°C). Text is the main parameter affecting their whisker orientation/distribution; an increase in Text leads to an increasing whisker alignment with extrusion direction. The RS were studied in the peak-aged (146°C/15 hours) conditions of materials. The results of the sin2y plots indicate that there is a non-hydrostatic stress state in all reinforced materials. This is expected in a whisker reinforced composite because of the deviatoric component of the RS tensor generated by the whisker geometry. This effect is associated with the Strength Differential Effect observed in uniaxial testing (tensile vs. compressive behavior) of these composites.

A-189 Residual stresses in friction stir welded Al sheets

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Friction stir welding (FSW) is a relatively new joining technique for aluminum alloys that are difficult to fusion weld. A potential field of application are aircraft structures where costs are to be reduced by using new joining techniques instead of riveting. Great effort is currently made to qualify FSW for this purpose. In this study, the influence of a coolant applied during welding of Al sheets was investigated. Liquid CO_2 coolant was poured near the weld seam for removing heat from the weld zone. The residual stresses across the weld were measured by neutron diffraction. Three sheets were used, one without cooling, and two with cooling, where two different distances of the coolant nozzles from the FSW tool pin were chosen. The results show that by applying a coolant, tensile stress in the center of the weld can be reduced significantly.

A-190 Stresses on two sets of Cu precipitates in $Fe_{95}Cu_5$ alloy

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Copper precipitates mostly on the grain boundaries of the main (Fe) matrix. The Cu(111) Bragg peak position and its broadening after different heat treatments has been investigated by neutron diffraction. Depending from the treatment the Cu(111) peak shows some peculiar character. On the one hand it shifts, on the other, however, broadens showing quite big elastic stresses and indicating plastic deformations, respectively. After some special cases the broadening even turns to splitting. The reason of both phenomena is the big difference in the thermal expansion coefficients of the constituents that strongly influence both the elastic and plastic stress conditions on the Cu precipitates. Stress calculations will be carried out for both cases. The possible sources of the splitting will also be explained.

A-191 Residual stress states before irradiations in ESS target welded structural materials

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The present work takes place in the framework of the European Spallation Source target R&D program. A wide spectrum of miniaturised, mechanical tests specimens of different structural materials were placed into the SINQ target (Paul Scherrer Institute Villigen, Switzerland) for post-irradiation investigations. One of the non-destructive techniques planned for evaluating the stress fields produced by irradiation is neutron diffraction. The measurements on irradiated specimens will be performed at SINQ using a new instrument designed for this purpose. This paper

presents the initial stress measurements performed at ILL (Grenoble, France) on specimens kept unirradiated in order to get the reference residual stress state.

A-192 Residual Strain Measurement of an Unidirectionally Solidified Al_2O_3/YAG Composites by TOF Laue Diffraction

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A high resolution time-of-flight (TOF) diffractometer *Sirius* at KENS has been utilized to measure the residual strain in the unidirectionally solidified $Al_2O_3/Y_3Al_5O_{12}$ (YAG) composite precisely. Since the sample is a double single-crystalline composite material, we developed a new technique combining Laue patterns and high-resolution TOF data of each Bragg spot, which enabled the detailed indexing of the Laue pattern as well as extracting residual strain precisely. We obtained residual strains of both Al_2O_3 and YAG phases precisely as we as their azimuthal relation.

A-193 Investigation of textures in titanium aluminides by neutron diffraction

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Among other methods neutron diffraction is an efficient tool to characterize texture developments in materials which are processed by casting, deformation and annealing. Three main advantages of neutrons against X-ray techniques were used to investigate textures in two-phase TiAl alloys. Depending on the relatively high penetration depth and a beam cross section of about 9 cm^2, the average texture of relatively large volumes can be measured and thus also coarse-grained materials can be handled with minor problems. Additionally, less-overlappings between the constituting phases, TiAl and Ti_3Al, occur based on the scattering behavior of Ti and Al. In this paper, the texture of hot-extruded TiAl will be presented.

A-194 SANS Study of ODS Ferritic-Martensitic Steels

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Iron oxide dispersion strengthened alloys are candidate for nuclear fuel cladding. Therefore, it is crucial to control their microstructure in order to optimize their mechanical properties at temperatures up to 700°C. SANS experiments allowed to characterize the oxide dispersion (size and chemical composition) in different ODS materials after high temperature thermal treatment and after phase transformations austenite / ferrite-martensite.

A-195 Development of Procedures for the Measurement of Residual Stress by Neutron Diffraction

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Neutron diffraction is a non-destructive method of determining residual stresses in crystalline materials. It is a relatively new technique and no standard is currently available for making these measurements. This presentation gives the background to research that has been carried out to develop a standard. It outlines the main findings and indicates the precautions that are required to achieve accurate positioning and alignment of specimens (and components) in a neutron beam and the analysis required to obtain reliable results. It also shows that special attention is needed in dealing with near-surface measurements because of surface aberration. It is demonstrated that, provided the recommended procedures are followed, a positional tolerance of ± 0.1 mm can be achieved with an accuracy in strain of $\pm 10^{-4}$ to give a resolution in residual stress of ± 7 to 20 MPa in most materials of practical interest.

A-196 Biophysical Applications of Neutron Compton Scattering

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The scattering of epithermal neutrons (≈1-100 eV) from organic molecules is dominated by the recoil response of protons together with, if partially deuterated, that of the deuterons in the sample. We have begun to use this neutron analogue of Compton scattering (NCS) to measure momentum distributions and potential parameters of protons in peptide models and polypeptides. The information accessible by NCS is of interest in particular for increasingly realistic *ab initio* simulations of the dynamics which go beyond conventional normal-mode or Newtonian MD approaches. In this contribution, we discuss the analysis of NCS spectra from peptide models, focusing on the characterisation of the amide proton dynamics in terms of the width of the hydrogen-bond potential well, its Laplacian, and the mean kinetic energy of the proton. The Sears expansion for the dynamic structure factor is used to quantify deviations from the high-Q limit (impulse approximation), and the resulting asymmetry parameters of recoil lineshapes are evaluated in terms of Hermite polynomials. Data from the Compton spectrometer EVS at ISIS (Rutherford Appleton Laboratory, Chilton, UK) on selectively deuterated acetanilide illustrate the results obtained.

A-197 SANS study of the structure of DPPC/NaDC aggregates as a function of lipid/detergent ratio

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Small angle neutron scattering, dynamic light scattering and isotermal titration calorimetry were used to study the different aggregation states in sodium deoxycholate-phosphatidylcholine systems at T=60°Celsius. The phosphatidylcholine concentration was fixed at 6 mM. The size and shape of the aggregates, occuring in the vesicle to micelle transition, were investigated in dependence of the bile salt concentration (1.5 - 10 mM). The interpretation of the small angle neutron scattering spectra was performed by Guinier approximation as well as the vesicles and micelles model spectra calculations.

A-198 All disordered regions of native cellulose are of the same nature

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The structural details of the biopolymer cellulose, specifically its morphology, are still debated. The classical two-phase-model of distinct amorphous and crystalline areas is considered to be too simple. Recent models treating natural cellulose as a composite material assume that the disordered regions are located primarily at the surface of the microfibrils. Inelastic neutron scattering gives new insight into this problem. The hydroxyl groups of cellulose are used as a probe to measure the dynamic response of the regions accessible to water. Identical INS spectra are obtained independent of the crystallinity and origin of the different cellulose types [1]. The spectra of these disordered parts differ distinctively from the spectra of non-accessible, crystalline regions inside the microfibrils and can be considered as a fingerprint of disordered cellulose. Apparently the disordered matrix of the composite material cellulose is made up to a large part by the microfibril surfaces. [1] M. Müller, C. Czihak, H. Schober, Y. Nishiyama, G. Vogl, Macromolecules **33**, 1834–1840 (2000).

A-199 Relaxational dynamics in dry and humid DNA

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The influence of hydration on the self correlation of DNA-hydrogens has been measured by using inelastic neutron scattering. The range from GHz to THz could be covered by combining back-scattering and time-of-flight data for long incident wavelength. The direct spectral response of water is masked by using D_2O. Similar to other hydrated biopolymers, a steep increase of the susceptibility at low frequencies indicates a dynamic transition around T∼200K. The difference for dry and humid DNA shows that the water molecules cause this transition.

A-200 Study of glass transition in DPPC/DMSO/water system at low temperatures

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Dimythyl sulfoxid (DMSO) is a widely used solvent in cryobiology and medicine. It creates a dehydration in the intermembrane space of biological model membranes [1]. Ice formation in the ternary phospholipid/DMSO/water systems mainly depends on the phase diagram of the binary DMSO/water system as has been established via X-ray small and wide-angle diffraction [2]. X-ray and neutron diffraction and differential scanning calorimetry were applied for the investigation of the glass transition below the temperatures of ice formation in the ternary DPPC/DMSO/water system in the range of temperatures from -40°C to -120°C. A new condensed phase of DPPC was found at and above a DMSO molar fraction of 0.05. [1] M.A. Kiselev, P. Lesieur, A.M. Kisselev, C. Grabiel-Madelmond, M. Ollivon. J. Alloys and Compounds 286 (1999) 195-202. [2] M.A. Kiselev, P. Lesieur, A.M. Kisselev, M. Ollivon. Nuclr. Inst&Method, A 448 (2000) 255-260.

A-201 Effect of Proteins on Dynamics of w/o AOT Microemulsions

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We showed previously that the hydrolysis of some esters catalyzed by chymotrypsins entrapped in w/o AOT microemulsions is greatly enhanced at low water/surfactant molar ratio w0 and that the oligomeric phase of the reversed micelle plays an important role for the acceleration of the metabolic turnover. Our other previous neutron spin echo (NSE) study showed that the effective diffusion coefficient relating to bending fluctuation of the AOT microemulsion is significantly enhanced at low w0 value. We will show some NSE results on the effect of the presence of proteins on the dynamics of w/o AOT microemulsion.

A-202 Neutron spectroscopy and QC modeling vibrational spectra of Kinetin

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Neutron inelastic scattering (INS) investigations of 6-furfuryloaminopurine, i.e. kinetin were performed on the NERA spectrometer at the IBR-2 pulsed reactor of JINR in Dubna. Molecular geometry was optimized for isolated molecule at the AM1 and PM3 semi-empirical levels, and the restricted Hartree-Fock level with the 6-31G* basis set using the Gaussian'98 program. As the results three sets of vibrational frequencies and their intensities in the INS and IR spectra have been calculated. The comparison of calculated frequencies and intensities with the experimental INS and IR spectra allow make out an assignments of the low frequency internal modes of kinetin molecule. The librations of furfuryl side chain and purine group mixed with the lattice vibrations below the frequency 150 cm^{-1}. Then subsequently, in the frequency range up to 600 cm^{-1} following vibrations may be observed: out of plane and in plane vibration in purine group, deformation of angles in furfuryl side chain, furan ring torsion around the C-C bonds, out of plane and in plane vibration in furan ring.

A-203 Diffusive Properties of DUTPASE/Water System by Neutron Scattering

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The ubiquitous dUTP pyrophosphatase (dUTPase) is an essential determinant of fidelity of DNA replication by effectively reducing the dTTP/dUTP ratio in cells undergoing active mitosis. The physiological importance of dUTPase renders the knowledge of its detailed catalytic mechanism important in order to understand the enzyme's exquisite specificity towards dUTP and its high turnover rate. The C-terminal protein segment is delocalized due to its flexibility and the crystallized complex contains the non-hydrolyzable dUDP substrate analogue. In this contribution we show neutron scattering findings on dUTPAse aqueous solutions. By the characterization of the translational, rotational and vibrational contributions to the motion of the protein as a whole and of the arm, we can estimate the strength of the dUTPase/water interaction and its influence on the diffusive dynamics of both dUTPase and water.

A-204 Density of phonon states in the light-harvesting complex II (LHC II) of green plants studied by inelastic neutron scattering

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The primary steps of photosynthesis comprise the generation of electronically excited states by light absorption in pigment-protein complexes which act as antenna and rapid excitation energy transfer (EET) to photoactive pigments of reaction center complexes. The major antenna of green plants is the light-harvesting complex II (LHC II). In antenna complexes, the coupling of the electronic transitions to low-frequency vibrations of the protein matrix (phonons) plays an essential role in the generation of a spectrally broad absorption as well as in the energy dissipation during EET. Thus, a detailed understanding of ultrafast EET requires a thorough analysis of electron-phonon coupling. Inelastic neutron scattering experiments provide valuable information on the phonon density of states which is indispensable for a consistent interpretation of complementary results from high-resolution optical spectroscopy (see e.g. Pieper et al., Chem. Phys. Lett. 310, 1999, 296).

A-205 Inelastic Incoherent Neutron Scattering studies of water in Biomolecules

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It is widely known that water is the key element of life. Our aim is to understand the dynamics of the water molecules that come in close contact with the biomolecules and compare their behaviour with the bulk ones, using Neutron Scattering. Here we present the results from our experiments with Photosystem II, a lipoprotein that is found in the green parts of the leaves of every plant (Nicholson et al., 1996), and DNA. We performed a series of experiments on the TOSCA and HET instrument of the ISIS facility using ascending amount of hydration for both substances. Our results indicate that there are significant differences between the dynamics of the water molecules associated with our substances and bulk ones. This water is strongly perturbed as judged by its energy transfer spectrum, with a much broader and lower energy transition than bulk water in the 50-75 meV ($\sim$400 -600 cm^{-1}) range (Li et al., 1997). Taking into account the differing geometry of (cylindrical) DNA and (planar) membranes, the number of water shells perturbed by each system was estimated. The data adds to the growing evidence that water structure in the vicinity of biological macromolecules is unusual and that the proximal water behaves quite differently compared to the bulk solvent. Nicholson, W.V. et al., 1996. Biochem. J. 315, 543-547. Li JC and Leslie M (1997) J. Phys Chem. 101, 6304-6307

A-206 Progesterone and testosterone studies by neutron scattering methods and quantum chemistry calculations

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Inelastic incoherent neutron scattering (IINS) and neutron diffraction (ND) spectra of progesterone and testosterone were measured simultaneously on the NERA spectrometer at the IBR-2 pulsed reactor in Dubna. Both studied samples do not indicate any phase transition in the temperature range from 20 to 290 K. The INS spectra have been transformed to the phonon density of state (PDS) in one-phonon scattering approximation. The PDS spectra display well resolved peaks of low frequency internal vibration modes up to 600 cm^{-1}. The assignment of these modes was proposed taking into account the results of calculations the structure and dynamics of isolated molecules of the investigated substances. Quantum chemistry (QC) calculations were performed by semi-empirical PM3 method and at the restricted Hartree-Fock level with the 6-31$\star$basis set. The lower internal modes assigned as torsional vibration of the androstane skeleton mix with the lattice vibrations. The intense bands in PDS spectra in the frequency range from 150 to 300 cm^{-1} are related to librations of structurally nonequivalent methyl groups.

A-207 Molecular Diffusion in Crowded Protein Solutions

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The diffusion of biomolecules inside living cells is impeded by the typically high concentrations of proteins present there, which may be either freely diffusing, or part of long cytosekletal fibers. The conditions for intracellular transport are thus likely to be quite different from the conditions most amenable to the simple classical analysis of self diffusion. The oxygen transport of in the muscle is facilitated by the self diffusion of oxymyoglobin at high concentrations. The neutron spin echo technique allows to study the interdiffusion of myoglobin and hemoglobin on microscopic to mesoscopic scale. We have measured the concentration dependence of myoglobin and hemoglobin interdiffusion up to 30 mM. At the highest concentrations we observe a finite intercept of the inverse correlation time at Q = 0. We present the experimental result and discuss possible explanation and relevance of different theories for the description of the data.

A-208 Influence of hydration and cation binding on the parvalbumin protein dynamics

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Due to structural characteristics, parvalbumin exerts a major role in intracellular Mg^{2+} and Ca^{2+} concentrations regulation during the muscular contraction-relieving cycle. This structure-function relationship being established, we are investigating the structure-dynamics-function relationship to take into account the protein dynamics. Because of the strong incoherent neutron scattering cross-section of protons and of the abundance of this element in proteins, incoherent inelastic neutron scattering is a unique probe to study vibrational and relaxational dynamics in biological macromolecules. We take advantage of the complementarities in resolution of various neutron spectrometers (time of flight, backscattering, spin-echo) to probe the parvalbumin dynamics from a fraction of picosecond to a few nanoseconds. Experiments carried out by neutron scattering and NMR highlight a strong coupling between dynamics of the protein and of hydration water. Influence of the nature of the cation on parvalbumin dynamics are discussed.

A-209 Elastic resolution spectroscopy: a new method to study molecular motions in small biological samples

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Few biological molecules are available in quantities,200-400 mg,that are required for quasi-elastic neutron scattering analysis.We show that the time correlation function of molecular motions can be extracted with much less material using only the strong scattering intensity at zero energy transfer.The time axis is introduced by varying the width of the instrumental resolution function,which is achieved using multiple-chopper time-of-flight spectrometers.The elastic intensity decreases with increasing energy resolution due to molecular motions in the sample.The technical feasibility of ERS is demonstrated with 23 mg of hydrated myoglobin in comparison with the standard approach.The intermediate scattering function of hydrated myoglobin is bimodal due to contributions of dihedral transitions and delocalized solvent dependent motions. The latter is absent in dehydrated samples.

A-210 Microstructural characterization of hydrated cement composites of varying compositions by small-angle neutron scattering

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Hydrated neat cement composites, varying in compositions with water to cement ratio of 0.3 and after 100 days of hydration, have been charaterised with the double crystal based SANS instrument at Trombay. The accessed range of wave vector transfer is 0.003-0.173 nm^{-1}. To account for the multiple scattering effects, each composite has been investigated with varying specimen thickness. All specimens exhibit volume or surface fractal behaviour with varying degree of fractal dimensions. The results could be related to the hydration characteristics of the neat cement composites. An attempt has been made to correlate the microstructure with the relative compaction of the hydrated cement matrix.

A-211 Quantitative analysis of UH_3 in U metal and UO_2 matrices by Neutron Vibrational Spectroscopy

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The ignition of UH_3 has resulted in uranium fires. Neutron spectroscopy is proposed to quantify UH_3 in fuel at risk. Spectra of UH_3 in UO_2 and U metal were obtained using the Low Resolution Medium Energy Chopper and Chemical Excitation Spectrometers at Argonne National Laboratory and the Filter Analyzer and Disk Chopper Spectrometers at the National Institute of Standards and Technology. Analytical standards were prepared with powders of UH_3, UO_2 and U metal. Linear relationships were found between the mass of UH_3 and the integrated intensity of the inelastic scattering peak. Variations in spectrometer design on the scattering spectra and analytical figures of merit will be presented.

A-212 Small-angle neutron scattering analysis of meso- and microporous activated carbons produced from paper-mill sludge

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Small-angle neutron scattering (SANS) and N_2-BET analysis were used to perform a detailed analysis of the microscopic pore structure of a series of activated carbons produced from paper mill sludge. The microscopic structural characteristics of these carbons determined from SANS were compared with the adsorption data results. Contrast-variation SANS studies demonstrated that although carbon particles have open structures largely accessible to the solvent, they also contain closed pores. The data suggest the existence of loose carbon clusters ornamenting the surface of the bulk, graphitic carbon particles. The trends observed demonstrate that the method of production can significantly and systematically influence the structural characteristics and hence the usefulness of the carbons for various applications.

A-213 Rietveld refinement of neutron, synchrotron and combined powder diffraction data of cement clinker

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An ordinary Portland cement clinker and a NIST standard reference material clinker were characterised using neutron and synchrotron powder diffraction. Rietveld analysis was performed using the program LHPM, with individual as well as combined data sets. Comparisons were made between phase quantifications from these different data sets, and with published results from other methods. Synchrotron refinements gave results consistent with the literature phase quantifications, while the lower resolution of the neutron data resulted in refinements with poor literature correlation for tricalcium and dicalcium silicates. Upon refinement of the atomic positions of triclinic tricalcium and dicalcium silicates, relatively minor adjustments to the structures resulted in significant improvement in the fit of the phases. This suggested that the published structures are not identical to those found in clinker materials.

A-214 Fatigue Investigations on Autofrettaged Steel Cylinders Based on Neutron Diffraction Measurements

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A material conditioning process called autofrettage generates compressive residual hoop stresses at the bore of thick walled steel cylinders to mitigate against unexpected failures. A series of cyclic internal pressurization fatigue experiments was conducted on partially autofrettaged cylinders with multiple internal radial elliptic shaped cracks machined with spark erosion. The neutron diffraction strain measurement method at the SAFARI-1 research reactor has been applied to identify the plastic boundary. Plastically strained material regions were clearly identified from the full width at half maximum of the Bragg peaks and found to correspond to maxima in the tensile hoop strain values, as well to be in agreement with theoretical modeling. Theoretical fatigue life predictions based on the actual measured plastic boundary were found to give more reliable results compared to calculations based on the autofrettage pressure.

A-215 2D modelling of small angle neutron scattering spectra for aged reactor vessel materials

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The neutron radiation and the high working temperature induce important changes in the microstructure of the metallic components of nuclear reactors. These changes result in weakening of the mechanical properties which is the primary interest for the safety and life-time of nuclear installations. In order to observe micro-structural changes separately due to radiation and high temperature, first we measured the thermaly aged 15Kh2mfa steel samples samples with and without magnetic field. The observed anisotropic scattering were fitted by a 2D model. We found

that the micro-structure of aged metals shows a strong dependence on the quality of the industrial treatment and the place of sampling too. This observation was important for the analysis of the irradiated samples by SANS and for the preparing of life-time control samples

A-216 SANS measurements of irradiated reactor vessel material sample series

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SANS technique has been used to study the micro-structural changes of reactor vessel materials under thermal ageing and neutron irradiation. We have carried out experiments on samples of irradiated reactor vessel material and welded components of VVER-440 type reactors on the SANS instrument at the Budapest Research Reactor. In our measurements irradiated as well as non-irradiated samples were compared and magnetic field was applied for viewing the magnetic structure effects of the materials. The samples were irradiated in BRR at high temperature and pressure. A clear modification of the structure due to irradiation was obtained. We processed the data sets with the ITP92 code, the inverse Fourier transform programme of O. Glatter. Our microstructural observations fit well to the hardness test results.

A-217 SANS study of sintering developed nanosized structures

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Before sintering, powder particles or agglomerates of a single phase solid material are surrounded by gas or vacuum. After the sintering, the sintered body can be close to the theoretical density, a lot of remaining pores are, however, always present in the matrix. This residual porosity consists of gas-filled bubbles or empty pores. During mechanical processing there is a great reduction in the average volume of pores or bubbles, which are filled e.g. with potassium. At room temperature the potassium covers the bubbles internal surface. Raising the temperature up to 1000-1020 K, this coverage will disappear, as the potassium forms a dense gas phase in the bubbles at elevated temperatures.SANS measurements were performed at 8 and 12 Angstrom neutron wavelengths. The samples, tungsten wires, were measured at 1000 K, 800 K, 600 K, 300 K temperatures. Analysing the anisotropic scattering patterns due to ellipsoidal shape of the bubbles, the variation of the potassium shall at various temperatures was derived.

A-218 Industrial Applications of Aggregation of Block Copolymers in Supercritical CO_2: a SANS Study

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Industrial applications of supercritical carbon dioxide rely upon the rather selective and easily adjustable solvent ability of carbon dioxide. CO_2 near the critical point is a poor solvent for high molecular weight hydrocarbon polymers, while being a very good solvent for fluorinated polymers. By increasing the pressure, CO_2 becomes a good solvent even for HMW hydrogenated chains. Specially engineered amphiphilic di-block copolymers, with a CO_2-philic and a CO_2-phobic portions, are expected to undergo trough a monomer-aggregate transition when the solvent density of the $scCO_2$ changes. Here results of SANS experiments will be presented for block copolymers in which for both the solvophilic and solvophobic moieties, different chemical formulation and molecular weights have been considered. The nature of the transition will be explained in terms of two important variables such as solute concentration and solvent density (critical micellization density).

A-219 Structural Investigation on Hybrid Nanocomposites

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A fast growing interest about polymer rubbers and their interaction with several inorganic fillers has been recently observed in scientific and industrial fields, thanks to the relevant technological application in tyre development. Small particle fillers, such as silica, are expected to modify morphological and mechanical properties when dispersed in the copolymer matrix. USANS, SANS and IANS techniques can span a wide range of the scattering vector investigating several domains rich of different information about the structural change in the modified polymer rubber, going from a fractal structure to the specific interface between hydrocarbon group and silica. Aging and thermal effects can be also exploited in order to show the mechanism of adhesion or detachment of silica aggregates from the rubber matrix. The copolymer rubber involved in this study is an elastomer Styrene-Butadiene where two kind of silica have been added in different amounts. Elastomer-silica system cointaining small amount of an organosilane coupling agent were also studied.

A-220 Neutron in the service of the combined material testings at the Budapest reactor

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Combined measurements have been carried out at the Budapest research reactor, where the dimension of the radiography station was extended for the purpose to control the condition of helicopter rotor blades in the different period of their life time. High resolution radiography pictures were taken to find anomaly in the distribution of resin materials at the core-honeycomb-hull interfaces, failure at the ”adhesive filling” and possible bondline flaws. Parallel to the radiographic visualisation vibration tests using the method of statistic energy analysis (focused on damping and energy distributions and propagation) served for control of dynamic behaviour of different aged structures. As a result of the work it is suggested that the combined application of the neutron-, X-ray radiography and vibration diagnostics might be a very useful method for the condition monitoring of helicopter rotor blades and other similar composite structures.

A-221 Neutron -, X-ray - and electron diffraction measurements for the determination of $\gamma/\gamma\prime$ lattice misfit in Ni-base superalloys

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In this contribution measurements of $\gamma/\gamma\prime$ lattice misfit in modern Ni-base superalloys will be presented. Misfit values are very important due to the influence on the precipitate morphology and stability. High resolution diffraction instruments using neutron, X-ray and electron radiation are suited for the determination of lattice parameter of the two phases. The methods are complementary because of different scattering sensitivity and allow comprehensive interpretation.

A-222 Gamma Neutron Transmutation as a Tool for Processing NPP High Active Waste

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The radwaste problem would be solved cardinally, if it was a success in finding a nuclear technique, which would allow transuranium actinides to be destroyed by fission and fission products to be transmuted into stable isotopes. Then, the nuclear fuel cycle could be altered so as to be ”wasteless”. A method proposed for the purpose is based on the photonuclear reactions induced by the magnetic bremsstrahlung γ-radiation of relativistic electrons. Photoneutrons to be produced thereby are used as another canal for transmutation. Method parameters, such as aproductivity, energy ”cost” of a transmutation event and so on, are considered. The method enables the above nuclides to be transmuted at the rate comparable to that of their build-up in NPP reactors under reasonable energy consumption. The method could be realized by some means depending on the γ-radiation nature.

A-223 Elaboration of the Effective Neutron Generator for Short-lived Isotopes Analysis

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Results of the elaboration of laboratory impulse laser generator for effective material activation analysis owing to short-lived isotopes (10 ms - 100 s) are delivered. The generator function is based on the acceleration of laser-produced plasma containing deuterium by external magnetic field that leads to plasma bundle velosity up to 3 km/s. Fast neutrons yield up to 1 billion in pulse duration not more than 100 ns is waiting. The main peculiarity of such neutron source is connected with substantial smaller x-ray background, accompanied a neutron irradiation process in traditional types of generators. It gives a good possibility to reduce the dead time of registration systems as well as to increase the sensibility of analysis. The neutron generator may be more effective for the content determination of isotopes such as carbon-12, nitrogen-14, oxigen-16, magnesium-26, gold-197 and some others.

A-224 Analysis of time structure of neutron-capture gamma-ray spectra, formed in the interaction of a nonsteady neutron flux and geophysical medium

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Presented is the analysis of time distribution of gamma-ray fluxes induced from neutron capture in studying geophysical media by pulsed neutron methods. Time structure of a gamma-ray field is presented as sum of decaying exponents and a constant background. In a case of a geophysical medium intersected by well, time decay of a far exponential curve brings information about the total neutron absorption cross-section of a geological formation and its mineralization. Reliable data about this important parameter, particularly, allows one to locate the water-oil contact in oil wells. Measurement results are presented as a random vector N with a dimensionality equal to the number of time channels. Overall, components of this vector form an integral count of events registered in each differential time channel. Time decay exponent of the gamma-ray flux is found from an overdetermined system of linear algebraic equations with coefficients depending on the components of vector N. An approximate solution of this system is found by the least square method with the use of Tikhonov regularization procedure.

A-225 Experimental Error in Measured Lattice Parameters due to Sample Displacement

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A significant error may occur in the measured lattice parameters when the sample is displaced from the diffractometer center. For time-of-flight

neutron diffractometers, this error gives rise to different lattice parameters not only for detectors at different 2Θ but also for detectors at the same 2Θ but on opposite sides of the incident beam. Tests made on GPPD at IPNS, Argonne National Laboratory, show that the source of this error largely comes from a change in the diffraction constant, L PsinΘ, where L is the total flight path. Modeling of the experimental data indicates that in order to achieve a precision of 10^{-4}, a typical requirement for strain measurements, for a wide angular range of detectors, the sample should be positioned to within 0.2 mm of the diffractometer center.

A-226 Structural Integrity Assessment based on the HFR Neutron Beam Facilities

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Neutrons are becoming recognized as a valuable tool for structural integrity assessment of industrial components and advanced materials development. Microstructure, texture and residual stress analyses are commonly performed by neutron diffraction and a Pre-Standard is under development for the latter. Furthermore neutrons provide for defects analyses, i.e. precipitations, voids, pores and cracks, through small angle neutron scattering (SANS) or radiography. At the HFR twelve beam tubes are installed for the extraction of thermal neutrons for such applications. Two of them are equipped with neutron diffractometers for residual stress and structure determination and have been extensively used in the past. Several other facilities are currently being reactivated and upgraded. These include the SANS and radiography facilities as well as a powder diffractometer. This presentation gives an overview of the status for the facilities and some recent applications of the installations are shown.

A-227 Time-of-flight neutron diffraction investigations of elastic and anisotropy strains in fatigued austenitic stainless steel

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The study of the elastic and plastic properties of the austenitic base matrix and martensitic precipitates induced during the high cycling tensile-compress loading was made in the in-situ stress rig experiment on the ENGIN instrument at the ISIS facility. It is observed that the elastic constants of the austenitic and martensitic phases for the axial and transverse directions determined by the Rietveld refinement are independent of the level of fatigue while the bulk value of the Young's modulus measured by a gauge extensometer decreases with increasing fatigue. The austenitic elastic response is linear throughout the measured stress range (0 - 500 MPa) while the martensitic response is linear only up to 180 - 200 MPa, the same level of stress at which bulk plasticity is observed. Herewith the ratio of the martensitic elastic constants in the axial and transverse directions is almost twice that expected based purely on the value of the Poisson's ratio.

A-228 The residual stress relaxation after fatigue in fine grained steels.

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Since the Residual stress (RS) relaxation during and after fatigue can be highly detrimental for the life of components, a study of their variation in weldings, after cyclic loading, simulating the operating conditions has been carried out. Plates of S96QL ferritic steel were investigated in as-welded conditions and subject to 350 MPa load for 100 cycles. Both have composition 0.17% C, 0.8% Cr, 2% Ni, a yield stress about 1000 MPa and were welded with manual TIG technique. The critical points for rupture are usually the two surfaces corresponding to the Heat Affected Zone (HAZ) were cracks can initiate if RS go tensile during relaxation. A transverse-to-weld scan was done at mid depth to identify the maximum RS, which turned out to be both in the HAZ and at the centre-weld. A through-depth scan was performed to follow the RS profile in both points. Results show that stresses decrease in the fatigued specimen: tensile stresses relax to moderate values below 300 MPa and compressive ones below 100 MPa. Especially in the weld the degree of triaxiality decreases after loading, which implies a lower risk to crack initiation and void formation.

A-229 Neutron diffraction study of martensitic transformation in austenitic stainless steel under low cycling tensile-compress loading

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The fatigue behaviour of austenitic stainless steel, in which a martensitic phase is formed due to plastic deformation, is of some interest for practical reasons. Before [1] we have reported results of the first stage of the in situ stress rig experiment on the ENGIN instrument at the ISIS facility with samples from steel X6CrNiTi1810 subjected to different tensile-compressive loading cycles at a frequency of 5 Hz. In this report we describe results of the second stage of the experiment in which a series of samples subjected at 0.5 Hz was studied. Extensive information about mechanical properties of the austenitic matrix and martensitic precipitates was obtained by the processing of mechanical and neutron experimental data. [1] M.R.Daymond, J.Schreiber, Yu.V.Taran, J.S.Wright. The ISIS experimental report RB 11007, April 2000.

A-230 Residual Stress Measurements in Thermally Sprayed Metallic Coatings

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The performance of thermally sprayed coatings is significantly influenced by residual stresses. In the present research project different spraying techniques were applied to manufacture metallic NiCrAlY deposits of diverse types of microstructures. Residual stress measurements on such deposits were performed by neutron diffraction at the strain scanner D1A at ILL, and by bending/curvature measurements using profilometry. The recently designed setup at D1A employing radially focussing collimators defines a gauge volume of $0.6 \times 1 \times 10 mm^3$. The high spatial resolution along the scanning direction allowed to obtain strain profiles even for deposits of about 1 mm thickness. Estimated average stresses deduced from the neutron strain profiles agree with results from bending measurements.

A-231 Distribution of radial strain in a disc braked railway wheel measured by neutron diffraction

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Disc brakes are applied in modern trains. In contrast to block braked railway wheels the tread of disc braked wheels is not heated during braking. The absence of this annealing effect can be result in flatten of wheel sectors and the formation of cracks below the tread. Residual stresses formed during service play an important role in the crack initiation and crack growth process. In order to investigate the structural reasons of this damages strain measurements were performed by neutron diffraction at the G5.2 facility of LLB Saclay (France). Three sectors of one wheel were investigated. At sector 1 no damages are visible or detected by ultrasonic measurements. Sector 2 was about 1mm flattened at the position of the minimum radius and sector 3 contains a macro crack some millimetres below the tread detected by ultrasonic measurements. In this contribution a detailed comparison of the strain distribution in the three sectors is given and possible reasons for it are discussed.

A-232 Stresses in a formed steel

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Springback phenomenon occurs in materials, such as mild steels, used for automotive sheet metal applications. In order to study this phenomenon, FEM simulations were used to theoretically evaluate the strain and stress fields inside the formed materials and neutron diffraction measurements were performed on U-formed steel specimens in order to experimentally evaluate strains and stresses in critical regions of the samples. The experimental results proved the ability of FEM simulations to describe the state of the formed components and thus also their ability to forecast the springback effect.

A-233 Neutron determination of the residual stress in a centrifuge bowl

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An experimental study of the stress field in centrifuges for food processing and for agricultural applications was undertaken. The model, the dimensions and the material of the sample are those of the most recent line of production of the Nuova Maip company. They are also some of the largest rotors produced by that firm. The residual strains and stresses were determined by using neutron diffraction techniques before centrifugation,in order to evaluate the residual stress after fabrication and forming, and also after centrifugation,in order to evaluate the changes of stress induced by centrifugation. The upper part of the rotating bowl is investigated where the highest stress field during centrifugation is theoretically forecast to occur.

A-234 Measurement and modelling of residual stresses in a TiG weld

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Residual stresses due to TiG welding have been determined using the neutron diffraction technique and the results compared with a Finite Element model calculation. Measurements were made on a single pass, autogenous, bead-on-plate TiG weld made along the centre line of an aluminium alloy rectangular plate of dimensions 172x150x3 mm. No filler material was used. The weld is 150 mm long with the start and end pools centred approximately 12 mm from the plate ends. There is an 8 mm wide fusion band on the crown surface of the weld. Measurements were made at a neutron wavelength 1.51 Åusing the aluminium (311) reflection and a gauge volume 2 mm wide. Scans were made along transverse lines at three longitudinal positions x = 0 (the centre of the weld), x = -35 (40 mm from the start) and x = 35 mm (40 mm from the end) at mid-thickness. Stresses were derived using strain data collected in the three symmetry orthogonal directions. A full 2-D FE model was generated using ABAQUS. The agreement between the calculated and measured results is good. The longitudinal residual stresses are strongly tensile (up to 200 MPa) in the weld falling to zero at around 15 mm from the weld line with balancing compression towards the edges.

A-235 Improved Structural Integrity through Advances in Reliable Residual Stress Measurement: the Impact of ENGIN-X.

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Residual stress fields are known to have significantly effect on fatigue crack growth and hence, structural integrity. Tensile stresses have detrimental effects on fatigue lives, whereas compressive stresses can be beneficial. So damage tolerant design and maintenance requires detailed knowledge of critical crack sizes; crack growth kinetics and accurate, reliable knowledge of residual stress distributions. This paper illustrates advances in neutron residual stress measurement using examples of both deleterious and beneficial residual stress fields and their impact on remnant life assessment. It will further describe how the novel software and hardware used in the construction of ENGIN-X, the new engineering diffractometer being built at ISIS, will provide an order of magnitude improvement over present instrumentation. Finally, the impact of the new instrument on the type and quality of residual stress measurement and consequent structural integrity assessment will be forecast.

A-236 Engineering applications of Bragg edge neutron transmission

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The transmission spectrum of thermal neutrons through a polycrystalline sample displays sudden, well defined increases in intensity as a function of neutron wavelength. The shape, magnitude and location of these edges can be accurately determined by the time-of-flight technique, and hence information about the stress state, texture and phases present in the material is readily available. An advantage of Bragg edge transmission over conventional neutron diffraction is that it can use a pixellated detector to map the strain in plane samples,producing images analogous to neutron radiography. Moreover, maps of the stress state and the unstressed lattice spacing for in-plane stress fields can be achieved by tilting the sample relative to the direction of the neutron beam. Examples of the application of this technique to typical engineering problems are presented in this work.

A-237 Residual Stress Instrument at the HANARO

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Residual stress measurements by neutron diffraction have been available since early 2000 at the HANARO, KAERI. Recently a newly developed linear position-sensitive detector of 100mm width $\times$ 200mm height was fabricated and tested, and will be put into operation in May 2001. Replacing the old detector by that new PSD with high vertical aperture, we expect to cover the vertically divergent scattering beam fully from the focusing monochromator and to get about two time higher measuring efficiency. We report here the installation of the HANARO residual stress instrument and its early measurements on the samples of VAMAS and some engineering works.

A-238 Neutron Strain Scanning of industrial components

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The neutron strain imaging technique is a non-destructive method for the determination of (residual) stresses deep inside matter. Often it is the only possible technique, particularly when the specimen is large or should not be modified by the measurement in order to perform other tests later on. Samples can be ”real” engineering components and no special sample treatment is necessary before a measurement. The strain imager at ILL shows high lateral resolution and is suitable as well to interface and surface problems. Several sectors of industry have used the facilities of the Institut Laue Langevin in Grenoble for their product research, control and development. The poster will describe the principle of the technique and some examples.

A-239 Residual stress in clinched joints of metals

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Diffraction methods are used for the determination of characteristic residual stress (RS) distributions in undismantled clinched samples for the assessment of the influence RS on the mechanical behaviour of clinched joints. While X-ray diffraction enables merely the determination of near surface RS distributions the higher penetration depth of neutron radiation allows the determination of triaxial RS states inside the material. In addition the complex geometry of clinched joints restricts the application of X-ray RS analysis. Therefore a combined RS determination by X-ray and neutron diffraction has been used to get an expressive assessment of the RS distributions in the immediate vicinity of clinched joints. Two different materials with different mechanical behaviour were used for clinching as well as two different common clinching techniques.

A-240 The study of remanent FeCoV/TiZr supermirrors.

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Polarized neutron reflectometry with phase analysis (PNRPA) technique can be realized by several methods. One of the possibilities is to use the scheme ïremanent polarizer - sample - remanent analyzer, provided the polarizer and the analyzer can be rotated about their surface normals. Thus, properties of remanent supermirrors is important for this scheme and have to be studied. For FeCoV/TiZr supermirror with a remanence the spin-up and spin-down neutron reflectivities were measured at different applied fields as a function of λ_{perp}=λ/sinθ. Different groups of layers are responsible for reflection at different values of λ_{perp} (thinner layers give rise to reflection at smaller λ_{perp}). Thus, magnetization processes in different magnetic layers of the supermirror can be studied. It was found that all magnetic layers remain to be magnetized opposite to the fields of magnitude up to 30 Oe.

A-241 Designing of Multilayered Systems with desirable properties

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We present an analytical method to deal with multilayered systems, which helps to prepare systems with desirable properties. In particular, we show how to prepare a system with forbidden second order Bragg diffraction.

A-242 Neutron standing wave and spatial beam splitting methods for neutron grazing incidence studies of layered structures

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The specific features of the formation of the neutron wave field at reflection, refraction, or channeling of polarized neutrons in layered magnetically noncollinear structures are studed theoretically and are demonstrated experimentally. The results of the investigation of magnetically noncollinear structures, such as the magnetic layer in an inclined magnetic field, magnetic-nonmagnetic interface locally noncollinear with the magnetic field, bylayer with an exchange interaction of atoms, and the domain structure in a thin iron layer by the method of neutron standing waves and the method of spatial beam splitting caused by spin flip are presented.

A-243 Neutron spatial beam-splitting for improvement of polarization analysis

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Reflection of polarized neutrons from the Co film on glass has been investigated. An external magnetic field is applied under an angle to the sample surface. The increasing of polarization degree of the beam in off-specular reflection region in comparison with the incident beam has been observed. It has been shown that joint using of reflection from magnetized mirror and the effect of spatial beam-splitting increases an efficiency of polarization analysis at investigations of layered magnetically non-collinear structures.

A-244 Inelastic scattering of neutrons on ultrasonic excitations in layered structures

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The effect of ultrasound on the grazing reflection of polarized neutrons in the conditions of neutron standing wave formation in the layered structures Cu/Ti/FeGd(Gd)/Ti/Cu is investigated. The dependence of the neutron reflection coefficient on the amplitude and frequency of the ultrasonic wave is obtained. Changes in the positions of the nodes and antinodes of the neutron standing wave field due to energy exchange between the neutron and the sonic phonon is first detected.

A-245 Magnetic Exchange Coupling in FeCr/Cr Superlattices

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To understand the mutual influence of the magnetic layers in a magnetic superlattice on each other it is desirable to continuously tune their magnetic properties. This allows to systematically study the resulting effect e.g. on the ordering temperatures in the system and on the exchange coupling. In practice this has hardly ever been achieved. Based on our previous studies of Fe/Cr superlattices [1] we have replaced the Fe layers by an $Fe_{x-1}Cr_x$ alloy in a wide concentration range x. Neutron reflectivity and MOKE measurements show that increasing x from zero to 0.8 reduces the Curie temperature T_C of the $Fe_{x-1}Cr_x$ to zero. Although a strong effect on the Néel temperature T_N of the Cr interlayers due to a proximity effect is expected, neutron scattering data only indicate a drop of T_N from 500 to 330 K at $x = 0.8$. The exchange coupling properties are dramatically modified compared to the Fe/Cr system. Whereas our Fe/Cr samples exhibited a strong non-collinear coupling mediated by frustrated spirals of antiferromagnetic Cr [1], the FeCr/Cr samples exhibit only strongly temperature dependent *collinear* ferro- or antiferromagnetic coupling. These results can be understood by considering the modification of the interface topology upon alloying highlighting the role of structural and magnetic roughness for the magnetic exchange coupling. [1] A. Schreyer et al., Phys. Rev. Lett. **79**, 4914 (1997).

A-246 Interlayer Coupling in Er|Tb superlattices

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We report on neutron diffraction experiments of Er—Tb multilayers. These have revealed different coupling phenomena depending on the layer thickness. In a sample $Er_{20}|Tb_5$ long ranged ordered helical and c-axis modulated structures as in bulk Erbium occur.The helical structure is stable up to temperatures higher than the bulk critical temperature. Measurements with synchrotron radiation have shown that the helical structure is also formed in the Terbium layers. Apparently proximity effects change the balance between anisotropy and RKKY interaction in a way to favour helical magnetic order. Additionally the neutron diffraction shows a doubling of the chemical superstructure as well for the $Er_{20}|Tb_5$ as for the $Er_5|Tb_{20}$ sample, pointing to antiferromagnetic coupling of subsequent layers of one element. Neutron reflectivity measurements are in progress to improve the understanding of this coupling.

A-247 Neutron Reflectivity Study of Gd/Cr multilayers

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In order to study the magnetic structure of Gd/Cr multilayers we have carried out polarized neutron reflectivity measurements on a Gd(30 Å)/Cr(30 Å) multilayer grown by MBE on a MgO(001) substrate. The reflectivity of the multilayer in the temperature range 12 to 300 K, with and without the application of a magnetic field perpendicular to both the incident and scattering wavevector, was measured. X-rays reflectivity data from a much wider Q-range, gives the thickness of about 37.5 and 30.0Åfor the Cr and the Gd layer respectively, while the interface roughness is limited to about 5 Å. At T=12 K, for zero magnetic field there is no net magnetization, whereas for an applied field of 2.34 kG simulation of the two spin polarizations gives a mean magnetic moment per atom of $3.30\pm0.12\mu_B$ for the Gd and $0.13\pm0.12\mu_B$ for the Cr. Both magnetic moments align ferromagnetically. At this temperature hysteresis exists and the saturation field is about 1.2 kG while the coercivity is about 0.6 kG. Magnetization measurements versus magnetic field have shown unambiguously in-plane anisotropy in the studied sample so we do not expect any perpendicular magnetization component.

A-248 Magnetic properties of $Fe_{1-x}Co_x$/Mn/$Fe_{1-x}Co_x$ trilayers as determined by polarized neutron reflectometry

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Epitaxial trilayers of the type $Fe_{1-x}Co_x$/Mn/$Fe_{1-x}Co_x$ (x=0 to 0.75) with interlayer thicknesses in the nm range show a strong interlayer exchange coupling that depends strongly on the Mn thickness and the ferromagnetic material (i.e. x). Using polarized neutron reflectometry, we have investigated the field dependent orientation of the magnetization in the individual layers. In the remanent state, a non-collinear orientation of the ferromagnetic layers is found; the orientations of the magnetic moments are found to be the result of competition between the crystalline anisotropy and the interlayer exchange coupling. For some stoichiometry of the ferromagnetic layers, a clear indication of a net magnetic moment within the Mn interlayer is found.

A-249 Neutron wave channeling in the structure Cu(10nm)/Ti(150nm)/Cu(100nm)/glass

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Experimental investigations of the channeling of neutron waves in the structure Cu(10nm)/Ti(150nm)/Cu(100nm)/glass are conducted. Three resonance modes detected at neutron tunneling through a 30 nm copper layer and at neutron radiation immediately out ofa 150 nm titanium layer are observed. The channeling length is determined. It is concluded that monochromatization increases as the wavelength of the channeled neutrons grows.

A-250 Polarised neutron reflection (PNR) study on the magnetisation reversal in ferromagnetic/antiferromagnetic Co/CoO multilayers

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Polarised neutron reflectometry (PNR) is used to investigate the magnetisation reversal in a ferro- antiferromagnetic Co/CoO film system exhibiting an extraordinarily strong exchange bias. After field cooling below the Néel temperature the present $[Co(16.4\ nm)/CoO(2\ nm)/Au(3.4\ nm)]_{20}$ multilayer is characterised by a shifted magnetic hysteresis loop with simple unidirectional properties. By PNR we observe an asymmetry of the in-plane projection of the net magnetisation in the reversal processes on opposite sides of the hysteresis loop. These results provide a detailed understanding of the observations made by SQUID magnetometry: A sharp transition from one magnetisation state to the other at decreasing fields (coming from the cooling field direction) as opposed to the reversal for increasing fields (returning to the cooling field direction) which is characterised by magnetisation rotation.

A-251 Total neutron reflection at ultrasonic excitation of the mirror

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The reflection of neutrons from a continuous mirror is investigated as a function of the frequency and amplitude of longitudinal and transverse ultrasonic waves excited in the mirror. The off-specular reflection of neutrons due to energy exchange between the neutron and the sonic wave is discovered. In the region of total reflection the intensity of the off-specular reflection of neutrons from the mirror is a periodic function of the neutron wavelength.

A-252 Analysis of Neutron Reflectometry Data by Monte Carlo Technique

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Neutron Reflectometry has been extensively used for obtaining layer densities and interfacial roughnesses with high resolution in thin films. Polarized neutron reflectometry can obtain magnetic moment density and magnetic structure in thin films of magnetic materials. One can generate the reflectivity pattern [R(Q)] by Parratts' formalism [1]. Model of the film structure is generated through χ^2 (fit between model and data in R(Q)) minimization in parameter space. In the present work we have tried a scheme for obtaining the structural parameters of a thin film by fitting reflectivity data through a Monte Carlo technique earlier used by us for amorphous systems. [1]L.G.Parratt, Phys. Rev., 95,(1954)359 [2]On deputation from Solid State Physics Division, Bhabha Atomic Research Centre, Mumbai 400 085, India

A-253 A Remanent Fe/Si Supermirror Transmission Polariser

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The material combination Fe/Si on a Si substrate was chosen because the contrast in the scattering length densities is high for spin-up neutrons and almost vanishes for spin-down neutrons and its neutron absorption is low. Remanence can be achieved for the Fe using DC magnetron sputtering. The problems inherent in this system are the interdiffusion of Si into the Fe, the non-ideal matching for spin-down neutrons and the presence of large stresses. However it is the anisotropic stress which causes the magnetic remanence. Optimisation of the sputtering parameters (e.g., partial pressures of Ar, N_2 or O_2) can deal with one of the problems but usually with an adverse effect on one of the others. A potential solution is to introduce a thin SiO_x-layer to prevent interdiffusion in the first place. The matching between Fe and Si worsens only slightly and the anisotropic stress is hardly affected.

A-254 Off-specular polarized neutron reflectometry from a periodic array of Co-disks

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Using off-specular polarized neutron reflectometry with neutron spin analysis, we determined the magnetic properties of a large array of in-plane magnetized ferromagnetic Co disks. Satellites are clearly observed in the off-specular reflectivity, due to the lateral periodicity of the disk array. Using polarized neutrons, the intensity of the satellites in the off-specular reflectivity was studied as a function of magnetic field applied in the sample plane. The magnetization reversal was investigated by spin analysis of the off-specular reflected neutrons.

A-255 Off-specular polarized neutron reflectometry study of magnetic dots with a strong shape anisotropy

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We have measured the off-specular polarized neutron reflectivity of a regular array of rectangular magnetic polycrystalline Co-dots, which were prepared by a combination of electron beam lithography, molecular beam deposition and lift-off processes. The dots have a length-to-width ratio of 4:1, imposing a strong shape anisotropy. The intensity of the off-specular satellite reflection was monitored as function of the magnetic field applied parallel to the dots and in the plane of the film, allowing us to analyse the magnetization reversal process using the four spin-polarized cross-sections.

A-256 Determination of the Temperature Dependence of the Coercivity in Fe/Cr(110)-Multilayers

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For single iron layers on a bulk chromium crystal it was shown that the coercivity as a function of temperature has a discontinuity at 130 K which is close to the temperature where bulk Cr exhibits a phase transition from a transverse to a longitudinal spin density wave. We prepared for our studies an [1.2 nm Fe/26 nm Cr]15 multilayer in (110)-orientation. In our experiment we determined the complete magnetic structure of the multilayer: the antiferromagnetic phases of the chromium with neutron diffraction and the ferromagnetic order of the iron layers with polarised neutron reflectometry. We did not detect any sign that the different magnetic phases in chromium affect the magnetic reversal of the iron layers. The coercive fields as a function of temperature can be perfectly fitted by an exponential.

A-257 Investigating magnetic proximity effects in NiO/Pd with polarized neutron reflectometry

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We investigated using polarized neutron reflectometry NiO/Pd heterostructures for the presence of a magnetic proximity effect, which is expected to produce an induced ferromagnetic moment in Pd. Using a specific isotope mixture of Ni in the preparation of NiO, the chemical contrast across the Pd/NiO interface was greatly suppressed, thus enhancing sensitivity to magnetic contrast at the reflecting interface. Despite enhanced sensitivity

for any induced magnetic moment, no evidence for a proximity effect was observed. If present, the magnetic moment per Pd atom could not be more than 0.01 mu_B, regardless of Pd layer thickness, crystalline interface orientation, and number of NiO/Pd bilayers.

A-258 Extracting buried twists with polarized neutron reflectometry

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Polarized neutron reflectometry can extract the depth-dependent magnetization of magnetic thin films more precisely than other techniques which are limited to a measurement of the average moment or the moment at the surface. Measuring the reflectivity first with neutrons glancing off the front surface and again with neutrons glancing off the back yields eight spin cross-sections instead of the usual four. Differences in the front and back reflectivities indicate the presence of magnetic twists in the sample. We have applied this method, as well as the more conventional front-surface-only reflectometry, to study a soft ferromagnet exchange-coupled to a hard ferromagnet in fields from 5 to 50 mT. Bulk magnetometry suggests spring-magnet behavior, and we find the twist in the soft phase extends into the hard phase at fields too small to induce irreversibility. This finding is at odds with simple mean-field models of spring-magnets.

A-259 Off-specular neutron scattering experiments with full polarization analysis on an Fe/Cr multilayer

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We present results of spin-resolved specular and diffuse neutron reflectivity experiments that were performed on a magnetic multilayer sample at room temperature at different magnetic-field magnitudes and orientations using a polarized ^{3}He gas spin analyser, the new wide-angle neutron spin filter. Owing to the correlated roughness structure of the sample, the obtained reflectivity maps possess very rich diffuse-scattering patterns. The results obtained with full polarization analysis allow us to separate the effects due to interface roughness of chemical from those of magnetic origin. The use of the recently proposed supermatrix method for data analysis [A. Rühm et al., Phys. Rev. B 60 (1999) 16073] is essential due to the magnetic non-collinearity of the studied Fe/Cr multilayer.

A-260 Surface excitations in thin helium film in silica aerogels.

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The interest to study the excitations in a 4He film adsorbed on silica aerogel is, that 4He films in the aerogel shows a different behavior compared to films on other substrates as e.g. graphite. Apart from this, bulk helium in aerogel, graphite or vycor shows a different exponent for the temperature dependence of the superfluid component near the lambda-transition. This may be related again to the different interface properties of the helium film excitations on the fractal aerogel surface. The results of the first experiment with thin helium films on the DIN-2PI spectrometer at IBR-2 reactor are presented here. Experiment shows the good performance of this spectrometer even without cold source. In particular the very low background is an absolute necessary feature to observe the very small signals.

A-261 Correlated interface roughness and domain structure in FeCoV/TiZr multilayers studied by off-specular neutron reflectometry

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In this work we present the results of time-of-flight polarized neutron reflection (PNR) experiments with spin-analysis (after the sample) on [FeCoV/TiZr] multilayers with applied fields parallel to the in-plane easy axis of magnetization. In the current study we used the reflectometer CRISP of ISIS in combination with a multichannel IRI-PNPI analyzer. Our measurements show magnetic off-specular neutron scattering which yields useful information on the field dependent magnetic domain structure and on the in-depth correlated interfacial roughness in magnetic multilayers.

A-262 Magnetization Reversal Processes in an Exchange Biased Co/CoO Film

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Polarized neutron reflectometry (PNR) is utilized to investigate the magnetization reversal processes in an exchange biased Co/CoO polycrystalline film. We demonstrate how specular PNR may be used to determine the mean square deviation of the lateral domain orientations. This quantity can be related to the mechanisms of the magnetization reversal processes. The reversal of the Co magnetization in the sample is studied as a function of field, temperature and field training. After field cooling the sample below the blocking temperature, the reversal of the untrained film occurs through uniaxial domain switching. In contrast, rotation of the magnetization in domains is observed during reversal of the trained film. Above the blocking temperature, uniaxial domain switching again dominates the reversal process. The results clearly indicate the importance of irreversible changes in the interfacial coupling. It is most likely these changes are a result of some reorientation of the antiferromagnetic domains. Work supported by US-DOE, Office of Science contract 31-109-ENG-38.

A-263 Domains and interface roughness in Fe/Cr multilayers: Influence on the GMR effect

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The non-colinear coupling of magnetic moments in a Fe-Cr multilayer stack is laterally constrained to domains seen by off-specular spin-flip half order Bragg-sheet scattering. The magnetic field dependence of the coupling angle, which scales with the GMR effect, is directly obtained through the cut-off of the off-specular Bragg-sheet scattering at the neutron spin-dependent critical angles. Interface roughness manifests in off-specular scattering through full order Bragg-peaks, is independent on a magnetic field and thus does not influence the magnetic field dependence of the GMR effect. 3-dimensional model calculations performed in the DWBA with supermatrix formalism allows for a detailed comparison of the data in particular in regions with interference from spin-dependent critical angle scattering with off-specular scattering of the multilayer structure. An excellent agreement of the 3-dimensional model calculation with the data is obtained.

A-264 Spin-flop transition in an antiferromagnetic superlattice

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Magnetic ordering in an antiferromagnetically (AFM) coupled uniaxial Fe/Cr(211) superlattice was studied in order to determine how the transition evolves as a function of the angle $alpha$ between the field H and in-plane easy axis and how the transition proceeds from one of the surfaces in this finite AFM system. Magneto Optic Kerr Effect measurements showed differences in the orientations of the Fe layer magnetizations as a function H for different values of α. Two regimes could be distinguished: α¡3° and α¿3°. While at high field all Fe layers are ferromagnetically ordered, on reduction of H, due to the coupling, an AFM ordering is achieved. Polarized neutron reflectometry (PNR) determined that depending on $alpha$ the top Fe layer becomes aligned either parallel or antiparallel to the field, when H decreases from the saturation to zero. With PNR the non-collinear magnetic ordering of the Fe layers was determined above the spin flop field. Work was supported by US DOE, Office of Science contract # W-31-109-ENG-38.

A-265 Spin density wave controlled by the superlattice period in Cr(001)/Sn multilayers with Sn monatomic spacer layers

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Exotic spin-density-wave (SDW) states in Cr(001)/Sn multilayers were studied by neutron diffraction. Four multilayers with different Cr thickness (t_{Cr} = 4 ,8, 12 and 16 nm) in Cr/Sn bilayers were synthesized on MgO substrates. Total thickness of Cr was kept constant to be 240 nm in all these samples. In the multilayers except t_{Cr} = 4 complicated incommensurate SDW states formed at low temperatures in which wave vectors of the SDW were determined by the superlattice period.

A-266 Spin-resolved unpolarized neutron off-specular scattering for magnetic multilayers studies

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We report on a new method using unpolarized neutron reflection for the investigation of magnetic structure in exchange coupled magnetic multilayers. Strong anomalies in the off-specular scattering are determined by the spin-flip process originating from magnetic fluctuations in the multilayer. This spin-flip selective process enables to use unpolarized neutrons. A complete 3-dimensional data analysis of specular and off-specular scattering has been employed to verify atomic spin correlations and the in-plane domain distribution in Fe/Cr multilayers, a typical system showing the GMR-effect.

A-267 The polarized neutron and X-ray reflectivity study of the interface diffusion of the annealed permalloy/NiMn thin film

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The neutron and X-ray reflectivity were used to study the interface diffusion of annealed permalloy/NiMn thin film. The polarized neutron reflectivity was performed at C5 beamline of the Chalk River Laboratory with (+,+) and (-,-) settings. The X-ray measurements were carried out at Spring-8 synchrotron facility. The X-ray data showed a strong interface diffusion between the buffer layer and NiMn layer after annealing the sample. However, the interface diffusion between the permalloy and NiMn layers cannot be determined accurately due to the low contrast of electron density, although 8 orders of magnitude had been collected under synchrotron radiation measurement. The polarized neutron measurement, although only four orders of magnitude had been done, reveals a better data to shed a light on the interface diffusion of this exchange bias sample.

A-268 Polarized Neutron Reflectivity Studies of the Exchange Bias in CoO/Co Multilayers

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The exchange bias (EB) phenomenon is associated with interfacial coupling between ferromagnetic and antiferromagnetic spin structures, resulting in an unidirectional magnetic anisotropy. The EB is expressed by a shift of the hysteresis loop (exchange field H_E) and an increase of the coercitivity field (H_c) by field cooling through T_N. We have investigated these two effects by MOKE and PNR. Neutron hysteresis loops were taken at 310 K (T_N (CoO) = 291 K) and 240 K by scanning the magnetic field and detecting the 4 reflectivities (R++, R+-, R-+, R–) at the position of the first multilayer peak. From such scans we infer that the magnetisation reversal does not occur by in-plane rotation but rather through domain wall movement. The reversal process remains the same for all temperatures and for both coercivity fields. Furthermore, diffuse scattering occurring during reversal supports the notion nucleation and wall movement instead of rotation.

A-269 Neutron Reflectometry and Magnetic Structure of Multilayers with Strong Interfacial Exchange Coupling

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Following our recent experiments at the neutron reflectometer (CRISP) at the Rutherford Appleton Laboratory we present a detailed study of the magnetic structure in the growth direction of multilayers made of Gd and Co layers. For the same purpose we analyse the magnetization determined by other techniques (SQUID). Such multilayer system is composed of two ferromagnetic layers that are antiparallel coupled at the interface and represents a giant artificial ferrimagnet. Comparison of experiments with profiles obtained from mean field simulations leads to a global picture of ferromagnetic layers with reduced moments and some interface effects.

A-270 Neutron Compton Scattering Studies of Stretched Polyethylene

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The mean kinetic energy of hydrogen and carbon atoms in unstretched and stretched polyethylene samples has been measured by neutron Compton scattering on the electronVolt spectrometer (eVS) at ISIS spallation source. Assuming that these atoms move in harmonic potential, the vibrational frequencies of the ground state and torsional energies have been calculated and compared with the existing data and calculations. The results obtained on deuterated and non-deuterated samples are compared.

A-271 Combining Cold Neutrons and Hard X-rays to Characterize Nanoscale Calcium-Silicate-Hydrate Gel in Cements

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Small-angle neutron and x-ray scattering (SANS and SAXS) have been combined to investigate the nature of solid calcium-silicate-hydrate (C-S-H) gel in hydrating cement. Using water and methanol SANS contrast variation methods together with absolute-calibrated double-crystal SAXS studies, we have resolved a discrepancy between published SANS and SAXS-measured surface areas in cement [1,2], determined a mean formula and density for solid C-S-H independent of existing models, and related these results to those of complementary quasielastic neutron scattering studies of free and bound water in cement [3]. [1] J.J. Thomas et al., Cem. Concr. Res., 28, 897 (1998). [2] J.J. Thomas et al., Concr. Sci. and Eng., 1, 45 (1999). [3] R.A. Livingston et al., Neutron News, 11 (4), 18 (2000).

A-272 Structure and Piezoelectric Effect in Ferroelectric PZN-PT Single Crystals

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$Pb(Zn_{1/3}Nb_{2/3})O_3$-$PbTiO_3$ (PZN-PT) are potentially valuable ferroelectrics with large piezoelectric strains (1.7%). Several mechanisms have been suggested, including a rhombohedral to tetragonal phase transition [1], two-phase coexistence [2] and micro-domain formation [3]. Each has a distinct diffraction signature including peak splitting, peak shifts, superlattice reflections and/or diffuse scattering. Neutron diffraction was conducted on D10 at ILL using PZN-4.5%PT cubic crystals ([111], [001] orientations). The structure was perturbed by (i) heating, and (ii) application of an in-situ electric field. No superlattice reflections or diffuse scattering were observed at any temperature or voltage (background $\sim 10^{-4}$). Heating experiments on a [111] crystal revealed a phase transition to tetragonal at 393K (± 3) and cubic at 420K (± 3). Electric fields up to 40kV/cm were applied to [001] crystals, causing an immediate, linear tetragonal distortion. [1] D-S Paik, S-E Park, S Wada, S-F Liu and TR Shrout, J. Appl. Phys., 85 1999, 1080. [2] U Belegundu, XH Du, LE Cross and K Uchino, Ferroelectrics, 221 1999, 67. [3] LE Cross, Ferroelectrics, 151 1994, 305

A-273 A Photon Neutron Source Driven by a Laser Synchronized with an Electron Beam

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The concept [1] needs to use of expensive electron beams with an energy up to 100 Gev. Advanced laser techniques allow the concept to be realized at electron accelerators with the energy $\sim$ 1 Gev. In view of that, a method is proposed to produce photons of high energy by synchronized interaction of electron bunches with a pulsed laser beam oscillating in a cavity. Cavities of infrared lasers could be used as "laser undulators" to apply the method with the best advantage. Spectra, intensities and polarization of hard X- and $gamma$-rays to be generated by such laser undulators and secondary photoneutrons to be produced in ^{9}Be and ^{13}C targets have been estimated for some new laser-electron-photon schemes and beam parameters designed for electron accelerators of the coming generation. [1] I.P.Eremeev, A.L.Barabanov, Nucl. Instr. Meth., A 405, 225 (1998).

A-274 Model of separated form factors for unilamellar vesicles

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Information about the internal membrane structure is mainly from X-ray diffraction experiments on multilamellar vesicles. The structure of most biological membranes is realised by a single bilayer membrane. Large unilamellar vesicles are a more biologically appealing model of the lipid bilayer than multilamellar vesicles. The SANS and SAXS experiment on the unilamellar vesicles from model biological membranes is a new prospective method for determination of the membrane structure [1,2]. The problem of accurate simultaneous evaluation of the vesicle radius, polydispersity and the internal membrane structure is not solved yet in SANS and SAXS experiment. New model of separated form factors is proposed to describe scattering curves from the unilamellar vesicles. 1. H. Schmiedel, P. Joerchel, M.Kiselev, G. Klose J. Phys. Chem. B 105 (2001) 111-117. 2. P. Lesieur, M.A. Kiselev, L.I. Barsukov, D. Lombardo. J. Appl. Cryst. 33 (2000) 623-627.

A-275 Atomic-partial vibrational density of states of i-AlCuFe quasicrystals

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We have previously studied the neutron-weighted vibrational density of states (VDOS) g(E) in isotopic-substituted samples of the i-$Al_{62}Cu_{25.5}Fe_{12.5}$ quasicrystal. Now the atomic-partial vibrational spectra Al-, Cu- and Fe-partial g(E) have been separated in a new self-consistent calculation. The partial Debye-Waller factors are calculated by means of an iteration process with starting values of g_i(E) (i = Al, Cu, Fe) as given in [R.A. Brand et al., Phys. Rev. B 62 8849 (2000)]. The partial contributions of 2- and 3-phonons scattering is simultaneously calculated. The sum of these contributions (after appropriate normalization) and the calculated background (due to frame-overlap) were subtracted from the raw data and one-phonon atomic-partial g_i(E) have been obtained for all three components. Thus the total g(E) has also been obtained and this has been compared with thermal properties.

A-276 Experimental Studies of Phase Transitions in Perovskites

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As part of an extensive experimental program on the phase transitions in perovskites neutron powder diffraction has been used to examine the structural changes of the rare-earth aluminates $La_{1-x}Pr_xAlO_3$ over a wide range of temperatures. At room temperature the aluminates adopt the rhombohedral perovskite structure in space group The rhombohedral structure is characterised by rotation of the oxygen atom octahedra about the threefold axis, the rotation decreasing as the temperature is increased, until a transition to the cubic structure is observed. The transition temperature is observed to increase as the Pr content increases. In the Pr rich compounds a further series of phase transitions are observed as the temperature is lowered to 20 K. For example at room temperature $PrAlO_3$, has a rhombohedral structure in space group R-3c which transforms to an orthorhombic structure in Imma at about 205 K and finally to a monoclinic, C2/c, structure at about 150 K. These are other experimental results will be presented.

A-277 The collective atomic dynamics of liquid Rb(x)Sb(1-x)

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The microscopic dynamics of liquids is very sensitive to a change of the interatomic forces. While in liquid alkali metals inelastic peaks (Brillouin peaks) or at least shoulders are visible in the dynamic structure factor S(Q,ω) up to Q=Qp where Qp is the position of the principle maximum of S(Q), such collective excitations have not yet been observed in liquids with mainly ionic and covalent bonds. To study the transition between different types of interatomic bonds inelastic neutron scattering experiments have been performed on the binary system Rb-Sb in the liquid state. For different concentrations dispersion relations of the longitudinal current correlation function and frequency spectra could be obtained. With increasing content of Sb a considerable shift of Qp to larger Q and higher characteristic frequencies could be observed on the alkali rich side towards the ionic composition Rb_3Sb. At equiatomic composition a well defined prepeak appears at Q=9.5 nm^{-1} indicating the existance of relatively long-lived covalently bonded polyanions in the liquid.

A-278 Neutron spin echo application for acoustic fields studies in solids.

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We have observed inelastic neutron scattering (INS) from acoustic phonons (AW) excited in solids by NSE methods. Results are in agreement with dynamical theory calculations. At the highest power in Si crystals we observed a cross-over to a chaotic phonon behavior. Rapid decay of NSE signal was described here by means of model of quasi mosaic crystal. For the case of classical mosaic crystal KBr we found new secondary extinction effect. Mosaic block is vibrated as a whole in the AW fields. The exchange of small energy between AW and cold neutron leads to the deviation of neutron impulse from the Bragg position. Elastic and one-phonon scattering are realized in the different parts of the impulse space in the one mosaic block. Results of secondary extinction calculations are in a good agreement with the results of our NSE experiment with KBr. We clearly observed INS in the strongly excited pyrolitic graphite. The effect of the AW at total intensity was absent due to the bad structure of PG. It seems that similar experiments could be applied for the estimate of the size of the mosaic blocks and for acoustic fields studies in solids.

A-279 Neutron scattering study of CDW in $NbSe_2$

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We report the observation of the quenched disordered CDW state in a quasi-two-dimensional CDW material 2H-$NbSe_2$ using neutron scattering. At lowest temperature, T=10.2 K, the CDWs doesn't develop to a 3D long-range order. Approaching the transition temperature about 30.2 K from below, the CDW condensates decompose to a localised state, and show a highly fluctuated behaviour in the critical range.

A-280 Prevention of Depoling in TGS Crystals by Alanine Substitution: An Interpretation Based on Neutron Diffraction Study

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Single-crystal neutron diffraction studies, conducted on L-alanine doped triglycine sulphate (TGS) in order to unequivocally establish the site of the alanine substitution in TGS, indicated that the most likely site is the zwitterionic G2, contrary to the general belief that the substitution is at G1. Prevention of depoling by the alanine substitution can be explained more convincingly by this result. An analysis, based on the hydrogen-bond criteria, clearly establishes the correlation between the reorientation of the amino group of G1 and the proton tunnelling in the H-bond between the carboxyl groups of G2 and G3. The exact nature of the ferroelectric phase transition in TGS is re-interpreted in the light of these results. A detailed account of the analysis and results will be presented.

A-281 Kinetic energies of Neon adsorbed on activated Carbon

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Neutron Compton scattering method was used to study the kinetic energies (KE) of Ne atoms, at 12 K, adsorbed on activated carbon (AC) where the specific adsorption surface area was 3000 m^2/g. At low T, a major part of the KE is contributed by the zero-point motion of Ne. This fact was used for deducing the pore sizes of the AC. The momentum distribution of adsorbed Neon atoms was measured by using the EVS spectrometer at ISIS, UK. The average KE of Ne at 12 K was: 78.9±5.4 K. This is much higher than that of solid Ne (49.0±2.4 K); the increase in KE is indicative of the occurrence of a confinement effect, where the Ne atoms are enclosed within the slit-shaped pores of the AC. Using this data, we deduced the average slit widths of the AC adsorber pores by assuming a LJ potential between Ne and the graphitic surfaces and applying the model of Ref. [1]. The average pore width obtained was ∼0.57 nm. Note that similar pore sizes were obtained when the non-local density functional theory [2] was employed for analyzing isotherm data of N_2 on AC. [1] D Nemirovsky, R Moreh, K Andersen, J Mayers, J Phys: Cond M 11, 6653 (1999). [2] M. El-Merraoui, M. Aoshima, K. Kaneko, Langmuir 16, 4300 (2000).

A-282 Isotopic quantum correction to liquid methanol at -30° C.

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Hydrogen/Deuterium (H/D) substitution of molecular liquids in neutron diffraction is a powerful tool for structure determination. However recent high energy x-ray studies have found observable differences in the structures of many H and D liquids at the same temperature. In some cases this isotopic quantum effect can be corrected for by measuring the D sample at a slightly different temperature to the H sample. The example of hydroxyl isotopic substitution in liquid methanol at -30°C is presented. The magnitude of the quantum effect is shown to be significant when compared to the size of the first order neutron difference function, when measured without applying the correction.

A-283 Hydrogen Bonding in the Neutron Structure of the mono-nucleotide 5" -UMP Disodium Salt

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A single-crystal neutron diffraction study on the structure of 5" -uridine monophosphate disodium salt has been carried out to obtain high-precision data on the hydrogen-atom stereochemistry and hydrogen-bond interactions in the crystal structure. This is the first neutron study of a nucleotide to be reported in the literature.The structure and conformation involving the non-hydrogen atoms is in broad agreement with those obtained from an earlier x-ray study. A detailed analysis of hydrogen bonding, and its influence on the molecular conformation has been carried out. The notable feature of this analysis is the correlation between the occurance of C6-H6—O5" intra-molecular H-bond and the base to sugar anti-conformation. A detailed account of the analysis will be presented.

A-284 Analysis and Interpretation of Hydrogen Bonding in High-Precision Neutron Studies on Amino Acids and Dipeptides

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Aim of this analysis is to obtain hydrogen-bond data from high-precision neutron studies on amino acids, etc., and to use this information to unambiguously interpret x-ray protein structures. H-Bonding in relevant structures from the Cambridge Structural Database has been analysed in terms of various functional groups, such as carboxyl and amino groups, peptide linkages, and side chains of polar, acidic and basic amino acids. Trends and propensities, specific to various groups, and the influence of H-bonding on the conformation of these groups have also been analysed. Results, obtained from an earlier phase of this analysis, have been successfully used in a limited interpretation of a few x-ray protein structures. A detailed account of the results of this analysis, and possible applications will be presented.

A-285 Changes in protein dynamics induced under Gdn-HCl denaturation

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We have here performed a study of protein dynamics in native and partially denatured horse heart met myoglobin (Mb-met) diluted in heavy water. Incoherent elastic neutron scattering scans have been recorded at the backscattering spectrometer IN13 (ILL). The temperature dependence of the mean square displacements indicates that protein dynamics is strongly solvent dependent. In particular, a sizeably change is induced on the protein stiffness under Gdn-HCl denaturation. The results confirms previous kinetic and structural investigations, which have shown that highly denatured state of Mb presents a more open active site structure with respect to the native case. On the other hand, the contributions from macromolecular diffusion and larger local diffusional motions, in the open structural conformation, is higher than the obstructing effect induced on the protein dynamics by the increasing solvent viscosity effect with higher Gdn-HCl concentrations.

A-286 Changes in the dynamics of oriented lipid bilayers induced by the MBP

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Myelin Basic Protein (MBP), one of the most prominent proteins of the myelin sheath, plays a key role in the demyelination diseases of nerve axons. To understand the mechanisms leading to MBP/lipid (DMPA) association and myelin membrane stabilisation we have performed incoherent elastic (using IN13 at the ILL) and quasielastic (QENS) (using IRIS at ISIS) neutron scattering experiments on oriented samples of DMPA with and without the addition of MBP (5% w/w). The temperature dependence of the elastic intensity, has been investigated in a wide temperature range, and for two orientations of the samples corresponding to a momentum transfer predominantly parallel or perpendicular to the membrane plane. The QENS scans have provided information on the motions responsible for the anisotropy of the DMPA dynamics, as well as on the effect of the MBP on the system, across the La-Lb phase transition of the lipids.

A-287 Protons in Proteins: Neutron protein crystallography at ILL

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Neutron diffraction offers unique advantages for molecular structural biology by enabling key and individual hydrogen atoms to be located in biological structures that cannot be seen by X-ray analysis alone. In the past, the problems associated with the relatively low flux of available neutron beams have restricted such application to only a few projects of specific technical interest. The field of neutron protein crystallography is now undergoing significant development as new detector technologies and parallel advances in molecular biology push the capabilities towards atomic resolution. At ILL, the most powerful neutron source in the world, these advances are being exploited on the joint EMBL/ILL instrument LADI, which provides dedicated facilities for neutron protein crystallography that allow high-resolution (1.5Å) data to be collected with 10-100 fold gains in efficiency compared with conventional neutron diffractometers. These advances make feasible studies of larger biological complexes and smaller crystals than previously possible and are being used to address specific questions in biology concerning enzymatic mechanism, ligand binding interactions, solvent effects, structure dynamics and their implications. Recent highlights will be presented.