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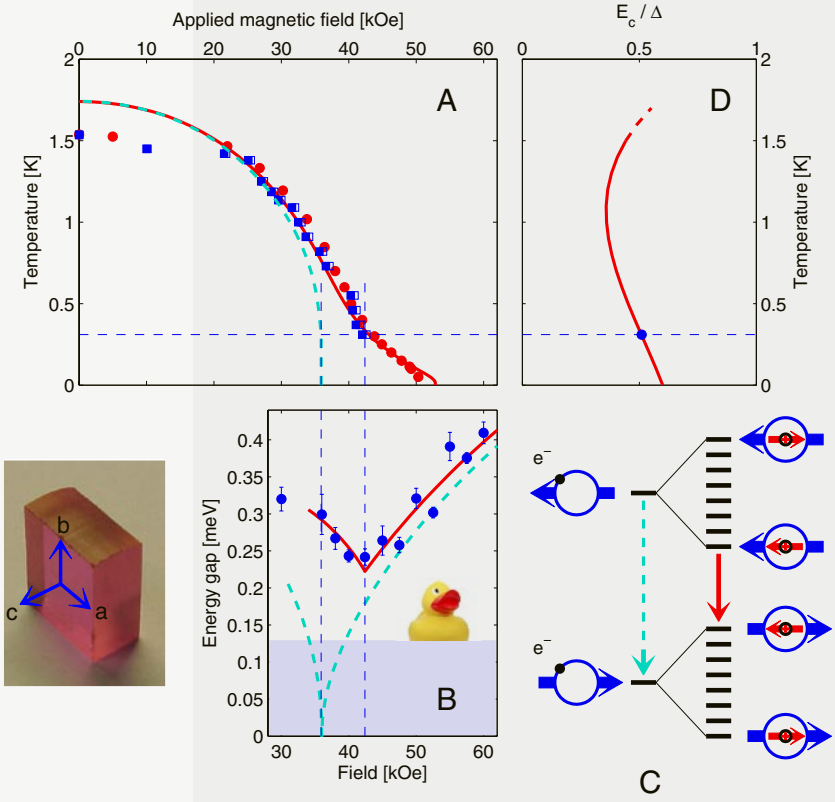
SWISS NEUTRON NEWS



Schweizerische Gesellschaft für Neutronenstreuung
Société Suisse pour la Diffusion des Neutrons
Swiss Neutron Scattering Society

Editorial:

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On the cover:

Summary of the quantum phase transition of the transverse field Ising system LiHoF4 coupled to the „spin bath" of Ho nuclear moments. (A) Magnetic field – temperature phase diagram. Measurements were performed at 0.3 K. (B) Electronic spin-gap as a function of transverse magnetic field. The softening anticipated (dashed line) in absence of hyperfine coupling is forestalled as the gap approach the bandwidth of the nuclear spin bath (shaded blue). (C) Hyperfine splitting of the crystal field doublet (left) facilitates mixing. (D) Spin gap at critical field as function of temperature, demonstrating that in this sense raising the temperature to 1 K would bring the system closer to the quantum phase transition.

For further information please have a look at the original reference:
H. M. Rønnow, R. Parthasarathy, J. Jensen, G. Aeppli, T. F. Rosenbaum, D. F. McMorrow, Science, Vol 308, 389 – 392 (2005)

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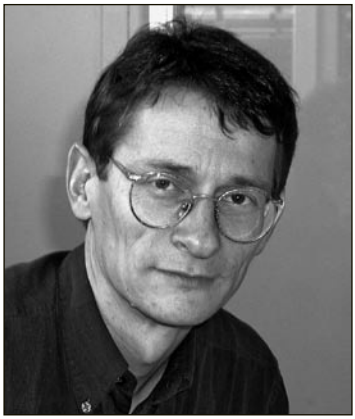

Dear members,

The answers to the ENSA-survey are presently being analyzed and some preliminary results can be accessed at: http://sgn.web.psi.ch/sgn/ensa.html. Here some comments: A total of 640 answers have been received from individuals and 120 groups representing 1170 scientists. Out of these, 46 came from people working in Switzerland. A quarter of these Swiss replies came from PhD-students and senior scientists respectively, and almost half from postdocs and instrument responsibles. This reply-rate for PhD-students is more than two times that for all individual answers and demonstrates the dynamics of the Swiss neutron user base. On the other hand, the Swiss neutron scene is still dominated by physicists and material scientists with 70% (while this is 59% for all answers). Chemistry with 14% (16%) and soft condensed matter with 12% (13%) are close to the average, while life sciences with 1% (5%) and earth sciences with 0% (3%) are underrepresented. 9% of the total beam time of all individuals and 5% of the groups is used for investigations at SINQ, but scientists from Switzerland measure 56% of their total time at SINQ, 20% at ILL, 8% at various continuous sources, 6% at HMI and 5% at ISIS. This tendency to measure at a home source is also obvious for other countries with home sources (mainly HMI and FRJ-2 for Germany, LLB for France). Scientists from countries without home sources (like Italy or Spain) or with a top-ranking source (like UK with ISIS) tend to perform between 60 and 85% of their experiments either at ILL and/or ISIS. The main reason for choosing a particular neutron instrument at a particular source is high flux followed by the available sample environment, local support (technical or scientific) and resolution (in this, the Swiss answers do not differ significantly from all the others). Finally, in case ESS will be built, 26% of the Swiss scientists plan to use it for more than 10% of their neutron measurement time and only one plans this for SNS. Without ESS 11% plan to use SNS for more than 10% of the time (all other users show similar results). The planned use of J-Parc for more than 10% of the neutron measurement time is negligible.

All proposals for the development and manufacturing costs for the actively shielded 16T magnet to be located at SNS have been approved with minor cuts. Here, I would

like to thank these sponsors (SBF, SNS, PSI, MaNEP) for their commitment. The project will start with a kick-off meeting at the end of June and should be finished by the end of 2007. For the users, of course, end of 2007 will be the start, since by then, SNS will also be in its commissioning phase. Hence, from the beginning we should be able to perform measurements in high magnetic fields at SNS. The whole development should not just result in a single magnet but in a family of magnets to be used when fringe fields are a problem at most existing instruments above a certain threshold field due to magnetic material or delicate equipment in their vicinity.

I hope all of you have submitted proposals to SINQ for the fall cycle. In this case you had your first encounter with DUO (digital user office). If you had problems to fill out or to submit proposals, we welcome your constructive feedback (stefan.janssen@psi.ch). The DUO-team will try to make the system as userfriendly as possible but at a certain point it becomes a question of philosophy and they will never be able to fulfill all wishes. Anyhow, some of the shortcomings (or: minor problems) are presently being worked on (e.g. the strict length restrictions which cuts off the references if you paste something into the main text). DUO is a PSI-project designed to unify the proposal process and user administration (SLS already uses DUO for quite a while and SμS will soon join). Hence, very soon the submission of proposals for beam time for photons, neutrons and muons at PSI will be very similar and I encourage you to use one of the other techniques once in a while.

Peter Allenspach

Minutes of the SGN/SSDN General Assembly on 27/01/2005

Locality: Paul Scherrer Institute, OSGA/EG6
Begin:17:17, End:17:56
Participants: 21 members of the society, 3 non-members

1. Welcome

The president of the SGN/SSDN, Dr. Peter Allenspach welcomes the participants to the general assembly 2005.

2. Minutes of the General Assembly 2004

The minutes of the general assembly of the SGN/SSDN from 22/01/2004 published in Swiss Neutron News 25 (June 2004) are accepted without objections.

3. Annual Report of the Chairman

The president P. Allenspach reports on the activities of the SGN/SSDN in the year 2004:

a) On October 6, 2004, on the occasion of his retirement colloquium the Swiss Neutron Scattering Society awarded the honorary membership to its first president Prof. Albert Furrer to honor his outstanding merits in the field of neutron scattering.

b) The society continued its activity of a Swiss participation at the American Spallation Neutron Source SNS: It is aimed to fund to a large extent a 16T vertical field cryomagnet for the SNS. The proposed financing scheme reads as follows:
– Swiss National Science Foundation: 1.0 MCHF
– Spallation Neutron Source SNS: 0.5 MCHF
– PSI: 0.4 MCHF
– MaNEP: 0.1 MCHF
The respective proposals have been submitted. The return would be assured beam time for the Swiss neutron user community, a key position for a post doc and an agreement on collaborations on instrument developments.

c) A welcome reception was offered by the society during the 'PSI Summer School on Condensed Matter Research 2004' in Zuoz.

d) Two new issues of 'Swiss Neutron News' were published, numbers 25 and 26. Both issues are on the web: http://sgn.web.psi.ch/sgn/snn.html

e) Actually the society has 195 members.

4. Report of the Treasurer

S. Janssen presents the annual balance sheet 2004:

Annual Balance 2002:

Assets SGN/SSDN on 1.1.2004:	**SFr 5446,85**	
	Revenues [SFr]	**Expensens [SFr]**
Membership-fees (cash box)	310,00	
Membership-fees (postal check acc.)	340,00	
Donations	0,00	
Advertisement Taylor & Francis	325,00	
Total expenses		1.435,40
– Apéro Zuoz (2003)		*600,00*
– Apéro Zuoz (2004)		*615,00*
– Presents		*220,40*
Credit for accrued interest	3,65	
Total expenses PC-account		58,20
Total	978,65	1.493,60

Net earnings 2004:	**SFr –514,95**
Assets SGN/SSDN on 31.12.2004:	**SFr 4.931,90**

Balance sheet 2004:

	Assets [SFr]	**Liabilities [SFr]**
Postal check account	3.970,00	
Cash box	961,90	
Assets on 31.12.04	**4.931,90**	

5. Report of the Auditors

Both Auditors (W. Fischer, K. Krämer) have examined the bookkeeping and the balance 2004. They accepted it without any objections. The participants therefore unanimously vote for a release of the SGN/SSDN board.

In addition W. Fischer criticizes the relatively low income of member fees in 2004 and the previous years. One reason might be the low member fee of CHF 10,-. He calls upon the members to pay their fees more regularly.

6. Budget 2005

The treasurer presents the following proposal for the budget 2005:

	Earnings [SFr]	**Expenses [SFr]**
member fees	800,–	
interests	5,–	
fees PC account		40,–
Zuoz Apero 2005		600,–
Total	805,–	640,–
balance 2005	**+ 165,–**	

The participants accept the budget proposal unanimously.

7. Change of statutes

The board of the SGN proposes two modifications of the statutes of the society. Both additions affect the topic 'membership' of the statutes.

a) So far there is no regulation on the procedure to award honorary memberships. The following addition is proposed to the members:

Article 3b (Membership):
The society can assign honorary memberships to persons, who made significant and outstanding contributions to the field of neutron scattering. On the appointment of honorary members the executive board decides.

b) The chairman figured out that - due to an unclear legal situation – members of Swiss societies, where the annual member fee is not explicitly mentioned within the

statutes, would be liable with their private property for the obligations of the society. To overcome this problem, the following addition (printed bold) is proposed:

Article 7 (Membership):
The amount of annual membership fees will be layed down/determined by the general assembly. ***Presently the annual membership fee amounts to CHF 10,–.*** *The members are not personally liable for the society's obligations.*

The members unanimously accept the modification of the society statutes.

8. News from ENSA

P. Allenspach reports on recent news from the European Neutron Scattering Organization ENSA:

a) The website of ENSA has been moved and restructured. The new address is: http://neutron.neutron-eu.net/n_ensa

b) During the ENSA meeting at PSI in October 2004 a new board was selected:
Chairman: H. Mutka, ILL
Vice-Chairman: P. Allenspach, PSI
Secretary: K. Mortensen, Risø

c) ENSA has launched a survey with the aim of gathering specific information on the use of neutrons in Europe. The online questionnaire is available from the ENSA webpage (see above link).

d) The next strategy meeting of ENSA will be organized in Paris on 28/02/2005.

9. News from the Institute Laue Langevin, ILL

The German associate ILL director W. Press reports on recent news from ILL:

REFIT programme:

a) Due to the reinforcement work of and around the reactor (REFIT program) the ILL had to reduce the number of reactor cycles per year from 4.5 to 3 also for 2005. The three cycles will run from 15/02–06/04, 15/04–04/06 and 15/06–04/08, respectively. Return to normal operation in 2007.

b) On level D of the reactor 550t (of 1000t total) of concrete already have been removed to improve seismic stability and considerable preparatory work has started in the guide hall ILL7. Still more work has to be done including a method of ancring level D at the reactor wall.

c) The programme aims to be completed in summer 2006. At that time ~25 M€ will have been spent, out of which 6.5 M€ already were spent in 2004.

d) Civil engineering work dominates in the near future: apart from the decoupling of the guide halls from the reactor building, the reinforcement of the office building ILL4 takes a lot of effort. Positive development: Finally two new building are being constructed (outside REFIT).

Millennium programme:

a) The upgrade of the polarizing thermal triple-axis spectrometer IN20 could be completed (e.g. new large focusing Heusler monochromator).

b) A new fast 2D-SANS detector has been developed for D22. The detector is installed and is capable to receive count rates of 1.5–2 MHz (former detector: 50 kHz).

c) A complete bank of polarizing analysers has been installed on D7.

d) Two new detectors for γ-spectroscopy, particularly of neutron-rich nuclei, have been mounted.

e) Three new area detectors for D19 were installed and the big new "banana" detector (with much larger solid angle) will probably start commissioning in the third reactor cycle 2005.

f) Sample environment: A Paris-Edinburgh type of pressure cell for low temperatures is almost ready.

g) A huge programme for the renewal of the H1-H2 neutron guide system supplying the ILL7 guide hall has been launched and must be completed during the next long shutdown.

h) Important further projects are the upgrade of the IN5 detector with a new flight chamber and increased solid angle range and the installation of a phase-space-transformer for IN16.

i) Several other new projects and pre-projects are planned to start in 2005.

Swiss use of ILL:

The Swiss use of the ILL is stable in the order of 3.5% of the allocated beam time.

Furthermore:

a) By the end of 2004 uranium from Russia has been delivered, securing the operation of the reactor for the years until about 2011.

b) Negotiations with several candidates for new partner countries are in progress.

10. Activities of the SGN/SSDN 2005

P. Allenspach informs briefly about the planned activities of the society in 2005:

a) The project of the Swiss SNS participation will be continued.

b) The SGN will play an active role during the analysis of the returned questionnaires of the ENSA survey.

c) Traditionally the society will again sponsor a 'welcome reception' during the next 'PSI Summer School on Condensed Matter Physics' in Zuoz from 14–21/08/2005. The topic 2005 will be 'Spectroscopy/Microscopy' and the school will be organized by the SLS.

d) The SGN encourages its members to actively participate in the 'International Conference on Neutron Scattering 'ICNS' in Sydney from 27/11–02/12/2005, where the 4th 'Walter-Hälg-Price' will be awarded.

11. Miscellaneous

The date of the next general assembly will be announced in due time on the SGN webpages: http://sgn.web.psi.ch

Inversion symmetry breaking magnetic structures in multiferroic oxides

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Abstract

We review the recent determination of magnetic structures in two multiferroic materials – $Ni_3V_2O_8$ and $TbMnO_3$ – where magnetic ordering is directly coupled to ferroelectric polarization. In both materials, the magnetic structures in the para- and ferroelectric phases have distinctly different symmetries, described by one and two irreducible representations respectively. Ferroelectricity arises when the magnetic structure breaks inversion symmetry. Through magneto-elastic effects such structures act as an effective electric field leading to electric polarization of the insulating material.

Introduction

Materials with ferroelectric and magnetically ordered ground states have been known for more than forty years [1]. Most of these materials are either (1) perovskite-type structures, (2) hexagonal rare-earth manganites, (3) boracite compounds or (4) barium fluorides in which the onset of ferroelectricity and magnetic order occurs at vastly different temperatures. In these materials, the redistribution of charge, which leads to ferroelectric polarization, is unrelated to the subsequent development of magnetic order at lower temperatures.

Large magnetoelectric coupling effects were recently discovered in $TbMnO_3$ [2] and $Ni_3V_2O_8$ [3]. These materials are different from previously known multiferroics in that their field-temperature phase diagrams consist of several magnetically ordered phases, some of which feature a ferroelectric polarization that vanishes at magnetic phase boundaries. By applying a magnetic field and traversing such phase boundaries, it is possible to change the direction of the electric polarization in $TbMnO_3$ [2], or to completely suppress it in $Ni_3V_2O_8$ [3].

Magnetic structures in $TbMnO_3$

$TbMnO_3$ has a distorted perovskite structure (Fig. 1) and is structurally similar to the prototype ferroelectric $BaTiO_3$ and other insulating pervoskites with non-linear magnetoelectric effects. $TbMnO_3$ however displays a sequence of phase transitions between incommensurate magnetically ordered phases as a function of temperature. Following an initial transition at 42 K to incommensurate order, there is a second transition at 27 K where the temperature dependence of the ordering wave-vector becomes so weak that it was initially interpreted as a lock-in transition. More recent experiments show that the distinguishing characteristic of the low temperature phase is transverse polarization, which leads to inversion symmetry breaking and ferroelectricity [1].

Following the discovery of magneto-electric coupling in $TbMnO_3$, neutron diffraction experiments revealed a sequence of incommensurate phases and provided evidence for competing interactions along the crystallographic **b-**direction [3,4,5]. To understand the origin of magneto-electric coupling in $TbMnO_3$, a comprehensive magnetic structure determination for the para-electric and ferro-electric phases was carried out using the four circle diffractometer TriCS at PSI. The goal of this work was to understand why only one of the two incommensurate phases is ferroelectric.

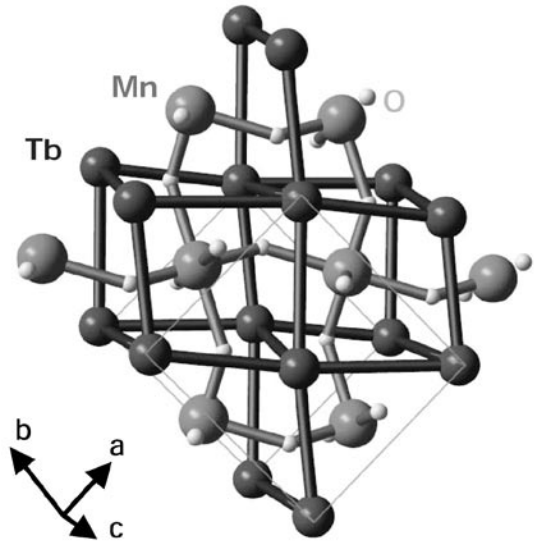

Fig. 1: Chemical structure of $TbMnO_3$, tilted to show the distorted perovskite structure

The complexity of magnetic structures in $TbMnO_3$ arises from the

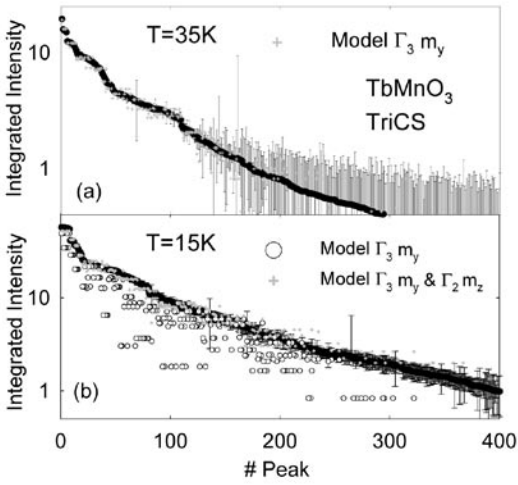

Fig. 2: Integrated intensities of magnetic Bragg peaks at T=15 K and T=35 K compared to various magnetic model structures. Peaks are sorted by decreasing measured intensity.

presence of two magnetic ions, Tb^{3+} and Mn^{3+}, and competing spin interactions. A unique determination of these ordered structures required over 900 magnetic Bragg intensities for each temperature and the use of magnetic representation theory to classify the possible structures.

Fig. 2 shows the intensity of the 400 strongest magnetic Bragg peaks of $TbMnO_3$ at T=15 K and 35 K, in the ferroelectric and paraelectric phases, respectively. The T=35 K structure is described by a single irreducible representation, Γ_3, with ordered magnetic moments along the **b-**axis.

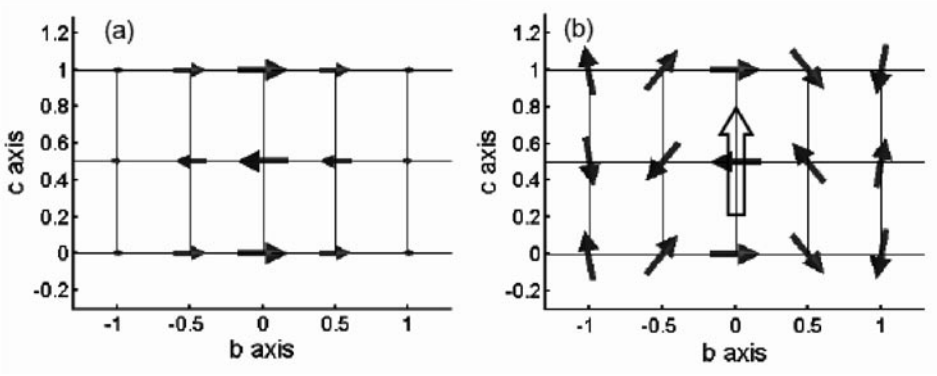

Fig. 3: Schematic of the magnetic structure of $TbMnO_3$ at (a) T=35 K and (b) T=15 K, projected onto the ***b-c*** plane. Filled arrows indicate direction and magnitude of Mn moments. The longitudinally-modulated phase (a) has a point of inversion while the spiral phase (b) does not. Electric polarization indicated by the unfilled arrow is hence allowed in (b) but not in (a).

The T=15 K magnetic structure on the other hand is a spiral described by a *y*-component of Γ_3 and an *x*-component of Γ_2 with a $\pi/2$ phase shift.

The structure at T=35 K, which is consistent with an earlier study [4], is illustrated in Fig. 3a. The absence of observable higher order magnetic diffraction peaks indicates a sinusoidal amplitude modulated structure. The low-temperature incommensurate phase at T=15 K is illustrated in Fig. 3b. Mn moments in this phase form an elliptical spiral.

Magnetic structures in $Ni_3V_2O_8$

$Ni_3V_2O_8$ contains anisotropic S=1 Kagomé planes [3] that can be described as dense magnetic spines running along the a-axis separated by cross-tie sites (Fig. 4a). The anisotropy of the lattice breaks the degeneracy of the Kagomé antiferromagnet and leads to long-range order at low temperatures. Due to the near degeneracy of different spin structures, small perturbations such as a magnetic field, spin-orbit interactions, and possibly magneto-elastic effects lead to qualitative changes in the spin structure. Indeed, $Ni_3V_2O_8$ adopts two different incommensurate magnetic structures – one of them ferroelectric – and at least one commensurate structure as a function of temperature at zero magnetic field [5].

Using neutron diffraction, it was found that as for $TbMnO_3$ the high-temperature incommensurate (HTI) magnetic structure is longitudinally modulated and can be described by a single irreducible representation [5]. In the low-temperature incommensurate (LTI) phase, which is ferroelectric, the magnetic structure is again a spiral with two spin components that belong to different irreducible representations. The LTI structure breaks inversion symmetry, which in an insulator implies that there will also be a finite electric polarization [1,3]. Below T=4 K, $Ni_3V_2O_8$ adopts commensurate (C and C') magnetic structures that restore inversion symmetry and are not ferroelectric.

Trilinear Coupling Theory

Harris et al. [3,6] recently showed that insulators with axial-non-axial parity breaking magnetic phase transitions must also be electrically polarized. The theory is based on a trilinear coupling of the magnetic and electric order parameters of the form

$$V = -\sum_{ij\gamma}\left(a_{ij\gamma}\sigma_{\Gamma_1 i}(q)\sigma_{\Gamma_2 j}(-q) + a^*_{ij\gamma}\sigma_{\Gamma_1 i}(q)\sigma_{\Gamma_2 j}(-q)\right)P_\gamma .$$

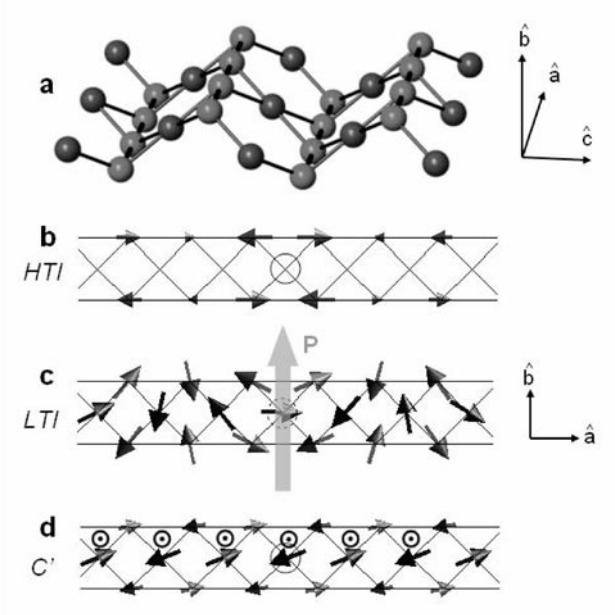

Fig. 4: Crystal and magnetic structures of $Ni_3V_2O_8$. (a) The crystal structure includes spin-1 Ni^{2+} spine sites forming chains and cross tie sites between the chains. (b-d) Simplified schematic representation of spin arrangement in the antiferromagnetic HTI, LTI, and C' phases [5]. The dotted circles in (d) indicate a small ferromagnetic moment. Only the HTI and C' phases have inversion symmetry relative to the indicated central lattice point.

Here $\sigma_{\Gamma_1 i}(q)$ is component i of the magnetic order parameter belonging to irreducible representation Γ_1, P_γ is the electric polarization along the γ crystallographic direction and $a_{ij\gamma}$ parametrizes the strength of the interaction between the electric and magnetic order parameters. This interaction is discussed in detail in Ref. 3, 6, and 7, where it is shown that at least two irreducible representations, Γ_1 and Γ_2, are needed to allow for a finite electric polarization. The direction of the electric polarization depends on the symmetry of the irreducible representations, making their experimental determination a crucial step in testing the theory. On the basis of the observed magnetic structures in $TbMnO_3$, the trilinear coupling theory predicts that there is no ferroelectric polarization at 35 K, but that there is an electric polarization along the c-axis in the low T phase. This is fully consistent with the electric polarization data. Likewise the tri-linear term for $Ni_3V_2O_8$ when combined with the observed magnetic structures correctly predicts the measured direction of the ferro-electric polarization. Taken together these results point to an interesting new class of multi-ferroics where inversion symmetry breaking magnetism induces electric polarization through a tri-linear magneto-electric interaction.

Acknowledgements

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Methods for neutron scattering under pressure

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Introduction

After having characterized a chemical system at various temperatures, varying the external magnetic field or the external pressure certainly stands at the top on any physicist or chemist's "to do list". The response of chemical systems in varying thermodynamic variables may indeed deliver a wealth of new information. In the case of pressure, interatomic distances may be changed and correspondingly the underlying interaction potential probed and at best fully parameterized. Once established, this interaction potential may be used to predict new phase transitions, properties at experimentally non-accessible conditions or properties of related systems. Hence for instance, many of the observed phase transitions in the phase diagram of water can be accurately calculated by means of classical or *ab-initio* molecular dynamics calculations, and benchmarked against experimental results (see e.g., the LDA-HDA transition in the next paragraph). Moreover, many exotic solid states at extreme conditions have been first predicted by theory and were later confirmed experimentally or still await to be confirmed. Thus the quest in providing clear experimental evidence of states, as e.g. solid metallic hydrogen [1, 2] (already predicted by Wigner in 1935), superconducting Li [3] or superionic and eventually metallic liquid water [4, 5], still represents major challenges in modern high-pressure physics. Other results from high-pressure physics may surprise the non-expert, as for instance the continuously increasing number of pure elements, which are found superconducting at high pressures [3] (e.g., oxygen [6] and iron [7]) or the synthesis of new materials at high pressures and high temperatures. Some of the latter products may be brought back to ambient conditions (i.e. "recovered"), where they show striking new properties of potential technological use (e.g., the synthesis of diamond and other super-hard materials, or the recent synthesis of polymeric carbon oxide, a so-called advanced energetic (i.e. highly explosive!) material [8]).

From the experimentalist's point of view pressure suffers a severe drawback in the fact that the repulsive forces in solids *per se* are quite strong and hence substantial pressures must be generated to reduce the interatomic distances by as little as 5 % (e.g., 3.4 GPa for ice VII, 16.9 GPa for Ge, 35 GPa for MgO). These pressures can only be realized at the expense of sample volume and often involve a considerable loss in data

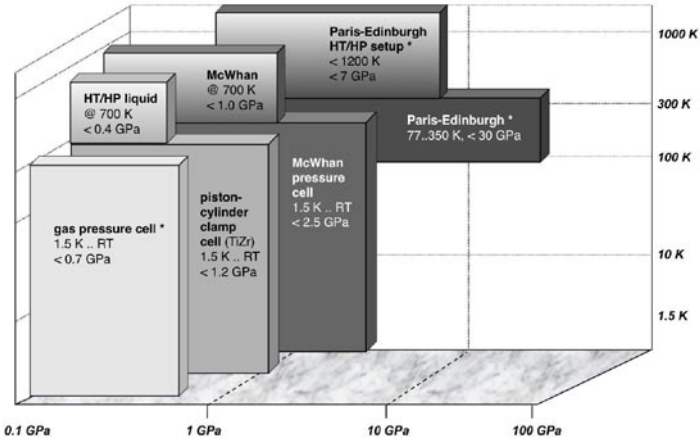

Figure 1*:* Pressure cells used for neutron scattering with their respective pressure and temperature ranges of application. The cells denoted with an asterisk allow the in-situ change of pressure at any given P/T-condition (adapted from Ian Bailey, ISIS)

quality.[1] Diamond anvil cells, as widely used for optical in-lab methods (Raman, Brillouin, infrared) and synchrotron techniques (XRD, EXAFS, dichroism, IXS), nowadays allow routinely measurements at pressures of a few Megabar on samples of a few 10 μm^3. However, in the case of neutron methods, sample volumes of the order of a few mm^3, currently minimally necessary, restrict the maximal obtainable pressure to a few 100 kbar.[2] The pressure cells used for neutron scattering may be roughly classified into three types (Fig. 1): (i) gas-pressure cells ($P < 0.7$ GPa, sample volumes of a few cm^3), (ii) piston-cylinder clamp cells ($P < 2$ GPa, a few cm^3) [e.g. also the McWhan pressure cell], and (iii) opposed anvil cells ($P < 50$ GPa, $1 < V < 100$ mm^3) [e.g. the Paris-Edinburgh pressure cell (see also inset of Fig. 5)]. As a matter of fact, all of these pressure cells must allow an *in-situ* characterization of the sample at the given P/T condition. This restriction is generally less of a concern in pure high-P/T synthesis of materials later to be recovered and analyzed at ambient pressure. On the other hand, some of the above neutron pressure cells also allow the *in-situ* change of pressure at any given temperature. This very feature is increasingly important, if the system under investigation itself represents a high-pressure phase, which first must be produced at non-ambient conditions and possibly via a well-defined path within the phase diagram. In this case the application of clamp cells is very restricted.

In what follows we illustrate the application of some of the above-mentioned pressure cells by reviewing some of our recent neutron scattering studies. These emphasize the importance of cells allowing the *in-situ* change of pressure and thus to synthesize and characterize high-pressure phases otherwise inaccessible.

[1] Sample volumes are often found to scale with $V \propto 1/P^n$, where $n > 1$.
[2] 10 kbar = 1 GPa

Polyamorphism in water

Despite the omnipresence of water on earth and in the universe, the probably most common liquid still poses many open questions. At ambient temperature and pressure, liquid water shows anomalies in almost all its thermodynamic properties including, e.g., minima in the isothermal compressibility, the isobaric specific heat, a negative thermal expansion coefficient as well as a point of maximum density [Ref. 9 and references therein]. Likewise the solid phase puzzles with as much as 13 crystalline ice phases (the latest discovered 7 years ago!) and at least 3 amorphous phases (Figure 1) [10]. Many of these high-pressure crystalline phases and in particular the amorphous phases are now believed to constitute the predominant form of water in outer space. The discovery of a high density amorphous ice phase (HDA) by compression of ordinary ice at low temperatures ($P > 1.5$ GPa, $T < 130$ K) in 1984 by Mishima et al. [11] constitutes one of the first examples of a pressure-induced transition from a crystalline to an amorphous phase (C-A transition) subsequently observed in a large group of other compounds, some of which important in geophysics, as e.g., α-quartz (SiO_2). The discovery of HDA also uncovered an important link to another form of amorphous water previously known by biologists from thermally quenched water droplets: If warmed at ambient pressure HDA transforms at ~ 130 K irreversibly to a low density amorphous phase (LDA) which shows similarities with the amorphous phase found from the quench experiments. Furthermore if compressed at T ~ 130 K, LDA transforms reversibly to HDA at P ~ 0.3 GPa. The question about the nature of this pressure-induced polyamorphous transition LDA↔HDA is crucial for explaining the above-mentioned thermodynamic anomalies

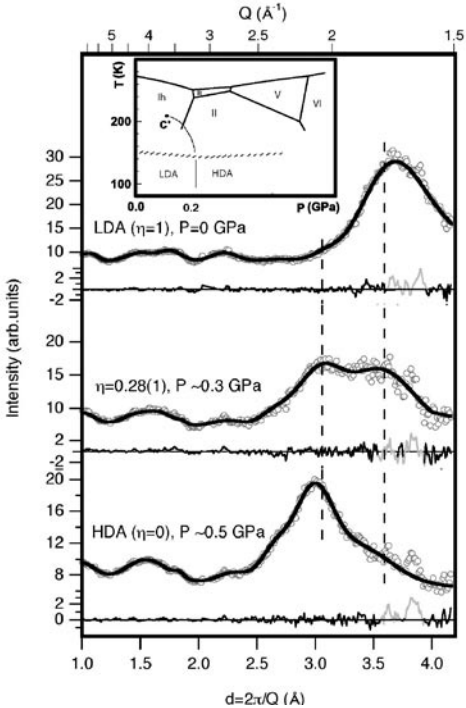

Figure 2: Neutron diffraction patterns taken as LDA (top) transforms to HDA (bottom) under reducing sample volume at 140 K (η denotes respective phase fractions, data measured at PEARL, ISIS, details in [13]). Inset: portion of the phase diagram of water, C' denotes the hypothesized second thermodynamic critical point in water.

of water. Theoretical models have been proposed suggesting the existence of two distinct phases of water which do phase-separate below a hypothesized second critical thermodynamic point at ~ 220 K, 0.5 GPa [12]. The observed polyamorphous transition LDA↔HDA has been tentatively proposed being linked to this two liquid scenario, which would require that the transition is first-order.

We have recently studied this transition in detail using the Paris-Edinburgh pressure cell [13]. Ordinary ice (D_2O ice Ih, ~ 100 mm^3) was compressed at 77 K into HDA and warmed at ambient pressure into LDA. The pressure-induced transition from LDA into HDA was then studied by neutron diffraction upon decreasing sample volume at 130 K. Figure 2 shows the initial LDA pattern together with a representative intermediate pattern at 0.3 GPa and a final pattern at 0.5 GPa. The latter can readily be identified to the pure HDA phase. We found that all intermediate patterns may be obtained by linear superposition of the initial (LDA) and final (HDA) patterns. The presence of phase-coexistence and the fact that the pressure remains constant during the volume-driven transition puts forward strong evidence for a first-order character of this phase transition. This important conclusion could be further confirmed by complementary Raman scattering experiments in a diamond-anvil cell showing spatial phase coexistence of LDA and HDA in the reverse transition from HDA to LDA upon increasing sample volume. This study highlights the importance of *in-situ* pressure techniques as all amorphous ice forms recrystallize into crystalline ice phases if warmed to above ~ 160 K. Hence these studies not only require the possibility of measuring *in-situ* the sample but also of *varying in-situ* the applied pressure. The latter is possible with both the Paris-Edinburgh pressure cell as well as membrane-driven diamond anvil pressure cells.

Phonon dispersion, negative thermal expansion and amorphisation of ordinary ice

The second example illustrating the need of *in-situ* pressure techniques involves another striking property of ice, namely the pronounced negative thermal expansion (NTE) of ordinary "household" ice (ice Ih) at ambient pressure below ~ 70 K [14]. Microscopically, for a non-magnetic compound, NTE requires a pronounced pressure-induced softening of some low frequency phonon branches. Furthermore, it was earlier theoretically suggested that a complete softening of an acoustic branch along a high-symmetry direction of the system may lead to amorphisation and thus may explain the mechanism of the above-mentioned pressure-induced amorphisation by so-called *mechanical melting* [15].

We have recently studied the phonon dispersion on a single crystal of D_2O ice Ih at 140 K up to 0.55 GPa (Figure 3) [16]. Many of the measured phonon branches show indeed pronounced softening upon pressure, which yield a NTE in excellent agreement with macroscopic measurements. Most notably, however, we identify an almost uni-

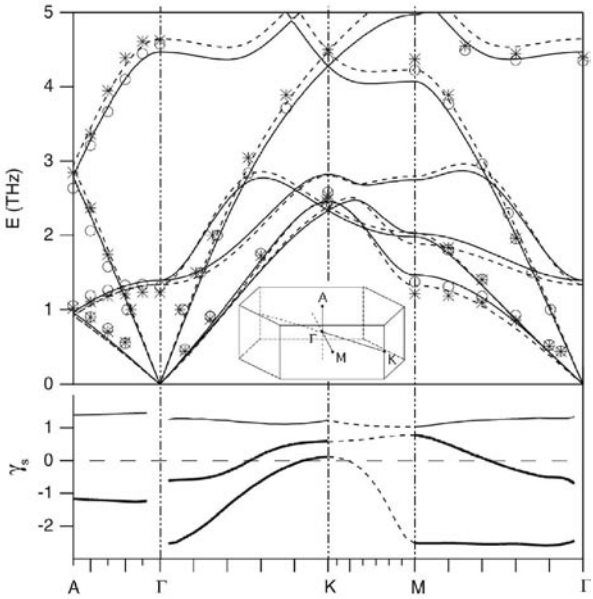

Figure 3: Phonon dispersion of ice Ih (ordinary ice, space group $P6_3/mmc$), together with corresponding mode-Grüneisen parameters. Pronounced pressure-induced softening is observed for all transverse acoustic branches, note the dispersionless Grüneisen mode parameter along Γ-M (data measured on 1T1, LLB Saclay, details in [16]).

form negative mode-Grüneisen parameter for the basal-polarized transverse acoustic branch along Γ-M, indicating that this branch will become entirely flat and thus triggers a lattice instability at an extrapolated pressure of ~ 2.5 GPa. This lattice instability can be regarded as the homogeneous limit to the experimentally observed C-A transition at 1.5 GPa, which occurs earlier due to density and thermal fluctuations. These measurements have been carried out in a gas-pressure cell thus allowing (i) the necessary sample volumes of a few cm^3, (ii) true hydrostatic pressure conditions (fragile single crystal) and (iii) the *in-situ* adjustment of pressure at temperatures below 150 K. The latter fact is mandatory due to the fact that for temperatures above 150 K ice Ih transforms to ice II at a pressure of 0.15 GPa (Figure 1).

Hot dense water

A dedicated high-temperature/high-pressure (HT-HP) setup of the Paris-Edinburgh cell has been developed for the study of liquid systems at temperatures up to ~ 1200 K and ~ 7-8 GPa [17]. A cylindrical graphite furnace allows the internal heating of the sample, which is filled into a zero-matrix (TiZr) capsule and latter electrically insulated to the former by a thin layer of MgO. The measurements are typically carried out on a TOF powder diffractometer which eases collimation of the axially incoming and at constant angle (typically 90°) scattered neutron beam, so that eventually only neutrons scattered by the sample are detected. The temperature is measured within an

accuracy of ~ 5 % by the Doppler broadening of neutron absorption resonance lines of Ta or likewise Hf. The pressure is derived from the known equation of state of a pressure calibrant. Heating of the sample is achieved by an electric current through the two anvils via the graphite furnace. The possibility of changing pressure *in-situ* is needed in such experiments, since the interruption of the current would lead to an abrupt cooling of the sample and in the case of water to its solidification with a breakage of the sample container. In Figure 4 we present radial distribution functions g(r) as well as spatial distribution functions (SDF) of water along its melting line up to 670 K and 6.5 GPa. A reversed Monte Carlo method (Empirical Potential Structural Refinement [18]) is applied in order to derive from the measured S(Q) of D_2O all partial angular and radial averages (g_{OO}, g_{OD}, g_{DD}) without isotope substitution. Our results are in excellent agreement to predictions by classical and *ab-initio* molecular dynamics simulations and preliminarily show that the structure of water under these high P-T conditions approaches the structure of other simpler liquids (e.g., Ar, supercooled Cu) despite an intact H-bonding within the first neighbor shell [19].

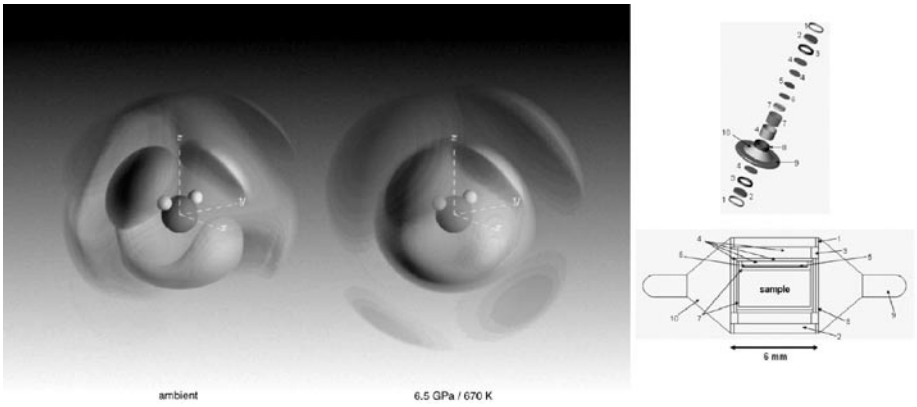

Figure 4: Left: Spatial distribution functions (SDF) of water at ambient condition (left) and at 6.5 GPa, 670 K (right) (data measured at PEARL, ISIS). Right: Sample-gasket assembly for HT/HP studies in the PE cell, TiZr capsule filled with D_2O water (7), internal graphite furnace (8) (details in [17,19]).

Perspectives

The above three examples illustrate the importance of flexible *in-situ* pressure techniques. With the exception of the inelastic study the above measurements were carried out in the Paris-Edinburgh pressure cell at the high-pressure beamline PEARL at ISIS. Here, collimation of the small sample inherently benefits from the TOF geometry,

whereas at constant-wavelength sources the beam has to pass equatorially twice through the gasket. On the other hand, TOF diffractometers generally suffer from less instrumental resolution than angular dispersive diffractometers. A new series of Paris-Edinburgh pressure cells is now available allowing angular apertures of 140° which in combination with novel neutron-absorbing boron-nitride anvils, promises to be a versatile tool also for constant-wavelength instruments, as e.g., HRPT at SINQ (Figure 5) [20].

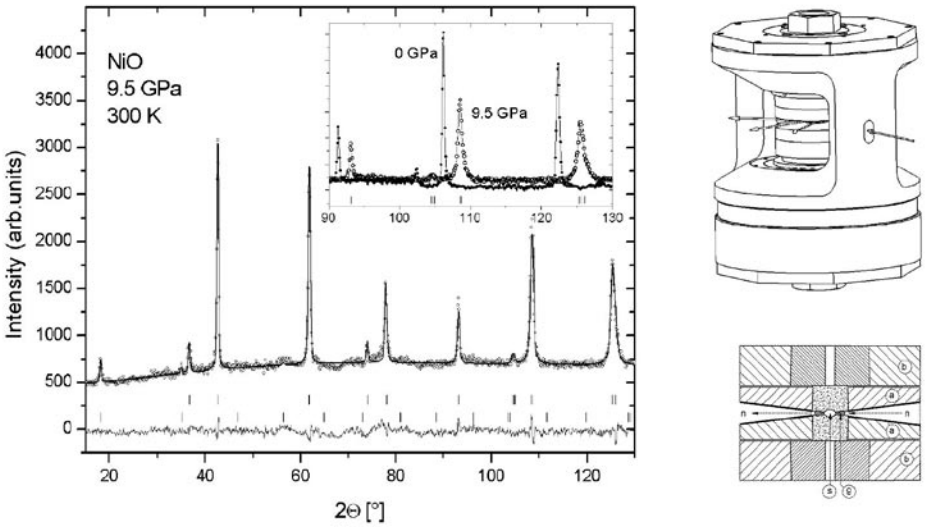

Figure 5: Left: Rietveld refinement pattern and difference plot of the neutron diffraction data of NiO obtained at a hydrostatic pressure of 9.5 GPa after 4 hours of data collection on HRPT at SINQ (λ = 1.494 Å, HI mode). Enhanced rhombohedral distortion is observed upon increasing pressure (e.g. peak at ~ 126°). Right: Paris-Edinburgh cell together with seat-anvil-gasket-sample assembly (details see [20]).

The intermediate-sized version of this cell (~ 30 kg) has been demonstrated to be cooled down to 4 K by a two-stage closed-cycle refrigerators [21]. This very recent development opens the way to studies below 77 K.

Acknowledgments

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Loveday, R.J. Nelmes, C. Bull, M. Guthrie and I am indebted to S. Klotz for careful reading of the manuscript. My post-doc stay has been made possible by an individual grant of the Swiss National Science Foundation and a Marie Curie Fellowship of the European Union.

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7th SINQ Users' Meeting

S. Janssen, PSI User Office,
NUM department, Paul Scherrer Institute

The 'User Office' team during the registration.

On January 27, 2005 the 7th SINQ Users' Meeting was organized at PSI. For the first time more than 100 participants (104) attended the one-day meeting, which was organized with two plenary sessions in the morning and four parallel sessions in the afternoon.

In the morning K. Clausen and J. Mesot first welcomed the participants on behalf of the SINQ management and presented briefly the new organization structure of the NUM department and the Laboratory for Neutron Scattering. After that four lively and highly interesting scientific presentations were given by Ted Forgan (Univ. Birmingham), Michel Kenzelmann (ETH Zürich), Anna Stradner (Univ. Fribourg) and Peter Günter (ETH Zürich).

PSI director Ralph Eichler.

Kurt Clausen, PSI, NUM department.

The director of PSI, R. Eichler then also welcomed the participants and gave a short overview about the other three user laboratories of the institute and the overall very positive prospects of PSI. The first session was closed by some actual information on the user programme.

Ted Forgan during his presentation on 'Flux line lattices and phase transitions in low- and high-T_c superconductors'.

Peter Günter: 'Soft modes and photonics'.

Mogens Christensen (left) and Kim Lefmann.

The second morning session was dedicated to the SINQ instrumentation. The respective group leaders informed about the status quo and foreseen upgrades on the instruments within their competence: J. Schefer (diffractometers), B. Roessli (spectrometers), J. Kohlbrecher (Small angle scattering instruments and reflectometers) and E. Lehmann (neutron imaging).

After the lunch break it was time to give the word to the users. No poster session was organized, instead the audience split up to attend four parallel oral sessions, where totally 36 talks were given by the users of the facility. The sessions were separated as follows:

- Magnetism and Superconductivity
- Structure and Dynamics
- Soft Condensed Matter
- Neutron Imaging

At the end of the day the feedback from the participants was overall quite positive: A lot of people had the opportunity to present their results and discuss them with the user community.

The next SINQ Users' Meeting will be held on **May 10, 2006.**

Coffee break!

Christian Rüegg
(foreground left)
and Karl Krämer

Talking about proteins?
Anna Stradner and Dieter
Middendorf

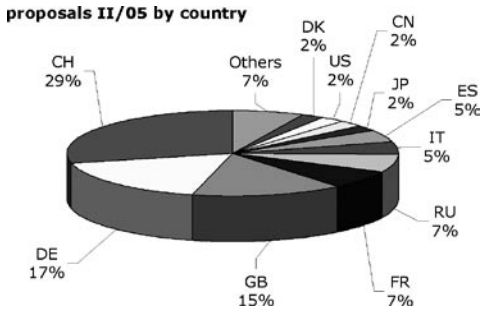
LNS ‘legend’ Peter Fischer with Penelope Schobinger and Oksana Zaharko

SGN/SSDN Members

The Swiss Neutron Scattering Society welcomes the following new member:
A. Naberezhnov (A.F. Ioffe Institute, Neutron Research Lab., St Petersburg, RU)
Presently the SGN has 194 members.

News from SINQ

The recent SINQ deadline on 15/05/2005 was the first one, which was completely processed by the new 'Digital User Office, DUO'. That software tool has originally been developed for the SLS user operation and now has been adapted to the SINQ requirements, such that the users of both these two PSI large scale facilities do have the same user interface. The next facility to be equipped with DUO will be the Swiss Muon Source SμS. So far the users very well accepted the new system and submitted a total of 87 new SINQ proposals.



Those proposals come from 17 different countries including overseas countries like USA, Japan, China or Australia. The largest share naturally emerges from Switzerland followed by Germany and Great Britain. The share of new long term proposals was again relatively low (9%). Most of the proposals were submitted for the SANS facilities (24), followed by TASP, HRPT and AMOR (14 each) and FOCUS (10). In mid July the authors of proposals will be informed about the result of the proposal evaluation and the beam time allocation, the new cycle II/05 starts on Sep 1, 2005.

Open Positions at ILL

To check the open positions at ILL please have a look at the ILL-homepage:
http://www.ill.fr following the link 'Job Offers'.

Spectroscopy/Microscopy: 4th PSI Summer School on Condensed Matter Research

Lyceum Alpinum, Zuoz, Switzerland
August 14-21, 2005

Purpose

The purpose of the Summer School is to give the participants an introduction to the basic principles of spectroscopy and microscopy at synchrotron and neutron facilities. The lectures will cover both theoretical and experimental aspects, with particular emphasis on the utilization of three prominent experimental probes available at PSI: synchrotron light, neutrons, and muons. For admission, no specialized knowledge of the subject is required, but a masters degree (or equivalent) in natural sciences is necessary. In addition to the lectures, there will be poster and discussion sessions in which the participants are welcome to present their own results in the field of condensed matter.

Topics

Angle-resolved photoemission, resonant inelastic soft X-ray scattering, inelastic neutron scattering, X-ray absorption spectroscopy, muon spin rotation, infrared spectroscopy, X-ray transmission microscopy, photo-emission microscopy, electron spectroscopy for chemical analysis, photoelectron diffraction, spectroscopy of orbital ordering, spectroscopy of correlated electron systems, superconductivity.

Further information

http://sls.web.psi.ch/view.php/science/events/Conferences/Zuoz2005/Scope.html

Conferences and Workshops 2005/06

(an updated list with online links can be found here: http://sinq.web.psi.ch/sinq/links.html)

July 05

ICNM-2005, International Conference on Nanoscale Magnetism
July 3-8, 2005, Istanbul, Turkey

SCES 05, International Conference on Strongly Correlated Electron Systems
July 26-30, 2005, Vienna, Austria

QAMTS, Quantum Atomic and Molecular Tunneling in Solids
July 27-31, 2005, Santiago de Compostela, Spain

Biological Membranes: Current Challenges
July 31 - August 19, 2005, Benasque Center for Physics, Spain

August 05

μSR 2005, 10th International Conference on Muon Spin Rotation, Relaxation and Resonance
August 08-12, 2005, Oxford, GB

Advanced Workshop on Strongly Correlated Electrons: Diverse Examples and Unifying Themes
August 8-20, 2005, Institut Scientifique de Cargèse, Corsica, France

Workshop on 'Probing complex fluids membranes and films with neutron spin echo'
August 14-17, 2005, Indiana University, Bloomington, IN, USA

4th PSI Summer School on Condensed Matter Research: Spectroscopy/Microscopy
August 14-21, 2005, Zuoz, Switzerland

7th United States National School on Neutron and X-ray Scattering
August 14-28, 2005, Argonne National Laboratory, USA

XX Congress of the International Union of Crystallography
August 23-31, 2005, Florence, Italy

Symposium to be Presented at the National Meeting of the American Chemical Society: Scattering from Polymers
August 28 – September 1, 2005, Washington, DC, USA

Microscopy Conference Davos 2005
August 28 – September 2, 2005, Davos, Switzerland

September 05

Magnetism, Neutrons and High-Pressure
September 1-2, 2005, University of Edinburgh, Edinburgh, GB

Neutrons in Biology
September 4-7, 2005, ILL, Grenoble, France

Risø Symposium 2005: Solid State Electrochemistry
September 4-8, 2005, Risø National Laboratory, Roskilde, Denmark

Perovskites - Properties and Potential Applications
September 5-7, 2005, EMPA Dübendorf, Switzerland

9th Oxford School on Neutron Scattering
September 5-15, 2005, University of Oxford, Mansfield College, United Kingdom

NSE 2005 - Neutron Spin Echo Workshop
September 7-10, 2005, ILL, Grenoble, France

9th Laboratory Course on Neutron Scattering
September 12-23, 2005, FZ Jülich, Germany

Workshop on Lattice Dynamics
September 19-21, 2005, University of Göttingen, Germany

Diffusion Fundamentals I
September 21-24, 2005, Leipzig, Germany

13th BENCS Users‘ Meeting
September 22-23, 2005, HMI Berlin, Germany

2005 Swiss Workshop on Materials with Novel Electronic Properties MaNEP
September 26-28, 2005, Les Diablerets, Switzerland

DYPROSO 2005, 30th International Symposium on Dynamical Properties of Solids
September 27 – October 1, 2005, Cesky Krumlov, Czech Republic

October 05

6th SLS Users‘ Meeting
October 17-18, 2005, PSI Villigen, Switzerland

MECA SENS conference on Stress Evaluation by Neutron and Synchrotron X-Ray Radiation
October 17-19, 2005, Santa Fe, USA

School on Pulsed Neutron Sources: Enhancing the Capacity for Material Science
October 17-28, 2005, Miramare, Trieste, Italy

November 05

ICNS 2005, International Conference on Neutron Scattering
November 27 – December 2, 2005, Sydney, Australia

January 06

SμS Users' Meeting
January 25-27, 2006, PSI Villigen, Switzerland

May 06

8th SINQ Users' Meeting
May 10, 2006, PSI Villigen, Switzerland

June 06

11th International Ceramic Congress and 4th Forum on New Materials
June 4-9, 2006, Acireale, Sicily, Italy

July 06

XIII International Conference on Small Angle Scattering
July 9-13, 2006, Kyoto, Japan

September 06

Polarised Neutron School
September 19-22, 2006, Berlin, Germany

PNCMI 2006, Polarised Neutrons in Condensed Matter Investigations
September 25-28, 2006, Berlin, Germany

Schweizerische Gesellschaft für Neutronenstreuung
Société Suisse pour la Diffusion des Neutrons
Swiss Neutron Scattering Society

Anmeldeformular / Registration Form

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Please submit to: **Paul Scherrer Institut, Secretariat SGN/SSDN,**
c/o Laboratory for Neutron Scattering, bldg. WHGA/147,
5232 Villigen PSI, Switzerland