Report on the ESS Science Symposium on Neutron Scattering at Extreme Conditions

5-6th July 2013 Centre for Science at Extreme Conditions , Edinburgh

Introduction

Extreme conditions neutron scattering is an important and growing area. It has had vital contribution to longstanding challenges like the behaviour of fundamental simple molecular systems like ice and hydrogen, the physico-chemistry of Earth's mantle, and the behaviour of planetary ices under in-situ conditions. Also, neutron scattering at extreme conditions is likely to have an important role in emerging new challenges like quantum critical phenomena and the search for new materials on which to base the hydrogen economy. As a result, all the current major international facilities (ISIS, SNS, J-PARC, ILL) have major programmes in this area and either operating dedicated EC beamlines or plans to build such beamlines. The aim of this symposium is to establish the important science drivers in the area over the period up to the opening of ESS, to identify the requirements for neutron instrumentation and for sample environment equipment, and to establish a interest group/user community for EC as the ESS.

The European Spallation Source (ESS) will be, when operating in 2019, the premier neutron facility worldwide. As part of the Pre-construction and Design-Update Phase leading up to the start of construction in 2013, we are currently evaluating the scientific opportunities that the ESS will offer, encompassing all areas of science.

The symposium was organised to bring together the European Extreme Conditions community to explore the likely important science drivers for EC science at ESS and to consider the needs for EC instrumentation to address these science drivers. The meeting was attended by participants from across Europe as well as from the U.S. and Japan. The talks covered a wide range of science including biology, earth and planetary science, magnetism and the physics of fundamental small molecules. Lively discussion was stimulated by the talks and the meeting produced the following recommendations.

General recommendations

a. Access to extremes of temperature, pressure and magnetic fields at ESS is crucial for a wide range of scientific communities (Biology, Earth Science, Materials Science, Fundamental Physics and Chemistry).

b. State of the art equipment to achieve these extremes on a wide range of instruments in the form of transportable and transferrable sample environment equipment will be required to satisfy some of this demand. This is likely to take the form of simple high-P equipment (gas cells, clamp cells, PE-cells), furnaces (including levitating furnaces), cryostats (including dilution fridges) and superconducting magnets. There will also be a need to access multiple extreme (for example, High-T High-P, High-P High-B, High-B and Low-T etc.)

2. Specific recommendations

a. For state of the art extreme conditions dedicated instruments are essential.

b. The requirements of very high fields and very high pressure are almost impossible to satisfy in a single instrument and we recommend that very high pressure and very high field instruments be pursued separately. Similarly, it is difficult to optimise an instrument for both elastic and inelastic scattering and hence these goals should also be accommodated on separate instruments.

c. There is a clear need for a high-intensity focussed beam instrument for medium to high resolution diffraction studies to megabar pressures. This will the development and use of diamond anvil techniques and the application to the instrument of experience gained from synchrotron x-ray HP work will be essential.

d. A dedicated high magnetic field instrument is advisable and the technical specification should be thoroughly investigated.

e. The advanced instruments above are technically challenging need a critical mass of skilled and motivated scientists. Strong links to university research groups and other facilities (neutron and synchrotron) including university staff based in outstations at Lund is essential as shown by the experience gained in the creation of all other dedicated high-pressure facilities around the world (ISIS, SNS, J-Parc).

f. There is a need to identify champions for these instruments to lead expressions of interest and to build links to the relevant communities. A present this appears likely to require the appointment of experienced staff to ESS to facilitate this process.

Programme

Friday 5th July 2013

Session 1 Chair: P Attfield, CSEC, University of Edinburgh

09.30 Welcome

09.40 Paul Henry, ESS, Lund

"What will ESS bring to Extreme conditions research?"

Session 2 *High Pressure Instruments Worldwide* Chair: J Loveday, CSEC, University of Edinburgh

11.00 Chris Tulk, Oak Ridge National Laboratory

"Successes and otherwise while building the SNAP high-pressure diffractometer at the Spallation Neutron Source"

11.40 Craig Bull, ISIS

"Advances in High Pressure Diffraction Techniques at the ISIS Neutron Facility"

12.20 Takanori Hattori, JPARC

"New High-Pressure Neutron Beamline PLANET at J-PARC"

Session 3 *Techniques: Low T, high P and high B* Chair: S Klotz, University Pierre et Marie Curie, Paris

14.00 Michael Meissner, ESS, Lund

"The Sample Environment Task: from Berlin to Lund"

14.40 Konstantin Kamenev, CSEC, University of Edinburgh

"Instrumentation development for neutron scattering at high-pressure and low-temperature"

15.20 Karel Prokeš, Helmholtz Zentrum Berlin

"Prospects for Neutron Research in Magnetic Fields"

Session 4 *Disordered and Biological Systems* Chair: P Henry, ESS, Lund

16.30 Phil Salmon, University of Bath

"Networks under Pressure"

17.10 Giovanna Giulia Simeoni, TUM - FRM II, Munich

"Neutron Spectroscopy under Extreme-Environment conditions"

17.50 Laura Spagnola, CSEC, University of Edinburgh

"Structural studies of proteins at extreme conditions"

18.30 Close

Saturday 6th July 2013

Session 5 *Geophysical and Earth Sciences* Chair: P. Salmon, University of Bath

09.30 Malcolm Guthrie, Geophysical Laboratory, Carnegie Institution of Washington

"Neutron Crystallography in the megabar range using diamond anvil cells"

10.10 Simon Redfern, University of Cambridge

"Rheology and anelasticity of Earth Materials: P/T/strain measurements"

Session 6 *Science Applications and Future Directions* Chair: P. Attfield, CSEC, University of Edinburgh

11.20 Takashi Saito, Kyoto University

"A-site-ordered perovskites: High-pressure synthesis and structural and physical property measurements under high pressure"

12.00 Christian Rüegg, Paul Scherrer Institute

"Extreme Magnetism"

12.40 Stefan Klotz, University Pierre et Marie Curie, Paris

Closing remarks & discussion

13:00 Close

Attendees

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Speaker Abstracts

Session 1

What will ESS bring to Extreme conditions research?

Paul F. Henry

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The European Spallation Source (ESS) will be a 5MW long-pulse spallation source, the first of its kind. The long-pulse offers unparalleled source brilliance, while at the same time a time-averaged flux commensurate with the most powerful research reactors available today.

Here, I will give an update on the status of the project, including how the characteristics of a long pulse source differ from reactor and short pulse spallation sources and how these characteristics can be harnessed by the extreme conditions community in Europe and further afield and to invite feedback from the future users. Engaging the community is important to us in order to design and build an instrument suite that reflects the needs of the scientist that will use the facility in the future. The reference instrument suite, as presented in the Technical Design Report (TDR) used for the costing of the ESS, will be presented and specific emphasis placed on the instruments most suited for extreme conditions materials research.

The ESS is a European large-scale facility project with 17 international partners based in Lund, Sweden, with construction planned to begin in 2013. It is scheduled to deliver its first neutrons to target in 2019 and have its full design complement of 22 public instruments by 2025. The ESS will offer new opportunities to all areas of scientific research, as well as complementing the existing neutron sources, both reactor and spallation-based, in Europe. The instrument suite is currently under development and provides an opportunity to investigate and evaluate novel instrument concepts that fully utilise the possibilities presented by a long-pulse source.Contact: paul.henry@esss.se

__Session 2__ High Pressure Instruments Worldwide

Successes and otherwise while building the SNAP high-pressure diffractometer at the Spallation Neutron Source.

C. A. Tulk,[a] A. M. dos Santos,[a] J. J. Molaison,[a] M. Guthrie,[b] R. Boehler[b]

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Recently there have been a number of breakthroughs in high pressure neutron diffraction techniques. A collaboration between the SNS at Oak Ridge National Laboratory, the SNAP instrument team and the Carnegie Institution of Washington has enabled high quality neutron powder diffraction data to be collected over an unprecedented pressure-temperature range. Bringing together high pressure cell design expertise and neutron instrumentation design expertise has been key to success. A broad overview for the prospects for future high pressure neutron diffraction will be presented in the ICNS Plenary sessions by H.-k Mao, with scientific results presented separately by M. Guthrie. In this talk I will review the design of the SNAP instrument and share some of the details that have made these breakthroughs possible, in addition, I will share ideas and efforts that did not 'yield fruit' as this project moved forward. I will finish by providing a few examples of systems that have been studied at SNAP.

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Advances in High Pressure Diffraction Techniques at the ISIS Neutron Facility

Craig L. Bull, William G Marshall, Matthew G Tucker
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The Pearl High Pressure Facility is a medium resolution high-flux neutron diffractometer optimised for data collection from the Paris-Edinburgh pressure cell. The instrument is situated on target station one of the ISIS Neutron Spallation Facility in the UK.

Pearl has recently been rebuilt following a major upgrade funded (€1.43M) by the CSIC (Consejo Superior de Investigaciones Cientificas) in Spain. Commissioning measurements on the new beamline have been completed and the user programme of experiments has resumed.

Taking examples from experiments performed on the Pearl instrument, we will present recent scientific highlights where the experiments have made use of new anvil materials, low and high temperature setups and the new instrument capabilities itself. We will also show developments which are currently in progress. Future envisaged high pressure developments will also be presented.

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New High-Pressure Neutron Beamline PLANET at J-PARC

T. Hattori[1,2], A. Sano-Furukawa[2,1], H. Arima[3], Y. Inamura[1], T. Nagai[4], Y. Katayama[2], T. Inoue[5], H. Kagi[6], T. Yagi[5]

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The PLANET is the high-pressure beamline newly constructed at J-PARC. The beamline aims at revealing the effect of water on the Earth's interior. The most characteristic feature is to possess the huge 6-axis press with the maximum load of 500ton/axis, which can generate high-PT condition of 10GPa and 2000K, simultaneously. By using the state-of-the-art neutron diffraction and radiography techniques, the beamline offers microscopic and macroscopic information of materials at high-PT conditions. The beamline is designed so as to analyze structures of both crystalline and amorphous materials. In designing the beamline, we focused on obtaining clear patterns even from tiny high-pressure samples. To reduce the background, incident beam is cut into the size relevant to the sample with a 4-dimensional slit placed just before the sample. To confine the scattering volume along the beam axis, 90 degree scattering geometry is adopted and the detector banks are equipped with radial collimators. This coupling enables us to obtain the information from 3mm cube in the high-pressure cell. The commissioning was finished at July 2012 and user experiments have started since November 2012. The resolution in the diffraction pattern was found to be almost equal to the designed value (Dd/d=0.6%), and the accessible d-range was 0.2-4.2A in the single frame mode (twice in the double frame mode). These specifications are sufficient to analyze structures of complex minerals as well as liquid/amorphous materials. The severe collimator system completely eliminated Bragg peaks from the sample surrounding materials even at high pressures, which enabled the observation of very weak magnetic peaks as well as the fine structure analysis of liquid and amorphous materials at 10 GPa. The beamline is now being used by project members and will be opened for general users in the next February.

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Session 3 *Techniques: Low T, high P and high B*

The Sample Environment Task: from Berlin to Lund

Michael Meissner

European Spallation Source, Lund and Helmholtz Zentrum Berlin

I will give a survey on 20 years of establishing the Sample Environment (SE) at HMI-BENSC (now HZB). The historical view might shade some light on how to start and develop this task at the ESS in Lund. At HZB we are able to offer to users a wide range of SE equipment and also we provide an intensive support to the neutron instruments and to the experiments, as well. With respect to extreme conditions in neutron scattering, starting in the early '90s, we developed dilution refrigerator inserts for fast sample changes operating down to 25 mK in split pair magnets up 15 T. Ten years later, at HZB the 30 Tesla hybrid magnet project was started and I will give an up-date on the present status with a foreseen delivery and start of the commissioning phase in early 2014.

However, during the last decade, the quest for new SE techniques (in-situ lab style measurements, time-resolved observations in materials under application of gases and/or liquids, and more) was limited by reduced man power and financial investments. Fortunately, in Europe with the NMI-3 program, a dedicated SE-JRA became to operation and since 2007 a variety of tasks and deliverables for new SE equipment has been defined. Today, from this shared effort of about five SE teams in European NSFs, new prototypes of humidity cells, furnaces for levitated samples up to 3000°C and various high-pressure gas devices are operating for user experiments, already. This share of knowledge and collaboration provides a good basis for building a wide ranged suite of Sample Environment at the European Spallation Source in Lund.

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Instrumentation development for neutron scattering at high-pressure and low-temperature

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Engineering approach to developing high-pressure cells and use of computer aided design (CAD) and finite element analysis (FEA) will be presented and illustrated with several examples. These include a large-volume piston-cylinder pressure cell with optimized transmission which has been used for inelastic neutron scattering studies of UGe_2 [1] as well as two opposed-anvil cells for neutron diffraction at cryogenic temperatures. One of them is a pressure cell developed for continuously changing pressure at low-temperatures. It incorporates a custom-built bellow capable of generating the load of 220 kN using compressed helium gas. The other cell is a miniature sapphire anvil cell which can fit into restricted space available in cryomagnets and used in SANS experiments on Nb crystals. Combined with experimental data, finite element analysis can also be used as a tool for deriving mechanical properties of materials at extreme conditions, and its use for optimizing the shape and dimensions of anvils in opposed-anvil devices will be presented.

References:

[1] W. Wang, D. A. Sokolov, A. D. Huxley, K. V. Kamenev, *Rev. Sci. Instrum.* 82, 073903 (2011).

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Prospects for Neutron Research in Magnetic Fields

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The Helmholtz Zentrum Berlin is known for its sample environment that is available for both internal and external users. By offering the highest static magnetic field of 17.4 T for neutron scattering, it offers unique experimental possibilities [1]. Presently, a project that combines dedicated scattering instrument (ExED) with a horizontal solenoid magnet with tapered cones is being realized. The magnet that utilizes hybrid (resistive insert and superconducting outsert) technology should be capable to produce 26 T consuming 4 MW of electrical power [2] with a possible upgrade to 33 T (8 MW). It is planned to become available in 2014. The ExED instrument is of time-of-flight (TOF) type and optimized for diffraction at extreme conditions.
In the contribution I first list few past and recent scientific activities that include neutron scattering experiments on (frustrated) magnetic systems, iron based superconductors and experiments under combined extreme conditions and discuss existing limitations and drawbacks. Then I outline the technical parameters of the ExED instrument and our future scientific prospects using the high field magnet at HZB and compare them with possibilities after the ESS is in operation. Being a spallation source, the extreme sample environment at ESS could be based on different approaches. For instance, pulse magnetic fields might be an attractive option. In my talk I will discuss few considerations that influence the selection of the future sample environment, mainly those connected with magnetic fields.

References
[1] P. Smeibidl et al., J. Low Temp. Phys. 159, 402 (2010).
[2] http://www.helmholtz-berlin.de/zentrum/perspektiven/hfm/index_en.html.
[3] K. Prokeš, et al., Physica B 312–313, 872 (2002).

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Session 4 *Disordered and Biological Systems*

Networks under Pressure

Philip S. Salmon[1], Kamil Wezka[1], Anita Zeidler[1], Keiron J. Pizzey[1], Dean A. J. Whittaker[1], James W. E. Drewitt[1], Ruth F. Rowlands[1], Stefan Klotz[2], Henry E. Fischer[3], Craig L. Bull[4], Matthew G. Tucker[5], Martin C. Wilding[6], Malcolm Guthrie[7]

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The structural changes in glasses and liquids induced by high-pressure and/or high-temperature conditions can alter substantially their dynamical and transport properties. A notable example is provided by so-called polyamorphic transitions where the variation of a state parameter such as pressure or temperature leads to an abrupt transformation between two phases having the same composition but different densities. Unravelling the mechanisms by which these transformations occur is, however, a formidable task owing to the nature of structural disorder and the experimental difficulties associated with the investigation of materials under extreme conditions. This talk will focus on recent advances to measure the structure of network-forming glasses using *in situ* neutron diffraction with a Paris-Edinburgh press at pressures up to 17.5(5) GPa [1-4]. In particular, the mechanisms of density-driven structural collapse in glasses such as GeO2 and GeSe2 will be considered, where the debate is informed by the results obtained from the first applications of the method of high-pressure neutron diffraction with isotope substitution. The diffraction data are complemented by the results obtained from new molecular-dynamics simulations.

References

[1] A. Zeidler *et al.*, J. Phys.: Condens. Matter 21, 474217 (2009)
[2] J. W. E. Drewitt *et al.*, Phys. Rev. B 81, 014202 (2010)
[3] P. S. Salmon *et al.*, J. Phys.: Condens. Matter 24, 415102 (2012)
[4] K. Wezka *et al.*, J. Phys.: Condens. Matter 24, 502101 (2012)

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Neutron Spectroscopy under Extreme-Environment conditions

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TOFTOF is a cold-neutron TOF spectrometer installed at the FRM II. Over the last two years, great effort has been dedicated to the instrumental improvement for the investigation of magnetic systems and small samples. This is the case of pioneering and avant-garde experiments employing extreme conditions sample environment, like high pressure cells or electromagnetic (EML) and electrostatic (ESL) levitator devices. For all these kinds of investigations an increased neutron flux over a small sample area is required.

The prototype of a focusing neutron guide, developed and produced completely in-home at the FRM II, has been recently installed at the TOFTOF instrument and came already routinely into operation.

It represents the first device in the world combining the leading-edge supermirror technology with the Adaptive Neutron Optics, suitable for Neutron Spectroscopy with a thermal-cold white neutron beam (1.4–14 Å).

We present a comparison between the simulated and measured beam cross sections, as well as the new performance of the instrument during QENS and INS experiments on molten and solid samples in levitators, high temperature vacuum furnaces and gas high-pressure cells [1].

[1] G.G.Simeoni et al., "*Focusing Adaptive Optics: Neutron Spectroscopy under Extreme-Environment conditions*", in preparation

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Structural studies of proteins at extreme conditions

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Proteins are complex polymers, which elicit diverse functions in cells. A key feature of proteins is their ability to adopt different conformations. In this talk, I will present recent work carried out in my laboratory on the Fen1 nuclease from the extremophile Pyrococcus abyssi. These include high-pressure crystallography work, as well as preliminary SANS experiments. I will comment on the conformational changes elicited by an increase of pressure and temperature in this protein.

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Session 5 *Geophysical and Earth Sciences*

Neutron Crystallography in the megabar range using diamond anvil cells

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Recent decades have seen dramatic technical advances in extreme conditions science revealing a wide range of unexpected and fascinating behaviour in condensed matter systems. The sensitivity of neutron crystallography to light atom positions and long-range magnetic order makes this a critical experimental technique for the study of these materials. However, to date, such studies have been confined to relatively low pressures (~25 GPa), severely constraining the range and applicability of neutron science.

Over the last 5 years, we have been collaborating with the SNAP beamline staff at the SNS to overcome this severe limitation on present neutron capabilities. By focusing on the extension of existing diamond-anvil cell (DAC) techniques we have successfully demonstrated quantitative neutron diffraction at close to 1 megabar (100 GPa) pressures. In addition to describing the new cell designs [1], we will also describe their implementation on the SNAP instrument and the development of specialised data-reduction procedures. This new capability has recently been used in a study of ice [2] up to unprecedented pressures, clearly demonstrating the potential of this important new technique.

References:

[1] R. Boehler, M. Guthrie, J.J. Molaison *et al.* “Large-volume diamond anvil cells for neutron diffraction above 90 GPa” High Press. Res. *In press*

[2] M. Guthrie, R. Boehler, C.A. Tulk et al. “Neutron diffraction observations of interstitial protons in dense ice”, Proc. Nat. Acad. Sci. 10.1073/pnas.1309277110 (2013).

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Rheology and anelasticity of Earth Materials: P/T/strain measurements

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The dynamic behaviour of Earth's mantle and core depends upon the time-dependent mechanical response of its component minerals. In turn, these depend upon viscoelastic processes which of bulk materials. Neutron scattering is uniquely placed to provide the essential probes to illuminate the mechanical properties of rocks and minerals. Phase transitions, microstructures, and deformation mechanisms all play a part in defining the viscoelasticity of polymineralic assemblages.

Realistic models of viscoelasticity in the deep Earth require probes that span a suitable range of pressures, temperatures, stresses and timescales. The development of high-P/T-stress apparatus for structural in situ studies of mineral mechanics at extreme conditions has been pioneered at neutron sources. Here, I will review the developments that have already taken place, and discuss how they might be applied for the solution of future problems.

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Session 6 *Science Applications and Future Directions*

A-site-ordered perovskites: High-pressure synthesis and structural and physical property measurements under high pressure

Takashi Saito and Yuichi Shimakawa

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A-site-ordered perovskites, in which transition metal cations occupy 3/4 of the A-site of the perovskite structure, provides an attractive playground in search for intriguing functions and physical properties. Many of the materials with this structure type are synthesized under high pressures in the GPa range, and we have recently discovered many new compounds. $LaCu_3Fe_4O_{12}$ is a newly synthesized compound at 10 GPa and its chemical formula is $La^{3+}Cu^{3+}{}_3Fe^{3+}{}_4O_{12}$ at room temperature and ambient pressure. The compound undergoes an inter-site charge transfer $3Cu^{3+} + 4Fe^{3+} \rightarrow 3Cu^{2+} + 4Fe^{3.75+}$ by increasing temperature (393 K) [1] and by increasing pressure (~3 GPa) [2]. The instability of the unusually high-valent $Cu^{3+}/Fe^{3.75+}$ cations gives rise to a rich phase diagram in a wide temperature-pressure range. Some other materials with this structure type will also be presented.

References:

[1] Y. W. Long, T. Saito, Y. Shimakawa, et al., *Nature* 458, 60 (2009).

[2] Y. W. Long, T. Saito, Y. Shimakawa, et al., *Chem. Mater*. 24, 2234 (2012).

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Extreme Magnetism

Christian Rüegg

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Neutron scattering is a prime experimental tool to study magnets. This is especially true for experiments at extreme conditions like high magnetic fields and hydrostatic pressures in combination with low temperatures, and for fully quantitative studies enabled by the neutron scattering cross section that is known exactly. Examples of recent experiments on a number of model quantum magnets using these specific advantages will be presented. These include studies of quantum critical points and of magnetic order and dynamics in the maximum magnetic field and pressure ranges that are currently available. The results will be discussed in the context of current limitations and future perspectives for studies of magnetism with neutrons at the ESS.

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Poster Presenters

Angel Arevalo-Lopez, University of Edinburgh

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Oliver Burrows, University of Edinburgh

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Charge, spin, vacancy and cation ordering in $Sr(Cr_{1-x}Fe_x)O_{3-\delta}$ *(x = 0, 0.5) perovskite superstructures. New hard-soft phases and substituted derivatives*

Craig Wilson, University of Edinburgh

On the possibility of ammonia-water solid solutions at high pressure

Field-induced spin-orders in 1-dimensional Co^{2+} chains in monoclinic CoV_2O_6 brannerite

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Metamagnetism and magnetization plateaus are among the unusual properties of low dimensional magnetic oxides based on spin-3/2 ions such as Co^{2+}. 1/3 (of ferromagnetic) magnetization plateaus have been predicted and observed experimentally in spin-3/2 antiferromagnetic uniform chains [1] and are known to occur also in spin-3/2 ferromagnetic uniform chains.[2]

Temperature dependent NPD at HRPD@ISIS shows that the monoclinic brannerite-type CoV_2O_6 orders antiferromagnetically at T_N=15 K with a *a* x *b* x 2*c* supercell in which Co^{2+} moments of magnitude 4.77(4) mB at 4 K lie in the *ac* plane and are ferromagnetically coupled within chains of edge-sharing CoO_6 octahedra parallel to *b*. Ferromagnetic chains are coupled antiferromagnetically to neighbouring chains in the *a* and *c* directions.[3]

Furthermore, applied magnetic field NPD from WISH@ISIS performed at H = 0, 2.5 and 5 T shows three collinear magnetic phases as field increases: an AFM state with propagation vector (0 1 1⁄2), a ferrimagnetic (–1⁄3 1 1⁄3) phase, and a (0 0 0) ferromagnetic order. In all cases, Co^{2+} moments of 4.4–5.0 μB have a large orbital component and are aligned close to the *c*-axis direction. Spin-lattice coupling leads to a magnetostriction and volume expansion as field increases. The ferrimagnetic phase accounts for the previously reported 1⁄3-magnetization plateau, and demonstrates that monoclinic CoV_2O_6 behaves as an accidental triangular antiferromagnetic lattice in which further frustrated orders may be accessible.[4]

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Hidden antiferromagnetism in a tungsten double perovskite with $5d^1$ local moments

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We studied the magnetic properties of the cubic rocksalt ordered double perovskite Ba_2YWO_6 with localised W^{5+} $5d^1$ electrons. Refinements of X-ray and neutron diffraction data confirm a cubic Fm-3m structure indicating that the $5d^1$ electron is in a triply degenerate t_{2g} orbital, which will be partially lifted by the strong spin orbit coupling yielding a $J = 3/2$ quadruplet. It had been predicted that the magnetic moments from the spin and orbital degrees of freedom cancel-out to give a (near-) zero effective magnetic moments. SQUID magnetometry and heat capacity data will be presented which confirm that while we have $J = 3/2$ moments that interact via the usual antiferromagnetic super-exchange, the effective moments from these spins is only 5% of the effective moment of an $S = 1/2$ spin and its contribution to the paramagnetic susceptibility is small compared to the diamagnetic suspectibility. Ba_2YWO_6 is the first material where this near-ideal cancellation is observed.

Explosives at Extreme Conditions: Polymorphism of 2,4-Dinitroanisole

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2,4-dinitroanisole (DNAN) is an energetic material, developed as an insensitive replacement for TNT in melt-cast explosive formulations. While DNAN-based formulations demonstrate greatly reduced sensitivity to accidental initiation compared to those using TNT, issues remain with the replacement of TNT with DNAN.[1] For instance, DNAN based formulations have demonstrated catastrophic levels of irreversible growth during heat-cycling, with volume increases of up to 15% reported. [2]

In order to investigate the role of polymorphism in the irreversible growth of DNAN, high-pressure and variable-temperature neutron and x-ray diffraction studies have been performed. Two polymorphs of DNAN have been found to exist at ambient temperature and pressure, the thermodynamic form, DNAN-I, and the kinetic form, DNAN-II.[3,4] The phase diagrams of both form-I and -II of DNAN have been explored for the first time.

In the case of DNAN-II, two high-pressure phase transitions were found. DNAN-II initially transformed to DNAN-III, which at higher pressures transformed to DNAN-IV. No further phase transitions were noted up to 5.88 GPa. In addition, variable temperature studies demonstrated that the DNAN-II to DNAN-III transition also occurs when DNAN-II is cooled below room temperature. The thermal expansion of the DNAN-II/III lattice was investigated from 150K to 363K, demonstrating that an abrupt change in the thermal behaviour of lattice parameters occurs at the DNAN-II/III transition. From these combined crystallographic studies, the structure of DNAN-III has been solved, showing it is closely related to DNAN-II.

In the case of DNAN-I, a transition to form III was not found during variable temperature studies, as may be expected given the dissimilarity between the DNAN-I and DNAN-II structures. High-pressure neutron powder diffraction studies, however, demonstrated that DNAN-I does transform at high-pressure to a new form (DNAN-V) that is distinct from DNAN-II,-III or -IV. Rietveld refinement of the high-pressure DNAN-I data also determined that the material exhibits negative linear compressibility, which is of interest given the use of DNAN as a shock-insensitive energetic material. Comparison of the behaviour of DNAN-I and –II under variable temperature and high-pressure conditions indicates that the kinetic form, DNAN-II, is the denser phase under all conditions studied.

The role of polymorphic transitions between DNAN-I and II during the melt-cast processing of energetic formulations has also been investigated. This work highlights the importance of crystallographic techniques in order to understand the polymorphism of energetic materials.

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Working with extremes, CAMEA – The Continuous Angle Multiple Energy Analysis Instrument Concept

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The CAMEA instrument concept is a neutron spectrometer designed for optimal efficiency in the horizontal scattering plane that enables detailed and/or rapid mapping of excitations. Optimization of a spectrometer in this geometry is ideally suited to studies of materials under extreme environments, the complex sample environs of insitu experiments, or where neutron studies of specific region of reciprocal space are required.

In in this presentation we will introduce the indirect geometry spectrometer CAMEA concept, and how CAMEA represents an evolution in neutron instrumentation. We will discuss the advantages of using the CAMEA concept on both pulsed and continuous neutron sources, and our progress on the realization of CAMEA in both cases. Finally we will highlight the experimental capabilities of CAMEA for measuring materials under extreme conditions.

Neutron Diffraction under Extreme Conditions

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The ability to obtain accurate structural data with neutron diffraction at high pressure has provided a wealth of new information [1,2]. The Paris-Edinburgh press can access pressures up to 30 GPa [3] and temperatures between 10 and 1000 K and its open Bridgman-anvil geometry allows a wide range of difficult samples (low-boiling point gases, liquids, air and water-sensitive systems, and liquid- and solid-gas mixtures) to be loaded and in some cases turned into single crystals [4,5]. This flexibility has allowed us to obtain valuable information on topics as diverse as the fundamental behaviour of hydrogen bonds [1] to the origin of the methane in Titan's atmosphere [2].

We have recently carried out studies of simple molecular systems and mixtures at high pressure. These include; the ordering of ice VI under pressure, the first high-pressure investigation of the expanded metal Li(NH3)4, the structures and transitions of amorphous ices, and new hydrogen and other gas storage materials based on water and other cage forming molecules. Results from a range of these systems will be presented.

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CAMEA- Prototype

M. Markó, PSI LNS

The CAMEA (Continuous Angle Multiple Energy Analyser) is a multi-analyzer back end for inelastic instruments. The CAMEA with time of flight front end enables to perform fast quasi-continuous mapping of the q-ω space. This concept is well suited for studies where the sample environment enables to detect the scattered neutrons only in a small vertical angular range around the horizontal plane (eg. Split-coil magnet or pressure cell) . We are developing such an instrument called extreme environment spectrometer for ESS. The prototype of the instrument was planned and built at DTU and KU and installed at MARS instrument at PSI. In this presentation we present the results of the first measurements on the prototype (resolution in the q-ω space, and an inelastic measurement on LiHoF4 sample) and compare the results with our analytical calculations and McStas simulations.

Charge, spin, vacancy and cation ordering in $Sr(Cr_{1-x}Fe_x)O_{3-\delta}$ (x = 0, 0.5) perovskite superstructures. New hard-soft phases and substituted derivatives

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Low temperature reduction of the high pressure perovskite SrCrO3 (hard-soft chemistry) allows us to obtain two new SrCrO3-d phases (d=0.2 and 0.25) with unusual superstructures and properties.[1] Both are re-oxidized to cubic SrCrO3 on standing air and form long-period Cr3+/Cr4+ charge-density waves. Reconstruction from octahedral geometry to tetrahedral environments in widely spaced (111) planes gives 15R and 6H repeat sequences for d=0.2 and 0.25 respectively. D20@ILL data reveal a long range spin order in the 15R phase where ferromagnetic layers are antiferromagnetically coupled to adjacent spin planes along c. Magnetic moments are parallel to the c-axis with refined magnitudes of 2.4(5), 1.3(3) and 1.1(2) μ_B and demonstrate that the Cr charge-density wave gives rise to a spin-density wave-type modulation of the magnetic moments with a long, doubled c-axis, periodicity (2c≈69 Å). The SrCrO3-d vacancy mechanism may be relevant to related SOFC anode materials since Fe substitution stabilizes the d=0.2 superstructure at ambient pressure. Substituted Sr(Cr-1xFex)O3-d phases with the same 15R structure were obtained by solid state reaction at high temperature. HRPD@ISIS data show that the 15R-SrCr0.5Fe0.5O2.8 has a partial cation ordering. The magnetic structure shows coexistence of two coupled magnetic propagation vectors (0, 0, 0) and (0, 0, 3/2) that vanish at Tc=263(2) K.[2]

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On the possibility of ammonia water solid solutions at high pressure

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The ammonia hydrates are some of the simplest systems that contain mixed hydrogen bonds, (O-H…N and N-H…O bonds) making them ideal systems to explore how these mixed hydrogen bonds are affected under high pressure conditions. Ammonia Hemihydrate II (AHH-II) is an important ammonia hydrate because it is the only ammonia hydrate that can be created from the liquid at room temperature [1]. Liquids of both ammonia monohydrate (AMH) and ammonia dihydrate (ADH) compositions crystallise to form a mixture of AHH-II and ice VII when pressurised at room temperature. The structure of AHH-II has recently been solved and has been found to have a molecular packing that is very similar to that of ice VII but with a drastically different hydrogen bonding network based exclusively on N-H…O, O-H…N and N-H…N H bonds [1]. In earlier work, we have shown that when compressed at liquid nitrogen temperatures to ~ 5 GPa and subsequently warmed to room temperature, AMH and ADH form a disordered molecular alloy (DMA) with a body-centred cubic structure, with substitutional disorder of the ammonia and water molecules [2,3]. We present here data that show that this DMA structure can also be produced by room temperature compression of AHH, AMH and ADH solutions. This confirms earlier speculation that a water-ammonia solid solution exists over a wide range of compositions at high pressure. This has important potential consequences for the outer planets which contain large quantities of both water and ammonia.

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