the framework occurs upon calcinations, thus explaining the negative thermal expansion.

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**Keywords: negative thermal expansion, trioxane silica sodalite, in situ time-resolved diffraction.**

MS05.P29

Structural study of $Eu_2(MoO_4)_3$ and $Sm_2(MoO_4)_3$ polymorphs

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Light RE molybdates (RE=La-Eu) can occur in two differently ordered scheelite-type ($CaWO_4$) structures: $La_2(MoO_4)_3$ [1] and α-$Eu_2(MoO_4)_3$ [2]. Intermediate molybdates (RE=Sm-Ho) show the β'-$Gd_2(MoO_4)_3$ ferroelectric-ferroelastic phase [3]. A sound knowledge of the dielectrical and optical properties of $RE_2(MoO_4)_3$ crystals correlated with their crystal structure is of fundamental importance for the application of these materials. Sm and Eu molybdates are particularly interesting because both phases (α and β') are stable at room conditions [4], [5]. In this work, we have studied the evolution of the four phases (α, β', β and amorphous) of $Eu_2(MoO_4)_3$ and $Sm_2(MoO_4)_3$ with the temperature, in order to explain a new anomaly detected in their dielectric permittivity and in the heat capacity around 275K. Such anomaly can be related with a phase transition between the ferroelectric phase β' and/or to the α phase, before the ferroelectric(β')-paraelectric(β) transition and the β-α transition at higher temperature. We have applied a new alternative method for treating distorted structures by means of symmetry mode analysis, performed using the program AMPLIMODES, developed in the Bilbao Crystallographic Server [6], and the Rietveld refinement of the amplitudes of such symmetry modes, instead of dealing with the atomic coordinates using the Fullprof program [7]. Moreover, the diffuse scattering has been analyzed and quantified to explain the dielectric and thermal anomalies.
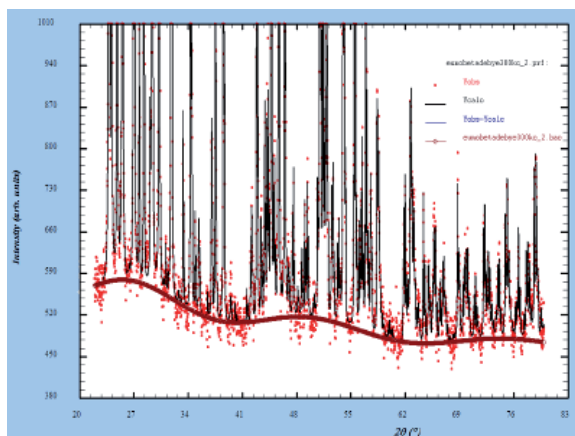
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Keywords: rare-earth molybdate, phase transition, polymorphism.

MS05.P30

Recent developments at the ECHIDNA high-resolution neutron powder diffractometer

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The ECHIDNA high-resolution neutron powder diffractometer at OPAL has been in routine and largely trouble-free operation for three years. Data from ECHIDNA have appeared in over 30 publications, covering a temperature range of 1.5K to 1900K and magnetic fields up to 7 T.

ECHIDNA data reduction is largely automated, with a user-friendly interface and a wide choice of output formats (hdf, pdCIF, xyd, GSAS). Data reduction implements a sophisticated approach to gain correction, described in a companion poster [1], and provides large amounts of metadata relating to the data reduction via the pdCIF output format.

The range of instrument configurations continues to expand. A low-temperature (<1K), cryogen-free top-loading cryostat is currently being commissioned and first results are reported. In addition, the maximum takeoff angle has been increased from 140 to 155 degrees without compromising our ability to use the robotic sample loader.

In addition to the regular calls for proposals twice a year, ECHIDNA operates a popular fast access mail-in program. In order to participate, applicants should fill in the simple web-based form accessed via http://neutron.ansto.gov.au. Following safety and technical review, applicants forward their samples to us and data are returned typically within one month of sample receipt. Mail-in sample environment is usually restricted to room temperature and/or 4K.

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Keywords: powder diffraction, instrumentation, neutron

MS05.P31

High-Pressure Neutron Diffraction Study of $Fe_{1.087}Te$

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The crystal and magnetic structures of $Fe_{1.087}Te$ were studied at high pressures and low temperatures. The experiment was performed on the DMC diffractometer at the SINQ spallation source at Paul Scherrer Institut, Switzerland. The sample was loaded into a clamp type pressure-cell using Fluorinert as the pressure transmitting medium. NaCl was used as an internal standard for pressure calibration and the

pressure-cell was loaded into a helium flow cryostate.

Analysis of the recorded data shows that $Fe_{1.087}Te$ is tetragonal with space group $P4/nmm$ at 80 K and ≈0.45 GPa. A commensurate antiferromagnetic monoclinic phase with propagation vector $\mathbf{k}$ = (½ 0 ½) was observed at ≈2K and 0.44 GPa. The magnetic moments of iron of 1.74(7) BM were found to be parallel with the b-axis. The magnetic structure observed at ≈2K and 0.44 GPa is closely related to the magnetic structure observed at ambient pressure and T < 63 K but with a reduced magnetic moment of iron [1]. The tetragonal $P4/nmm$ structure was preserved at a pressure of 1.15 GPa and 80 K. Cooling at this pressure revealed the existence of a new magnetic phase between ≈60 and ≈48 K. The analysis of data recorded at 55 K shows that the magnetic phase is incommensurate with propagation vector $\mathbf{k}$ = (0.445 0 ½), while the chemical structure was found to be orthorhombic, space group $Pmmn$. The data could be fitted with both a sinusoidal (magnetic moments parallel to the b-axis) and a cycloidal (magnetic moments rotating in the a,c-plane) model of the magnetic structure. This magnetic phase seems to be stable between 48 and 60 K. This phase corresponds to the HP1 phase observed by Okada *et al*. by resistivity and magnetization measurements [2]. An incommensurate antiferromagnetic structure with propagation $\mathbf{k}$ = (0.495, 0, 0.486) was observed at ≈2K and 1.15 GPa. The observed magnetic structure is closely related to the commensurate antiferromagnetic structure observed at ≈2K and 0.44 GPa but with a sinusoidal modulation with an amplitude of 1.98(8) BM and it develops gradually upon cooling from 48 to 2 K.

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Keywords: magnetic structure, neutron diffraction, high-pressure.

MS05.P32

Magnetic ionic liquids 1-ethyl- and 1-butyl-3-methylimidazolium tetrachloroferrate(III). Crystallization monitored by powder x-ray thermodiffraction

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Ionic Liquids (ILs) are salts with melting points below 100ºC, i.e. they are liquids comprised entirely of cations and anions. ILs exhibit a unique set of physical properties that make them interesting for different applications: non-volatility, high thermal stability, wide liquid-state temperature range, good solvents for synthesis, etc. [1]. In addition, ILs containing transition metal complexes show very interesting magnetic properties [2]. It is known that the physicochemical properties of the ILs can be tailored by the selection of both cation and anion components, but the origins of their unique behavior are still relatively poorly understood [3]. Previous studies demonstrate that a correlation exists between the structure of the ILs in the solid and liquid states. Therefore the elucidation of the crystal structures of these compounds would provide a strong basis from which the ILs properties can be further rationalized. ILs display very complicated thermal and crystallization behavior, such as supercooling and glass formation, pre-melting or solid-solid phase transitions [4]. Usually, ILs thermal behavior studies have been performed by differential scanning calorimetry but, to our knowledge, no thermodiffraction studies have been reported yet.

One of the most prominent cation-type is the N-N'-dialkylimidazolium. Their counterion may range from a simple halide to more sophisticated anions. We have investigated the thermal behavior of the magnetic room temperature ionic liquids (MRTIL) 1-ethyl-3-methylimidazolium tetrachloroferrate(III), EMIM[FeCl$_4$], and 1-butyl-3-methylimidazolium tetrachloroferrate(III), BMIM[FeCl$_4$], by using X-ray powder thermodiffraction and differential scanning calorimetry. EMIM[FeCl$_4$] displays thermal polymorphism and exhibits reversible and irreversible solid-solid crystalline transitions with the formation of metastable phases. On the other side, for BMIM[FeCl$_4$] homogeneous crystallization from the melt is inhibited and glass formation occurs on cooling. This amorphous solid crystallizes below room temperature by devitrification on heating from the cooled glassy state. Both systems show a very complex behavior with formation of metastable phases depending on the materials thermal histories.

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Keywords: ionic liquid, thermodiffraction, thermal polymorphism

MS05.P33

Dynamic and crystallographic neutron studies of $La_{1-x}Ba_{1+x}GaO_{4-2/x}$: toward a better understanding of the proton diffusion in new fuel cell materials

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Fuel cells convert chemical energy directly to electrical energy cleanly and efficiently. A crucial part of a fuel cell is the electrolyte, a material that conducts ions. On the way towards practicable solutions, one of the biggest problems at the moment is the lack of stable and effective ionic conductor at the desired intermediate temperature range (100–600°C). Solid oxide proton conductors present many advantages compared to the other candidates as they offer higher efficiency than conventional oxygen ion materials.

Novel oxide structures such as $La_{1-x}Ba_{1+x}GaO_{4-2/x}$ containing tetrahedral units and high proton and oxide ion conductivities have been shown to be potential candidates at intermediate temperatures ionic conductors [2,3]. Proton conductivity dominates in wet atmospheres below 700 °C, giving rise to as high protonic conductivity as 10^{-4} S cm^{-1} at 500°C [3,4]. In these systems water can be incorporated into the structure as protonic defects from humid atmospheres. However, despite the importance of proton conduction for energy applications, our understanding of the complex mechanisms driving the proton transport is still not well understood [1].

High-resolution neutron powder diffration as well as quasi-elastic measurements will be presented to highlight our knowledge of the migration/diffusion of protons into the material and therefore understand the dynamic of the phenomenon.

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