[1] A. Aebischer, M. Hostettler, J. Hauser, K. Krämer, T. Weber, H.U. Güdel, H.-B. Bürgi, *Angew. Chem. Int. Ed*. **2006**, *45*, 2802-2806. [2] G. Oszlányi, A. Sütő, *Acta Cryst.* A, **2007**, *63*, 156-163. [3] L. Palatinus, G. Chapuis, *J. Appl. Crystallogr*. **2007**, *40*, 786-790.

Keywords: twinning, modulation, diffuse_scattering

MS30.P04

Acta Cryst. (2011) A**67,** C416

Short range order in ferroelectric triglycine sulphate

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Triglycine sulphate (TGS) $[(NH_2CH_2COOH)_3H_2SO_4]$ is a hydrogen-bonded ferroelectric with a phase transition temperature of 322K [1]. The phase transition is reversible and second-order, order disorder type, making TGS of fundamental interest in the field of phase transitions [2]. Above the critical temperature, one of the glycine molecules is disordered across a mirror plane and below the critical temperature, it chooses a side, breaking the symmetry. The ferroelectric state is obtained through the ordering of the glycine orientations on neighbouring sites, but the mechanism for the phase transition is not well understood.

We have investigated the short range order in TGS by collecting single crystal diffuse scattering data on TGS (x-ray) and fully deuterated TGS (neutron). We have also developed a model of the short-range order using the program ZMC [3] by creating a model crystal in which the molecules can interact and bringing it to equilibrium using a Monte Carlo algorithm. The validity of the model crystal was assessed by calculating the x-ray and neutron diffuse scattering patterns and comparing them with the experimental data.

This approach has yielded new insights into the behaviour of the disordered glycine molecule. For example, above the critical temperature, the correlations in the orientation of the disordered glycine molecule persist over short range rather than being completely random as suggested by the average structure. This is important information because understanding the behaviour of the disordered glycine molecule is crucial to understanding the phase transition and influences the ferroelectric properties of TGS.

[1] S.G. Zhukov, V.A. Tafeenko, G.V. Fetisov, *Journal of Structural Chemistry* **1999**, *31* 84-90. [2] K. Itoh, A. Nishikori, H. Yoklomize, E. Nakamura, *Japanese Journal of Applied Physics* **1973**, *24* 235-251. [3] D.J. Goossens, A.P. Heerdegen, E.J. Chan, T.R. Welberry, *Metallurgical and Materials Transactions A* **2010**, *41* 1110-1118.

Keywords: short range order, diffuse scattering, modelling

MS30.P05

Acta Cryst. (2011) A**67,** C416

Diffuse scattering and superstructures in brownmillerites and related materials

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Brownmillerite-type materials exhibit a wide range of structures, which vary in the arrangement of tetrahedral chains. In brownmillerites perovskite-like layers of octahedra alternate with layers of tetrahedral chains. The tetrahedral chains can adopt two mirror-related configurations, which allow different inter- and intra-layer ordering patterns to form. The simplest of which result in space groups *Pnma* and *I2mb*. More complicated ordering patterns show commensurate or incommensurate intra-layer ordering sequences. Variants with alternating intra-layer order exhibit stacking faults [1] and commensurate or incommensurate stacking sequences. Apart from that, domain structures (anti-phase and twin domains) are possible and cause diffuse scattering to appear.

Diffuse scattering related to stacking faults is observed in layered brownmillerites $Ca_4(Fe,Al)_2(Mn,Ti)O_9$, which exhibit separated blocks (OTO) of the brownmillerite structure. Similar to $Sr_2Fe_2O_5$ [1], the layered brownmillerites show a certain degree of order according to superstructures with different stacking sequences, as evident by electron diffraction experiments. All possible superstructures can be described in superspace group $A2_1/m(0\beta\gamma)0s$ utilising different modulation wave vectors.

In order to explain the observed diffuse scattering intensities, computer simulations were performed. For example, Fig. 1 shows the (3kl) layer of the reciprocal space of a layered brownmillerite. Experimental data is displayed on the right, whereas the left side shows diffuse scattering calculated [2] from a computer model with random stacking faults. The scattering of the average structure (Bragg peaks) is removed from the calculated pattern.

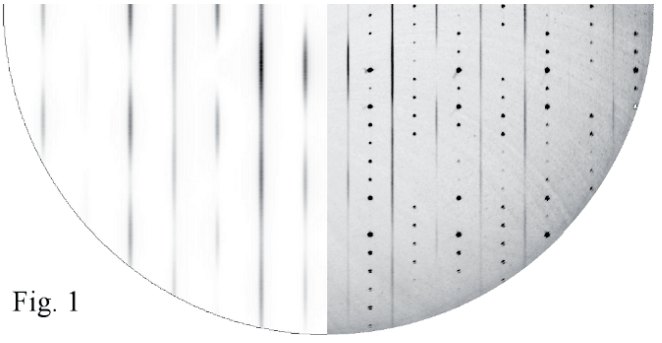

Fig. 1

[1] H. D'Hondt, A. M. Abakumov, J. Hadermann, A. S. Kalyuzhnaya, M. G. Rozova, E. V. Antipov and G. V. Tendeloo, *Chemistry of Materials* **2008**, 20, 7188-7194. [2] B. D. Butler and T. R. Welberry, *Journal of Applied Crystallography* **1992**, 25, 391-399

Keywords: diffuse, scattering, brownmillerite

MS30.P06

Acta Cryst. (2011) A**67,** C416-C417

Hydrogen storage characteristics of melt spun $Ti_{45}Zr_{38}Ni_{17}$ nano-quasicrystalline alloys

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The present study describes the microstructural changes with respect to the quenching rate and their correlation with hydrogen storage characteristics of $Ti_{45}Zr_{38}Ni_{17}$ quasicrystalline alloys. The ribbons of the alloy have been synthesized at different quenching rates obtained through different wheel's surface speeds (35, 40, 45 and 50

m/sec) and investigated for their hydrogen storage characteristics. The lower cooling rate obtained through low wheel speed (35 m/sec) produces, i- phase grains whose size ranges from 300-350 nm. Whereas higher cooling rates obtained through high wheel speed (45 and 50 m/sec) promote the formation of nano-quasicrystalline grains with size ranges from 50-150 nm in $Ti_{45}Zr_{38}Ni_{17}$ ribbons. It has been found that the ribbons synthesized at 35 m/sec absorbed ~2.0 wt%, whereas ribbons synthesized at 50 m/sec absorbed ~2.84 wt.% of hydrogen. Thus the hydrogen storage capacity of ribbon increases with increasing quenching rate. One of the salient features of the present study is the improvement of hydrogen storage capacity obtained through higher quenching rates (~45 to 50 m/sec wheel speed) lead to the formation of lower grain size.

Keywords: quasicrystals, hydrogen storage, microstructure

MS30.P07

Acta Cryst. (2011) A**67,** C417

Diffuse scattering and phason modes in the Zn-Sc icosahedral quasicrystal

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Recently, a new binary icosahedral quasicrystal $Zn_{88}Sc_{12}$ has been obtained by Canfield and co-workers [1]. Because of the chemical order and the x-ray contrast between Zn and Sc, this phase is a nice system for the structure refinement. On the other hand, a large amount of diffuse scattering can be seen on the x-ray diffraction pattern [1]. In this study, we carried out an absolute scale measurement of the x-ray diffuse scattering of the $Zn_{88}Sc_{12}$ to study the possible presence of phason modes (phason diffuse scattering) and estimate phason elastic constats K1 and K2.

Millimeter size single grain of the $Zn_{88}Sc_{12}$ was obtained by slowly cooling from the melt. The sample was polished with a surface perpendicular to 5-fold axis. Systematic *Q*-scans and diffuse scattering maps have been measured on the D2AM beamline (ESRF) using an incoming x-ray energy equal to 9.3 keV.

Compared with ZnMgSc quasicrystal on an absolute scale [2], we find that the amount of diffuse scattering is larger in the $Zn_{88}Sc_{12}$ sample. Also, the maximal Q_{perp} value necessary for indexing the diffraction pattern was found to be less than 3 (r.l.u.) i.e. much smaller than for ZnMgSc for which it was found to be 7 [2]. In addition, as for other quasicrystals [3] a characteristic diffuse intensity distribution due to phason fluctuations around strong Bragg reflections is clearly visible on the systematic reciprocal space map. The ratio K2/K1 is found to be close to the three-fold instability limit, which results in the strong elongation of the diffuse scattering along directions parallel to a three-fold axis. Finally, the simulation is carried out based on the elastic theory and reproduces well the observed anisotropic shape of the diffuse scattering.

[1] P.C. Canfield, M.L. Caudle, C.-S. Ho, A. Kreyssig, S. Nandi, M.G. Kim, X. Lin, A. Kracher, K.W. Dennis, R.W. McCallum, A.I. Goldman, *Phys. Rev. B*, **2010,** 81, 020201. [2] M. de Boissieu, S. Francoual, Y. Kaneko, T. Ishimasa, *Phys. Rev. Lett.*, **2005,** *95*, 105503. [3] T. Janssen, G. Chapuis, M. de Boissieu, *Aperiodic Crystals. From modulated phases to quasicrystals,* Oxford University Press, Oxford, **2007**, 466.

Keywords: quasicrystal, diffuse scattering, phason

MS30.P08

Acta Cryst. (2011) A**67,** C417

Quantitative modeling of diffuse scattering from a relaxor ferroelectric

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The lead-based relaxor ferroelectrics like $Pb(Zn_{1/3}Nb_{2/3})O_3$ - $PbTiO_3$ (PZN-PT) are of pressing applied interest due to their exceptional piezoelectric properties [1]. X-ray single-crystal diffuse-scattering techniques have recently yielded insights into the local structures of these compounds, where marked changes have been observed upon the application of a strong electric field [2]. We will present high-resolution reciprocal-space volume reconstructions from PZN-PT from X-ray CCD images, both with and without an in-situ electric field, and also describe quantitative fits of structural models against our diffuse scattering data. The results facilitate a better understanding of the influence of an applied field on the local structure of this material.

[1] H. Fu, R.E. Cohen, *Nature* **2000,** *403***,** 281. [2] G.Y. Xu, Z. Zhong, Y. Bing, Z.G. Ye, G. Shirane, *Nat Mater* **2006,** *5*, 134.

Keywords: relaxor ferroelectric, diffuse scattering, soft phonon

MS30.P09

Acta Cryst. (2011) A**67,** C417

Framework defects in alumino-phosphate ALPO-5

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ALPO-5 is a well-known microporous alumino-phosphate framework compound[1] with a one-dimensional channel system consisting of 12-ring tubes formed from 6-ring sheets. Several metal cation-substituted forms of this AFI framework type are important due to their superior shape-selective catalytic properties. Single-crystal x-ray diffuse scattering data from ALPO-5 reveal oddly-structured rods of diffuse scattering parallel to the hexagonal axis. Diffuse scattering is not expected in this plane due to the constraints of strict Al-O-P linkage ordering. We will present a defect structure model that explains the observed scattering pattern.

[1] J.M. Bennett, J.P. Cohen, E.M. Flanigen, J.J. Pluth, J.V. Smith, *ACS Sym. Series* **1983**, *218*, 109-118.

Keywords: aluminophosphate, diffuse scattering, faulting

MS30.P10

Acta Cryst. (2011) A**67,** C417-C418

Ab-initio lattice dynamics and thermal diffuse scattering in $CaTiSiO_5$

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