with others indicating larger spacing. Samples reach in PCL (% ≥ 80) exhibit also smaller spacings. Examination of SAXS curve for clay intercalated PHB suggests the existence of thin platelets, constituted by single layers or a stacking of few layers. No evidence of similar behavior has been observed in NC prepared with other polymers proportions, in spite of the observed improvement of their characteristics.

[1] A. Botana et al. *Applied Clay Science* **2010**, *47,* 263–270. [2] S. S. Ray & M. Bousmina, *Progress in Materials Science* **2005**, *50,* 962–1079.

Keywords: Biodegradable polymers, Polimers Clay Nanocomposites

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MEM electron density study of $NaGaH_4$

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The search for hydrogen storage materials has received massive attention during the past decade in hope that hydrogen, in the future, may replace fossil fuels as energy carrier. Among the considered compound, it is worth mentioning $NaGaH_4$ that is formed of almost isolated GaH_4^- anions and spherical Na^+ moieties. It has about 4.2 wt% hydrogen, therefore not fulfilling the requirements for being a candidate hydrogen storage material for mobile applications. However, the compound reveals peculiar structural features that deserve further examination. In particular, a phase transition around 280 K has been pointed out by an anomaly in heat capacity measurements. [1] XRPD analysis likewise revealed a discontinuity in atomic displacement parameters when going from low to high temperatures. [2] V. P. Tarasov et al., on the basis of NMR data, implied that the phase transition can be attributed to changes in the orientation state of the distorted $Ga(H,D)_4^-$ anion. [3] Despite all the hints of a phase transition, structural knowledge is still lacking.

We studied $NaGaH_4$ in the temperature range 90 K – 390 K by synchrotron X-ray powder diffraction data collected at SPring8, Japan. Complementary synchrotron neutron powder data were collected at PSI, Switzerland, on the deuterated sample, $NaGaD_4$. For each of the considered temperatures, the Maximum Entropy Method (MEM) is utilised to maximise the information contained in the extracted structure factors and to determine the corresponding electron density. The MEM charge density at 90 K (fig. 1) is analysed within the quantum theory of atoms in molecules, [4], and compared to theoretical charge density obtained from periodic ab initio DFT calculations.

The Rietveld refinements and MEM densities of $NaGaH_4$ and $NaGaD_4$ do not show any apparent, structural indication of the expected phase transition. A possible explanation is provided by Raman scattering studies which imply a symmetry reduction with increasing temperature. [5] This is supported by structural NMR results which up to the phase transition demonstrate a temperature movement towards axial symmetry for the GaH_4 tetrahedron. [3] Therefore, starting from the *Cmcm* space group (63) of $NaGaH_4$, we explored its maximal non-isomorphic subgroups through Rietveld refinements of the 300 K neutron and X-ray data. Of the symmetry reduced space groups, $P2_1/m$ (11) is the only one capable of describing the structural NMR results.

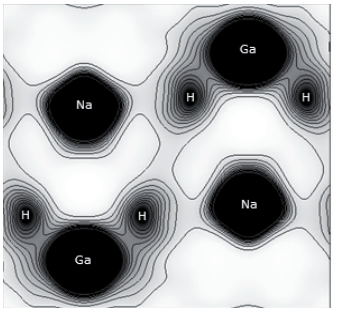

Figure 1 (200) contour plot of the MEM charge density of $NaGaH_4$ at 90 K. The density starts at 0.4 eÅ^{-3} and has contour level 0.1 eÅ^{-3}.

[1] V.E. Gorbunov et al., *Zh. Neorg. Khim*. **1982**, *27*, 1915-1920. [2] A.V. Irodova et al., *Zeitschrift Fur Physikalische Chemie Neue Folge* **1989**, *163*, 239-242. [3] V.P. Tarasov et al., *Russ J Phys Chem B* **2007**, *1*, 653 – 660. [4] R.F.W. Bader. *Atoms in Molecules: An Quantum Theory.* **1990**, Oxford University Press. [5] K.S Gavrichev, *Inorg Mater* **2003**, *39*, 89-112.

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Controlled annealing of nanocrystalline Y_2O_3

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Well crystallized cubic Y_2O_3 turns out to be an excellent material for calibration purposes in powder diffraction. In various respects it clearly supersedes the well established standard materials Silicon (SRM 640d) [1] or Lanthanum hexaboride (SRM 660b) [2]. On the other hand Y_2O_3 is a well known and commercially available nanomaterial. We have studied two different batches (30 – 50 nm and <50 nm nominal particle sizes) with respect to time- as well as temperature dependence of annealing process. Heat treatment was performed in platinum crucibles in air. Special care was taken to keep conditions for both samples most consistent. Powder diffraction patterns were taken in transmission mode using a Huber G670 Guinier camera applying Cu-$K_{\alpha 1}$ radiation. Modelling of peak shapes along with derived lattice parameters are critically evaluated. Clearly such evaluation needs to keep in mind that applicability of standard powder diffraction methods on nanocrystalline materials is controversially debated [3].

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[1] SRM 640d; *Silicon Powder Line Position and Line Shape Standard for Powder Diffraction*; National Institute of Standards and Technology; Gaithersburg, MD **2010**. [2] SRM 660b; *Lanthanum Hexaboride Powder Line Position and Line Shape Standard for Powder Diffraction*; National Institute of Standards and Technology; Gaithersburg, MD **2010**. [3] B. Palosz, E. Grzanka, St. Gierlotka, S. Stelmakh, *Z. Kristallogr.* **2010,** *225,* 588–598.

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Preparation and structural characterization of HFMOD-WO_3 thin films

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Tungsten oxide films have been successfully deposited by hot-filament metal oxide deposition (HFMOD) technique under atmospheric pressure and an oxygen atmosphere. Although several techniques were used to characterize the WO_3 layers, this work emphasizes the results