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X-ray diffractometer with ellipsoidal multilayer mirror for convergent optics

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Rigaku SmartLab is a multipurpose, fully-automated horizontal X-ray diffractometer that allows many types of applications. SmartLab's expandable system and the Cross Beam Optics (CBO) enable the realization of various optical configurations. The optics of the CBO system permits easy switching of incident X-rays by simply changing the selection slit. The Cross Beam Optics mechanism allows user selectable switching between two incident X-ray beam without resetting or realigning the optical system. Both system geometries are permanently mounted and permanently aligned to allow easy changeover for different applications.

In the Rigaku SmartLab two Cross Beam Optics systems are available: CBO and CBO-E. The CBO lets the user select the Bragg-Brentano focusing method or parallel beam method using the parabolic multilayer mirror, while the CBO-E lets the user select Bragg-Brentano focusing method or convergent beam optics using an ellipsoidal multilayer mirror. The convergent beam optics enables high resolution measurements by the transmission method. This method is used to measure samples with low absorption coefficients and preferred orientation, such as pharmaceuticals.

The Cross Beam Optics systems (CBO and CBO-E) can be combined with a Johansson type Ge crystal for monochromatization of the incident X-rays to the Kα1 (Kα1 system) [1]. One of the main applications of Kα1 X-rays is powder pattern indexing of unknown crystal structures. Since the diffracted X-rays are not obstructed in the Kα2, the separation of the overlapping Bragg reflections becomes easier in the Kα1 system. The Kα1 system is designed as the position of the focal point of the Johansson type crystal is corresponding to the conventional X-ray source. Due to the design of the Kα1 system it acts like a virtual X-ray source. Therefore, the CBO or CBO-E system can be used in the same way as the conventional Kα system. Consequently, the Kα system and the Kα1 system can be easily switched.

The diffracted X-rays can be focused on the detector plane by means of using Kα1 monochromatized X-rays together with convergent-beam optics (CBO-E). If we consider this optical system, high-speed and high-intensity measurement can be realized, if the high-speed one-dimensional position sensitive X-ray detector D/teX Ultra is used.

In this presentation, we report outline of the optics on the Rigaku SmartLab system and discuss high-resolution measurement results by using CBO-E with and without CuKα1 system.

[1] *The Rigaku Journal (English version),* **2010,** *26(2),* 29-30.

Keywords: X-Ray diffractometer, convergent-beam optics, powder diffraction

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Investigation structural of the new solid solution series: $Sr_{10-x}Nd_x(PO_4)_{6-x}(SiO_4)_xF_2$ ($0 \le x \le 3$) with rietveld method

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Apatitic phosphosilicates, generally called "britholites" [1] have been the object of a great interest because of their potential use for the conditioning of nuclear wastes [2]. They crystallize mainly in the hexagonal system (space group $P6_3/m$).

In this work, we present study structural of the new solid solution series of $Sr_{10-x}Nd_x(PO_4)_{6-x}(SiO_4)_xF_2$ ($0 \le x \le 3$). The structure has been determined at room temperature from X-ray diffraction by the Rietveld method analysis. A variation of the *a* and *c* lattice parameters in the solid solutions was observed, with a decrease of a and c parameters, related to the radius of the corresponding substituted ions. The structural refinement using the Rietveld method [3(a, b)] indicated that Nd^{3+} ions were located into the two sites with a strong preference for M(2) sites especially for low contents. A progressive shift of the F^- position along the *c*-axis outside the centre of the triangle formed by M(2) atoms was observed with the increase of *x*. Fourier maps were performed. These maps of electron densities expressed the existing possibility of each atom composing britholites. In particular, columnar neodymium and screw-axis neodymium were clearly revealed.

[1] J. Felsche, *J. Solid State Chem.* **1972**, *5*, 266. [2] E. Du Fou de Kerdaniel and al., *J. Nucl. Mater.* **2007**, *362*, 451-458. [3] a- H.M. Rietveld, *Acta Cryst.* **1967**, *22*, 151. b- H.M. Rietveld, *J. Appl. Crystallogr.***1969**, *2*, 65.

Keywords: britholites, rietveld method, sites

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Biodegradable polymer-clay nanocomposites

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The use of conventional plastics in the last years has been broadened to new areas. Due to their low degradation rates, they can cause ecological problems when disposed as solid waste. Biodegradable polymers (BP) offer an alternative to keep sustainable development economically and ecologically attractive. Polyhydroxybutyrate (PHB) is an example of a BP, obtained from renewable resources. It has been found that polymers with nanostructurated natural minerals enclosures lead to nanocomposites (NC) with highly improved stiffness, thermal stability, decreased flammability and gas-barrier properties at low loading (<5vol%). Addition of 5% of a functionalized commercial clay (30B-cloisite, 30BC, Southern Clay), by melt intercalation has yield a PHB NC with improved tensile modulus and better fire properties [1]. In spite of its many possible applications, its high melting point, brittleness, and low toughness, make their use difficult. Polycaprolactone (PCL), a synthetic BP, has a low melting point and is ductile at room temperature. The physical mixture of these polyesters is an alternative to obtain new materials with desirable properties. Blends of PCL and PHB are inmiscible. Here we present the characteristics of NC produced by addition of 30BC to mixtures of PHB with PCL in different ratio. Samples were prepared in an intermeshing twin screw extruder. Films were obtained in a single screw extruder with flat die and take off unit. NCs were investigated by X ray diffraction (XRD) and differential scanning calorimetry (DSC). The results indicate that some degree of polymers insertion between the clay layers has been achieved. According to the literature [2] simultaneous small angle X-ray scattering (SAXS) and XRD studies yielded quantitative characterization of nanostructure and crystallite structure in some NC. Data from synthesized materials present the (001) diffraction peaks of the phyllosilicate almost at the same position as in the neat clay, along

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.

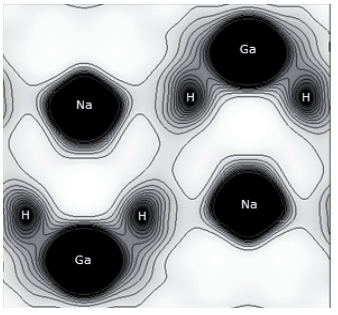

Figure 1 (200) contour plot of the MEM charge density of $NaGaH_4$ at 90 K. The density starts at 0.4 eÅ^{-3} and has contour level 0.1 eÅ^{-3}.

[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