

actinide-bearing pyrophosphates and found that all of them exhibit this unusual high temperature behaviour above 673K. Besides we also found that their room temperature structure is not cubic as previously reported [6] but triclinic (distorted cubic) and transform into a cubic form around 150°C. The phase transition has been characterized by various experimental techniques, X-ray diffraction, solid-state NMR and Raman spectroscopy. The room temperature structure has been determined by from synchrotron radiation data obtained at the ESRF. Our presentation will report the experimental results and discussed the structural aspect related with the contraction of the materials at high temperatures.

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Keywords: actinides, phosphates

MS05.P26

Acta Cryst. (2011) **A67**, C248

Crystal Structure and phase transitions of mechanoactivated manganese oxides

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One of the features of the Mn-O system is the formation therein of two Mn₂O₄ spinel-type polymorphs: γ -Mn₂O₄ with a spinel structure (sp.gr. *Fd3m*) and β -Mn₂O₄ with a hausmannite structure (sp.gr. *I4/amd*) often considered as a tetragonally distorted spinel with lattice parameters $a^* = \sqrt{2} a, < c (c/a^* > 1)$. The tetragonal distortion of the spinel lattice is due to the cooperative Jahn-Teller effect, and is caused by Mn(III) ions on octahedral sites. Transformation between the two structures is observed both under equilibrium non-ambient conditions (at 1172°C in air) and on quenching the oxide from a high temperature state to room temperature where only a hausmannite structure exists. In this work effects of mechanical activation on the crystal structure and phase transitions of the treated manganese oxides were studied by high-temperature X-ray diffraction.

Highly activated manganese oxides (MnO₂, Mn₂O₃, Mn₃O₄, and MnO) were prepared by high-energy ball milling ($g > 60$). The as-prepared mechanically activated manganese oxides were characterized as single-phased, nanoscaled or ultra-fine-grained compounds.

Ball milling gave rise to a direct phase transition from α -Mn₂O₃ to γ -Mn₂O₄ omitting β -Mn₂O₄ phase during heating. This result was obtained both for the as-treated nanoscaled α -Mn₂O₃ oxide and for the coarse-grained powder after multiple thermal recycling. The same effects were observed upon thermal study of the other activated manganese oxides. A significant increasing in the temperature of the phase transition from Mn₂O₃ to Mn₃O₄ during heating and an easier reversibility of the Mn₃O₄ to Mn₂O₃ phase transition upon cooling were also detected. It was proposed structural defects due to the mechanical activation are responsible for the anomaly in phase transition behaviour of mechanoactivated manganese oxides. Therefore the structure and microstructure of the phases involved were refined depending on treatment conditions and temperature.

The work is supported by Ministry of Education and Science of the Russian Federation (02.740.11.0641) and RFBR (10-03-96016).

Keywords: oxide, temperature, transition

MS05.P27

Acta Cryst. (2011) **A67**, C248

Hybrid instrument for in situ studies at the ESS

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The decision to build the European Spallation Source (ESS) came in the spring of 2009. The ESS will be built in Lund, Sweden and become the most powerful neutron source in the world. The ESS will give unprecedented possibilities for neutron scattering in crystallography and materials science. Investigations of materials at multiple length scales are getting increasingly important for understanding and improving materials performance such as hydrogen storage, solid oxide fuel cells and lithium ion batteries.

A new concept for a neutron scattering instrument will be presented. The new idea combines powder neutron diffraction (PND), small angle neutron scattering (SANS), and neutron imaging (NI) in the same instrument. The PND potentially allows extraction of pair distribution functions for looking at amorphous systems. By combining these techniques it will be possible to look at length scales 0.01-5 nm using diffraction, 1-1000 nm using SANS, and 0.01-100 mm using NI. In addition to the different techniques a high time resolution is envisioned to study processes at the subsecond level. In this way it is possible to study materials *in situ* or *in operando*.

Through the presentation it is intended to start discussions with the potential user community of the suggested hybrid instrument. The ESS is soon to determine the instrument suite which will be built in the first round. Since this instrument presents new concept there is no existing user community. The IUCr is therefore a great opportunity for engaging with the potential users.

Keywords: neutron scattering, materials, in situ

MS05.P28

Acta Cryst. (2011) **A67**, C248-C249

Negative thermal expansion in trioxane silica sodalite (TRSS)

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The thermal dehydration and template burning process of a silica sodalite (SS), synthesized in the presence of boric acid and trioxane (TR), Na_{0.15}[B_{0.07}Si_{11.93}O₂₄]-1.8TR, were studied *in situ* by synchrotron powder diffraction up to 900°C. Time-resolved diffraction data were collected at the GILDA beamline at ESRF (Grenoble). Thermal analyses (TG, DTA and DTG) of the as-synthesized samples were carried out in the same temperature range under a constant flux of air (heating rate 5°C/min).

The cell volume of TRSS does not change remarkably until the expulsion of the template, which occurs above 380°C, as inferred by TG analysis weight loss and by the structure refinement of trioxane molecule occupancy. When trioxane molecule decomposition begins, volume expansion becomes negative. This result is unusual for sodalite type materials which typically show positive thermal expansion [1-3]. However, in all these studies the extraframework content is given by cations or anions (such as Cl⁻, F⁻) which cannot be expelled during heat treatment. In our case, extraframework content is represented by organic molecules which are expelled upon heating. A similar result was observed only in a silica sodalite synthesized in the presence of ethylene glycol [4]. These results suggest that when the organic molecules are neutral complex and only act as spacer, a contraction of