



Upon complex thermolysis in hydrogen the nanoalloy $\text{Pd}_{0.5}\text{Rh}_{0.5}$ is formed ($Fm\text{-}3m$ space group, $a = 3.847 \text{ \AA}$). The thermolysis final temperature is $400 \text{ }^\circ\text{C}$, the average particle size is 20 nm .

The work has been supported by RFBR Grant 08-03-00603, Presidium SB RAS interdisciplinary project No 112 and State contract № P960 of Federal target program «Scientific, Research And Teaching Specialists In Russia» 2009–2013.

[1] Sheldrick, G.M., *Acta Crystallogr.* 2008, A64, 112

Keywords: crystal structure analysis, X-ray phase determination, thermal decomposition.

FA2-MS16-P53

High resolution X-ray diffraction experiments for selected minerals. Marcin Stachowicz^a, Maura Malinska^a, Jan Parafiniuk^b Krzysztof Woźniak^a.

^aFaculty of Chemistry, University of Warsaw. ^bFaculty of Geology, University of Warsaw.

E-mail: marcin.stachowicz1@gmail.com

Jarosite is used as a by-product of the metal-processing industry as well as a common supergene mineral in ore deposits, and is associated with acid-mine waste. Jarosite is thought to exist on Mars, and its presence suggests that water existed on Mars in the past [1]. Celestine is the most abundant strontium mineral and is the principal commercial source of strontium [2].

The high resolution X-ray diffraction experiments have been carried out for series of natural minerals. Natrojarosite $\text{NaFe}_3(\text{SO}_4)_2(\text{OH})_6$ had been mined in Greece and celestine SrSO_4 in Poland. These compounds crystallize in the trigonal R-3m and orthorhombic Pnma space groups, respectively.

The multipole model within the Hansen-Coppens formalism [3] is applied for crystals containing disorder and heavy atoms. In American Mineralogist Crystal Structure Database are four structures of Celestine [2,4-6] all ordered, however fitting of the multipole models of electron density leads to identification of small degree of disorder. Several multipole model refinement strategies have been applied and compared to find the most suitable one. Topological analysis of electron densities estimated from multipole models gives opportunity for better understanding intermolecular interactions and bonding properties in these minerals.

[1] Basciano L.C., Peterson R.C., *American Mineralogist*, 2008, 93, 853. [2] Jacobsen S.D., Smyth J.R., Swope R. J., Downs R.T., *The Canadian Mineralogist*, 1998, 36, 1053. [3] Hansen N.K., Coppens P., *Acta Cryst.*, 1978, A34, 909. [4] Miyake M., Minato I., Morikawa H., Iwai S.I., *American Mineralogist*, 1978, 63, 506. [5] Hawthorne F.C., Ferguson R.B., *The Canadian Mineralogist*, 1975, 13, 181. [6] Garske D., Peacor D.R., *Zeitschrift für Kristallographie*, 1965, 121,204

Keywords: X-ray crystallography of minerals, charge density inorganic materials

FA2-MS16-P54

Structural investigations of $\text{Li}_3\text{Ti}(\text{MoO}_4)_3$. A. Thomas^a, D. Mikhailova^{a,b}, H. Ehrenberg^a

^aInstitute for Complex Materials, IFW Dresden, Germany, ^bInstitut für Materialwissenschaft, Technische Universität Darmstadt, Germany.

Email: a.thomas@ifw-dresden.de

Complex molybdates with 3d transition metals crystallizing in a NASICON-type structure are promising materials for Li-storage during Li-insertion and deinsertion. Recently, a new molybdate with V(III), $\text{Li}_3\text{V}(\text{MoO}_4)_3$, was prepared and characterized. It was shown that Li-ions can be reversibly intercalated and deinserted with formation of $\text{Li}_2\text{V}(\text{MoO}_4)_3$ and $\text{Li}_4\text{V}(\text{MoO}_4)_3$ [1]. In the related system Li-Ti-Mo-O, only the composition $\text{Li}_3\text{Ti}_{0.75}(\text{MoO}_4)_3$ with Ti(IV) and the same crystal structure type is known [2]. There are no examples in the literature for the coexistence of molybdenum(VI) and titanium(III) in one phase. Every stable product which contains titanium and molybdenum was synthesized from titanium(IV) and molybdenum(IV/VI) compositions, for example $(\text{Ti}_x\text{Mo}_{1-x})_5\text{O}_{14}$ [3] and TiMo_2O_8 [4]. If titanium (III) and molybdenum(IV) were used as educts, molybdenum(IV) was reduced to metal and titanium(III) was oxidized to titanium(IV) [5].

A new phase with the composition $\text{Li}_3\text{Ti}(\text{MoO}_4)_3$ was synthesized by solid state reaction. It crystallizes in the space group *Pnma* and is isostructural to $\text{Li}_3\text{V}(\text{MoO}_4)_3$. Similar electrochemical properties as for $\text{Li}_3\text{V}(\text{MoO}_4)_3$ are expected [1]. The material shows the same plateau at ca. 1.8 V during galvanostatic cycling against a Li-anode, which indicates the same two-phase mechanism during first Li-insertion. The similarity of Li^+ and Ti^{3+} radii for octahedral oxygen coordination leads to mixed cation occupancy in the structure, which can be varied by different synthesis conditions. A difference in the lattice parameters for two samples, prepared at different temperatures, between $a = 5.05876(8) \text{ \AA}$, $b = 10.4833(1) \text{ \AA}$ and $c = 17.5653(2) \text{ \AA}$ to $a = 5.0715(1) \text{ \AA}$, $b = 10.5096(2) \text{ \AA}$ and $c = 17.6050(3) \text{ \AA}$ confirms this assumption. In comparison the lattice parameters of $\text{Li}_3\text{Ti}_{0.75}(\text{MoO}_4)_3$ are: $a = 5.0467(12)$, $b = 10.454(3)$ and $c = 17.538(4)$ [2].

[1] Mikhailova, D.; Sarapulova, A.; Voss, A.; Thomas, A.; Oswald, S.; Gruner, W.; Trots, D. M.; Bramnik, N. N.; Ehrenberg, H.; *Chem. Mat.* (in press). [2] Smit, J. P.; McDonald, T. M.; Poeppelmeier, K. R.; *Solid State Sci.* 10 (2008) 396. [3] Ekström, T.; *Acta Chem. Scand.*; 26 (1972) 1843. [4] Krishnamurty, R.; Chincholkar, V. S.; *Current Sci.* [India] 41 (1972) 36. [5] Kirshenbaum, A. D.; Beardell, A. J.; *Thermochimica Acta*, 4 (1972) 239.

Keywords: NASICON-type structure, Li-insertion, cation mixed occupancy

FA2-MS16-P55

Synthesis and structural studies of ammonium-cobalt-nickel phosphates, $\text{NH}_4[\text{Co}_{1-x}\text{Ni}_x\text{PO}_4]\cdot\text{H}_2\text{O}$.

Laura Torre-Fernández, Camino Trobajo, José R. García, Santiago García-Granda. *Departamentos de Química Física y Analítica y Química Orgánica e Inorgánica, Universidad de Oviedo, Spain.*

E-mail: torrelaura@uniovi.es