

**FA5-MS06-P07**

**Pressure Induced Phase Transformations in the Partially Filled Clathrate  $\text{Rb}_{6.15}\text{Si}_{46}$ .** Denis Machon<sup>c</sup>, Pierre Toulemonde<sup>b</sup>, Paul F McMillan<sup>e</sup>, Mónica Amboage-Castro<sup>a</sup>, Alfonso Muñoz<sup>d</sup>, Plácida Rodríguez-Fernández<sup>d</sup>, Alfonso San-Miguel<sup>c</sup>.

<sup>a</sup>European Synchrotron Radiation Facility, Grenoble, France. <sup>b</sup>Institut Néel, CNRS and Université Joseph Fourier, Grenoble, France. <sup>c</sup>LPMCN, Université Lyon-I and CNRS, France. <sup>d</sup>Universidad de La Laguna, La Laguna, Tenerife, Spain. <sup>e</sup>University College London, London, UK.

E-mail: [sanmiguel@lpmcn.univ-lyon1.fr](mailto:sanmiguel@lpmcn.univ-lyon1.fr)

Group 14 clathrates constitute prototype systems for the study of host-guest interactions at the nanoscale [1]. Type-I clathrates are made of the crystalline assemblage of silicon nanocages of 24 and 20 atoms having the possibility for the endohedral inclusion of guest atoms. The high pressure evolution of group 14 clathrates include a number of intriguing phase transitions including an homothetic isostructural volume collapse or a pressure induced amorphisation [2]. One method to better understand the importance of host-guest interaction in their pressure evolution is to consider the study at partial nanocage filling. The type I clathrate  $\text{Rb}_{6.15}\text{Si}_{46}$  with partly empty cages has been studied up to 36 GPa using Raman spectroscopy, synchrotron x-ray diffraction in diamond anvil cells, and ab initio total-energy and lattice-dynamics calculations. A first phase transition is observed at  $13\pm 1$  GPa, and a “volume collapse” transition within the clathrate structure is then observed at  $24\pm 1$  GPa. Pressure-induced amorphisation into a high-density amorphous (HDA) state occurs above  $P = 33\pm 1$  GPa. The HDA form transforms into a low-density amorphous polymorph (LDA) during decompression. During the compression study using angle dispersive synchrotron x-ray diffraction techniques we measured bulk modulus parameters for rocksalt-structured TaO for the first time, included adventitiously in the clathrate sample ( $K_0 = 293(3)$  GPa and  $K_0' = 5.4(3)$ ).

[1] A. San Miguel, *Chemical Society Reviews*, 35, 876-889, 2006.

[2] A. San Miguel and P. Toulemonde, *High Pressure Research*, 25, 159, 2005.

**Keywords:** high pressure; clathrates

**FA5-MS06-P08**

**Microstructure of Yb Substituted  $\text{Y}_2\text{O}_3$  from HRTEM and X-ray Diffraction Approaches.** Bratislav Antic<sup>a</sup>, Aleksandar Kremenovic<sup>a</sup>, Milica Vucinic-Vasic<sup>a</sup>, Aleksandar S. Nikolic<sup>b</sup>. <sup>a</sup>The “Vinca” Institute, Belgrade, Serbia. <sup>b</sup>Faculty of Chemistry, University of Belgrade, Belgrade, Serbia.

E-mail: [bantic@vinca.rs](mailto:bantic@vinca.rs)

The mixed sesquioxides,  $\text{Y}_{2-x}\text{Yb}_x\text{O}_3$  ( $x=0.06, 0.10, 0.20, 0.40$ ) with particles of 5 nm were studied by HRTEM and X-ray diffraction technique approaches. Rietveld refinement

of occupation numbers shows a preferential  $\text{Yb}^{3+}$  toward to  $C_3$  sites in the Ia3 space group for concentration  $x\leq 0.20$ , and become random for  $x=0.40$ . From optical point of view, site distribution of active centers in host matrices is important. The sample with  $x=0.20$ , where a random distribution of  $\text{Yb}^{3+}$  was found, is most suitable sample for optical applications. Electron transmission microscopy is used in order to obtain information about sizes, size distribution, defects and morphology of nanoparticles. Distribution of the particles is narrow and most of particles are approximately 5 nm in diameter. The particles are homogeneous, without visible shell in particle or amorphous phase in sample. At nanoscale, particles can be described by core/shell model. The particles are probably without shell, or with very tiny shell which are not detected by the used experimental techniques. Crystallite size distribution obtained from Breadth computer program is compared from one determined by HRTEM. Although, both of them show the similar values for the large number of particles/crystallites, there is a discrepancy between two distribution for small particles/crystallite values. The disagreement is based on the basic principles of methods and mathematical models that were used. Crystallite size determined by X-ray diffraction technique is close to particle size determined from HRTEM, indicating that particles are composed from one crystallite in average. X-ray line broadening analysis show negligible anisotropy of X-ray line broadening provoked by the strain and size effects.

In conclusion, we develop a method for production of rare earth oxides based nanophosphors using thermal decomposition of acetylacetonato (AcAC) complexes and it was applied here for formation of monodispers nanoparticles. Crystallographic study show that the method could be used in commercial production of rare earth based nanophosphors.

**Keywords:** microstructures; nanoparticles; oxides

**FA5-MS06-P09**

**Effect of Sign Reversal by Misorientation Vector along Block Boundaries and its Detection.**

Vyacheslav Malkov<sup>a</sup>, Victor Strekalovsky<sup>a</sup>, Andrey Malkov<sup>b</sup>, Oleg Malkov<sup>b</sup>, Vladimir Puchin<sup>c</sup>. <sup>a</sup>Institute of High-Temperature Electrochemistry, Ural Branch RAS. <sup>b</sup>“ROSNA” Scientific and Production Center. <sup>c</sup>Institute of Metal Physics, Ural Branch RAS, Ekaterinburg, Russia.

E-mail: [mvb@ihte.uran.ru](mailto:mvb@ihte.uran.ru)

Studies performed in the field of nanotechnologies necessitate exploration and testing of nanomaterials. Similarly to microscale materials, nanomaterials can be in the elastodeformed state [1]. Such nanomaterials include flat nanothin diamond-shaped crystals of hexagonal selenium growing in amorphous films at crystallization temperatures of  $180\div 160^\circ\text{C}$ . The lattice of these crystals has a nonuniform rotational distortion about the C-axis whose direction coincides with the short diagonal of the diamond-shaped crystal. Relaxation of the elastodeformed state in some area of the diamond-shaped nanothin crystal results

in formation of a twist block boundary.

Twist block boundaries in nanothin selenium crystals were studied by methods of transmission electron microscopy. Extinction bend contours along twist block boundaries were indexed using bright- and dark-field microdiffractions. Indexing of the bend contours was followed by examination of the alternation order of contours in crystal blocks along the boundaries. It was found that twist block boundaries of two types – with the invariable alternation order of bend contours and with a different alternation order of bend contours – were formed in nanothin selenium crystals (Fig. 1).

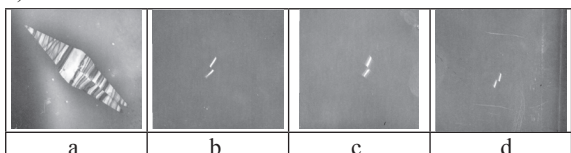


Fig. 1. An electron microscopic image of a nanothin selenium crystal with a block boundary, along which the alternation order of bend contours and, hence, the sign of the misorientation vector  $\omega$  changes: bright-field image (a) and dark-field image in the reflection (201) (b), (202) (c) and (203) (d).  $\times 5000$ .

The analysis of the mutual alternation of bend contours along twist block boundaries in crystal blocks demonstrated that the mutual alternation order of contours changed due to the effect of the sign reversal by the misorientation vector  $\omega$ . A model describing the formation of twist block boundaries in nanothin selenium crystals is proposed for explanation of the effect of the sign reversal by  $\omega$ . This model takes into account that relaxation of a nonuniform elastic torsional distortion of the crystal lattice about [001] takes place in both blocks of a nanothin crystal.

[1] Bolotov I. E., Prilepo V. L. *Phys. Stat. Sol (a)*, **1983**, V. 67, № 1, p. 67-70.

**Keywords: selenium; crystals; boundaries**

#### FA5-MS06-P10

**Synthesize of Sillenite  $\text{Bi}_{24}\text{AlO}_{39}$ ;  $\text{As}^{3+}$  Nanocrystal by Co-precipitation Method.** Abdolali Alemi<sup>a</sup>, Aliakbar Khandar<sup>a</sup>, Samira Hajibabae<sup>a</sup>. <sup>a</sup>*Department of Inorganic Chemistry, Tabriz University, Tabriz, Iran.*

E-mail: [abdolalialemi@yahoo.com](mailto:abdolalialemi@yahoo.com)

Sillenite single crystals have a nominal chemical formula  $\text{Bi}_2\text{MO}_2\text{O}_{20}$ , where (M=Ge, Si, Ti, Al, B...). [1] Sillenite compounds display a variety of advantageous physical properties that make them very important technological materials. The current applications of sillenites are mainly in the fields of electro optics, acoustics, and piezoelectrics, where their physical properties such as photo refractivity, optical activity, photoconductivity, piezo-modulus, the velocity of ultrasound wave propagation, etc., can be exploited. Recently, however, sillenites have begun to be considered for use as dielectrics in the field of electronics. [2] The overall structure of the  $\text{Bi}_{12}\text{MO}_{20}$  crystal may be described in terms of the 7-oxygen Coordinated Bi

polyhedra and with  $\text{MO}_4$  tetrahedral. This body centered cubic structure of sillenite crystals facilitate the solubility of novel metal ions in the crystal, because the tetrahedron of oxygen atoms surrounding the M atoms is able to expand or contract without a major effect on the remaining atomic arrangement. [3]. In this paper the sillenite crystal of  $\text{Bi}_{24}\text{AlO}_{39}$  were doped with  $\text{As}^{3+}$  prepared by a co-precipitation method. In a typical process, Bismuth nitrate  $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$  was initially dissolved in  $\text{HNO}_3$  solution. Aluminum nitrate  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  was dissolved in a mixture solution of  $\text{HNO}_3$  and anhydrous ethanol. And Arsenic oxide  $\text{As}_2\text{O}_3$  was dissolved in  $\text{HNO}_3$  solution.  $\text{Bi}(\text{NO}_3)_3$  solution and  $\text{Al}(\text{NO}_3)_3$  solution and  $\text{As}_2\text{O}_3$  solution were mixed under stirring for 30 min. The pH value of the above solution was adjusted to ca 8.5-9.5. The resulting white precipitate was washed with twice distilled water for several times, and then dried, smashed and calcined at  $600^\circ\text{C}$ .

The prepared powders were examined by the XRD, FTIR and scanning electronic microscope (SEM).

[1] Suzuki E., Iyi E., Kitamura K., *Korean. Physical. Society*, **1998**, 32, S173. [2] Valant M., Suvorov D., *Chem. Mater*, **2002**, 14, 3471. [3] Yao W.F., Xu X.H., Zhou X.N., Zhang y., Shang S.X., *Molecular. Catalysis. A*, **2004**, 212, 323.

**Keywords: sillenite;  $\text{Bi}_{24}\text{AlO}_{39}$ ; co-precipitation**

#### FA5-MS06-P11

**Hydrothermal Growth and Characterization of Boehmite ( $\gamma\text{-AlOOH}$ ) Nanoparticles.** Aliakbar Khandar<sup>a</sup>, Abdolali Alemi<sup>a</sup>, Zahra Hoseinpour<sup>a</sup>. <sup>a</sup>*Department of Inorganic Chemistry, Tabriz University, Tabriz, Iran.*

E-mail: [abdolalialemi@yahoo.com](mailto:abdolalialemi@yahoo.com)

The synthesis of one-dimensional (1D) nanoscale inorganic nanoscale materials with special properties have long been attracting great attention in material science in the past few decades due to their distinctive geometries, novel physical and chemical properties and potential application in numerous areas[1]. Material properties are mainly determined by their composition, structure, crystallinity, size and morphology [2], [3]. Boehmite nanoparticles ( $\gamma\text{-AlOOH}$ ) with the orthorhombic phase, Cmc space group and cell constants of about  $a=3.7^\circ\text{A}$ ,  $b=12.29^\circ\text{A}$ ,  $c=2.84^\circ\text{A}$  have been successfully synthesized by a hydrothermal method at  $180^\circ\text{C}$  and 24h using Aluminum nitrate  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  as the aluminum source and sodium metaborate  $\text{NaBO}_2 \cdot 4\text{H}_2\text{O}$  as the controlling agent. The size and morphology of boehmite nanoparticle could be controlled by adjusting the pH value of the reaction mixture. The size of boehmite can be varied in the range of 50 and 120nm. The resulting products were characterized by X-ray diffraction (XRD), fourier transform infrared spectra (FTIR), Scanning electron microscopy (SEM) and Uv-vis spectra.

The present nanoparticles with distinctive size and shape features might open up promising application in many fields.