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Preparation and characterization of Sb_2S_3 nanorods and nano particles via hydrothermal condition.

Abdolali Alemi, Younes Hanifehpour Firouzsalari, *Department of Inorganic Chemistry, Faculty of Chemistry, University of Tabriz, Iran*
E-mail: abdoalialemi@yahoo.com

Single-crystalline antimony trisulfide (Sb_2S_3) nano materials with nano particle and rod-like morphologies were successfully synthesized via hydrothermal method by the reaction of antimony trichloride ($SbCl_3$) and carbon disulfide with high yield in 24h at 180 °C .The powder X-ray diffraction pattern shows the Sb_2S_3 crystals belong to the orthorhombic phase with calculated lattice parameters a=1.120nm, b=1.128nm and c=0.383nm.The quantification of energy dispersive X-ray spectrometry analysis peaks give an atomic ratio of 2:3 for Sb:S. Scanning electron microscopy (SEM) images show that diameter of Sb_2S_3 nano particles is around 80-150nm, and rod- like Sb_2S_3 possess a diameter around 70-140nm and length up to 3µm, respectively. X-ray powder diffraction, scanning electron microscopy, atomic force microscopy, optical measurements, UV-Vis analyses were used to characterize the products. UV-Vis analysis and emission spectra indicates that band gap of Sb_2S_3 is around 2.82ev, indicating a considerable blue shift relative to the bulk. The effects of reaction time and reaction temperature on the growth of nano materials with different morphologies were also investigated.

Keywords: antimony sulfide, nanorods, nano particles , Hydrothermal

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Synthesis of nanocrystalline intermediate phase between cancrinite and sodalite.

Corinna Grader[a], Lars Robben[b], Josef-Christian Buhl[c], [a,b,c]*Institut für Mineralogie, Universität Hannover, Callinstr.3, D-30167Hannover, Germany*
E-mail: grader@mineralogie.uni-hannover.de

Syntheses in the system Na_2O-SiO_2-Al_2O_3-Na_2CO_3-H_2O were carried out under various experimental conditions with the aim to synthesize nanocrystalline cancrinite, a mineral with zeolite-like behaviour.
After synthesis of a cancrinite reference from the educt zeolithe A and well-known high-temperature conditions at 200°C, an alumosilicat-gel was used to produce amorphous aluminosilicate precursor phases for crystal growth at 60°C. This low-temperature hydrothermal synthesis resulted in the formation of an intermediate phase between sodalite and cancrinite with one dimensional stacking disorder of alumosilicate layers along the [001] direction, first described in [1]. A second series of syntheses with the addition of the Al^{3+}-complexing additive triethanolamine (TEA) was performed to investigate the effect on crystal growth and structure-upgrade at 60°C [2]. Additionally the hydrothermal stability of the nanocrystalline intermediate phase was tested. Therefore the material was treated in water at 80°C for times up to 24h.
The products were analysed by FTIR-spectroscopy, X-ray powder diffraction and scanning electron microscopy.
Temperature dependent FTIR-spectoscopy as well as heating experiments in a muffle furnace up to 600°C and thermogravimetry (TGA) with differential-thermoanalysis (DTA) up to 1400°C supplied further informations.
The nanocrystalline intermediate phase shows interesting zeolitic behaviour. Properties like water content and thermal stability are higher than in pure-phase carbonate cancrinite, whereas the hydrothermal stability of the intermediate phase is less than those of the cancrinite-phase.
Furthermore the crystal growth under addition of TEA yield to formation of aggregates of nanocrystalline material of intermediate phase [3]. Beside the time dependent deceleration of nucleation of aluminosilicate by TEA a recrystallisation of big Na_2CO_3 crystals occured. Later nanoparticle formation of the intermediate phase by heteregenous nucleation on the surface of the Na_2CO_3 crystals is responsible for the aggregate formation (Fig.1).

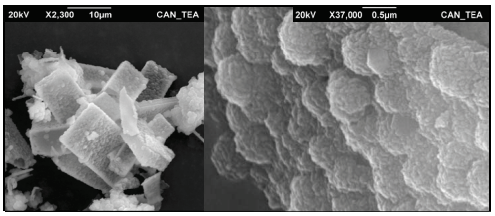

Fig. 1: Nanoparticles on Na_2CO_3-crystals, left: magnification 2300x, right: 37000x.

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Keywords: nanocrystallites, intermediate zeolite, hydrothermal stability

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Hydrogen Thin structure of nano-dispersed powders of rare earth oxides and fluorides produced from amorphous precursors.

Ivan Shmytko, Galina Strukova, Elena Kudrenko, *Institute of Solid State Physics, Chernogolovka, Moscow distr., 142432, Russia.*
E-mail: shim@issp.ac.ru

The nano-dispersed rare earth simple oxides Lu_2O_3, Gd_2O_3, Y_2O_3, La_2O_3 and Eu_2O_3, garnets $Y_3Al_5O_{12}$ and $Y_3Ga_5O_{12}$, perovskites $YAlO_3$ and $LaAlO_3$, borates $LuBO_3$, $GdBO_3$ $(Lu_xGd_{(1-x)})BO_3$ and YBO_3, molibdate $Eu_2(MoO_4)_3$ and fluorides LuF_3 and $Na_5Lu_9F_{32}$ were produced by different methods.
X-ray investigations have shown that independently of synthesis method the first stages of crystallisation of such compounds are characterized by new three phenomena.
First phenomenon is the formation of very unusual two-phase state of simple rare earth oxides Eu_2O_3, Lu_2O_3, La_2O_3, Gd_2O_3, garnet $Y_3Ga_5O_{12}$ and $Na_5Lu_9F_{32}$ at early stages of the nano-crystallites formation. These phases are isomorphous and have different lattice parameters. Detailed x-ray investigations have shown that both phases are realized in the same crystallite. This two-phase state transforms then into one-phase state in process of growth of the dimensions of the crystallites. It permits us to conclude that along the early stages of nano-crystallization the nano-grains consist of surface and core phases. The surface phase has enlarged lattice parameters with