

crystals also form in boric-fluoride solutions; they show hexagonal and trigonal prisms. As a rule, analogous (-) ends of crystals are broken or monohedron. Crystals formed in boric acid solutions show higher number of faces. The trigonal pyramid {02-21}, the trigonal prism {10-10} and the trigonal pyramid {02-21} are formed in boric acid solutions. Habit of crystals is from short to long prismatic. Unlike natural tourmalines the crystals synthesized in boric-bearing hydrothermal solutions show fewer numbers of faces, which exclusively develop the following main habit forms: trigonal {10-10} and hexagonal {11-20} prism and trigonal pyramids {10-11}, {02-21} and {02-2-1}.

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[1] T. Setkova, Y. Shapovalov, V. Balitsky, *J. Crystal Growth*, **2011**, 318, 904-907.

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Synthesis of ¹³C - diamonds in CaCO₃-C system

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Isotope-pure ¹³C-diamond was synthesized in melts of the Ca¹³CO₃ - ¹³C system at 8.5 GPa, 2000 K (in anvil-with-hole apparatus) and 20 GPa, 2500 K (in multi-anvil press). Starting carbonate and graphite (metastable phase) materials were based on 100%-rich ¹³C-carbon isotope. A "solution-melt" mechanism of ¹³C-diamond crystallization was realized. The mechanism is characterized by formation of oversaturated in respect to diamond labile carbon solution in carbonate melt (under experimental PT conditions, melting of Ca-carbonate is congruent). Diamond synthesis with the use of mainly ¹²C -based starting Ca-carbonate and graphite at 8 – 9 GPa, 2100 – 2300 K has been reported previously [1]. Colorless, transparent diamond single crystals of octahedral habit and 5-30 micron size have been grown and are shown in the fig. 1 together with the quenched Ca-carbonate melt. Raman spectra of the synthesized diamond (fig. 2) contain a narrow intensive peak 1280 cm⁻¹, which is characteristic for ¹³C-diamond based on 100%-rich ¹³C-carbon isotope.

The experimental results are in agreement with phase diagram of CaCO₃. A stable phase region of congruent Ca-carbonate melts within 2300 and 3500 K is determined at 20 GPa by the newest experimental studies [2]. This is of specific interest for the problem of "super-deep" diamond genesis in carbonate-rich growth media of the transition zone and lower mantle of the Earth.

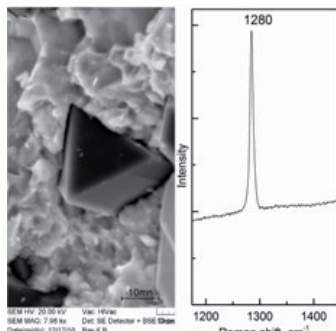


fig.1

fig.2

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[1] A.A. Shul'zhenko, *Get'man. German Patent* **1971**, 04, 2032083 [2] A.V. Spivak, L.S. Dubrovinsky, Yu Litvin. A. Doklady, *Earth Sciences*, **2011** (in press).

Keywords: carbonate-synthetic ¹³C-diamond, carbon, isotope

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Growth and morphology of eulytite crystals synthesized in hydrothermal solutions

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Eulytite (Bi₄(SiO₄)₃) is one of the rarest minerals in the nature. It forms as single crystals, aggregate of crystals and radial fiber spherulites. The crystals usually have tetrahedral habitus. Faces of crystal (2-11) and (211) are most developed. Less developed faces are (100), (110), and (111) and rarely developed faces are (511). [1]

Crystals of bismuth orthosilicate (eulytite) are using for scintillator in the high-energy physics, computer tomography, and dosimetry. Eulytite is one of the most promising materials for these purposes, but the problem of production still remains unsolved due to crystals growing from high viscosity melt.

Previously eulytite was synthesized in NaOH solution [2], but subsequent researches in this field did not carry out then.

We have performed hydrothermal synthesis of the fine crystalline bismuth orthosilicate (eulytite) at temperature 250 - 260°C and pressure 500 bars in solutions of sodium hydroxide (NaOH), ammonium fluoride (NH₄F), and hydrogen peroxide (H₂O₂). The stoichiometric mixture of standard Bi₂O₃ and SiO₂ chemicals has been used as a starting material. Fine crystalline eulytite has been obtained in the experiments with duration from 7 to 12 days. In our experiments eulytite crystals were growing in ammonium fluoride and hydrogen peroxide solutions for the first time in the world.

Analysis of the run products has shown that hydrogen peroxide is the most perspective solvent for eulytite synthesis since in the system there are no elements strange in composition to eulytite.

SEM investigation of obtained crystals has shown that eulytite single crystals are growing in different solutions have tetrahedral habitus, and crystal aggregates generally grow with vague marked faces.

The crystals growing in oxygen peroxide were most analogous to natural eulytite.

[1] V.V. Shulgina, T.I. Polupanova, A.V. Kruzhalov, V.M. Scorikov. *Bismuth orthogermanat*. Ekaterinburg, **1992**, 8. [2] B.N. Litvin, U.V. Shaldin, I.E. Pitovranova. *Crystallography*. **1968**, 13, 6, 1106

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Keywords: morphology, bismuth, growth

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Conformational polymorphism in the crystal structure of HgBr₂ adducts of dppf

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Polymorphism, the ability of a molecule to crystallize in more than one packing arrangement, is a popular chemical and crystallographic phenomenon [1]. Polymorphs can be generally recognized by virtue of their different unit cell parameters and often from the different crystal symmetries. The occurrence of polymorphs implies that the free energies of the various crystalline forms are comparable. The crystallization process to lead a polymorph is generally sensitive to variation in the conditions such as temperature, type of solvent, pressure and the manner in which the crystals are obtained. Polymorphism results when different (but energetically similar) packing interactions are operative during crystal growth. Here, we present two X-ray crystal structures of $\text{HgBr}_2 \cdot \text{dppf}$ adduct, briefly, which exhibit conformational polymorphism. The dimorph (1) and (2) are supramolecular adducts bearing ferrocene moiety and two bromide ligand. Crystallization of the complex in THF led to orange crystals whereas recrystallization of the compound in DMSO resulted a mixture of orange (1) and red crystals (2). Polymorphs may be identified from differences in the relative orientation of phenyl rings of diphenylphosphine groups of flexible dppf ligand. This unique phenomenon is not known for the other coordination complexes of mercury(II) halides (chloride and iodide) with dppf as ligand [2-3]. Single crystal X-ray diffraction study of the polymorph (1) shows a monoclinic ($C2/c$) system. Red crystals of the polymorph (2) crystallizes in triclinic ($P\bar{1}$) system.

The independent molecules observed in the dimorphs differ in their angle between the P-C-Fe mean planes of coordinated dppf ligand. The conformational differences between the two polymorphic modifications are depicted in Figure 1.

The conformation of each dppf ligand with respect to the central iron may be defined according to the PC-Fe-PC angle. Conformational changes from one structure to the other lead to formation of two different polymorphs. In order to establish whether conformational changes were reason for the polymorphism.

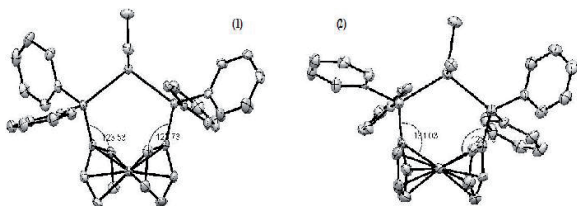


Figure 1. The P-C-Fe bond angle in dimorph (1) (left) and (2) (right).

[1] A. Nangia, *Acc. Chem. Res.* **2008**, *41*, 595-604. [2] Y.Y. Niu, Y.L. Song, S.X. Liu, X.Q. Xin, *Z. Anorg. Allg. Chemie.*, **2002**, *628*, 179-182. [3] J. McGinley, V. McKee, C.J. McKenzie, *Acta Cryst.* **1998**, *C54*, 345-347.

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Deposit of ZnO films by SS-CVD to atmospheric pressure,
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ZnO films were deposited by single source chemical vapour deposition (SS-CVD) technique. The zinc acetate dihydrate was used as a precursor of Zn and oxygen as oxidant agent. ZnO thin films

were grown on glass substrates at different deposition temperatures (250–400 °C in steps of 50 °C) at atmospheric pressure. The structural properties were investigated by X-ray diffraction (XRD) and atomic force microscopy (AFM). Low temperature deposition is provide by SS-CVD system, this characteristic generate high deposition rate which facilitate the ZnO films formation. X-ray diffraction indicates that all deposited films were polycrystalline. The films deposited shown a preferred orientation depending of the substrate temperature. This feature determines the shape of the structure of the ZnO films. The lattice parameter strain decrease in the ZnO films by the increase of the deposit temperatures. The texture coefficient for (002) plane is 2.25. Our samples showed a transmittance in the range of 75 % to 90% in the visible region.

Keywords: CVD, zinc oxide, X-ray

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Structural characterization of a new series of (Gd,R)-cuprate. R=Rare Earth

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Great advances have been made in achieving superconductivity at high temperatures. Superconductivity up to 120 K has been found in $\text{Ti}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$, with tetragonal structure with $a=3.85 \text{ \AA}$, $c=35.9 \text{ \AA}$ and S.G. I4/mmm [1, 2]. On the other hand, rare earth systems have had a considerable attention because they could give interesting electric and magnetic properties [3].

In this work, we present preliminary results in making ionic substitutions on $\text{R}_2\text{Ba}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}:\text{Li}$ (R = Gd, Dy, Ho) mixing the appropriate stoichiometric amounts of reactants for perform a conventional solid state reaction. The synthesis was nearly followed by X-ray diffraction, in order to reach the best conditions for synthesis. To observe the thermal stability, a combination of Thermo-Gravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) were carried out. The characterization was completed by the study of morphology by Scanning Electron Microscopy (SEM) and chemical analysis by Electron Dispersive X-Ray Spectroscopy (EDX).

[1] C.C. Torardi, M.A. Subramanian, J.C. Calabrese, J. Gopalakrishnan et al, *Science*. **1988**, *240*, 631-634. [2]C.C. Torardi, M.A. Subramanian, J.C. Calabrese, J. Gopalakrishnan et al, *Phys.RevB:Cond.Matt.*. **1988**, *38*, 6624-6630. [3] C bryn-Jacobsen, D.F Mc Morrow, R.C.C Ward, M.R Wells *J. Phys: Condens. Matter* **1997**, *9*, 8727-8735.

Keywords: x-rd, synthesis, rare-earth

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Synthesis and structural characterization on RESrMnO_3 (RE=Rare Earth) system

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