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Phase equilibria and $\beta\mbox{-}BaB_2O_4$ crystal growth in the Ba, Na, B // O, F system

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Due to the phase transition at 925°C, nonlinear optical crystals of low temperature modification of β -BaB₂O₄ (BBO) are commonly grown from high-temperature solution by top-seeded solution growth technique. Main solvents are some compositions in the BaO-B₂O₃-Na₂O ternary system, the BaB₂O₄ – NaF system, the BaB₂O₄ – BaF₂ system and also some mixed sodium oxide-fluoride compositions. The information in the available literature has been often contradictory and segmental. We believe that it's more appropriate to consider mentioned solvents as one four-component mutual system Ba, Na, B // O, F. It represents a trigonal prism with the corresponding oxide (BaO-B₂O₃-Na₂O) and fluoride (BaF₂-BF₃-NaF) concentration triangles at the bases.

Our investigation of the $BaB_2O_4 - NaF$ system has shown that it includes a primary crystallization area of the new compound $Ba_2Na_3[B_3O_6]_2F$ (hexagonal system, $P6_3/m$, a=7,346(1) Å, c=12,637(2)Å) [1,2]. Faceted bulk crystals of the new compound have been grown. Thus, $BaB_2O_4 - NaF$ section belongs to the Ba, Na // BO₂, F $(BaB_2O_4-(NaBO_2)_2-(NaF)_2-BaF_2)$ ternary mutual system, which is the prism section. Comprehensive investigation of the phase equilibria and crystal growth has been carried out. It has been shown that the BaB_2O_4 - $Ba_2Na_3[B_3O_6]_2F$ system is suitable for BBO crystal growth. The fast pyrohydrolysis of BaF2 in air causes gradual transformation of the BaB2O4–BaF2 system into the BaB2O4–BaO system and results in the co-crystallization of BaB2O4 and Ba₅B₄O₁₁ phases, making the growth of large high-quality β-BaB2O4 crystals in air impossible [3]. The Ba₅B₄O₁₁ phase (PDF 00-058-0115) was earlier found in the BaO-B₂O₃-Na₂O system [4].

High-quality crystals of about 400 g in weight with the convex form of the crystallization front, which is an evidence of the stability of the crystallization process, are reproducibly obtained. Currently the maximum aperture of NLO element cut from the crystals is $30 \times 30 \times 6 \text{ mm}^3$ [5].

 A.E. Kokh, N.G. Kononova, T.B. Bekker, P.P. Fedorov, E.A. Nigmatulina, A.G. Ivanova, *Crystallography Reports* 2009, 54, 146-151. [2] T.B. Bekker, A.E. Kokh, N.G. Kononova, P.P. Fedorov, S.V. Kuznetsov, *Crystal Growth & Design* 2009, 9, 4060-4063. [3] T.B. Bekker, A.E. Kokh, P.P. Fedorov, *CrystEngComm* 2011 DOI:10.1039 /C1CE05071K. [4] A.E. Kokh, N.G. Kononova, T.B. Bekker, Y.F. Kargin, N.G. Furmanova, P.P. Fedorov, S.V. Kusnetsov, E.A. Tkachenko, *Russian Journal of Inorganic Chemistry* 2005, *50*, 1749-1753. [5] A.E. Kokh, T.B. Bekker, V.A. Vlezko, K.A. Kokh, *J. Crystal Growth* 2011, *318*, 602-605.

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Transition metal sheet-like systems based on diacetic ligands <u>Antonio Diego Lozano-Gorrin</u>, Pau Díaz-Gallifa, Óscar Fabelo, Jorge Pasán, Catalina Ruiz-Pérez, *Laboratorio de Rayos X y Materiales Moleculares, Department of Fundamental Physics II, Facultad de Física, Universidad de La Laguna, Tenerife (Spain).* E-mail: adlozano@ull.es

The preparation, the structure determination by means of X-ray

diffraction and the study of magnetic properties of different transition metals sheet-like systems based on diacetic ligands have been done.

1,1-Cyclohexanediacetic and 1,1-cyclopentanediacetic acids, dicarboxylic acid-substituted C-6 and C-5 alicyclic hydrocarbons, can exhibit typical carboxyl group chemistry in variety of industrial applications. There are almost infinite esters obtained from thousands of potential starting materials. Esters are formed by removal of water from an acid and an alcohol, e.g., carboxylic acid esters, phosphoric acid esters, and sulfonic acid esters. Carboxylic acid esters are used as in a variety of direct and indirect applications. Lower chain esters are used as flavouring base materials, plasticizers, solvent carriers and coupling agents. Higher chain compounds are used as components in metalworking fluids, surfactants, lubricants, detergents, oiling agents, emulsifiers, wetting agents textile treatments and emollients.

Both acids are metal chelators. Chelation is applied in metal complex chemistry, organic and inorganic chemistry, biochemistry, and environment protection. It is used in chemotherapeutic treatments for metal poisoning. An example of end product derived from 1,1-cyclohexanediacetic acid is gabapentin, an anti-epileptic agent, chemically described as 1-(aminomethyl)cyclohexane acetic acid.

Polynuclear compounds of high-spin cobalt(II) ions bridged by carboxylate groups exhibit different magnetic behaviors depending on the bridgingmode of the carboxylate and on its coordination number [1-3]. Concerning the magnetic properties, a relationship between the nature of the magnetic coupling and the type of bridge that links the metal center has been proposed.



1,1-Cyclohexanediacetic acid 1,1-Cyclopentanediacetic acid

(a) H.-P. Jia, W. Li, Z.-F. Ju, J. Zhang, *Eur. J. Inorg. Chem.* 2006, 4264-4270. (b) D. Ghoshal, G. Mostafa, T.-K. Maji, E. Zangrando, T.-H. Lu, J. Ribas, N.-R. Chaudhuri, *New J. Chem.* 2004, 28, 1204-1213. [2] (a) H. Kumagai, Y. Oka, K. Inoue, M. Kurmoo, *J. Phys. Chem. Solids* 2004, 65, 55-60. (b) J. M. Rueff, N. Masciocchi, P. Rabu, A. Sironi, A. Skoulios, *Eur. J. Inorg. Chem.* 2001, 2843-2848. [3] (a) F. S. Delgado, J. Sanchiz, C. Ruiz-Pérez, F. Lloret, M. Julve, *CrystEngComm.* 2003, 5, 280-284. (b) H. Kumagai, Y. Oka, K. Inoue, M. Kurmoo, *Dalton Trans.* 2002, 3442-3446. (c) E. W. Lee, Y. J. Kim, D. K. Jung, *Inorg. Chem.* 2002, 41, 501-506.

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Peculiarities of crystal growth and morphology of carbonatesynthetic diamonds

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Diamond formation in multicomponent K-Na-Ca-Mg-melts oversaturated with dissolved carbon in respect to diamond is studied at 7.0 - 8.5 GPa. Experiments on diamond spontaneous crystallization and seeded growth were carried out in the "anvil-with-hole" high-pressure apparatus. Diamond-forming processes are high-effective [1] and characterized by estimated density of nucleation of no less than $3 - 5 \cdot 10^3$ nuclea in cm³ for individual crystal of up to 200 µm size, spinel-law and polysynthetic twins, polycrystalline aggregates of