compound $Li_{3.17}(P_{0.69}Ge_{0.24}Mo_{0.07})O_4$ has been grown on Pt–rod. For the structure determination there was chosen the single crystal with size 0.13x0.17x0.20 mm.

Crystal structure of analyzed compound is similar to the structure of $Li_{3+x}P_{1-x}Ge_xO_4$ (x=0.31). The partial substitution of $Ge^{4+}(P^{5+})$ for Mo^{6+} follows with the decrease of Li-atom content. In spite of it a significant amount of Li occupy 3 additional sites - 2 tetragonal (very close to 2 main Li-positions which are partially vacant) and 1 octahedral. The fourth additional Li-site found in $Li_{3+x}P_{1-x}Ge_xO_4$ (x=0.31) in our compound is empty.

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Keywords: crystal growth, conductors, electrocrystallization

P.16.04.2

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Phenomenon of Polytypism in Melt Grown Layered Crystals of CdI_2, PbI_2 and $CdBr_2$

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Polytypism has been observed in a large number of materials where the nearest neighbor relationship between identical two-dimensional layers of atoms can be satisfied in more than one way . The phenomenon has posed interesting problem for the Scientists, since the nature of force that causes ordering over the scale ranging from few angstrom to few thousands of angstrom units is not known.

The theoretical and experimental advancements made in the study of polytypism in melt- grown crystals of CdI_2, PbI_2 and $CdBr_2$ in the last few decades have been reviewed. The past work done in this field by us (using optical, Lasers and X-ray diffraction techniques) and update on the aspect of polytypism in the above crystals has been outlined with special reference to the role of:

(1) Purification and effect of impurities (known and unknown);
(2) Solid state phase transitions in the above crystals;
(3) Temperature dependence and their thermodynamic stability.

Keywords: polytypism, X-ray diffraction, melt growth

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Nonstationary Heat field as a new Approach in Bridgman Crystal Growth

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A traditional and axiomatic approach in crystal growth is the creation of a stable and stationary heat field with desired axial and radial temperature gradients. However, an obvious progress in application of Heat Field Rotation Method [1] is reached by development of the non-linear crystal growth technologies of β-BaB_2O_4 (BBO) in Czochralski configuration, LiB_3O_5 (LBO) and $CsLiB_6O_{10}$ (CLBO) in Kyropulos method.

The experiments of $AgGaS_2$ crystal growth by Bridgman method were performed using modified furnace which allows to create cyclic temperature oscillations. Obtained results suggests that moderate temperature oscillations (up to 4°C) favor the crystal quality and are likely to affect generally the hydrodynamic situation in the melt according to Curie principle. More complete mixing resulted to the crystals free of crack, twins and inclusions. Such defects often accompany crystals grown in stationary heat field where the mixing generated by natural convection is slowed due to "stabilized" axial temperature distribution in the melt.

[1] Kokh A.E., Popov V.N., Mokrushnikov P.W., *J. Crystal Growth*, 2001, **230**, 1-2, 163.

Keywords: crystal growth apparatus design, convection, nonlinear optical materials

P.16.04.4

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Growth and Crystal Structure of Bismuth Octaborate, α-$Bi_2B_8O_{15}$

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The objects of this search were a study of $Bi_2B_8O_{15}$ crystallization in the melts of near stoichiometric compositions, a determination of bismuth octaborate solid solutions range and a refinement of crystal structure of low-temperature phase of bismuth octaborate, α-$Bi_2B_8O_{15}$.

The bismuth octaborate crystals were grown from the melts of stoichiometric (20mole% Bi_2O_3 /80 mole% B_2O_3) and near stoichiometric (21.9mole% Bi_2O_3 / 78.1mole% B_2O_3) compositions. The grown crystals of a plate like form were of (5-7) mm in thickness, 27 x 27 mm^2 in cross-section. Comparison of lattice parameters of grown α-$Bi_2B_8O_{15}$ crystals (a=4.3191(9), b=22.175(7), c=6.4739(19)Å, β=105.44(2)°, sp.gr. $P2_1$, z=2) with the data presented in [1, 2] indicates that the phase of non-stoichiometric, Bi_2O_3-rich, composition exists unlike to the α-$Bi_2B_8O_{15}$ phase studied by authors of [1, 2]. Structure was refined as a racemic twin with components 0.80 and 0.20. Range of solid solutions having the 78.1mol.% B_2O_3 – 84.7mol.% B_2O_3 boundaries exists for the $Bi_2O_3{\cdot}4B_2O_3$ compound.

[1] Teng B., Yu W. T., Wang J. Y., Cheng W. F., Dong S. M., Liu Y. G., *Acta Cryst*, 2002, **C58**, i25. [2] Egoryusheva A.V., Kanisheva A.S., Kargin Yu. F., Gorbuova Yu. E., Mikchailov Yu. N., *Journ. Inorganic Chemistry*, 2002, **47**. 1961.

Keywords: bismuth octaborate, crystalline solid solutions, crystal structure

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Flux Growth and Characterization of Gallium-substituted $YAl_3(BO_3)_4$ Crystals

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Non-centrosymmetric $YAl_3(BO_3)_4$ (YAB) crystals of huntite structure, especially, doped with Tm, Yb, Eu, Er are of most interest as promising multifunctional solids for lasing and non-linear optical applications [1]. So far, no borate of this family is found in the nature, although Italian mineralogists have recently discovered anhydrous rare earth (RE) metaborate with closely spaced chemical composition [2]. For this reason, investigations of crystallogenesis in complex flux systems based on high-temperature RE borates are important from both scientific and technological viewpoints. The present work is focused on flux growth and characterization of $Y(Ga_xAl_{1-x})_3(BO_3)_4$ (x = 0, 0.05, 0.15, 0.2, 0.4) (YGAB) single crystals. YGAB crystals were obtained by top seeded solution growth technique as wel as spontaneous nucleation under different conditions using a $K_2Mo_3O_{10}$ based flux. As a result, transparent and homogenous YGAB single crystals with size up to 3 mm have been grown. From ASEM data, it was found that the average Ga distribution coefficients in YGaAB crystals slightly rises from 0.84 to 0.98 with increase of Ga concentration in the initial borates from 5 to 15 at %.

This research was supported, in part, by RFBR grant № 04-05-64709.

[1] Leonyuk N.I., *Prog. Crystal Growth and Charact.*, 1995, **31**, 279. [2] Ventura G.D., Parodi G.C., Montana A., Chaussidon M. *Eur.Y.Miner.*, 1993, **5**, 53-55.

Keywords: boron compounds, crystal growth from solution, crystal characterization

P.16.04.6

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Characterization of Profiled LiNbO3 and SBN Crystals by X-ray Diffraction

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The x-ray diffraction experiments were performed on $LiNbO_3$(LN) and $Sr_xBa_{1-x}Nb_2O_6$(SBN) crystals grown by modified Stepanov technique in bulk-profiled configuration using dies of capillary type with different cross-sections. The lattice defects were visualized by x-ray topography. The experiments show the presence in LN samples mosaic blocks drawn out along pulling direction with sizes 5-20 mm in this direction and 0.3-2.0 mm in perpendicular to growth axis. Adjacent blocks were also misoriented with respect to each other with average angles of ~6 arc min. Structure distortions for c-cut of bulk-profiled LN have a character of concentric rings, those form and sizes match to the die construction. The picture of structural imperfections depends on growth conditions forming of crystal-melt interface. For profiled LN grown in high temperature gradients the phase interface was inhomogeneous: flat over die plates, concave to the capillaries. The position of rocking curve maximum depends on x-ray incident angle and displaces together linear scanning along LN sample surface. It indicates the presence of crystallographic plane bend of 0.6+-0.1 degree. Low thermal conductivity of SBN crystals leads to formation of convex to the crystal crystallization front what allows to eliminate such lattice defects as small angle grain boundaries and as a result to obtain crystals of high optical quality. Atomic structure of SBN (x=0.33; 0.61;0.75) was investigated. Peculiarities of distribution of Sr and Ba ions as well as Ce, Tm doping ions in lattice channels are determined.

Keywords: Stepanov technique, X-ray topography, crystal defects

P.16.04.7

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Growth, Magnetic Behavior and Structure of Single Crystals of pure and Mg doped $SrCu_2(BO_3)_2$

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High quality single crystals of $SrCu_2(BO_3)_2$ pure and doped with Mg have been grown by the optical floating zone image furnace. Selected crystals were grown using highly enriched B^{11} isotope to ensure low neutron absorption. Only self flux was used. Problems related to growing high quality crystals doped with Na and Mg will be discussed in details. Magnetic susceptibility measurements were done on the single crystal samples oriented by Laue method and show relatively complex behavior, confirming the Sutherland-Shastry model.

The obtained single crystals were characterized by the X-ray diffraction at room temperature and by high resolution, inelastic neutron scattering.

Keywords: optical floating zone technique, magnetic susceptibility, neutron diffraction

P.16.05.1

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JAXA-GCF Project--- High-quality Crystals Grown in Space for Structural Biology

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Japan Aerospace Exploration Agency has been conducting the project (JAXA-GCF) for obtaining high-quality protein crystals to contribute to the progress in structural biology twice a year since 2003 using microgravity environment.

In this project, we construct a user-friendly space experimental frame work and provide regular flight opportunities. In technical point of view, we contrived gel-tube method [1] which worked as an effective crystallization device both in space and on the ground, based on the counter-diffusion technique [2]. We also provide techniques for efficient preliminary optimization of crystallization conditions using 1-dimensional simulation and for harvesting and cryoprotecting crystals before X-ray diffraction experiment. As a result, the success rate of the crystallization has become increased significantly.

We conclude that, using space environment, we have developed technologies for growing high-quality protein crystals for understanding 3-dimensional protein structure.

[1] Tanaka H. , et al., *J. Synchrotron Rad*, 2004, **11**, 45-48. [2] Garcia-Ruiz JM., Moreno A., *Acta Cryst*., 1994, **D50**, 484-490.

Keywords: space experiment, microgravity crystal growth, high quality protein crystal

P.16.05.2

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Atomic Resolution Crystals Obtained in Viscous Crystallizing Condition in Space

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Alpha-amylase, a glycoprotein derived from *Aspergillus oryzae*, has been used as a technical verification protein for Japan Aerospace Exploration Agency project (JAXA-GCF). We obtained crystals of alpha-amylase which diffracted beyond 0.89Å at SPring-8 beamline BL12B2 using polyethylene glycol (PEG) 8000 as a precipitant. Furthermore, they did not form cluster-like morphology which was usually observed on the ground experiment.

From our numerical analysis, viscosity of the crystallization solution, caused by PEG, might result in growing highly-ordered protein crystals depending on depletion zone formation around a crystal especially under microgravity.

Based on this, lysozyme crystallization experiment was performed using NaCl as a precipitant in which PEG 8000 was added to increase viscosity of the crystallization solution to enhance the effects of microgravity. The crystal diffracted beyond 0.88Å at SPring-8 beamline BL12B2 was obtained.

Therefore it may be expected that viscous crystallization solution is preferable to enhance the effect of microgravity on crystal growth. Other high viscous chemicals were verified as well.

Keywords: microgravity crystal growth, viscosity, polyethylene glycol

P.16.06.1

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***In-situ* X-ray Diffraction during Pulsed Laser Deposition**

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Pulsed Laser Deposition (PLD) has become a widespread technique for fabrication of thin films. A powerful pulsed laser is used to create a plasma off a target material, which is subsequently epitaxially deposited on a heated single crystal substrate. The PLD process can take place at relatively high oxygen pressures (up to 100 Pa), thereby making it especially suited for the deposition of High-T_c superconductors. For the purpose of studying the crystalline structure of the film during growth, a special sample chamber has been constructed to be used with synchrotron X-rays. The first results of deposition of thin films of $Yba_2Cu_3O_{7-x}$ on $SrTiO_3$ substrates were obtained at the European Synchrotron Radiation Facility. From intensity oscillations of the specularly reflected X-ray beam it is concluded that growth proceeds in a layer-by-layer fashion. Deposition was interrupted several times, which allowed for detailed structural characterization of the grown film at the deposition temperature of 780 °C, where pronounced Kiessig fringes show that