Institute, Russian Academy of Science,[b.]Technomash, Moscow, Russia. E-mail: ivleva@ran.gpi.ru

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

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

Hanna A. Dabkowska[1,2], S. Dunsiger[2], Antoni B. Dabkowski[1], Graeme M.Luke[1,2], John S. Preston[1,3], Bruce D. Gaulin[1,2], *[1]Brockhouse Institute for Materials Research. [2]Department of Physics., McMaster University, 1280 Main Street West, Hamilton, Ontario L8S 4M1, Canada. [3]Department of Engineering Physics, McMaster University, 1280 Main Street West, Hamilton, Ontario L8S 4M1, Canada*. E-mail: dabko@mcmaster.ca

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

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

Masaru Sato[a], Hiroaki Tanaka[b], Koji Inaka[c], Shinichi Shinozaki[b], Ari Yamanaka[b], Sachiko Takahashi[b], Mari Yamanaka[b], Erika Hirota[b], Shigeru Sugiyama[c], Mitsuyasu Kato[a], Chie Saito[a], Satoshi Sano[a], Moritoshi Motohara[a], Tai Nakamura[a], Tomoyuki Kobayashi[a], Susumu Yoshitomi[a], *[a]Japan Aerospace Exploration Agency. [b]Japan Space Forum. [c]Maruwa Food Industires, Inc.* E-mail: sato.masaru@jaxa.jp

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

Hiroaki Tanaka[a], Koji Inaka[b], Masaru Sato[c], Sachiko Takahashi[a], Shigeru Sugiyama[b], Mari Yamanaka[a], Satoshi Sano[c], Moritoshi Motohara[c], Tomoyuki Kobayashi[c], Susumu Yoshitomi[c], *[a]Japan Space Forum. [b]Maruwa Food Industries, Inc. [c]Japan Aerospace Exploration Agency.* E-mail: PXW01674@nifty.ne.jp

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

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

Vedran Vonk[a], Kurt Driessen[a], Mark Huijben[a], Sybolt Harkema[a], Heinz Graafsma[b], *[a]Science and Technology, University of Twente, The Netherlands. [b]European Synchrotron Radiation Facility, Grenoble, France.* E-mail: v.vonk@tnw.utwente.nl

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

the surface is particularly smooth [1].

[1] Vonk V., et al., *ESRF Highlights*, 2004 (2005).

Keywords: laser ablation, superconductor films, synchrotron X-ray diffraction

P.16.06.2

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Crystallization of $RAl_3(BO_3)_4$ and $R:YAl_3(BO_3)_4$ Single Crystal Layers

Elena A. Volkova[a], N.I. Leonyuk[a], A.V. Mokhov[b], E.A. Golubev[c], [a]*Geological Faculty, Moscow State University, Moscow, Russia.* [b]*Institute of Ore Deposits Geology, Petrography, Mineralogy and Geochemistry, Moscow, Russia.* [c]*Institute of Geology, Komi Science Center, Ural Branch of RAS, Russia.* E-mail: volkova@geol.msu.ru

Current development in miniature optical components leads to extensive study of single crystal layers because of a number additional benefits such as ability to use materials with high doping levels or to obtain high gain with modest pump powers. New crystals of solid solutions based on the $YAl_3(BO_3)_4$-$RAl_3(BO_3)_4$ (YAB-RAB) systems are promising solids for self-frequency doubling lasers [1].

In this report, our recent results on liquid-phase epitaxy (LPE) of RAB and R:YAB single crystal layers are discussed. Variations of growth rates of these layers were determined, in order to control crystal growth mechanism. Relations between the growth rate V and relative supersaturation β were found. It is also shown that primarily volume and surface processes occur simultaneously although evidence is presented for kinetic limitation of the growth rate on the later stage.

Besides, growth spirals epilayers frequently exhibit irregularities such as cusps and corrugations, but flat areas may also present on the surface. Micromorphological features as well as growth kinetics greatly depend on the substrate perfection.

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

[1] Dorozhkin L.M., Kuratev I.I., Leonyuk N.I., Timchenko T.I., Shestakov A.V., *Sov. Tech. Phys. Lett.*, 1981, **7**, 555.

Keywords: liquid epitaxy, epitaxial layers, kinetics and mechanism of crystal growth

P.16.06.3

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RHEED Observation of c-GaN on 3C-SiC/Si(001) Template Grown by RF-MBE

Tadashi Ohachi, T. Kikuchi, A.S. Somintac, M. Wada, *Dept. of Electric Eng., Doshisha University, 1-3 Miyakodani, Tatara, Kyotanabe, Kyoto 610-0321, Japan.* E-mail: tohachi@eml.doshisha.ac.jp

Cubic gallium nitiride, c-GaN, can be used to grow on a cubic substrate with a suitable latice constant for lattice matching. Cubic silicon carbide, 3C-SiC, which was formed by the carbonation of Si surface using a C_2H_2 jet nozzle[1], was used as a substrate for the epitaxial growth of c-GaN. The grown c-GaN layer was analyzed by reflection high energy electron diffraction (RHEED), electron microscopic techniques, and X-ray diffraction (XRD) techniques.

For the growth of the GaN layer, a specially designed RF-ECR type N radical source of 13.56 MHz was used to efficiently eliminate N ions and electrons from the surface [2]. The initial carbonization and initial growth of a LT buffer layer of c-GaN were monitored using RHEED during growth. The GaN was found to have the (2x2) surface structure. A GaN layer 1.5-mm thick grew epitaxially on the (001) face. The relative intensity ratio between cubic (002) and hexagonal(h) (10-11) XRD peaks from the GaN was 0.95:0.05.

[1] Kikuchi T., et. al, *J. Crystal Growth*, 2005, **275**, *in press*. [2] Ohachi T., et. al., *J. Crystal Growth*, 2005, **275**, *in press*.

Keywords: epitaxial layers of c-GaN, in situ observations by RHEED, RF-MBE

P.16.06.4

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Photoluminescence Study of Selenium Doped GaSb Layers Grown by Liquid Phase Epitaxy

Javier Olvera[1], J. Olvera-Cervantes[2] , M. Rojas-López[3], F. de Anda[4], E. Momox-Beristain[1], A. Olvera[2], [1]*Centro de Investigación en Dispositivos Semiconductores, BUAP, 72000, Puebla, Pue. México.* [2]*Universidad de Cádiz, Puerto Real, Cádiz, Spain.* [3]*Centro de Investigación en Biotecnología Aplicada, IPN, 72160, Tlaxcala, Tlax. México.* [4]*Instituto de Investigación en Comunicación Óptica, UASLP, 78100, San Luis Potosí, S.L.P., México.* E-mail: jolvera@siu.buap.mx

We analyzed the photoluminescence spectra of selenium-doped GaSb grown by liquid phase epitaxy at several temperatures from 10-160 K. The growth was performed using the same solution on GaSb substrates at 450 °C. The obtained layers showed only a small variation of carrier concentration. Photoluminescence measurements at 10 K showed a dominant transition near 777 meV associated to the residual acceptor. The dominant residual acceptor has been attributed to the native defects caused by antimony deficiency, usually due to the Ga antisite or Ga antisite defect in combination with the Ga vacancy. Also at this temperature, there are observed several bands associated to the presence of selenium shallow donors. As the measurement temperature increases, the photoluminescence band associated to the GaSb energy bandgap dominates the spectrum and its temperature dependence agrees with those for the case of tellurium and sulphur doped GaSb.

Keywords: GaSb, photoluminescence, liquid phase epitaxy

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Substitutional and Interstitial Inclusions of Mn Additivies onto the KDP Lattice

Luis Humberto Avanci[a], C.M.R. Remédios[b], F.C. Nascimento[c], M.H. Tabacniks[a], J.M. Sasaki[b], L.P. Cardoso[c], S.L. Morelhão[a], [a]*Inst. de Física, USP, CP 66318, 05315-970, São Paulo, SP, Brazil.* [b]*Dep. de Física, UFCe, CP 6030, 60455-760, Fortaleza, CE, Brazil.* [c]*Inst. de Física, UNICAMP, CP 6165, 13081-970, Campinas, SP, Brazil.* E-mail: lhavanci@if.usp.br

In this work, pure and Mn^{3+} doped potassium di-hydrogen phosphate (KDP and KDP:Mn) were studied through Rutherford back-scattering (RBS), Rietveld refinement (RR) and X-ray n-beam diffraction (nBD). RBS results allows the determination of KDP:Mn stoichometric formulae as $KH_2PO_{3.8}Mn_{0.4}$ while, from the RR results, it was possible to determine that the Mn^{3+} atoms are substitutional to the K ones. The lattice parameters were determined for both pure and KDP:Mn by using RR and nBD and, besides agreeing very well, they indicate the better accuracy of the results from nBD. This fact comes from the high sensitivity of the nBD technique in determine micro-crystallographic variation. According to the lattice parameter results, all values for KDP:Mn are smaller than those for KDP. Those results are also compared with a previous one, were it was determined that Mn^{3+} in concentration of 2-5 x 10^{-4} mol are occupying interstitial sites [1] and located 0.66 from (200) plane and 0.21 from (112) plane [2]. Rietveld refinement was performed from X-ray high-accuracy single crystal measurement and the nBD measurements were carried out at beam line XRD1 of the Brazilian Synchrotron Light Laboratory. All samples were grown at the same pH of 1.5.

[1] Lai X., Roberts K. J., Avanci L. H., Cardoso L. P., Sasaki J. M., *J. Appl. Cryst.*, 2003, **36**, 1230-1235. [2] Lai X., Roberts K. J., Sasaki J. M., Cardoso L. P., Bedzyk M., Lyman P. F., 2005, *in preparation*.

Keywords: X-ray difraction techniques, inclusion phenomena, Rietveld structure analysis

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New Routes in Carbon Nanotube Synthesis by Means of a Modified Hot Filament Chemical Vapor Deposition Technique

Angelamaria Fiori[a],Silvia Orlanducci[a], Vito Sessa[a], Emanuela Tamburri[a], Maria Letizia Terranova[a], Marco Rossi[b], [a]*Dept. of Science and Chemical Technol. Tor Vergata University, Rome, Italy and*