CRYSTAL GROWTH: TECHNIQUES, INSTRUMENTATION AND APPLICATIONS

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

The x-ray diffraction experiments were performed on LiNbO₃(LN) and Sr_xBa_{1-x}Nb₂O₆(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 xray 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. Dunsiger², Antoni B. Dabkowski¹, Graeme M.Luke^{1,2}, John S. Preston^{1,3}, Bruce D. Gaulin^{1,2}, ¹Brockhouse Institute for Materials Research. ²Department of Physics., McMaster University, 1280 Main Street West, Hamilton, Ontario L8S 4M1, Canada. ³Department 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 B¹¹ 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

<u>Masaru Sato</u>^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, ^aJapan Aerospace Exploration Agency. ^bJapan Space Forum. ^cMaruwa 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

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Atomic Resolution Crystals Obtained in Viscous Crystallizing Condition in Space

<u>Hiroaki Tanaka</u>^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, ^aJapan Space Forum. ^bMaruwa Food Industries, Inc. ^cJapan 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

<u>Vedran Vonk</u>^a, Kurt Driessen^a, Mark Huijben^a, Sybolt Harkema^a, Heinz Graafsma^b, ^aScience and Technology, University of Twente, The Netherlands. ^bEuropean 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 Yba2Cu3O7-x on SrTiO3 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