babaryk@bigmir.net

Langbeinite-type (α-$K_2Mg_2(SO_4)_3$[1]) phosphates has been intensively investigated over the last decade. In particular, to continue the search of the relationship with large family of {$[M_2(PO_4)_3]^{n-}$}; anionic framework, which covers NASICON (Na Supersonic Conductor), $ScWO_4$, Garnet structure types, is being of great interest. In this work the new synthetic approach is applied for single crystal obtaining, following structure investigation of Langbeinite-type phosphates $K_2Ln_{1.5-x}Nb_{0.5+x}(PO_4)_3$, where *Ln* = Sc, Y, In, Ho-Lu is subject of this report. The corresponding compounds were grown in spontaneous crystallization mode from fluxes of melted systems KPO_3-$K_4P_2O_7$-LnF_3-K_2NbF_7. All synthesized tetragonal-shaped crystals belong to the cubic system (sp. gr. *P*213). Single crystal structure determination revealed correlation between the composition of target compounds and radii of trivalent metal cations. Analysis of potassium, lanthanide and phosphorus bonding parameters (BVS method) allows consider crucial role of phosphate tetrahedron on total stability of the structure.

[1] Zemann, A., Zemann, J. (1957) *Acta Cryst*., **10**, 409-413.

Keywords: phosphates, X-ray crystal structure analysis, crystal synthesis

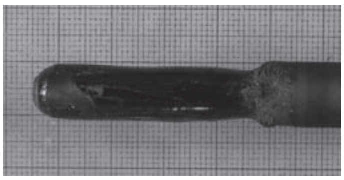
P16.04.30

Acta Cryst. (2008). A**64**, C589

Growth of large single crystals of high-Tc superconductor using a tilted-Lamp floating zone furnace

A.T.M. Nazmul Islam[1,2], I. Tanaka[2], S. Watauchi[2]
[1]Hahn-Meitner-Institut, SFN1, 100 Gleinicker strasse, Berlin, Brandenburg, 14109, Germany, [2]Center for Crystal Science and Technology, University of Yamanashi, Miyamae 7-32, Kofu, Yamanashi 400-8511, Japan, E-mail：nazmul.islam@hmi.de

The intimate connection between superconductivity and magnetism in high-Tc cuprates is believed to be fundamental to the superconducting mechanism. So, extensive neutron-scattering measurements had been carried out on $La_{2-x}Sr_xCuO_4$. Since $La_{2-x}Sr_xCuO_4$ has small neutron scattering cross-section, large volume of single crystals were needed for these measurements. Typical single crystals grown by Traveling Solvent floating zone method are 5 mm in diameter and few centimeters in length. To accommodate large volume of single crystal in the neutron beam several single crystals are aligned in the beam. This process also leaves empty spaces in between and hence unused part of neutron beam. To overcome these problems here we have grown La_2CuO_4 single crystal of diameter about 10mm, almost twice of that grown until now (Fig. 1). A specially modified floating zone furnace, Tilted-Lamp Floating zone furnace, where the ellipsoidal mirrors could be tilted downward up to 30 degrees was used for this experiment. Optimum tilting of the mirror-lamp systems modifies the heating profile of the molten zone in a way that we believe, is favorable for growth of single crystals with larger diameters.



Keywords: tilted-lamp floating zone, single crystal La214, high-Tc Superconductor

P16.04.31

Acta Cryst. (2008). A**64**, C589

Solid-liquid interface in floating zone growth of rutile crystal with variation of focusing angle

Md. Abdur Razzaque Sarker, Watauchi Satoshi, Tanaka Isao
University of Yamanashi, Center for Crystal Science and Technology, Miymae 7, Kofu, Yamanashi, 400-8511, Japan, E-mail：g08df004@yamanashi.ac.jp

In the floating zone (FZ) method, the molten zone is kept between a feed and a grown crystal by the balance in gravity and surface tension of the melt. Therefore, stable molten zone is necessary for good crystal growth. In infrared convergence type FZ, the molten zone is heated from the surface. So the shape of the solid-liquid interface is usually convex for many materials, which often makes it difficult to grow large crystals in diameter, because the interface becomes more convex as the diameter increases. In our experiment, we focused on the locations of heating lamps and the molten zone. In conventional heating system, the locations are in the same horizontal plane. In our experiment, these locations were systematically changed. Heating lamps and reflecting mirrors were tilted from the horizontal configuration to 20 degree by 5 degree as step for the crystal growth of rutile (TiO_2). To examine the interface shapes, molten zones were quenched. However, the shapes of solid-liquid interfaces were not clear by polarized optical microscopy. To evaluate the shapes by chemical analysis of EPMA, Yttrium was added in the molten zone because Yttrium is very hard to dissolve in the grown crystal of rutile. By using this technique, we could successfully investigate the interface shapes precisely. Focusing angle dependence of the interface shapes would be reported.

Keywords: float zone growth, oxides, interface characterization

P16.04.32

Acta Cryst. (2008). A**64**, C589-590

Growth of Nd-doped YVO_4 single crystals by anisotropic heating floating zone method

Dal Hoy Kwon, Satoshi Watauchi, Isao Tanaka
University of Yamanashi, Center for Science and Technology, Faculty of Engineering, Miyamae 7, Kofu, Yamanashi, 400-8511, Japan, E-mail：kwondalhoy@hotmail.com

Nd-doped YVO_4 single crystals was grown along the a-axis by anisotropic heating floating zone (AHFZ) method. YVO_4 single crystals has strong tendency to grow along the c-axis. For normal floating zone (FZ) method, the grown crystals show a flat shape, longer in the c-axis. The average aspect ratio shows 1.4 (c-axis diameter / a-axis diameter) in normal FZ method. To make the shape of crystals round type, AHFZ method was tried. The powder of Y_2O_3, V_2O_5 and Nd_2O_3 was mixed with V_2O_5 richer than the stoichiometric composition. Nd of 3at% was substituted for Y sites in YVO_4. The mixture was calcined at 600℃ for 15 hrs, and sintered at 1500℃ for 6 hrs. In case of AHFZ machine, the electric power of two pairs of lamps (A and B lamp pairs) located at the diagonal positions can be controlled independently. The c-axis of seed crystal was positioned along B lamp pair direction. The a-axis of seed crystal was positioned along A lamp pair direction. The voltage ratio [c-axis voltage / a-axis voltage] (=B lamp pair voltage / A lamp pair voltage) was changed from 0.7 to 1.3. As the voltage ratio was changed from 0.7 to 1.3, the aspect ratio of crystal (c-axis diameter / a-axis diameter) changed from 1.05 to 1.88. In case the electric power applied to c-axis of crystal is stronger than the electric power applied to a-axis of crystal,