

Synthesis of materials by chemical vapour transport, Czochralski and floating zone methods

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Chemical Vapor Transport (CVT) Method



The Chemical Vapor Transport (CVT) method is a technique used to grow single crystals of various materials. It involves the use of a transport agent, which reacts reversibly with source material creating volatile species which transports the material from a source region to a growth region, where the crystals are forming with the release of transport agent.

Furnaces with 2, 3 and 4 heating zones were built for correct temperature gradient needed in CVT processes.

Crystallization of Ga₂O₃ by floating zone in optical furnace

In 2024, research on production of ${\rm Ga_2O_3}$ crystals by zone melting in an optical furnace was continued. Due to the high evaporation of gallium oxide and its deposition on the quartz cover of the crystallization chamber during the crystallization process, a constant air flow in the chamber and its further filtering were used.

A series of processes were performed in the 010 growth direction, each subsequent crystal used to produce seeds with as few defects as possible.



Photos of Ga₂O₃ crystal grown in (010) direction

Sample preparation

Samples with oriented walls in three crystallographic directions were cut from the crystal.



Optical observation in polarized light







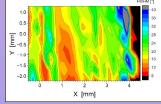
Natural cleave **a** plane

Twins and cleaving plane are visible

c plane

X-ray High-Resolution 2D-Mapping

(collaboration with SL1.3)



Precision X-ray measurements (OmTA scans with a 0.2x0.5 mm² beam) conducted by dr. Jarosław Domagala in SL1.3 on the natural a (100) schist surface at altered sample positions showed that almost over the entire surface the OmTA - curves are very narrow, FWHM<30 angular seconds (for reflex 12 0 0). See also poster SL1.3

Photoluminescence (PL) from β -Ga₂O₃ crystals

(prof. K.P. Korona, Faculty of Physics, University of Warsaw)

Measurements of bulk crystals have shown that the PL was anisotropic: PL intensity changed for different crystallographic directions and it was polarized. The emission was strongest in [100] direction. Light emitted in [100] and [010] directions was polarized along [001] direction. Such polarized emission means that related centers have dipole moments closely to [001] direction. Light emitted in [001] direction was weaker and was polarized mainly along [100] direction.



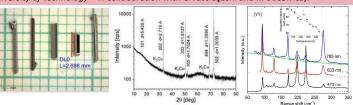




The polarized PL spectra of three samples with different planes a) (100), b) (010) and c) (001)

Results were published: Krzysztof P. Korona, Jan Fink-Finowicki, Aliaksei Bohdan, Roman Stępniewski, Henryk Teisseyre, Polarized UV–Visible Photoluminescence from Bulk θ -Ga $_2$ O $_3$ Crystals, Physica Status Solidi (b), DOI: 10.1002/pssb.202400566

ZrAs₂ single crystals were grown in few CVT processes using iodine as a transport agent. Obtained crystals were characterized using ARPES (*Solaris Synchrotron Centre – in collaboration with B. Kowalski and M. Rosmus*) and Raman Spectroscopy (*Warsaw University of Technology – in collaboration with C. Jastrzębski and R. Bacewicz*).



ZrAs₂ single crystals: photo, XRD data and Raman spectra

Research on ZrAs₂ resulted in 2 publications in Phys. Rev. B ("Emergent impervious band crossing in the bulk in topological nodal line semimetal ZrAs₂" and "Temperature and excitation energy dependence of Raman scattering in the nodal-line semimetal ZrAs₂")

Using our experience, we performed synthesis of crystals of analogous $\mathbf{ZrP_2}$ compound. Grown crystals were sent to Raman and XRD measurements.



ZrP₂ single crystals: photo, XRD data and preliminary Raman results

 $\mathbf{MnTe_2}$ single crystals were grown in several different CVT processes which resulted in crystals in varying size. Obtained samples were sent for Hall and Raman measurements.



MnTe₂ single crystals: results of different grown processes and Raman spectra

Czochralski Method

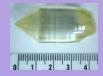
A series of crystallization processes of **SrLaAlO**₄ single crystals using the Czochralski method were performed. The obtained single crystals were oriented, cut, polished and submitted as substrates for research in teams dealing with epitaxy growth (prof. Marta Cieplak, IF PAN).





Optical image of grown SrLaAlO₄ single crystal

The crystallization process of a $PbMo_{0.5}W_{0.5}O_4$ was carried out from $PbMoO_4$ – $PbMO_4$ (1:1 mol) solution in a Pt crucible with a diameter of 40 mm. Process was carried out in air atmosphere, using a Pt as a seed with a growth rate of 2.5 mm/h and a rotation of 20 rpm.



Optical image of $PbMo_{0.5}W_{0.5}O_4$ single crystal

The crystallization processes of YAlO₃:Bi were carried out in a nitrogen atmosphere using irydium crucible and with iridium seed using the Czochralski method. Due to the intense evaporation of $\rm Bi_2O_3$, the process was carried out from a specially prepared starting composition of the melt - 140 g of previously synthesized Y_{1.01}Al_{0.99}O₃ pellets were melted and homogenized and then supplemented with 25 g of a powdered YAP single crystal with mixture of $\rm Bi_2O_3/Y_2O_3$ (1:1 mol) containing $\rm 5\%_{mol}$ of $\rm Bi_2O_3$ in respect to the entire melt. The crystal growth rate was 4 mm/h. Due to the high vapor pressure of $\rm Bi_2O_3$ at the crystallization temperature, it was not possible to obtain the proper Bi concentration in the produced crystals.



Photos of obtained crystals of YAP doped with Bi

Samples cut from obtained crystals with trace concentrations of Bizmuth were sent for further studies (Prof. Zhydachevski).