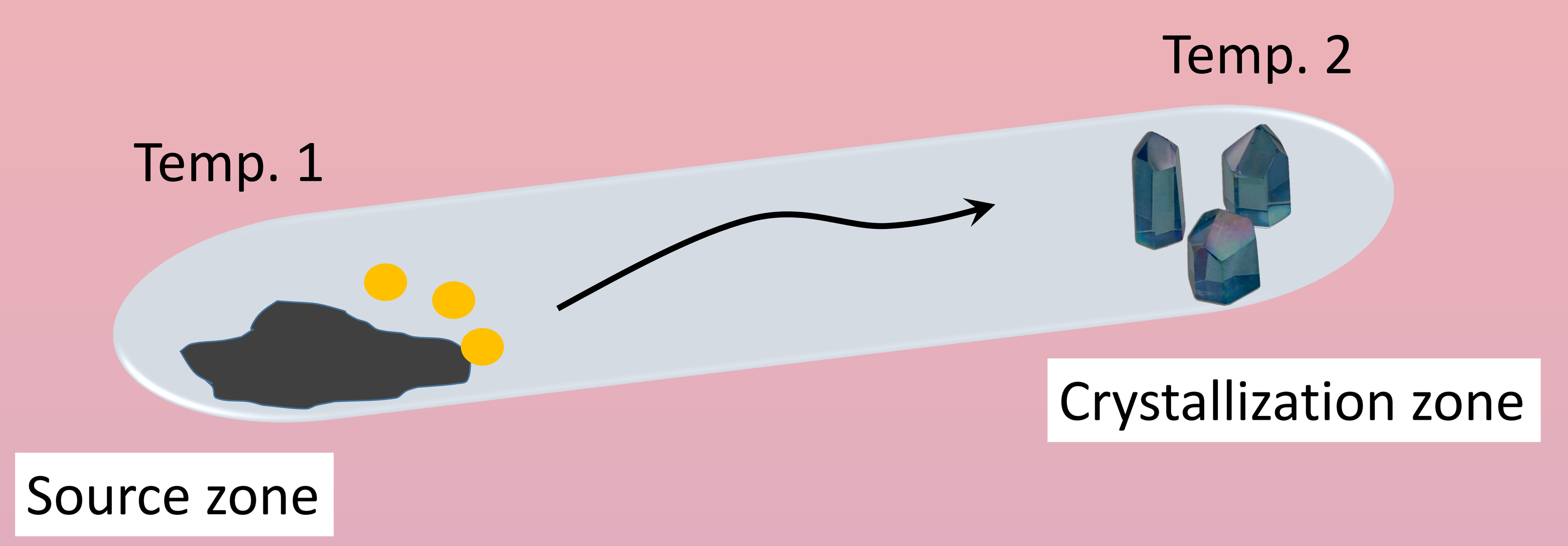
Synthesis of materials by chemical vapour transport and Czochralski method, properties of selected materials

D. J. Jastrzębski, K. Cieślak, A. Wadge, A. Hruban, J. Fink-Finowicki, P. Iwanowski, R. Diduszko, T. Wojciechowski, M. Berkowski, A. Wiśniewski

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 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.

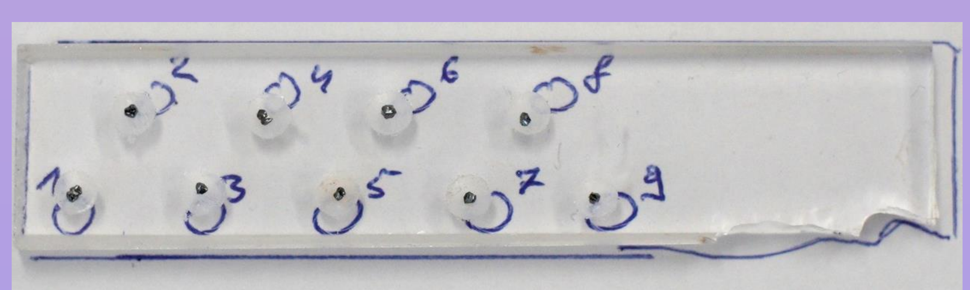
Boron Arsenide synthesis and crystallization

Cubic boron arsenide (BAs) is a material with the second, after diamond, ultra-high thermal conductivity, of the order of 1100 W/mK. It is a chemically inactive material and has a thermal expansion coefficient of $3.0 \times 10^{-6} K^{-1}$, similar to Si ($2.6 \times 10^{-6} K^{-1}$). As a substrate, it enables the growth of materials with a zinc blende structure, such as GaN and GaAs. It is a semiconductor with an energy gap of ~1.5eV. The use of the chemical vapor transport (CVT) method in 2018 opens up a wide field for the development of the synthesis and growth of bulk BAs single crystals.

Synthesis and crystallization of BAs were carried out using the CVT method using iodine or BI_3 as a transport agent. As a result of the process, numerous BAs single crystals were obtained in the deposition zone (with good parameters confirmed by XRD and Raman measurements).

Optical images of BAs crystals obtained from CVT process

In the next stage, an attempt was made to further grow of BAs on seeds (boron arsenide crystals selected from previous process) in order to obtain crystals of larger sizes and the desired orientation.



Quartz plate with BAs crystals used as a seeds

$NbSe_2$

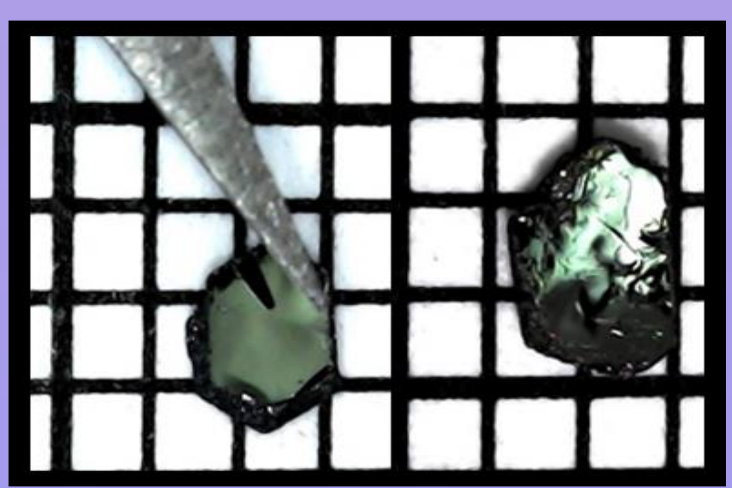
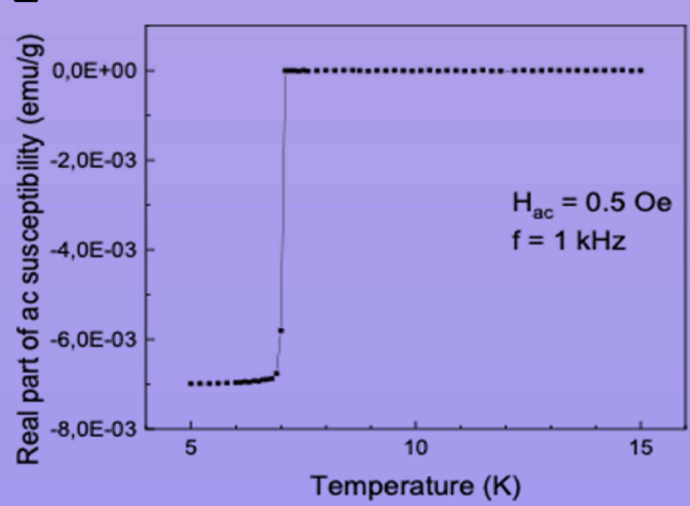


Photo of single crystals of $NbSe_2$

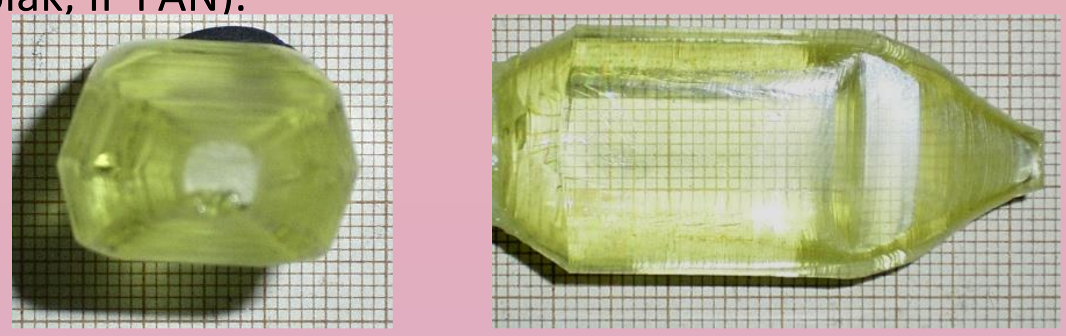


SQUID measurements of $NbSe_2$

Single crystals (susceptible to exfoliation) with a diameter of up to 3 mm were obtained. The quality of the crystals was confirmed by XRD measurements. Preliminary SQUID magnetic testing were conducted by Artem Lynnyk.

Czochralski Method

A series of crystallization processes of $SrLaAlO_4$ single crystals using the Czochralski method were performed on substrates for epitaxy growth of superconductors. The obtained single crystals were oriented, cut onto substrates, polished and submitted for research in teams dealing with epitaxy on these substrates (Prof. Marta Cieplak, IF PAN).



Optical image of produced $SrLaAlO_4$ single crystal

The crystallization process of a $PbMo_{0.5}W_{0.5}O_4$ was carried out using the Czochralski method from $PbMoO_4$–$PbWO_4$ (50:50 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.

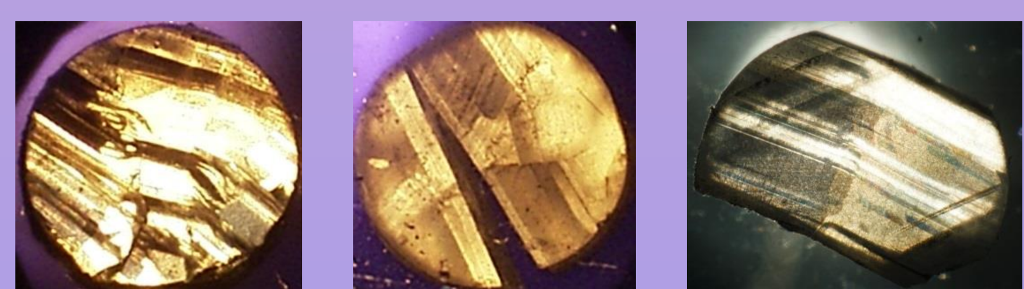
Optical image of $PbMo_{0.5}W_{0.5}O_4$ single crystal

The trial crystallization process of a solid solution with the starting composition $PbMo_{0.6}W_{0.4}O_4$ was carried out under N_2 pressure of 0,4 MPa with a growth rate of over 5 mm/h and a rotation speed of 16 rpm. For crystallization, a mixture of $PbMo_{0.5}W_{0.5}O_4$ and $PbMoO_4$ crystals was used as the starting material in appropriate proportions. The process was carried out to check the activated equipment (ADL puller).

Crystallization of Ga_2O_3 by zone melting in optical furnace

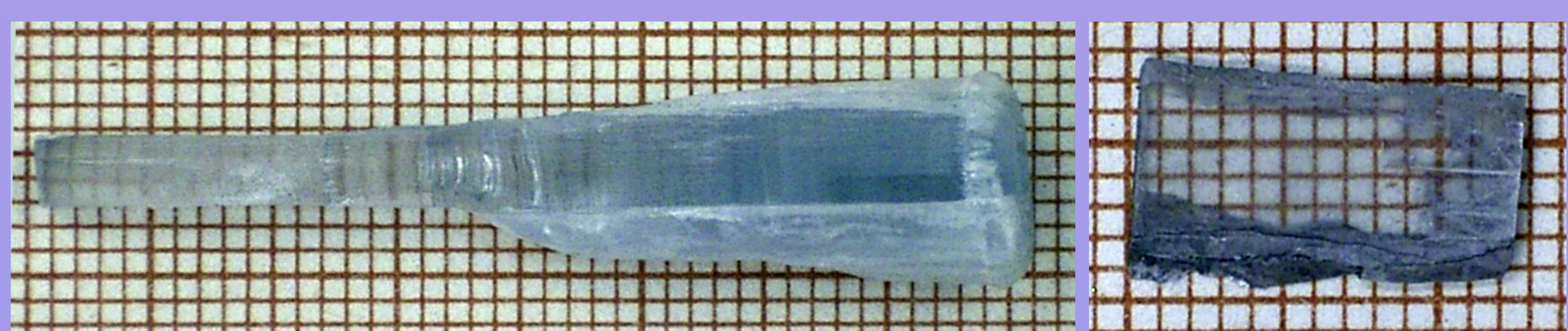
In 2023, research on production of Ga_2O_3 crystals by zone melting in an optical furnace was continued. Due to the high evaporation of gallium oxide and its deposition on the quartz cover of the crystallization chamber during the crystallization process, it was necessary to modernize the station to ensure a constant air flow in the chamber and its further filtering. Currently, this system allows the process to last several hours without significant clouding of the chamber cover.

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



Twinning visible in polarized light of Ga_2O_3 cross-section for subsequent crystals

On selected seeds growth of gallium oxide dopped with rare metals for optical research was carried out. As a first dopant, due to its optimal ionic radius, yterbium was selected.



Optical image of obtained Ga_2O_3:0.5%Yb crystal and sample from the crystal sent for luminescence measurements in Faculty of Physics University of Warsaw

Presenter: Daniel Jastrzębski (Email: djastr@ifpan.edu.pl)