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Purpose	Customer report				

Status of growing of PWO II.

at Crytur

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1 Crystal growth

It was proved that the technology developed in Crytur can produce crystals which meet the requirements for PANDA (Report June 2015).

Raw material

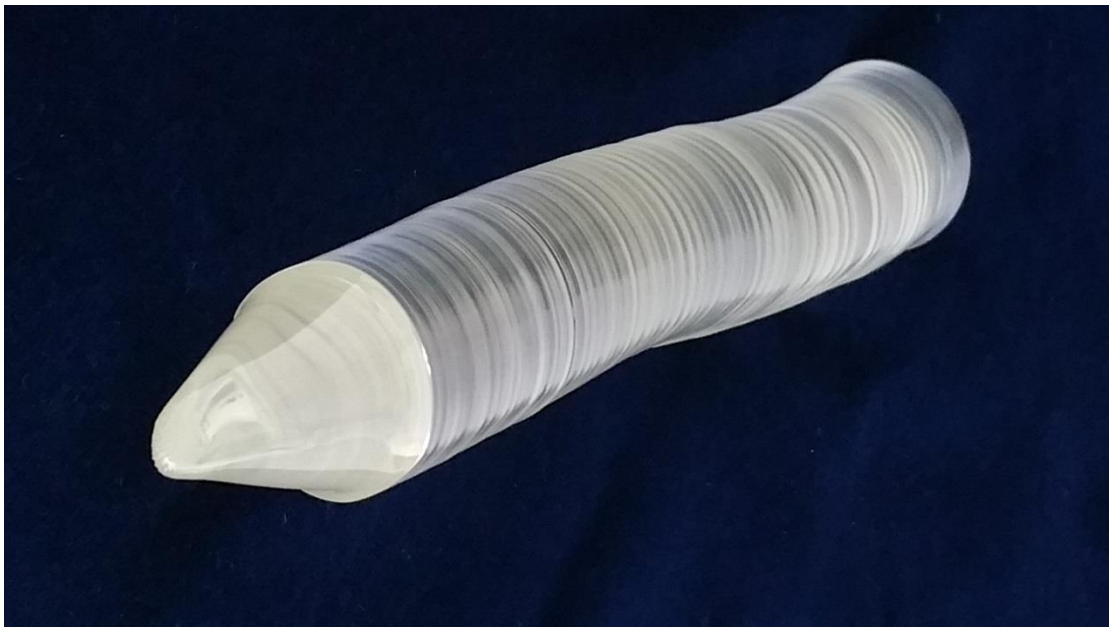
After the campaign of crystals grown from remelted crystals we started to grow crystals from different composition of raw material, closer to real production: we started with a clean crucible and fill it with 70 wt. % of cracked crystals and 30 wt. % of annealed powder. After each growth cycle the crucible is being filled with 30 wt. % of total batch mass of annealed powder, pre-melted because of higher volume, and filled in to the total batch mass with cracked crystals.

There is a positive influence of pre-melting for properties of crystals, but negative for efficiency of growth cycle.

Because of the fact that cutting on single wire saw has not been started in large scale yet, there is a lack of pieces of crystals for further melting. We are testing to grow crystal only from annealed (and because of volume pre-melted) powder to get crystal with sufficient parameters for production of prisms or as a semi-product for next growing. It is repeating and testing of technology from BTCP under our conditions.

Atmosphere

Because of changed design of the new furnaces we are able to measure content of O2 during the growth. A nitrogen generator was installed and is running, so we have strong source of N2. Measurements showed, that content of O2 in the first prototype of growth furnace is about 8 % and that we can reach less than 2 % with new furnaces and with N2 generator. Influence of amount of O2 will be evaluated and the lower concentrations of O2 will be tested, our current value of O2 content is cca 2% on new the furnaces. At this time, only the colour of as grown crystals can help us to consider amount of O2, samples for transmission measurements are in production, but they are relatively light, not yellow – see picture no.1



Picture no. 1: The first crystal grown in new furnace

Defects of as grown crystals mentioned in Report no.1

Curved and/or cracked beginning of crystals (seed end) still occurs on crystals produced on the prototype of furnace, although we tested an addition of insulation at the end of pulling period, and changed direction of input of N2 to the crucible. Larger rearrangements of N2 inflow are being done now.

The first crystal grown after heating tests and measurements of thermal gradients of new furnaces is on picture number 1. It seems better from these two points of view, but it is necessary to confirm this by further measurements.

There are two main reasons for this difference: the first is the way how N2 is delivered near to the crucible (rearrangement on prototype of furnace) or slightly different thermal properties of insulation on new furnaces, which influence speed of cooling – it is lower and takes more time on new furnaces. The difference in this property is known, because it was impossible to get identical insolation bricks. Probably it is useful for crystals, but it makes growth cycle longer.

Checking of chemical composition

Our current supplier of analysis of dopants is not suitable for us, because of its long and irresponsible delivery times, so we are looking for a new supplier.

XRF analysis of stoichiometry is used satisfactorily. Pressing of powder for production of samples is used now. Nevertheless we also bought a new induction furnace for smelting of samples and after testing period it will be used for XRF samples preparation. Advantages should be better quality of sample and complete “in house” analysis in Crytur.

2 Machining

Cutting

Mechanical holder for cutting of regular prisms was designed, made and tested. It enables cutting of all sides and small change of its design enables also cutting of ends. It works without gluing so there is no danger of thermal shocks during glue removal, there is no need of cleaning of crystals from the glue and it saves time. Because of more experience with the cutting process we reduced the addition for grinding and polishing from 1 to 0,5 mm.

Picture no. 2: Holder for cutting of crystals

We are planning to work on the holder modification for cutting of irregular prisms (type 11, right).

Experiment with cutting of sample no. 18 described in previous report showed that there is no influence of orientation of cleavage planes and that a bigger influence has the quality of the saw.

Grinding and polishing

Results presented in previous report came from “in hand” process. Now grinding and polishing is carried out in a holder for one piece of crystal, but still on the same machines. There will be three regular prism machined by this way till the end of September, they will be measured and sent to the customer.

Experiments with this holder for one piece of crystal on new production machines are planned at the end of September. Lapping machine is ready to use and we are finishing the preparation of polishing machine now.

Last step, which means to use currently three holders for six, resp. three pieces of crystals on production machines will follow. Delivery time of these holders is beginning of October.

3 Physical parameters of samples

Several crystals were grown under the conditions described in the Chapter 1. In the Fig.1, the absorption coefficient was measured for crystals grown using different filling of the crucible :

1) Annealed powder (PWO2-PWO9)
2) Cracked Crystals (PWO13-PWO19)
3) Annealed powder + cracked crystals (PWO26)

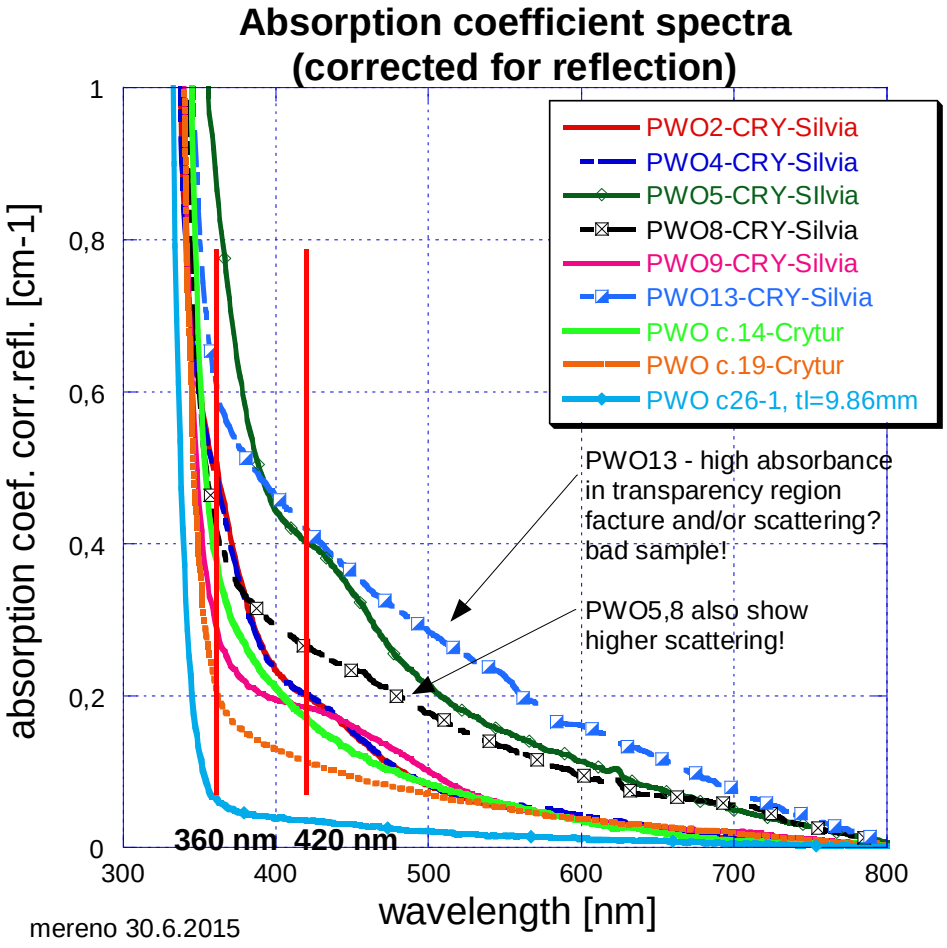


Fig. 1 Absorption coefficient spectra.

In the Fig.2 the absorption spectrum before and after irradiation for the crystal PWO26 was measured.

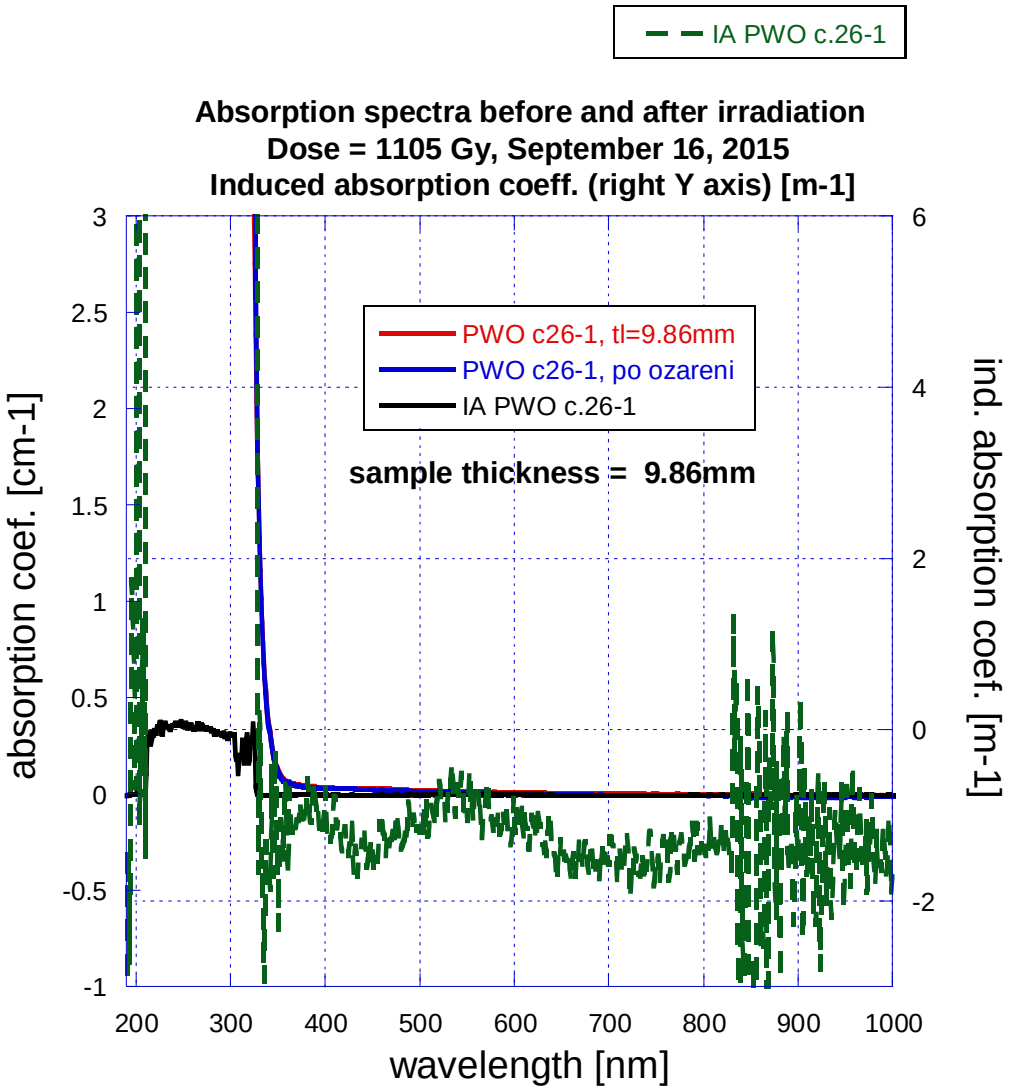


Fig. 2: Absorption coefficient spectra (left Y axis) and induced absorption coefficient (right Y axis).

4 New premises and equipment

Extensive rebuilding of new premises is being finished, we are working on heating and ventilation, but it doesn´t have any influence on production.

Three new growth furnaces are on their places – see picture no. 3. Two of them are after growing tests and they are ready to produce crystals. Growing test on the last furnace have started.

Picture no.3: Crystal growth furnaces in new premises

Lapping and polishing machines are installed and the vented boxes were ordered and the production is ongoing.

Picture no. 4: Lapping and polishing machines

Measurements of the Pb content in the air during growing was performed. Further actions to reduce the amount of Pb were done and new measurement is ongoing.

The content of Pb in the air during cutting and polishing will be performed after the installation of the vented boxes.

5 Schedule of next steps

Crystal Growth

Production on new furnaces have started at the beginning of September.

Tasks to solve:

- tune process to grow crystals with required geometry (shape, length)
- tune process to grow crystals with required physical properties (number of growth cycles, concentrations of dopants)
- develop technology of pre-treatment of raw material
- find-out ways of shortening of growth cycle

Cutting

- tune holder for cutting for irregular prisms (type 11 right)

Grinding and polishing

First tests on production lapping and polishing machines have started at the beginning of September.

Task to solve:

- building the vented boxed for the grinding and polishing machines
- tune holders for mass machining of regular prisms
- tune holder for grinding and polishing for irregular prisms (type 11 right).