

Status of Phase II Detector production TG02

Max-Planck-Institut
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The logo of the Max-Planck-Institut für Physik (Werner-Heisenberg-Institut) is a circular emblem. It features a central figure, likely a historical or scientific symbol, surrounded by text in a circular border. The emblem is rendered in a light blue or teal color.

L. Bezrukov, A. Caldwell, J. Janicskó, M. Jelen, D. Lenz, J. Liu, X. Liu,
B. Majorovits, V. Kornoukhov
Consultant: I. Abt



Reminder



- 37.5 kg Enriched Germanium with 87% ^{76}Ge first delivered to Munich, now stored underground in the HADES UGL (Geel).
- 50 kg of depleted GeO_2 also delivered to MPI is being used for purification and crystal pulling tests
- In 2007 new purification test started at PPM Pure Metals (Langelsheim, DE)
- June 2007: first test with depleted Ge completed
- October 2007: start of crystal pulling R&D contract with IKZ
- December 2007: a second purification test completed



Purification tests at PPM



- The first purification test at PPM Pure Metals GmbH (Langelsheim) was performed in May-June 2007. Analysis of the resulting *Ge* metal completed. The goal was to produce 6N material and analyze for possible contaminations
- The test was completed. The results are summarized in a report: GSTR-08-001
- Total yield of 6N material 77%, no isotopic dilution effect, no dangerous contamination levels



Aims of the Second Test



- Purification test combined with underground storage of the Ge in order to minimize cosmic exposure
 - Intermediate storage in a mine around Langelsheim: organized by PPM
 - Ge will be above ground only for processing and transportation
 - will give us a precise estimate of the exposure during the purification
- Maximize the yield of 6N material with a third pass of zone refinement, 80% and above expected
- Test completed in December 2007.



Results of the Second Test



- There were no mass spectrometry measurements this time, the test was focused on increasing the yield
- 7.3 kg of 6N material produced from depleted Ge
- After 3 iterations of Zone-Refinement \approx 90% yield was achieved:

Reduction

| batch | GeO ₂ | expected | Ge metal |
|-------|------------------|----------|----------|
| #1 | 6400 g | 4441 g | 4355 g |
| #2 | 5514 g | 3827 g | 3792 g |

combined yield 98.5 %

Zone-refinement

| Run | input | output > 50 Ω cm | yield |
|-------|--------|-------------------------|-------|
| #1 | 8069 g | 4388 g | 54% |
| #2 | 3651 g | 1867 g | 51% |
| #3 | 1784 g | 1091 g | 61% |
| Total | 8069 g | 7346 g | 91% |

There are 693 g Ge left, which are below 50 Ω cm.



Underground storage



- The problem of underground storage was solved in a nearby mining museum
- Minimizing the time was partial success: transporting the material forth and back is feasible, but PPM requests help (manpower) to do it
- Minimum time above ground was about 7 days, could be no more than 2 days.

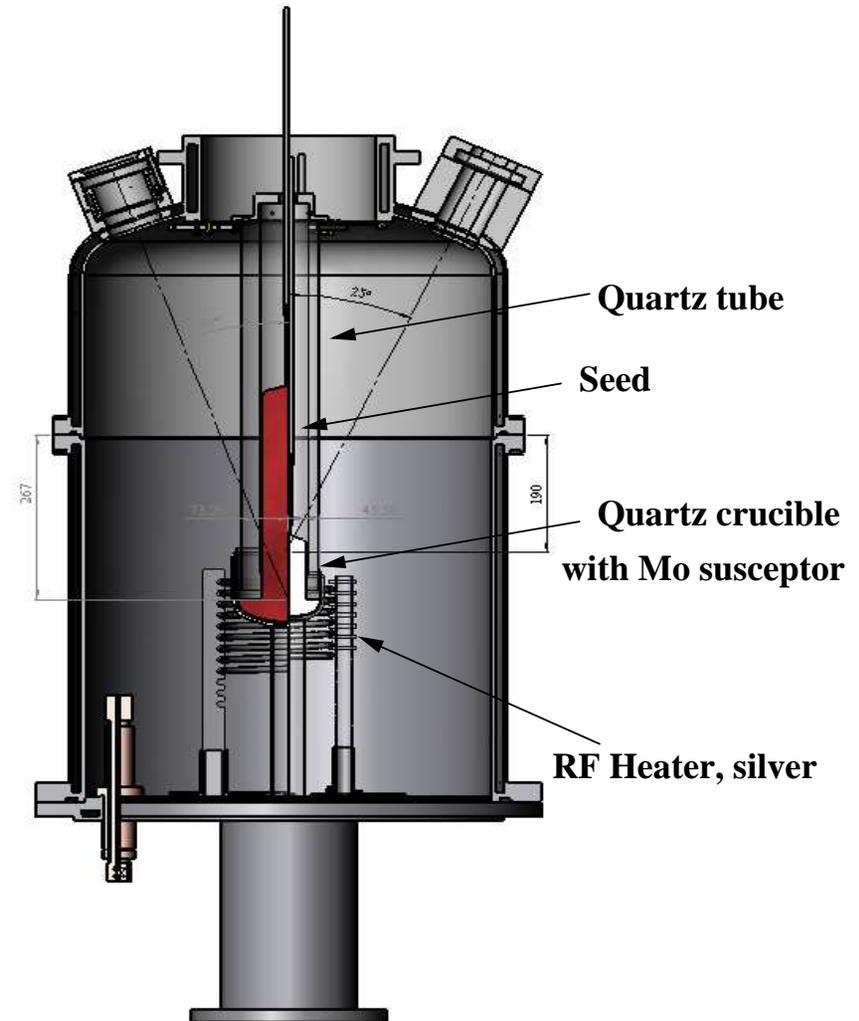
Third test

- The third test will focus on improving the purity to detector grade material
- For planning of the third test we need the results from Hall-effect and PTIS measurements (IKZ, Berkeley)

- Czochralski puller modified for inductive heating (from resistive)
- Vacuum test of the Cz. puller done, up to 10^{-5} mbar
- 4" quartz crucible purchased (for up to 2" crystal) and they are waiting offers for 6" crucibles
- IKZ purchased nat. Germanium and they will start pulling test crystals soon
- IKZ will also help us with the characterization of the crystals



- Molybdenum susceptor, 4" crucible, silver coils, quartz tube delivered
- RF feedthrough assembled
- Copper is replaced with silver in RF coils, no copper inside the puller !
- 4" equipment is delivered, being assembled, first 2" crystals expected within 4 weeks





Crystal pulling - New developments



- there was a delay caused by extended delivering times of the molybdenum susceptor crucible, silver parts (for the RF coil), and manufacturing the coaxial RF feed-through → done ✓
- They have one offer for 6" diameter crucible. Delay only because of the tendering process.
- 6" crucible with Molybdenum susceptor will be delivered in April
- First 3" crystals expected by the end of April
- Successful test of float-zone crystal growing with natural Ge, might be useful for the last purification step (Al removal) and test crystal growth



Crystal characterization (reminder 2007)



- MPI purchased detector grade crystal samples from Canberra and we delivered them to IKZ.
- Hall-effect measurement was done at low temperature (15K) in order to measure the net concentration of electrically active impurities: they found 10^{10} impurities/cm³
- In addition Photothermal Ionization spectroscopy is needed in order to identify the impurities. IKZ will set up a PTIS spectrometer.
- IKZ will grow sample crystals from ZR material from PPM and will help us to achieve detector grade purity



Test of ZR material



- IKZ succeeded to grow (with the pedestal method) a 7 mm thick and about 60mm long crystal from the dep. Ge, directly from the ZR ingot from the first test at PPM
- Hall-effect measurement was already done at 77K. Preliminary results:
 - Measured net concentration of the charge carriers: 10^{11} at the seed side (middle of ZR ingot) but typically 10^{12} impurities/cm³
 - Reminder: detector grade material 10^{10} /cm³
 - ZR Ge is sold as 6N material, we think we have 10N purity. Consistent with resistivity measurement on polycrystalline ZR ingot.
- New float-zone crystal being grown (now). Waiting for results ...
- Samples sent (or will be sent) to Berkeley for confirmation
- PTIS under development: IR lamp, cryogenic equipment upgrade needed for operation at 8K. First results promised for next week



2007 Dec. 13 visit of Prof. Haller (Berkeley) in Munich

- He agreed to analyze our samples (Hall-effect, PTIS)
- He shared with us many technological details about ZR and crystal pulling
- We can test a small radiation detector even before pulling a large crystal



Summary



Since the last collaboration meeting:

- Total yield of 6N (or better) material of **90%** was achieved during ZR
- Test crystal successfully grown from ZR material for Hall-effect and PTIS measurement
- Purity achieved so far (only with ZR) 10^{12} impurities/ cm^3
- Third purification test is planned (depending on the PTIS results)
- Progress has been made with the preparation of the Czochralski puller: first crystals expected in a few weeks