Status Report TG2

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No news on prototype detectors. Focus on Phase II materials.
Recall:

-Have 37.5 kg of ^{enr}Ge (87% ^{76}Ge)
-Next steps:
- -Purification (6N) PPM ?
- -Monozone refinement (8-9N) PPM ?
- -Crystal pulling IKZ ?
- -Detector production (Canberra demonstrated can make high quality 18-fold segmented true coaxial detectors.)

Recall: PPM is candidate for purification of our ^{enr}Ge. Perform tests with ^{dep}Ge to determine yield and isotopic dilution. Contract signed, first tests underway.

Purchased 50 kg of $^{dep}GeO_2$ (compound mass) at start of the year from ECP - 'waste' product from our enrichment.

Minutes of visit to PPM Pure Metals on 25.4.07

Goal: determine yield for 6N quality material, n-type, high resistivity starting with GeO_2 from ECP plant. Starting product is isotopically shifted Ge (depleted in ^{76}Ge) in the form of GeO_2 powder (10.6 kg). Steps:

1. $^{dep}GeO_2$

Take samples from each of six bags, mix, and produce 2 • 1 gm samples for isotopic measurements (TIMS), and 2 • 2 gm samples for ICPMS measurements. Samples should be sent to Dr. Vasily Kornoukhov, INR. ***Done, measurements are under way.***

Determine water content of starting compound. ***0.07%. Material lost on ignition 0.12%.***

PPM visit minutes - continued

2. Reduction (note - no chemical purification, go straight to reduction & sone refinement)

Determine mass of GeO_2 in each boat. Keep track of contents of each boat separately. ***Density of GeO_2 from ECP (0.75 gm/cm^3) almost factor 2 lower than standard. Requires more boats, more time for reduction. Also seen at Germaniy. So far, 7 boats reduced.***

After reduction, resistivity measurement of each regulus with 1 cm spacing. Each regulus should also be weighed. Keep track of mass loss in etching & grinding processes. ***Resistivity low (typically $\leq 1\ \Omega$-cm). Mass loss at per mil level. One boat lost due to malfunctioning of equipment. Decide to zone refine all reduced material independently of resistivity.***

Choose 1 regulus, sample at head, center, tail (about 1 mm thickness)* for ICPMS. Send labeled samples to Dr. Kornoukhov, INR.

Produce 3 • 1 gm samples (head, center, tail) for isotopic measurements (TIMS).

(*) Dimensions of Ge metal sample for SS MS: 2 x (10÷20) x (10÷20) mm, and mass of Ge and GeO2 for ICP MS: 2 gm, for TIMS (isotopic measurement): 1 gm.

PPM visit minutes - continued

3. Zone Refinement

Determine the starting mass in each boat. Label each for bookkeeping purposes. ***So far, two ingots have been zone refined (combination of several bars from reduction). Only one measured.***

Final mass of each ingot after zone refinement.

Resistivity for each ingot, measured at 20^{o} C in 1 cm steps ***See chart***

Sign of conductivity at different points along ingots.

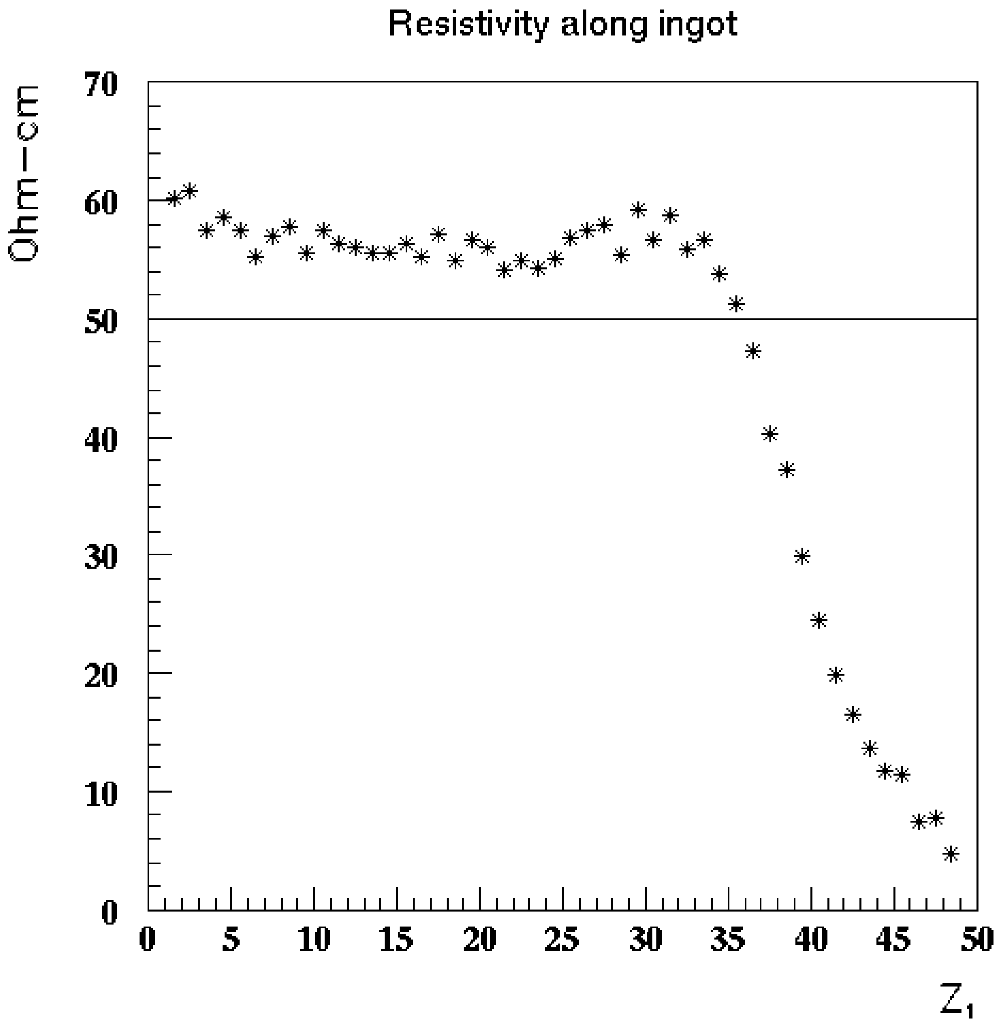
Keep track of time needed for the different steps.

Pick one ingot (in discussion with MPI), sample high resistivity region (>50 Ω-cm) every 2cm (ca. 1 mm)* for ICPMS and Spark Source spectroscopy. Label samples according to location on the ingot and send the Dr. Kornoukhov.

Produce 3 • 1 gm samples (head, center, tail) for isotopic measurements (TIMS).

Determine final mass of high resistivity material, mass of low resistivity material in ingots, and amount of recoverable waste (in principle) from the different steps.

First results:



Yield ~70%. Plan is to combine the low resistivity tails from several ingot an zone refine again.
Germanii found 58% yield for ‘direct reduction’

Conclusion from FSUE Germaniy (2)

1. Yield of Ge metal with > 47 Ohm*cm **is 58%** reached only after poly zone refinement of regulus.
2. Purity of this metal and standard GPZ is the same at DL of ICP MS measurements (we are still waiting for results of SS MS measurement), but concentration of majority carriers is $2*10^{13}$ cm^{-3}.
3. GeO_2 produced according to the ECP technology **<u>is not suitable</u>** for production of GPZ/HP germanium in direct technology

“GeO_2 → reduction&directional crystallization →Ge regulus”

because of:

- low resistivity of regulus ($\rho < 5$ Ohm*cm along a whole ingot (for standard regulus $\rho > 30$ Ohm*cm for L > 70% of total length);
- High concentration of majority charge carriers n = $2*10^{13}$ cm^{-3} (for standard regulus n = $1.5*10^{12}$ cm^{-3}).

2. They propose to carry out “full” cycle of $^{76}GeO_2$ reprocessing, chemistry including. They can tune their equipment and complete “42 kg” experiment during 24 days (12 days for preparation and 12 days for experiment, sampling and certification including).

IKZ visit on 26.4.2007

Milestones:

1. Installation of CZ puller and pulling of first Si crystals, then Ge (15/6/07)
2. Refurbish motor, controls, vacuum, crucible & other graphite parts of CZ puller (1/10/07).
3. Acquire ultra-high purity 6in crucible (1/1/08). ***What is best material for inductively heated crucible (susceptor) - high purity graphite, high purity Mo ?***
4. Demonstrate crystal characterization capability & sensitivity (measurement of crystal axis, charge carrier density as function of T, resistivity at LN temperature, variation in electrical parameters, dislocation density, photothermal ionization or photoluminescence to measure shallow depth impurities). MPI will acquire samples of high purity Ge crystals (1/8/07).
5. Set up hall for Hydrogen atmosphere in CZ ? Determine if forming gas adequate (1/2/08). ***Idea is to use Ar+2%H_2 mixture instead of pure H_2. Issue is whether pure H_2 is needed. According to Haller, need dislocations to capture H_2. H_2 used as buffer gas to capture impurities so not gettered in Ge melt.***
6. Demonstrate that crystals of desired dimensions can be pulled (1/2/08).
7. Acquire 6N material, fully characterize before pulling. Then pull crystal, and demonstrate improved in purity (2 N) and net charge carrier density (1 order of magnitude) with yield 30% (1/8/08).
8. Starting with monozone refined material of specified properties (purity, resistivity, charge carrier density, …), grow three crystals with all specified characteristics for detector grade (1/10/08).
9. Make working detector at Canberra (1/1/09)

EKZ 2000, LEYBOLD, 1983

Ready for first test runs with Si in 14 inch crucible
Then first test runs with Ge

20 kg of 6N ^{nat}Ge ordered

Numerical simulation of thermal fields for Ge growth are in progress. First results (heat consumption) in the next days

After mid-August:

- state-of-the-art electronic control will be installed
- new drive motors, oil-free turbo pump, process gas supply system
- installation and implementation of 40 kW, 15 kHz RF generator and heating coil
- inductive heating chosen upon our advice (E. Haller writes best experience)
- video camera based diameter control system under development
- steps should be ready by 1/10/07

E. Haller will visit in later October, November