Status of Phase II Detector production TG02

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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
- Previous purification test at FSUE Germaniy (Russia):
  - Total yield of high purity material 58%, in ZR 72% yield
  - Isotopic dilution effect was seen in ”wet” chemistry procedure, no dilution during reduction and ZR
- New purification test started at PPM Pure Metals (Langelsheim, DE)

First purification test 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.
- For the test 10.6 kg of depleted GeO_2 was used (leftover of the enrichment)
- The purity was measured with mass spectrometry methods and resistivity measurements were done
- Isotopic content was measured after each phase of the processing
- Main steps of the purification:
  - Reduction: In H_2 atmosphere and at high temperature the GeO_2 is reduced to metallic Ge
  - Mono-zone refinement (ZR): A molten zone is pulled over (slowly) the Ge ingot

Analysis after reduction

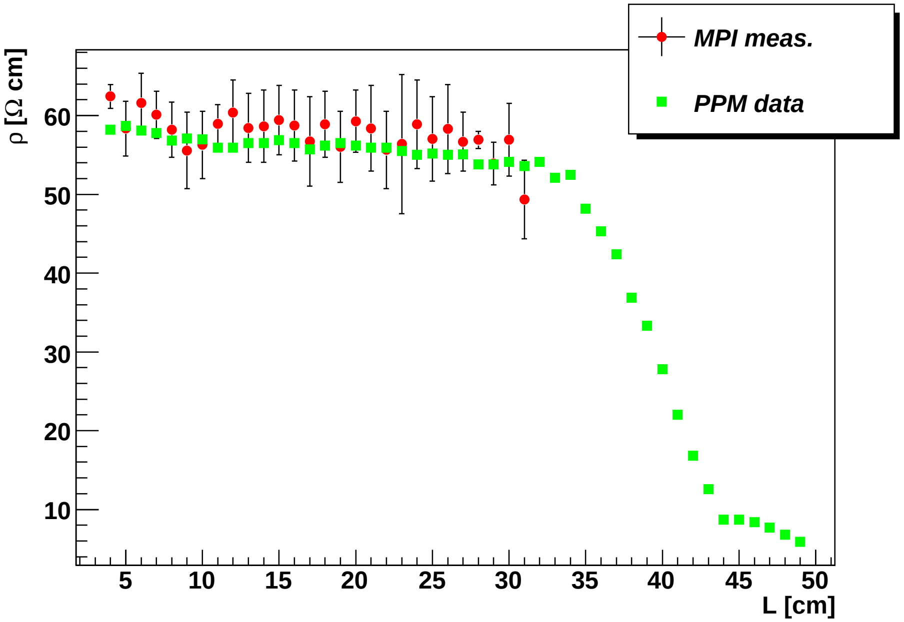
- Samples of GeO_2 and Ge metal (after reduction) were sent to Russia for two different MS measurements: Spark Source Mass Spectrometry (SSMS) and Inductively Coupled Plasma Mass Spectrometry (ICP-SM)
- Conclusion: no serious contamination, good quality starting material (4N or better).
- Samples for isotopic content measurement were taken.
- Resistivity measurements: in avarage 1 Ωcm, far from the intrinsic resistivity of 50 Ωcm
- Resulting Ge metal melted to fill a reduction ”boat“ and PPM started the zone-refinement

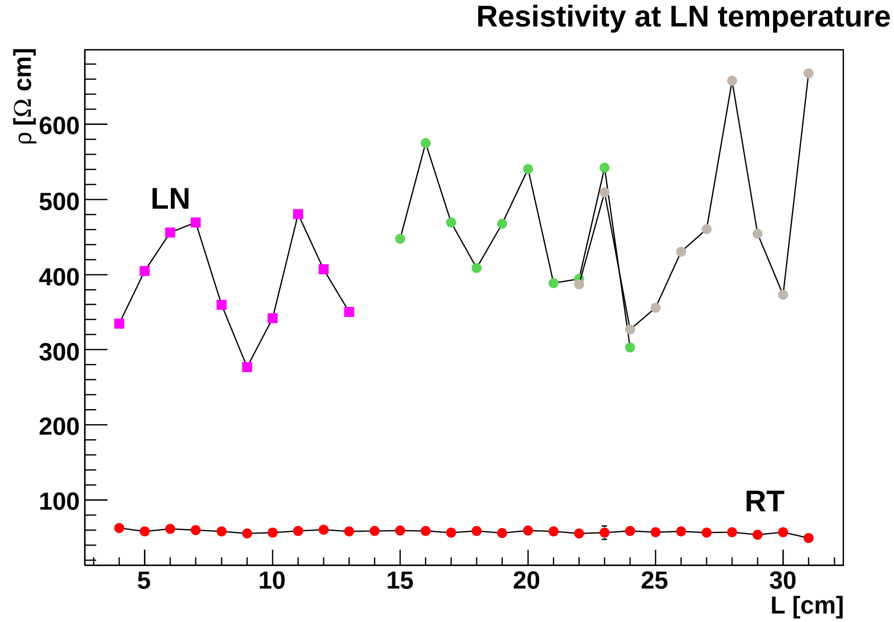
Example: results of ICP-MS

Element	DL ppm	GeO_2	1/4	2/4	3/4	Element	DL ppm	GeO_2	1/4	2/4	3/4
Li	0,006	<DL	<**0.01**	<DL	<DL	Sb	0,03	<DL	<DL	<DL	<DL
Be	0,0005	<DL	<DL	<DL	<DL	Te	0,006	<**0.02**	<DL	<DL	<DL
B	0,1	<DL	<DL	<DL	<DL	Cs	0,001	<DL	<DL	<DL	<DL
Na	20	<DL	<DL	<DL	<DL	Ba	0,5	<DL	<**7**	<**1.8**	<DL
Mg	1	<DL	<DL	<DL	<DL	La	0,003	<DL	<DL	<DL	<DL
Al	2	<DL	<DL	<DL	<DL	Ce	0,01	<DL	<DL	<DL	<DL
K	6	<DL	<DL	<DL	<DL	Pr	0,001	<DL	<DL	<DL	<DL
Ca	7	<**10**	<DL	<DL	<DL	Nd	0,002	<DL	<DL	<DL	<DL
Sc	0,02	<DL	<DL	<DL	<DL	Sm	0,0003	<DL	<DL	<DL	<DL
Ti	0,4	<DL	<DL	<DL	<DL	Eu	0,0002	<DL	<DL	<DL	<DL
V	0,5	<DL	<DL	<DL	<DL	Gd	0,0009	<DL	<DL	<DL	<DL
Cr	2	<DL	<DL	<DL	<DL	Tb	0,0001	<DL	<DL	<DL	<DL
Mn	0,2	<DL	<DL	<DL	<DL	Dy	0,0003	<DL	<DL	<DL	<DL
Fe	5	<DL	<DL	<DL	<DL	Ho	0,0001	<DL	<DL	<DL	<DL
Co	0,02	<DL	<DL	<DL	<DL	Er	0,0001	<DL	<DL	<DL	<DL
Ni	0,3	<DL	<DL	<DL	<DL	Tm	0,0001	<DL	<DL	<DL	<DL
Cu	0,2	<DL	<DL	<DL	<DL	Yb	0,0005	<DL	<DL	<DL	<DL
Zn	2	<DL	<DL	<DL	<DL	Lu	0,0002	<DL	<DL	<DL	<DL
Ga	0,1	<DL	<DL	<DL	<DL	Hf	0,01	<DL	<DL	<DL	<DL
As	0,2	<DL	<DL	<DL	<DL	Ta	0,01	<DL	<DL	<DL	<DL
Se	0,3	<DL	<DL	<DL	<DL	W	83	<DL	<DL	<DL	<DL
Rb	0,009	<DL	<DL	<DL	<DL	Re	0,003	<DL	<DL	<DL	<DL
Sr	0,04	<DL	<DL	<DL	<DL	Ir	0,0004	<DL	<DL	<DL	<DL
Y	0,001	<DL	<DL	<DL	<DL	Pt	0,06	<DL	<DL	<DL	<DL
Zr	0,02	<DL	<DL	<DL	<DL	Au	0,01	<DL	<DL	<DL	<DL
Nb	0,02	<DL	<DL	<DL	<DL	Hg	0,6	<DL	<DL	<DL	<DL
Mo	0,07	<DL	<DL	<DL	<DL	Tl	0,0002	<DL	<DL	<DL	<DL
Rh	0,01	<DL	<DL	<DL	<DL	Pb	0,2	<DL	<DL	<DL	<DL
Pd	0,04	<**0.35**	<DL	<DL	<DL	Bi	0,002	<DL	<DL	<DL	<DL
Ag	0,04	<DL	<DL	<DL	<DL	Th	0,0006	<DL	<DL	<DL	<DL
Cd	0,004	<DL	<DL	<DL	<DL	U	0,0002	<DL	<DL	<DL	<DL

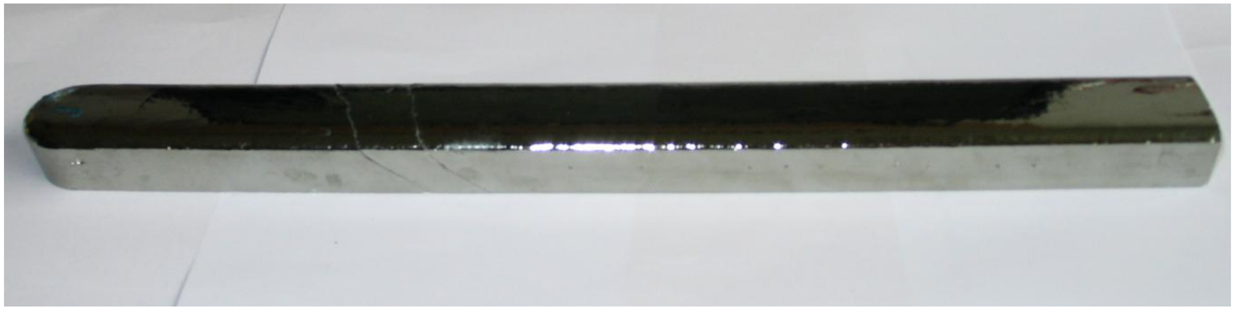
Analysis of the ZR material

- According to the specification given by PPM, the purity of the ZR material is 6N or better. Material with purity better than 6N cannot be analyzed only with MS
- Resistivity measurement: intrinsic resistivity of pure Ge is around 50 Ωcm $\Longrightarrow$ if the resistivity at room temperature is 50 Ωcm than the purity is 6N or better. ZR ingots are cut where the resistivity drops below 50 Ωcm
- Yield of high resistivity material is 60%
- The low resistivity tails are cut off and ZR once more $\Longrightarrow$ Total yield of 77%
- Estimated from the resistivity measurement at 77K, the net concentration of electrically active impurities is around $10^{11}\ \frac{imp}{cm^3}$, only one order of magnitude higher than the detector grade material

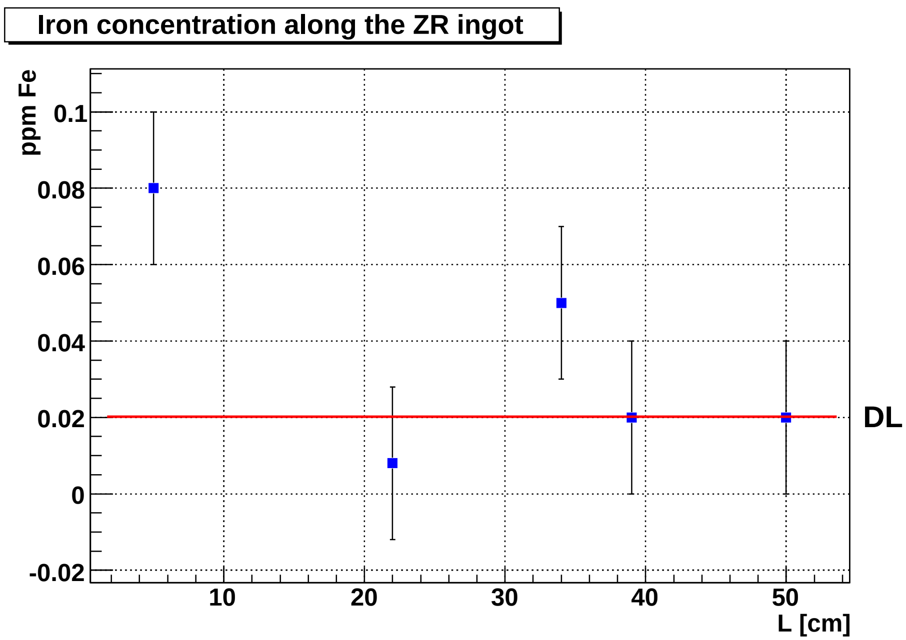




Resistivity at room temperature (left) and at LN temperature (right)



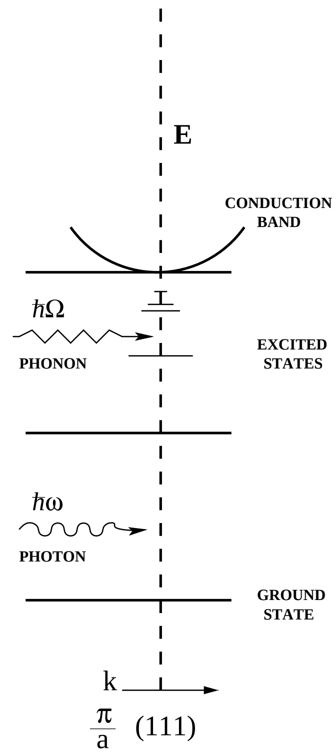
- MS measurements are difficult to interpret:
  - the two measurements show higher contamination for the samples from the low resistivity tail
  - they disagree on the quantity
  - difficult to follow the effect of ZR on one particular element

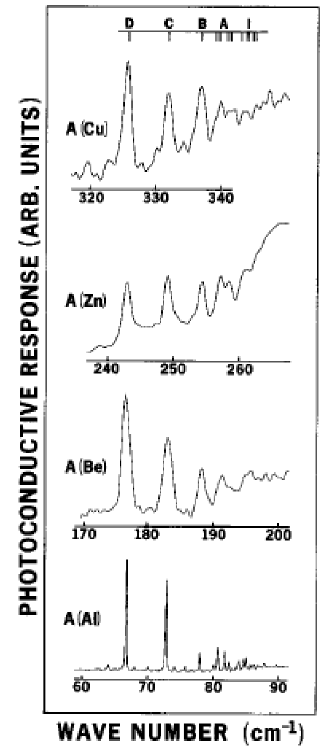


Element	ppm weight	Element	ppm weight	Element	ppm weight
H	ND	Zn	<0.02	Pr	<0.05
Li	<0.001	Ga	<0.01	Nd	<0.2
Be	<0.001	Ge	OCHOBA	Sm	<0.1
B	<0.001	As	<0.1	Eu	<0.04
C	ND	Se	<0.2	Gd	<0.2
N	ND	Br	<0.1	Tb	<0.1
O	ND	Rb	<0.1	Dy	<0.3
F	0.4	Sr	<0.2	Ho	<0.06
Na	<0.02	Y	<0.1	Er	<0.04
Mg	<0.01	Zr	<0.2	Tm	<0.05
Al	0.03	Nb	<0.1	Yb	<0.2
Si	0.02	Mo	<0.3	Lu	<0.1
P	<0.005	Ru	<0.1	Hf	<0.2
S	0.04	Rh	<0.05	*Ta	ND
Cl	ND	Pd	<0.3	W	<0.2
K	<0.02	Ag	<0.05	Re	<0.1
Ca	<0.01	Cd	<0.2	Os	<0.4
Sc	<0.01	In	<0.05	Ir	<0.2
Ti	<0.01	Sn	<0.1	Pt	<0.2
V	<0.05	Sb	<0.05	Au	<0.1
Cr	<0.01	Te	<0.4	Hg	<0.3
Mn	<0.01	I	<0.05	Tl	<0.1
Fe	0.05	Cs	<0.1	Pb	<0.3
Co	<0.01	Ba	<0.2	Bi	<0.1
Ni	<0.05	La	<0.1	Th	<0.1
Cu	<0.02	Ce	<0.1	U	<0.1

Sample **4.3**, ^{dep}Ge after ZR at PPM Pure Metal, *Ta is material of the ion source.

- Hall-effect measurement can give the exact number of impurities and PTIS (Photo-thermal ionization spectroscopy) can identify the chemical elements
- Photothermal ionization setup is being prepared at IKZ, they will perform the measurements and we were promised help from Berkeley
- Single crystal is needed for this measuremenmts, they will be grown at IKZ





Isotopic composition

- Isotopic composition was measured after each step of the processing with surface ionization mass-spectrometer at the Institute of Microelectronics Technology and High Purity Materials RAS (Chernogolovka, Moscow).
- No isotopic dilution effect was observed at the level of $\pm$ 0.01% (accuracy of measurements).

	Ge1a	Ge1b	Ge2b	Ge3b	Ge4b	Ge_i1	Ge_i3	Ge_i4	Ge n
70	22.8	22.7	22.8	22.8	22.8	22.74	22.75	22.70	21.2
72	30.1	30.0	30.00	30.00	30.00	30.07	30.05	30.08	27.8
73	8.32	8.30	8.33	8.33	8.32	8.32	8.30	8.29	7.75
74	38.2	38.4	38.3	38.3	38.3	38.27	38.30	38.34	35.9
76	0.59	0.60	0.59	0.59	0.60	0.60	0.60	0.59	7.35

Ge1a and Ge1b are depleted GeO2, Ge2b - Ge4b are depleted Ge metal after reduction, samples Ge_i1 - Ge_i4 are Ge metal after zone-refinement, Ge n - natural germanium

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
- Test should have been already done; waiting for news from PPM
- A third test is planned to improve the purity, will be a function of the previous results

Crystal pulling

Last meeting at Institut für Kristallzüchtung (IKZ) 01.10.2007. Summary:

- 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, results coming soon ...

Crystal characterization

- 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 concentration of electrically active impurities: they found 10^{10}impurities/cm^3
- In addition Photothermal Ionization spectroscopy is needed in order to identify the impurities
- IKZ will grow sample crystals from ZR material from PPM and will help us to achieve detector grade purity

Summary

- After zone-refinement 6N purity (or better) was achieved
- Yield of 77% achieved after two steps of ZR (60% in one step). Further improvement still possible with more iterations.
- We have ongoing discussions (negotiations) about fine tuning of the ZR for improving yield and purity and reducing cosmic exposure
  - A second test is being done now to test the time needed above ground
  - Third test with depleted Ge is planned for improving the purity
- We are studying different measurement methods below the detection limit of mass spectrometry methods
- Sample crystals for analysis will be grown at IKZ (Berlin)
  - Setting up PTIS and Hall-effect measurements