

Status of Phase II Detector production TG02



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Reminder



- April 2006: 37.5 kg Enriched Germanium with 87% ^{76}Ge first delivered to Munich, now stored underground in the HADES UGL (Geel).
- April 2006: 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.
- Second test August-December 2007
- Both tests were completed. The results are summarized in a report: GSTR-08-001
- After 3 steps of ZR total yield of 6N material 90%, no isotopic dilution effect, no dangerous contamination levels
- Solution was found for underground storage during purification
- Remaining 27kg of depleted Ge zone-refined at PPM (almost done)
- We are negotiating the reduction and zone-refinement of the enriched material



Crystal growing



Last meeting at IKZ 29.10.2008

- Since last collab. meeting 6 new crystals produced:
 - CZ4: from PPM material
 - CZ6: from 6N standard Ge
 - CZ7: The cleaner part of two previous crystals recycled
 - FZ-V3105: Float-zone crystal from PPM material
 - Ge-409: grown with a different Cz. puller
 - CZ8: from PPM material and using Ultra High Purity crucible
- They were all measured with Hall-effect and PTIS at IKZ



Crystal characterization



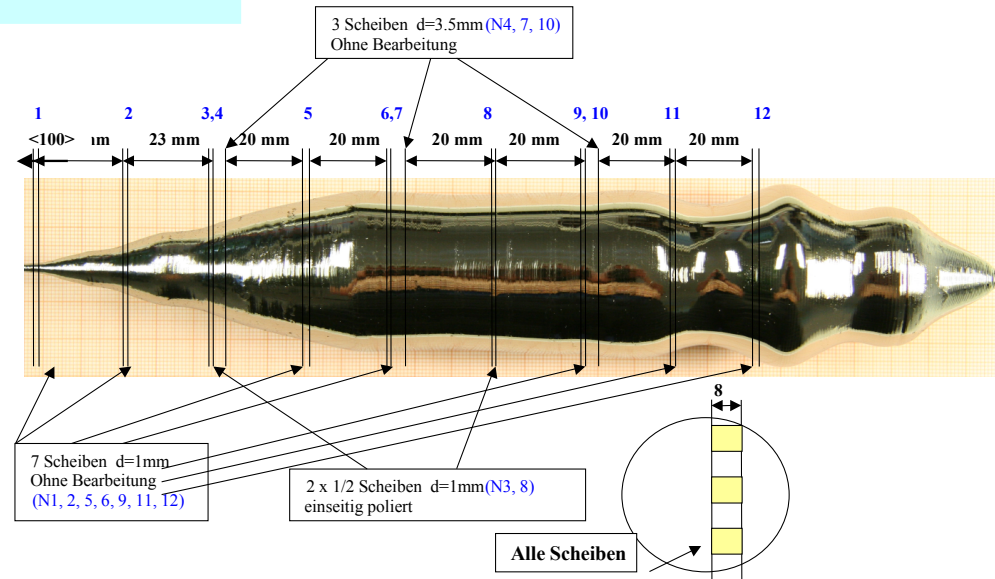
- CZ4 (PPM material) was cut every 2cm to produce a longitudinal impurity profile
- 2-3 samples cut from all other crystals
- Analysis completed:
 - All Cz. crystals have an impurity level typically of $10^{13}/\text{cm}^3$
 - Float zone crystal seed end has 10^{11} impurity/ cm^3
- Samples from CZ4 analyzed in Dresden with Photo-Luminescence spectroscopy
- Samples are prepared for further measurements at IKZ and Dresden

PPM –Material (3 experiments)

Crystal - CZ4 (Ge-HP-4)

Crucible – qsil PN

Atmosphere – Ar+2%H₂

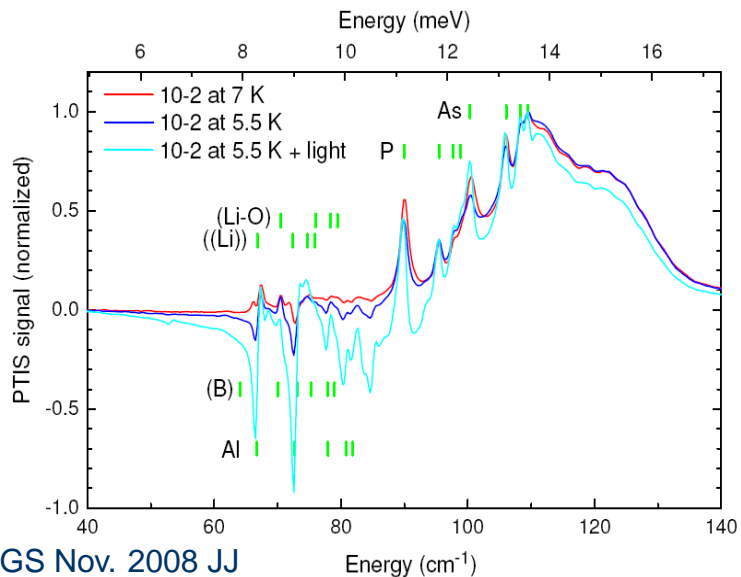
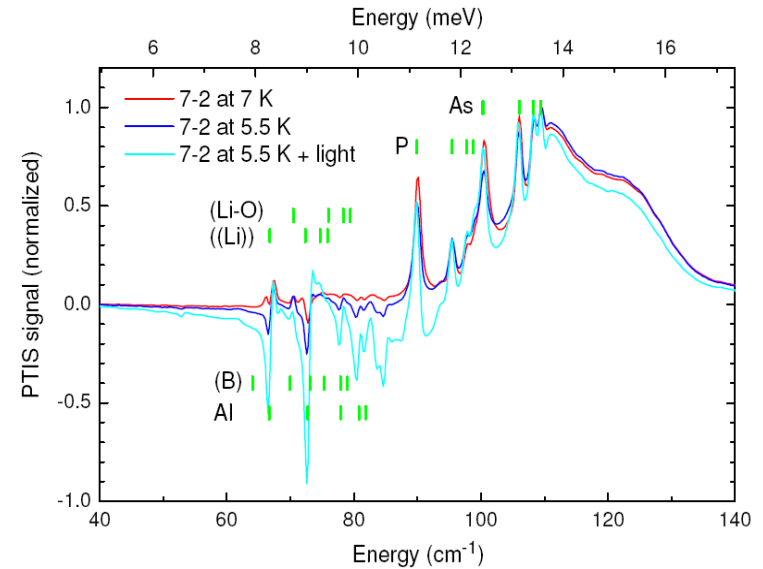
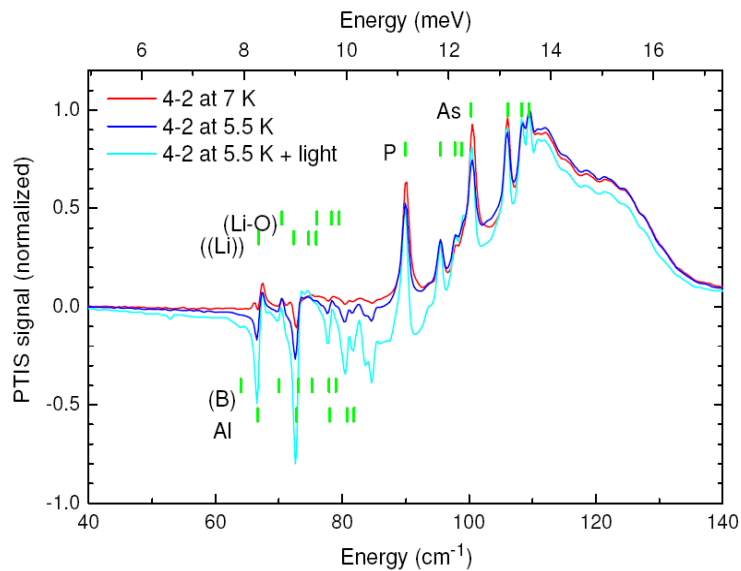


Conductivity and Hall effect results:

| | Resistivity (Ωcm) | | Electron conc. (10 ¹³ cm ⁻³) | | Mobility (cm ² /Vs) | |
|--------------------|-------------------|-------------|---|-------------|--------------------------------|-------------|
| <i>Temperature</i> | 297 K | 77 K | 297 K | 77 K | 297 K | 77 K |
| CZ4_1-2 | 46.9 | 11.8 | 5.20 | 1.44 | 2561 | 36600 |
| CZ4_2-2 | 51.6 | 11.5 | 4.14 | 1.50 | 2921 | 36090 |
| CZ4_3-2 | 54.3 | 9.7 | 3.55 | 1.78 | 3238 | 36190 |
| CZ4_5-2 | 44.2 | 7.8 | 4.60 | 2.22 | 3066 | 36120 |
| CZ4_6-2 | 42.7 | 6.9 | 4.60 | 2.58 | 3182 | 35100 |
| CZ4_8-2 | 30.2 | 4.3 | 6.36 | 4.11 | 3246 | 34970 |
| CZ4_9-2 | 25.6 | 3.2 | 6.89 | 5.57 | 3539 | 34620 |
| CZ4_11-2 | 13.4 | 1.6 | 12.3 | 12.24 | 3772 | 32170 |
| CZ4_12-2 | 5.8 | - | 45.3 | - | 2366 | - |

Crystal - CZ4 (Ge-HP-4), PTIS

Photothermal ionization spectroscopy



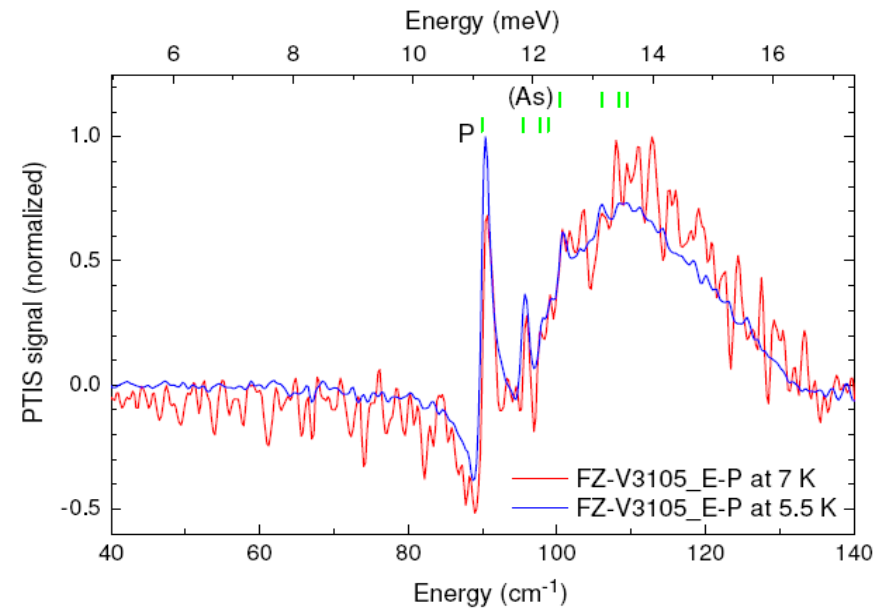
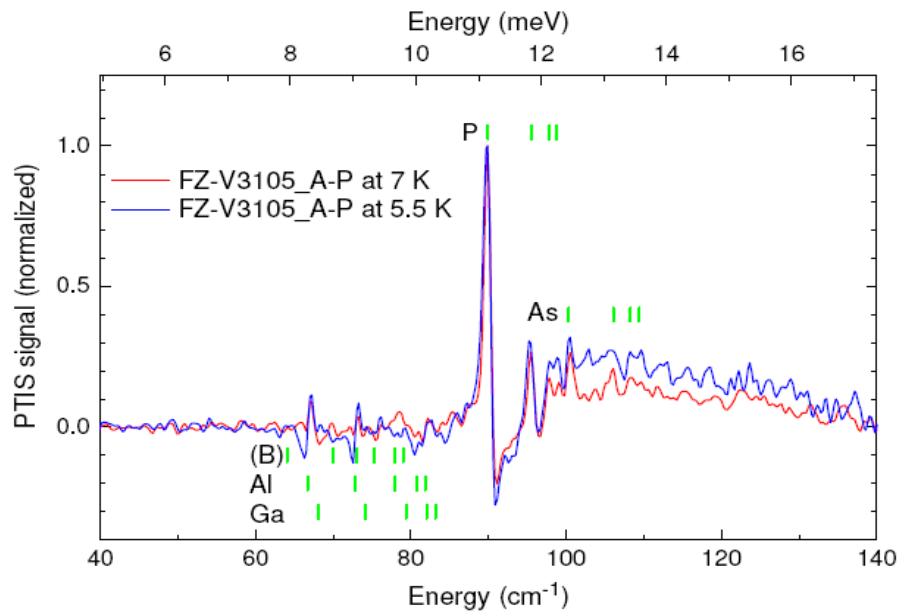
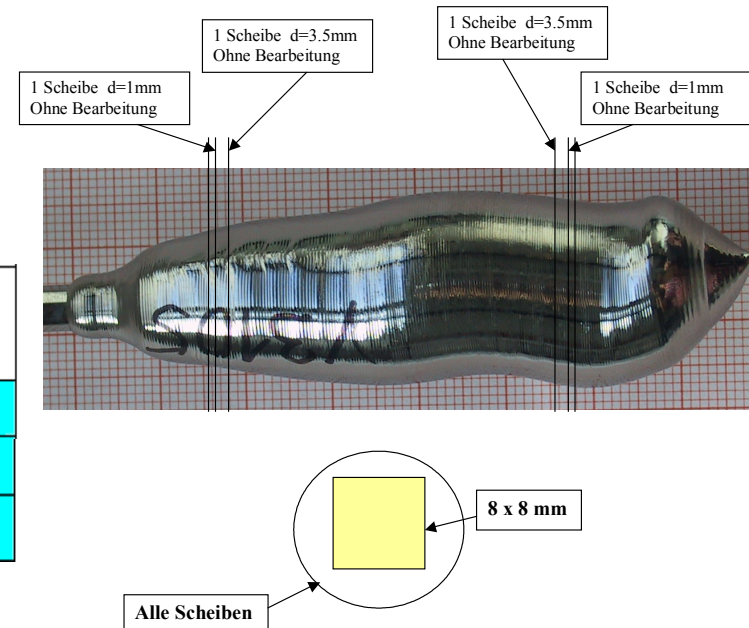
Results

- (i) The main donor impurities in this crystal are phosphorous and arsenic with a concentration ratio $[P]/[As] \approx 1$ in sample 4-2 that seemingly changes to $[P]/[As] \approx 2$ in sample 10-2. Additionally, there may be traces of lithium (isolated lithium donor and lithium-oxygen complex).
- (ii) Compensating acceptors are aluminum and, to a much lesser extent, boron.
- (iii) The net donor concentration $N_D - N_A (= [P] + [As] + [Li] + [Li-O] - [Al] - [B])$ increases from about $1.4 \times 10^{13} \text{ cm}^{-3}$ near the crystal's seed to $1.2 \times 10^{14} \text{ cm}^{-3}$ near the tail (sample 11-2).

PPM –Material (3 experiments)

Ge-FZ-V3105

| | Resistivity (Ωcm) | | Electron conc. (10^{13}cm^{-3}) | | Mobility (cm^2/Vs) | |
|---------------|--------------------------------------|------|---|------|---|-------|
| Temperature | 297 K | 77 K | 297 K | 77 K | 297 K | 77 K |
| Ge-FZ-V3105_A | 57,9 | 3379 | 7,18 | 0,01 | 569 | 25130 |
| Ge-FZ-V3105_E | 49 | - | 12,9 | - | 990 | - |



- main impurities: $[P] \gg [As], [Al]$, (traces of B, Ga)
- low concentration $N_D - N_A = 1 \times 10^{11}\text{cm}^{-3}$ near the seed
- no reliable Hall measurement at the crystal tail end up to now

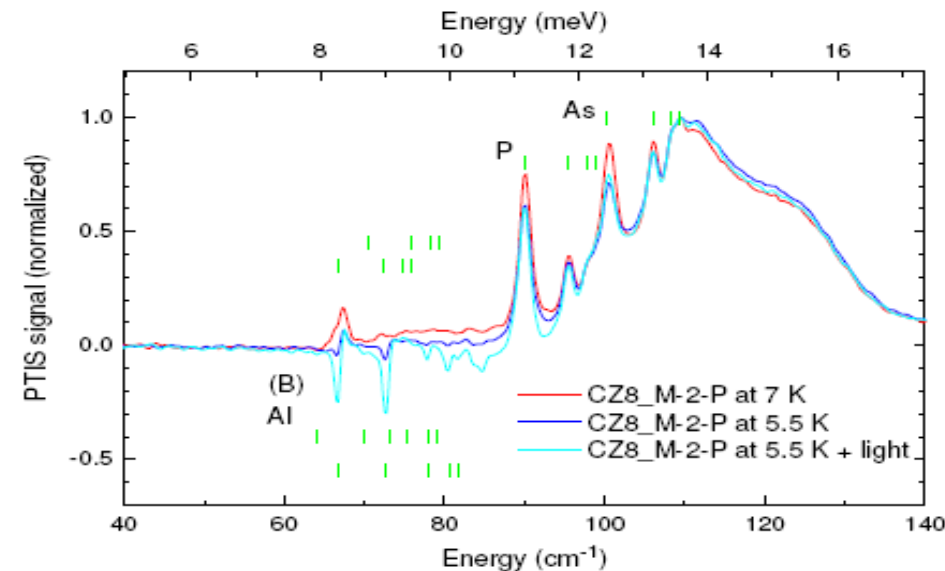
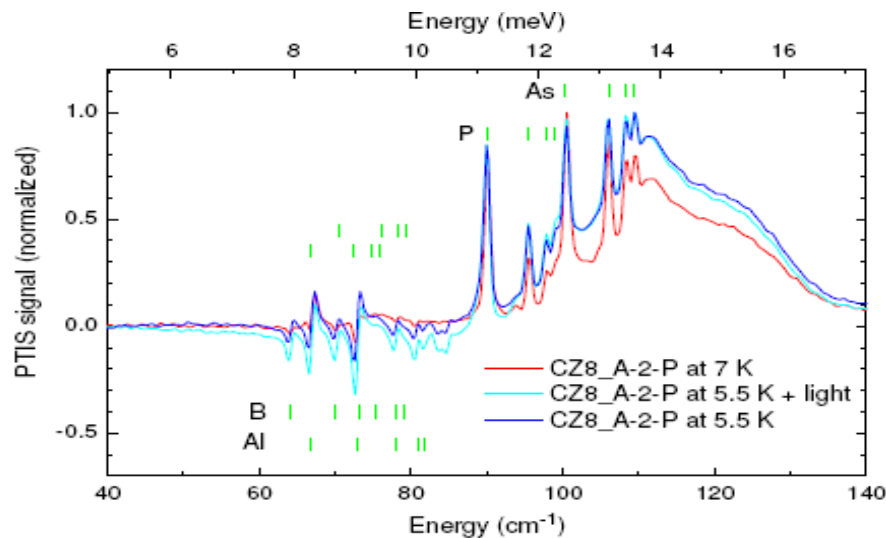
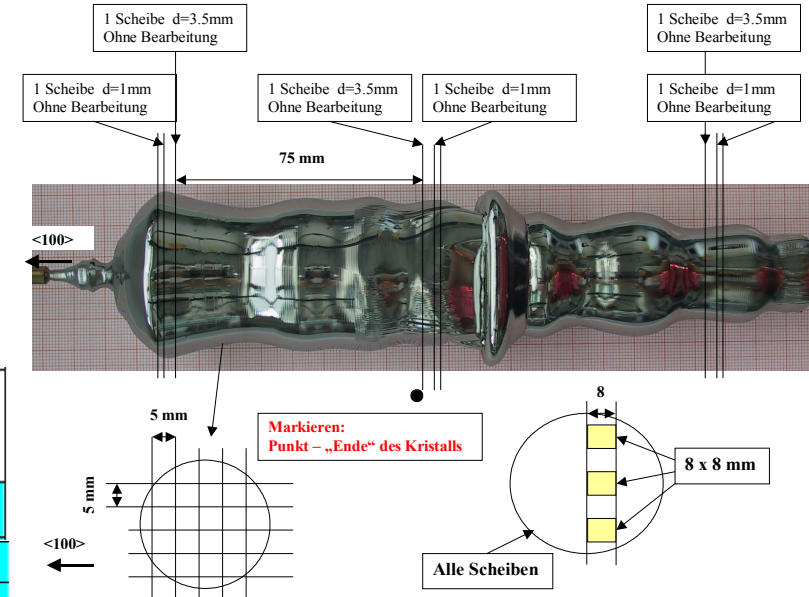
PPM –Material (3 experiments)

Crystal – CZ8 (Ge-HP-8)

Crucible – suprasil

Atmosphere – Ar

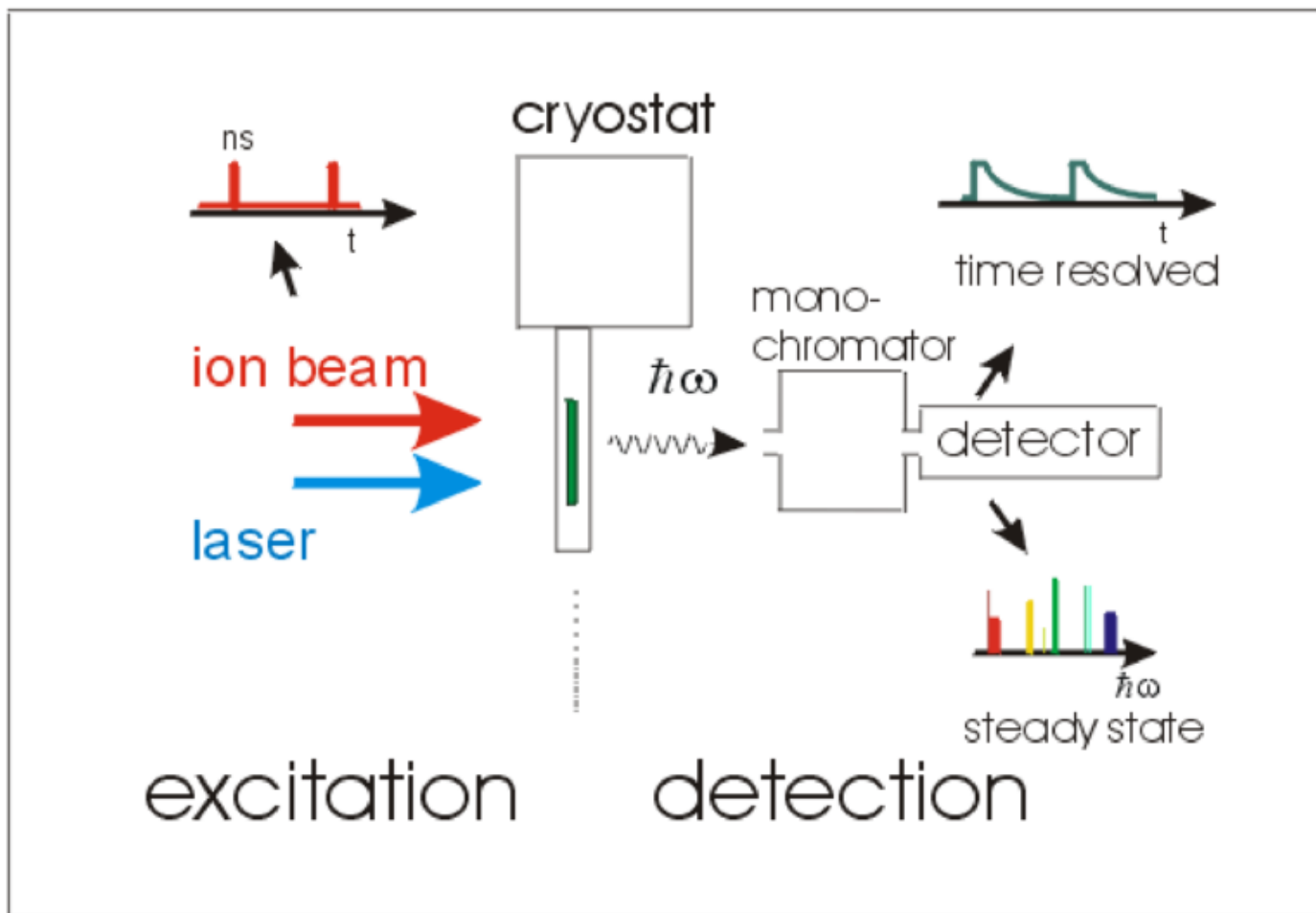
| | Resistivity (Ωcm) | | Electron conc. (10^{13}cm^{-3}) | | Mobility (cm^2/Vs) | |
|-------------|---------------------------------------|------|---|------|---|-------|
| Temperature | 297 K | 77 K | 297 K | 77 K | 297 K | 77 K |
| Ge-CZ8_A-2 | 50,8 | 21,1 | 5,5 | 0,81 | 222 | 36500 |
| Ge-CZ8_M-2 | 37,1 | 6,29 | 5,57 | 2.97 | 3020 | 33400 |
| Ge-CZ8_E-2 | nicht messbar / elektrisch inhomogen! | | | | | |



- main impurities: $[P] \geq [As]$; $[Al] > [B]$
- $N_D - N_A = 8 \times 10^{12}\text{cm}^{-3}$ near the seed
- $N_D - N_A = 3 \times 10^{13}\text{cm}^{-3}$ near axial center
- no results near the tail due to electrical inhomogeneity

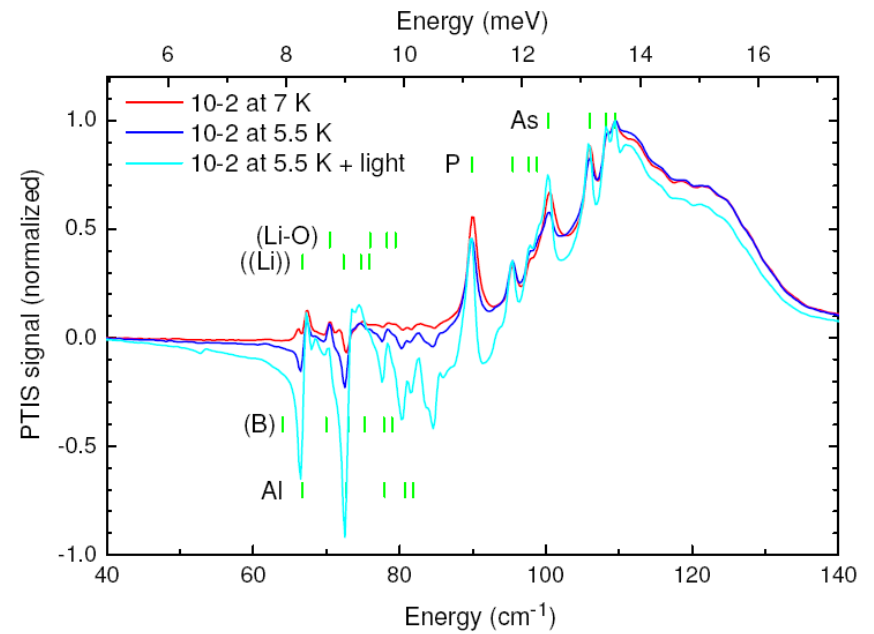
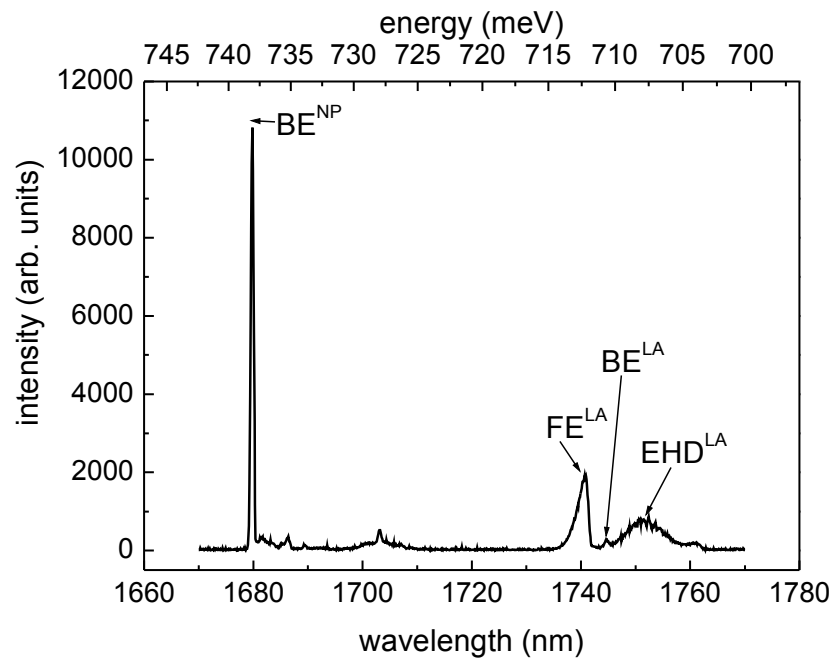


Photoluminescence





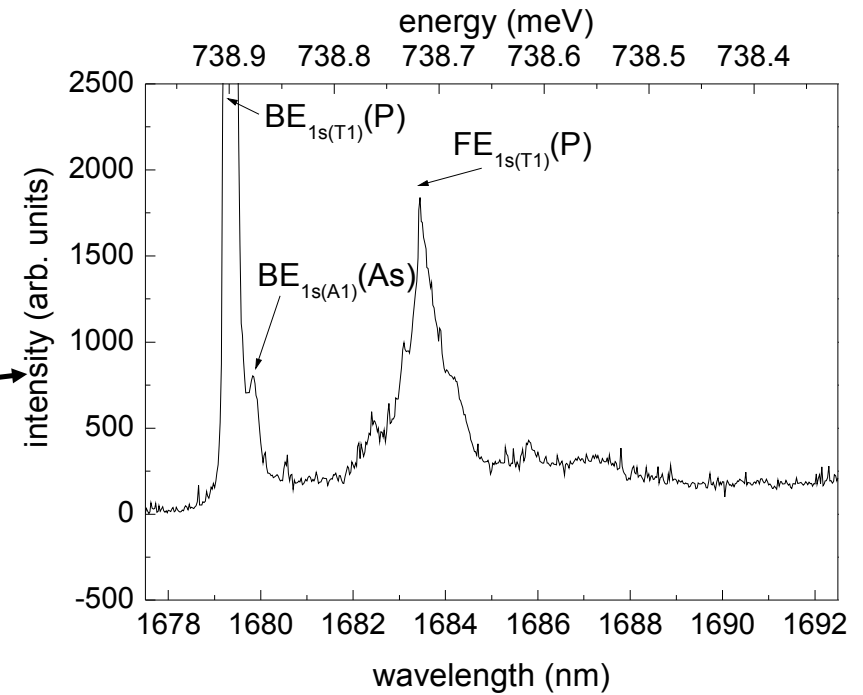
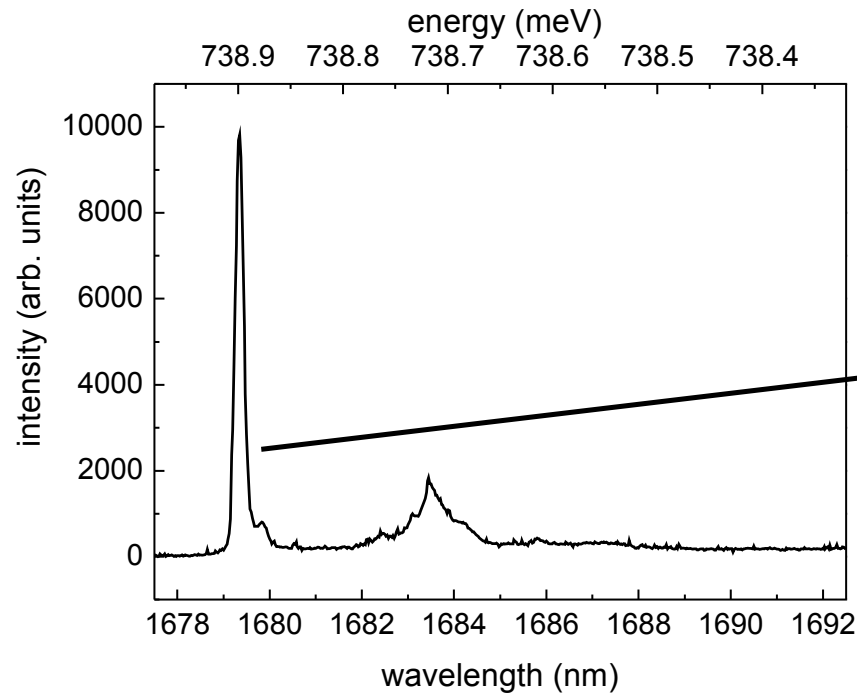
PL vs. PTIS



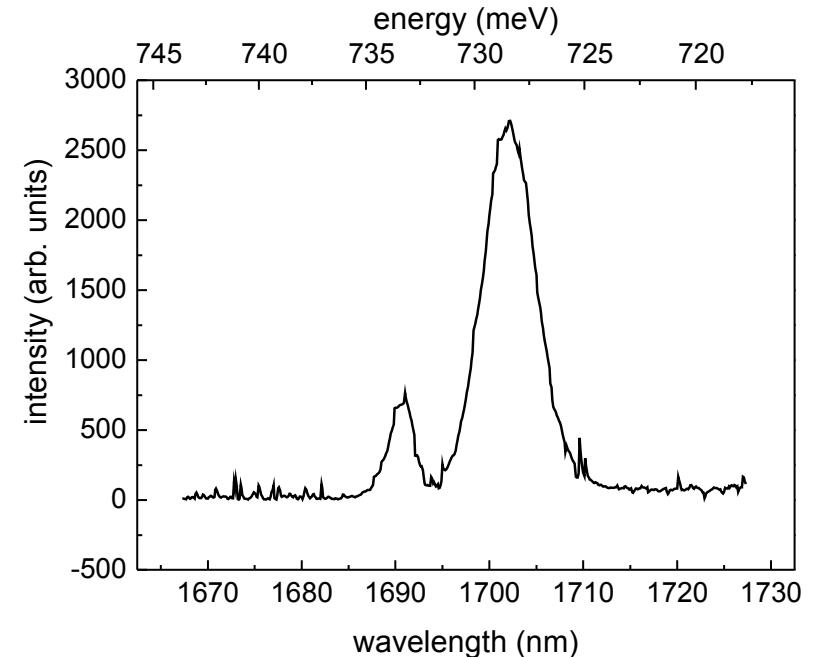
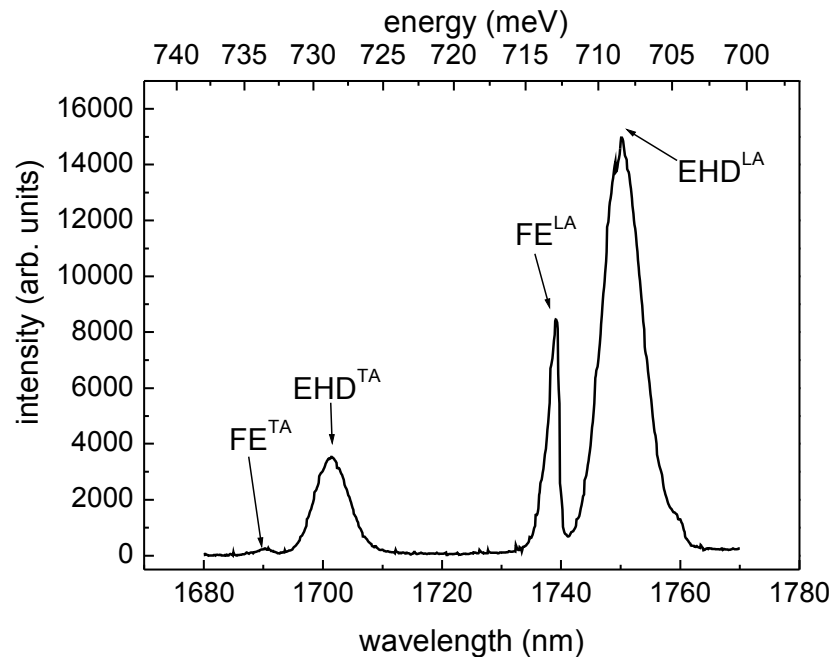
TU Dresden, 04/11/08

slide 5

Sample Ge#4-7/3



→ Phosphorous exciton much stronger than arsenic exciton (ratio 10:1)



→ No bound excitons visible → low impurity concentrations

→ very pure material



Crystal pulling - near future - conclusion



- Many crystals pulled and analyzed
- The source of impurities identified: is the Czochralski puller itself
- IKZ will upgrade the Cz. puller. First results in 4-6 weeks
- New analytical method from TU Dresden
- More detailed analysis of the existing crystals being done
- We are planing to make a small detector from the float-zone crystal
- The goal of having detector grade crystals in early 2009 is unlikely but still within reach