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
- November 2008: third 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
- Third test: the remaining 27kg of depleted Ge zone-refined at PPM: finished Nov.2008
- We are preparing the reduction and zone-refinement of the enriched material

Results of the third test

- Goal: provide IKZ with enough 6N Ge to test crystal pulling
- 26710 g of depleted GeO2 reduced and zone-refined
- Yield after reduction = 99.3 %
- Total yield after three steps of zone-refinement = 83.9% (15554 g 6N metal)

Run	$> 50\Omega$cm (g)	yield
1	7919	43.0%
2	5172	49.3%
3	2463	46.3%
total	15554	83.9%

- Remaining material from Test 1. recycled with all the tails
- At the end 21.3 kg 6N germanium produced and shipped to IKZ

Summary of Reduction and Zone-refinement

	GeO_2 (g)	69.73%	Ge metal	6N Ge	ZR yield	Total yield
Test 1	10609.6	7398	6333	4913	77.57%	66.4%
Test 2	11918.9	8311	8147	7346	90.16%	88.38%
Test 3	26689.8	18610	18416	15554	84.46%	83.57%
Test 3′	–	–	7843	5870	74.84%	–
Total	49218.3	34319.9	32896	30212*	91.84%	88.03%
left	781.67	–	–	–	–	–
Total	49999.97	–	–	–	–	–

- almost all 30 kg 6N germanium handed over to IKZ
- 780 g (a small bag) GeO_2 left for later sampling
- 400 g 6N germanium is still in Muenchen
- without the reduction that went wrong total yield would be 90.6%

Reduction of the enriched material

03.03.2009 we had a meeting at PPM in Langelsheim.
Contract for the enriched material being prepared.

- PPM will guarantee an 80% yield + we pay a bonus for every kg or % above
- The material will be insured while at PPM
- Underground intermediate storage organized by PPM
- Precise book keeping is required
- Possible starting dates middle of April or July
- Transport to and from Langelsheim will be done by MPI

Crystal pulling status

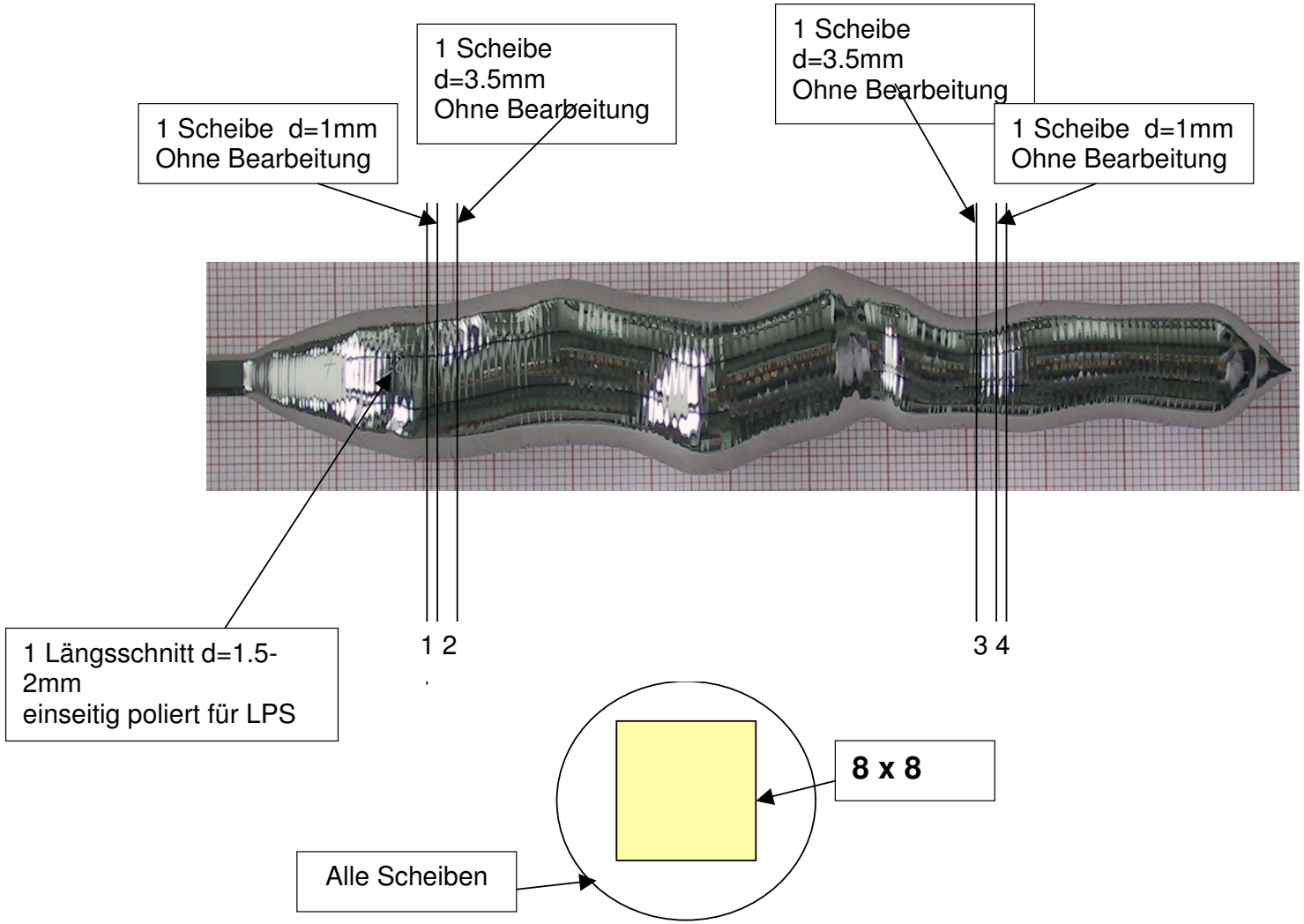
A dedicated Czochralski puller is set up at IKZ, Berlin. Fully operational, many components have been modified:

- Operated with success first on April 7, 2008
- 10 successful crystal pulling test already done
- Purity of the crystals typically 10^{13} impurity/cm^3 (detector grade 10^{10})
- Improvments are made continuously: quartz tube to protect the melt, gas purification system and more to come ...

Crystal growing

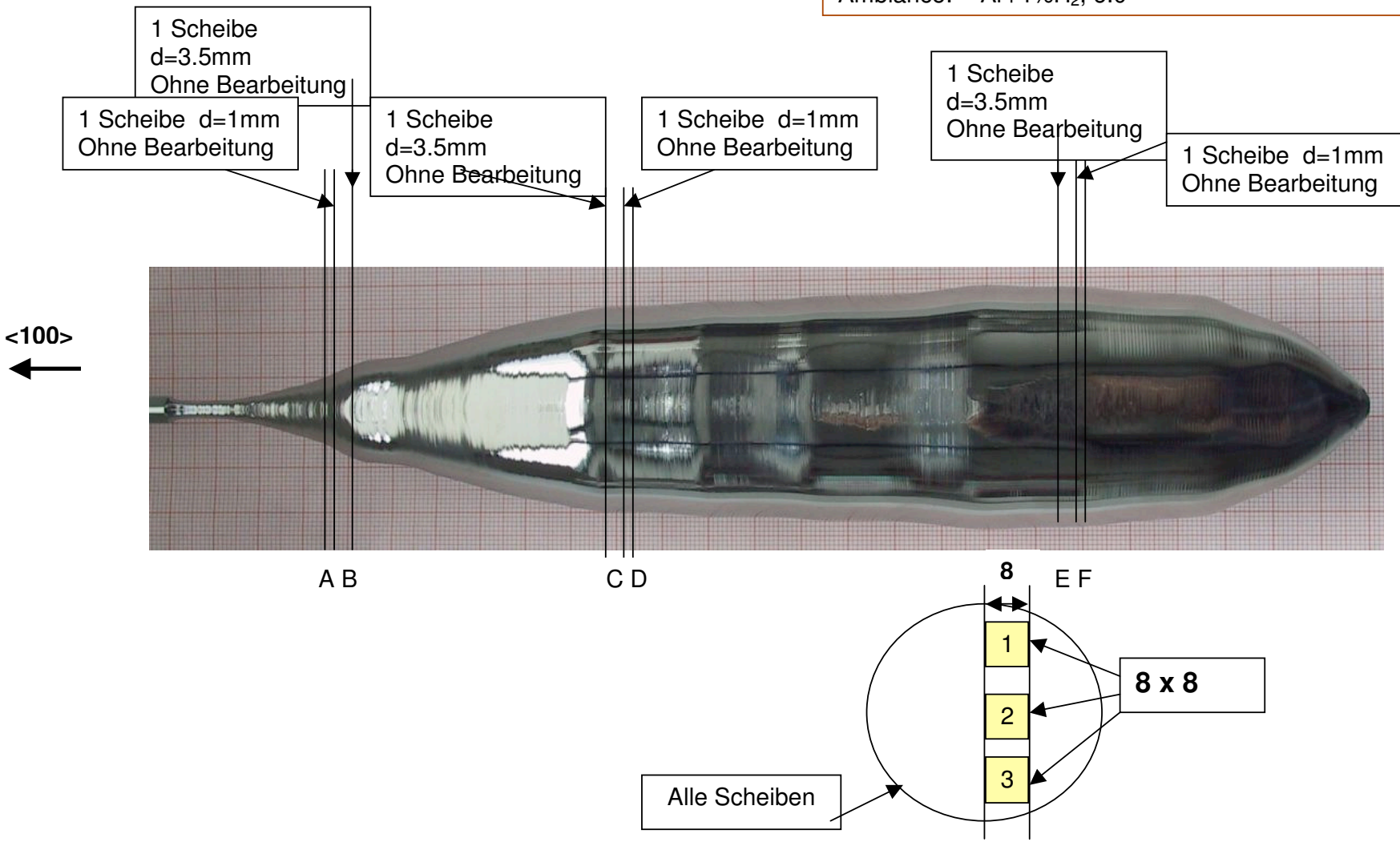
- We suspected that the source of impurities is the Cz. puller (As)
- Modifications done to reduce the contamination: a quartz tube protecting the melt and a gas purification system was installed
- 3 new crystals pulled:
  - CZ9: PPM material, quartz tube around the growing crystal
  - FZ-V3127: New float-zone crystal from PPM material
  - CZ10: PPM material, quartz tube, gas filter
- They were all measured with Hall-effect and PTIS at IKZ
- Cz9 and FZ-V3127 measured with PLS in Dresden

Ge-FZ-V3127 (feedstock - Gerda Poly-Ge; Ar-atmosphere)



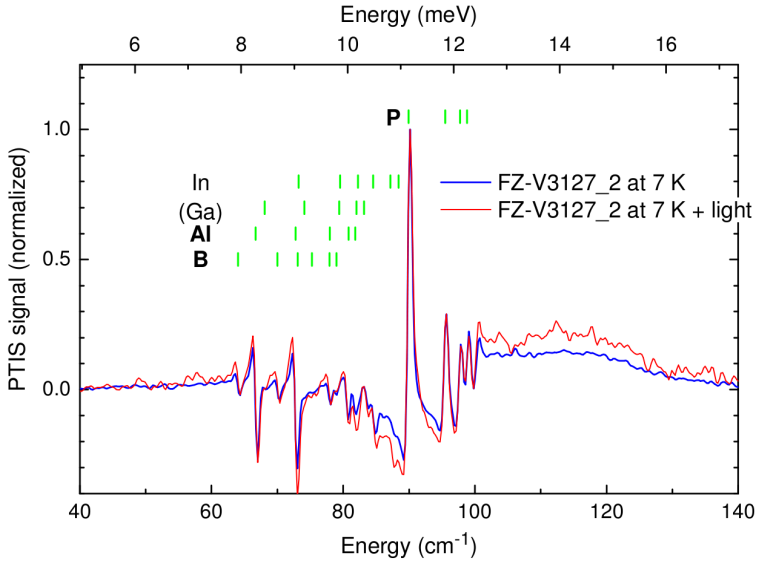
Cz-9 (Ge-HP-9)

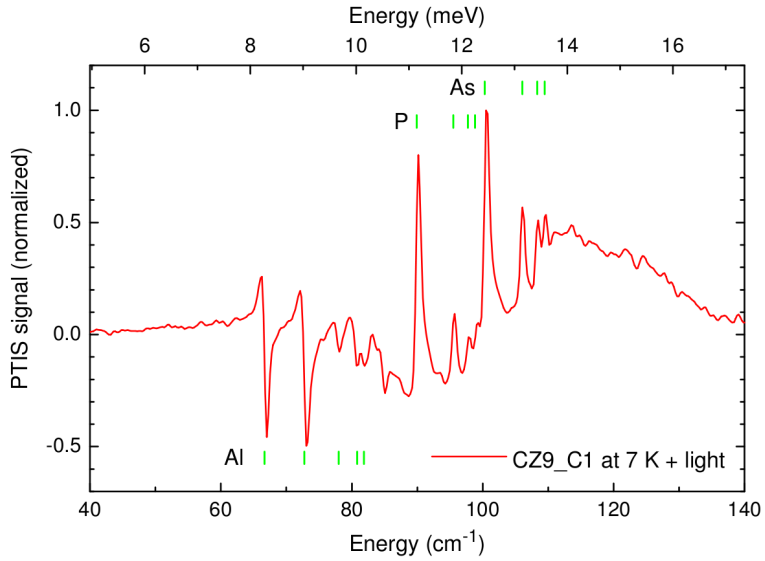
Furnace:	EKZ 2000
Feedstock:	PPM (^{76}Ge-depleted Ge), 1.410 kg
Crucible:	Suprasil, neu
Ambiance:	Ar+4%H_2, 6.0



Conductivity and Hall effect results:

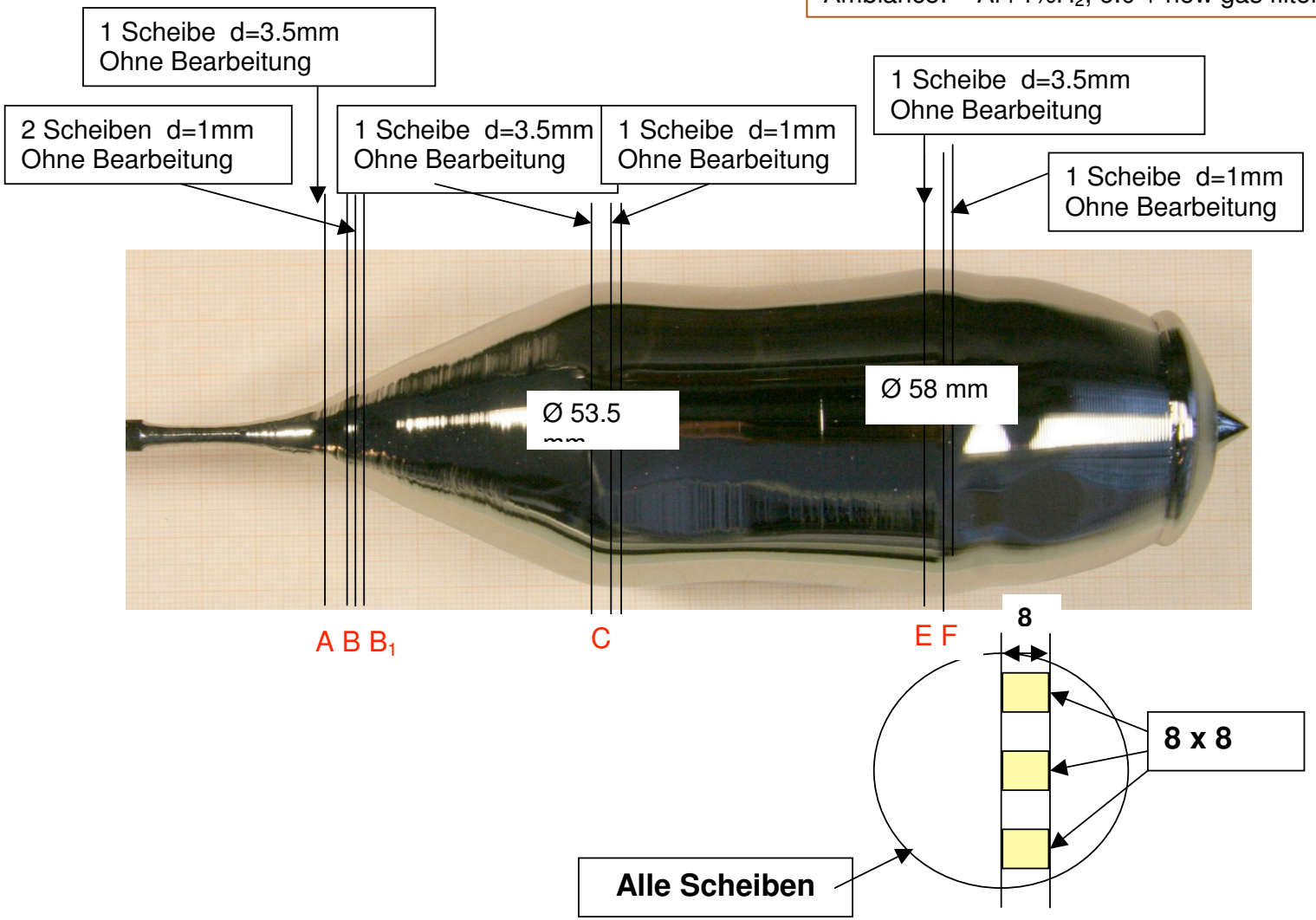
	Resistvity (Ωcm)		carrier concentration (cm^{-3})		Mobility (cm^2/Vs)	
Temperature	**297 K**	**77 K**	**297 K**	**77 K**	**297 K**	**77 K**
FZ-V3127_1	61.5	3290	-6.6×10^{13}	$+4.1\times10^{10}$	1540	45900
FZ-V3127_4 Sym≈5: inhom.!! not reliable!!	86.1	15500	$+2.1\times10^{14}$	$+3.7\times10^{11}$	348	1070
Ge-CZ9_A	59.6	332	-6.3×10^{13}	-6.4×10^{11}	1650	29400
Ge-CZ9_D-1	69.8	287	-5.5×10^{13}	-6.3×10^{11}	1640	34200
Ge-CZ9_F-2	59	29	-5.2×10^{13}	-6.3×10^{12}	2020	34600





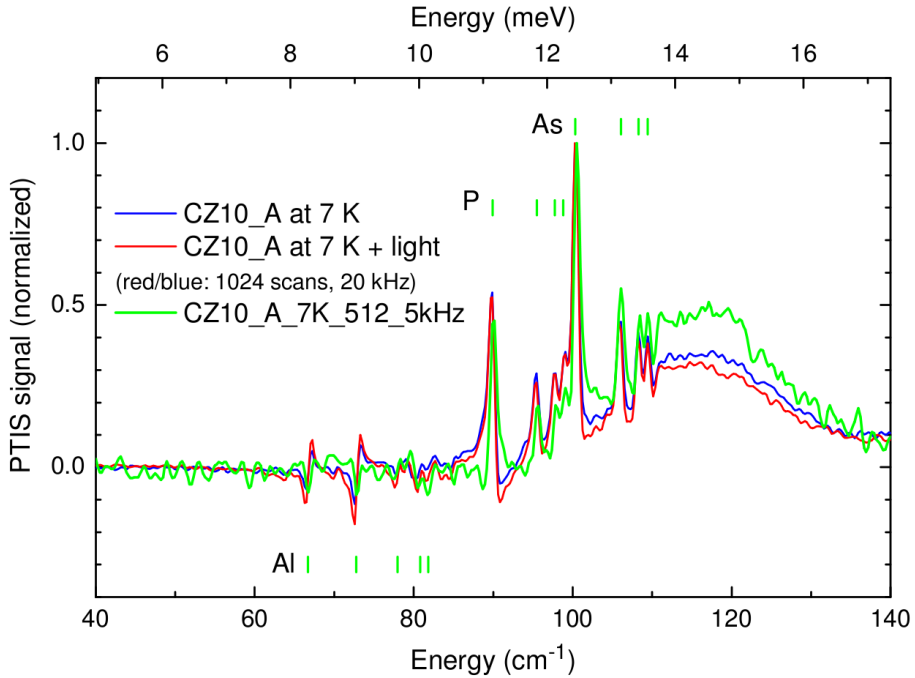
Cz-10 (Ge-HP-10)

Furnace:	EKZ 2000
Feedstock:	PPM (^{76}Ge-depleted Ge), 1.399 kg
Crucible:	Suprasil, 2nd experiment
Ambiance:	Ar+4%H_2, 6.0 + new gas filter



Conductivity and Hall effect results:

	Resistvity (Ωcm)		carrier concentration (cm^{-3})		Mobility (cm^2/Vs)	
Temperature	**294 K**	**77 K**	**297 K**	**77 K**	**294 K**	**77 K**
Ge-CZ10_B	60.9	190	-6.4×10^{13}	-1.0×10^{12}	1600	32500
Ge-CZ10_D-1	60.2	194	-6.7×10^{13}	-1.1×10^{12}	1540	29800
Ge-CZ10_F-1	38.1	69.5	-9.7×10^{13}	-3.0×10^{12}	1700	29500



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Results from PL measurements of Cz#9 and FzV3127 samples

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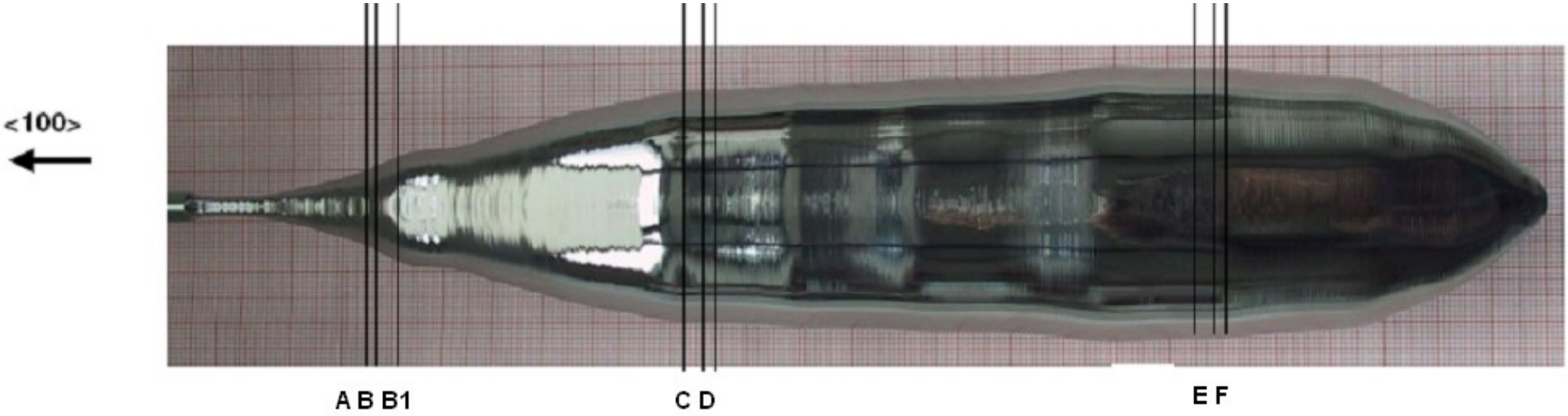
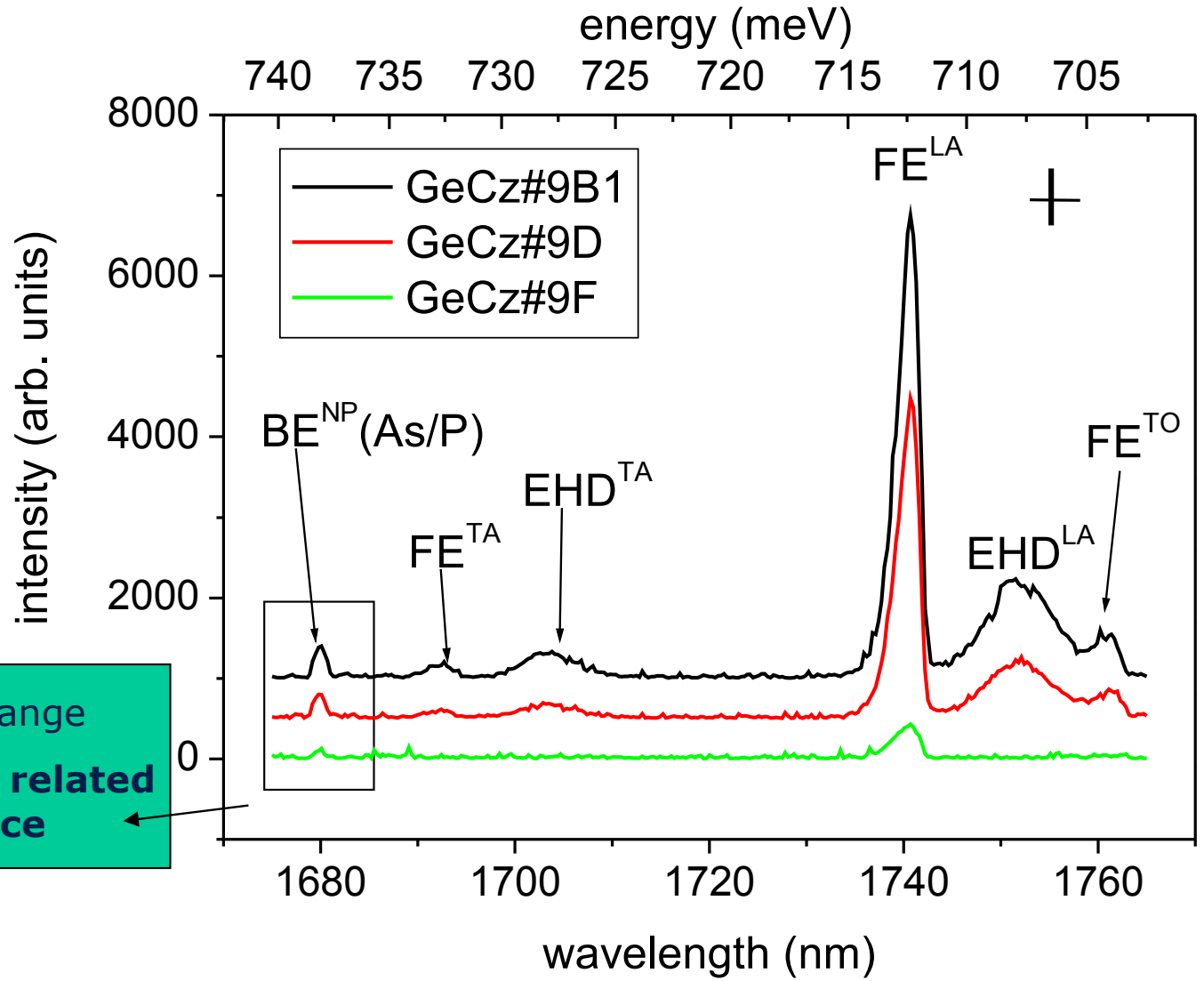
Cz#9 crystal



- investigated samples from positions B, D and F :

Ge#9B1 Ge#9D Ge#9F

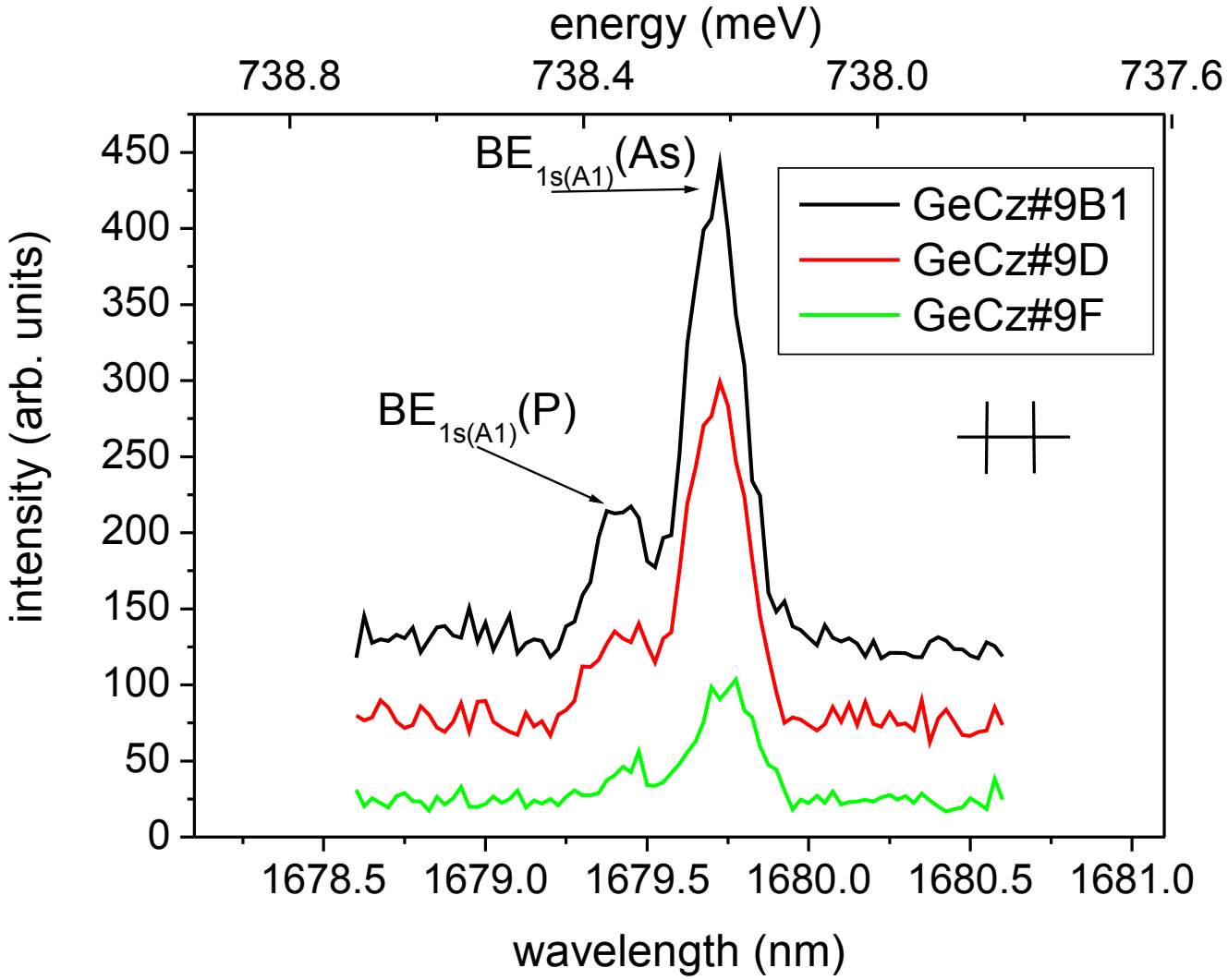
PL spectra of Cz#9 samples



Interesting range

→ **Impurity related luminescence**

PL spectra of Cz#9 samples



→ arsenic exciton stronger than phosphorous exciton (ratio 3:1) but no direct correlation to the impurity concentration ratio

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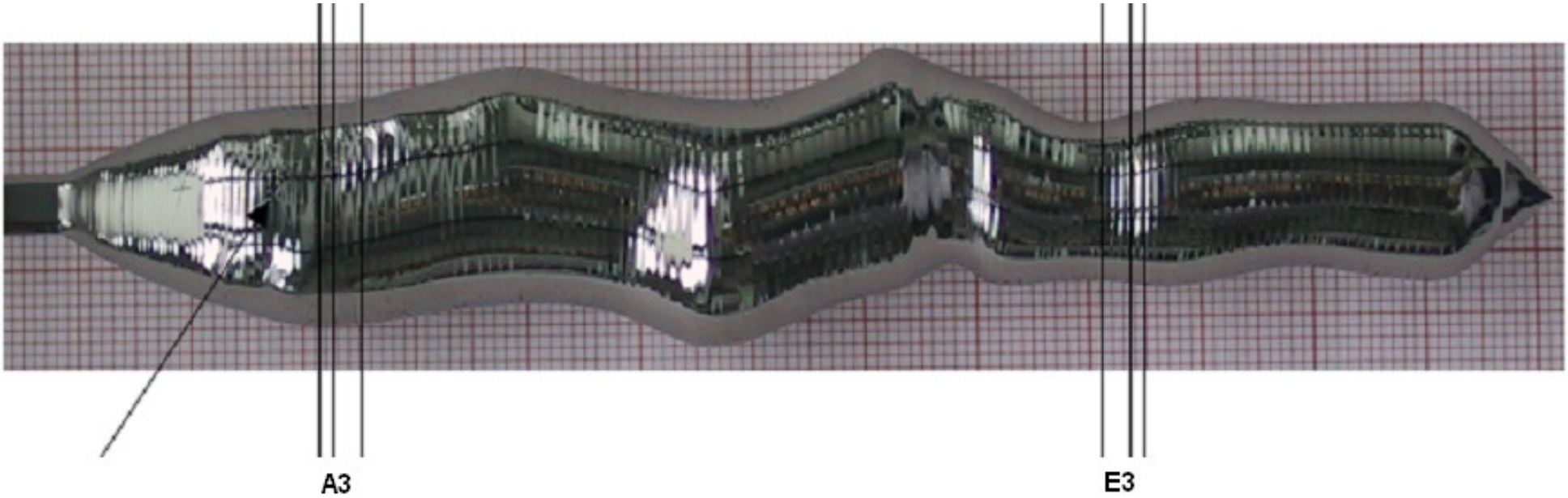
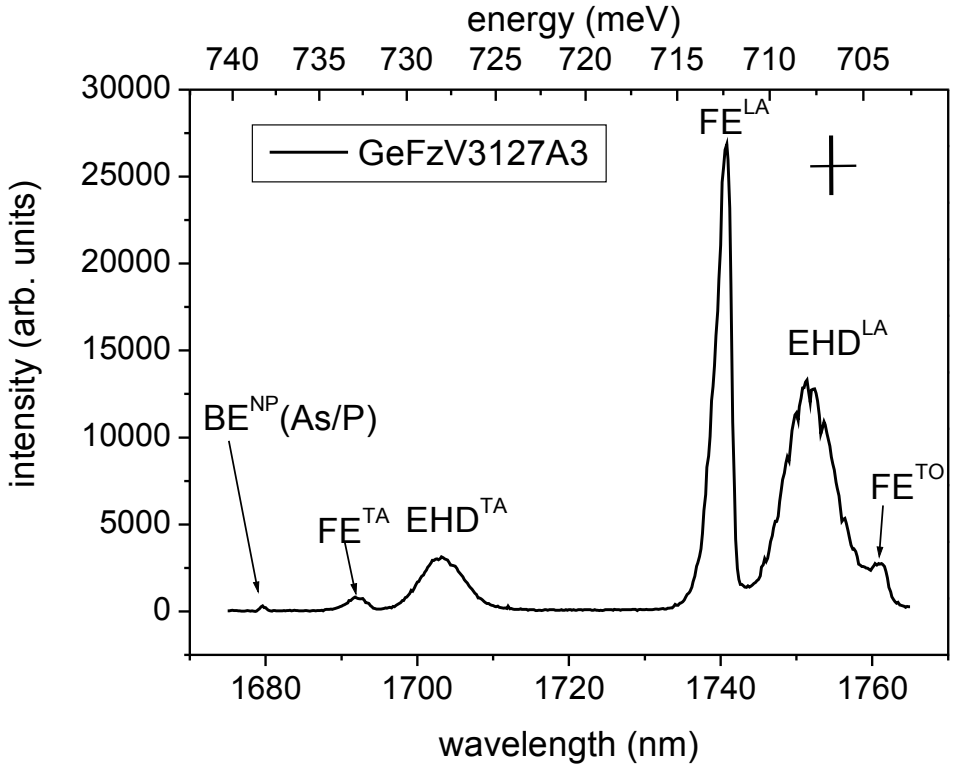
FzV3127 crystal

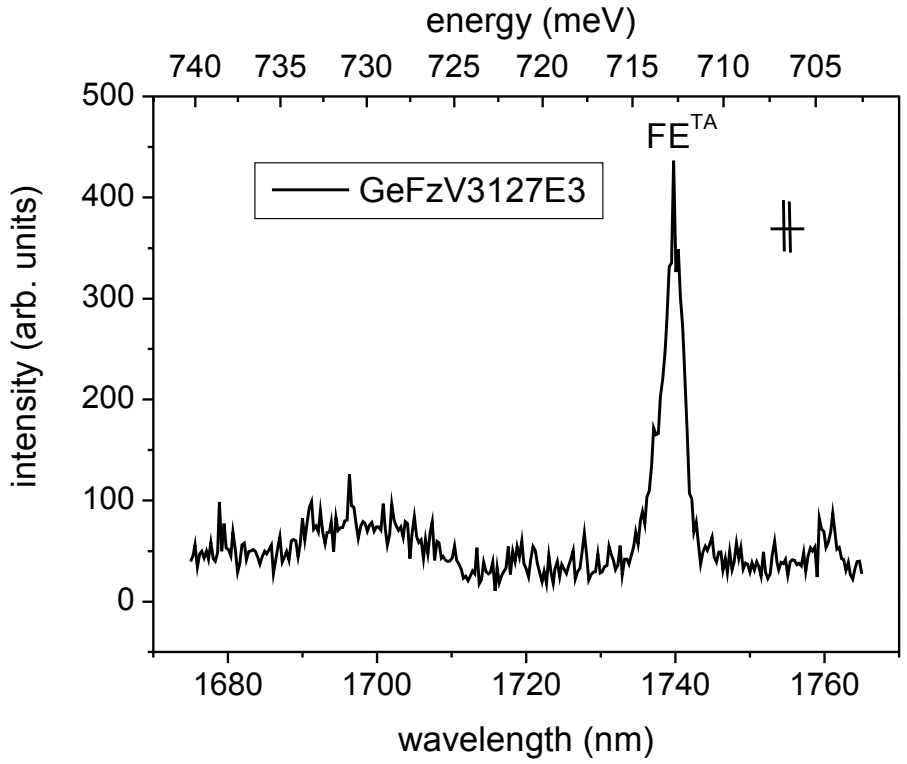


- investigated samples from positions A3 and E3 :

GeFzV3127A3 GeFzV3127E3

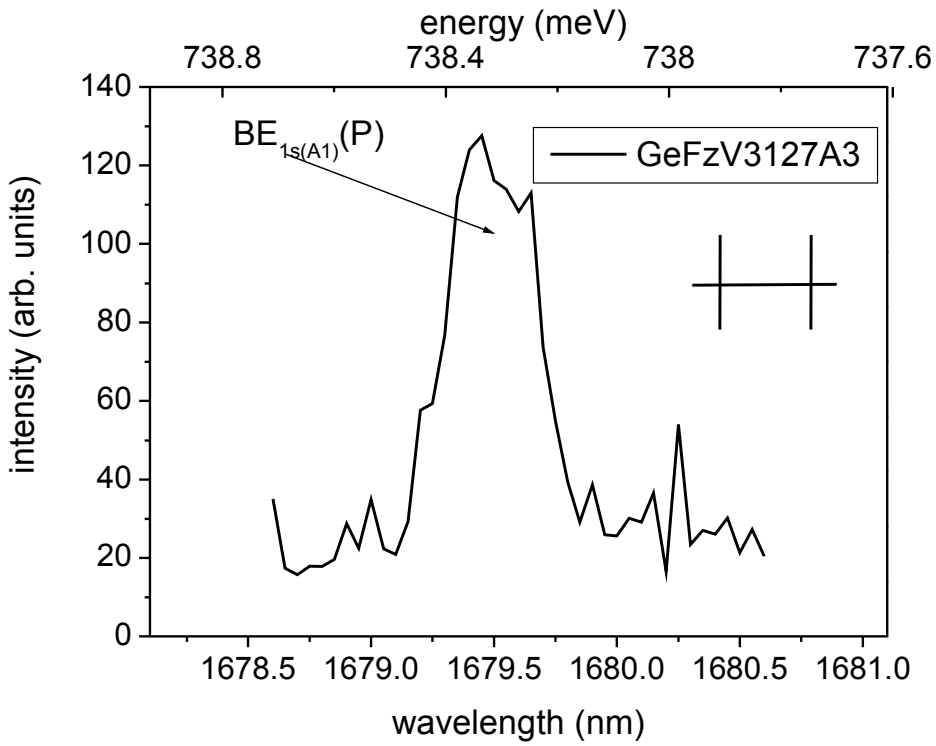
PL spectra of FzV3127 samples

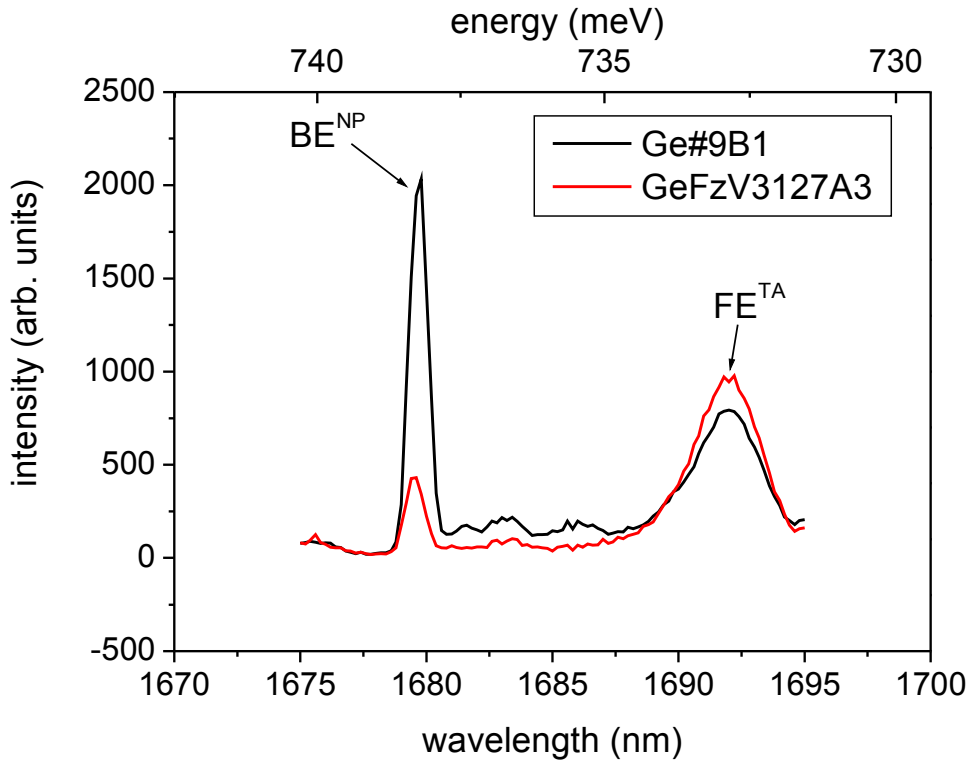




- strong decrease in luminescence intensity of the tail end sample compared to the seed end sample

PL spectra of FzV3127 samples





-only phosphorus bound exciton detectable in sample GeV3127A3

-bound exciton luminescence roughly 5x stronger in sample GeCz#9B1 under comparable measuring conditions

Summary

- Main donors in Cz#9 crystal are phosphorus and arsenic

→ bound exciton luminescence intensity is roughly 2 orders of magnitude lower compared to Cz#4 crystal

- Unfortunately aluminium seems not be able to detect by PL

- Main donor in FzV3127 crystal is phosphorus
- bound exciton luminescence roughly 5x stronger in sample GeCz#9B1 under comparable measuring conditions

- PL results of donor impurities consistent with IKZ results from Hall- and PTIS measurements

Crystal pulling - near future - conclusion

- For the first time 10^{10} impurity / cm^3 was measured (detector grade)
- One order of magnitude improvement with the Cz. puller
- Many ideas how to improve the purity:
  - New susceptor crucible made of graphite
  - Electro-polishing the whole puller
  - Reducing the active volume of the puller etc.
- PL measurements show good agreement with the PTIS done at IKZ
- Next crystal pulling in two weeks time