Czochralski crystal growth in IKZ: Current results of HP-Ge crystal growth

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Application of Germanium detectors in fundamental research
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Introduction

- High purity (HP) Ge growth at the IKZ was initiated by the GERDA project (2007)
- Goals: HP crystals of ^{76}Ge for detectors
  - 3“ <100> with low carrier net-concentration ($\approx 10^{10}$ cm^{-3} at 77K)
  - well-adjusted dislocation density ($\approx 10^{-2}$-10^{-3}cm^{-2})
  - 10 crystals with usable lengths of ≥70mm
  - modified HP-Czochralski(CZ) technique

- Essentials of a HP Ge crystal growth
  - pure starting material
  - “clean” furnace
  - pure growth conditions

Starting material

- Material preparation for GERDA
  - ~50kg of $^{76}GeO_2$, isotope enriched to (3.5N) (Krasnojarsk, RU)
  - Reduction to Ge and multizone purified (6N) at PPM Pure Metals(Langelsheim/Germany)
  - For process development: residual depleted Ge, free of ^{76}Ge

Czochralski Method

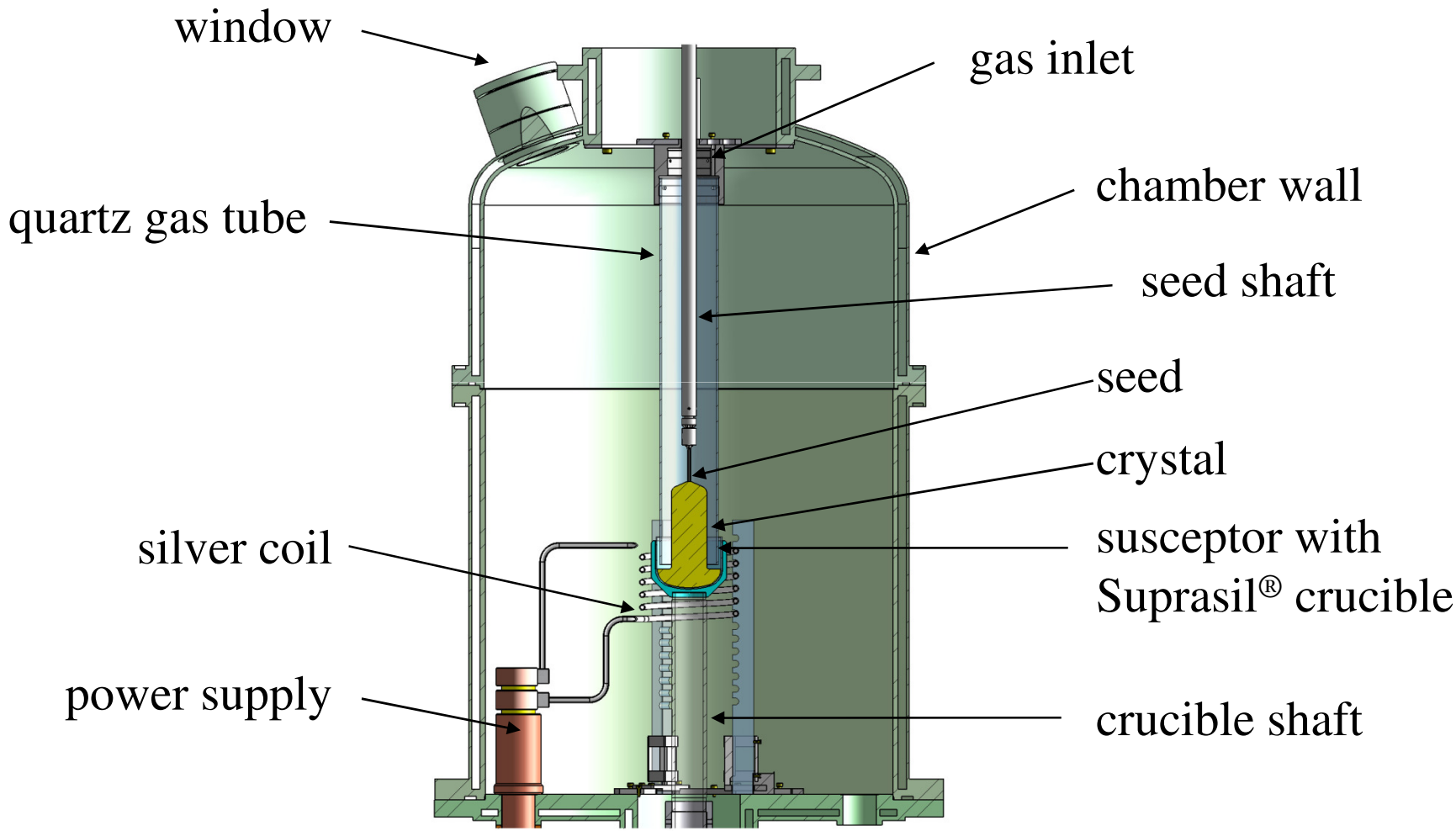
- Modernized puller EKZ 2000 (build in 1982)
- Crucible made of quartz Suprasil® (≤0.05ppm for Alkali and metals) (95mm and 150mm)
- **Molybdenum susceptor** inductively heated with f=16kHz 30KW generator
- Pull speed around 0.5-0.8 mm/min
- Surrounding gas **Argon 6N with 4% H_2 - dried**
- Normal pressure (10.0 l/min)

EKZ 2000 growth machine

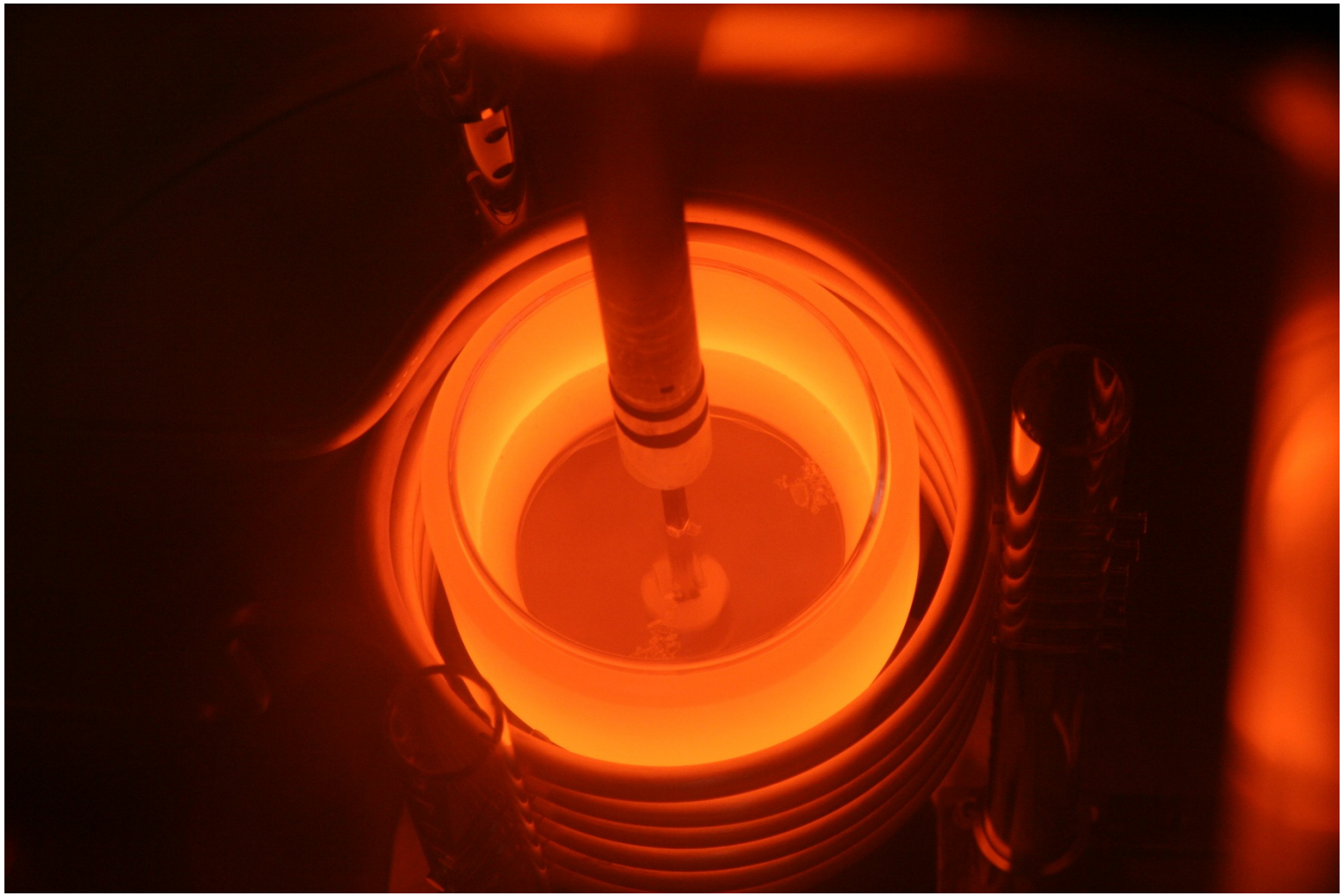
- **Modernized 2007-2008**
  - Controlling
  - New vacuum system (turbo molecular pump)
  - New heating system (RF heating)
  - New seals
- **16.04.08** – first crystal growth experiment (6N material)
- **09.10.08** – first growth experiment with depleted germanium

Picture before modernization

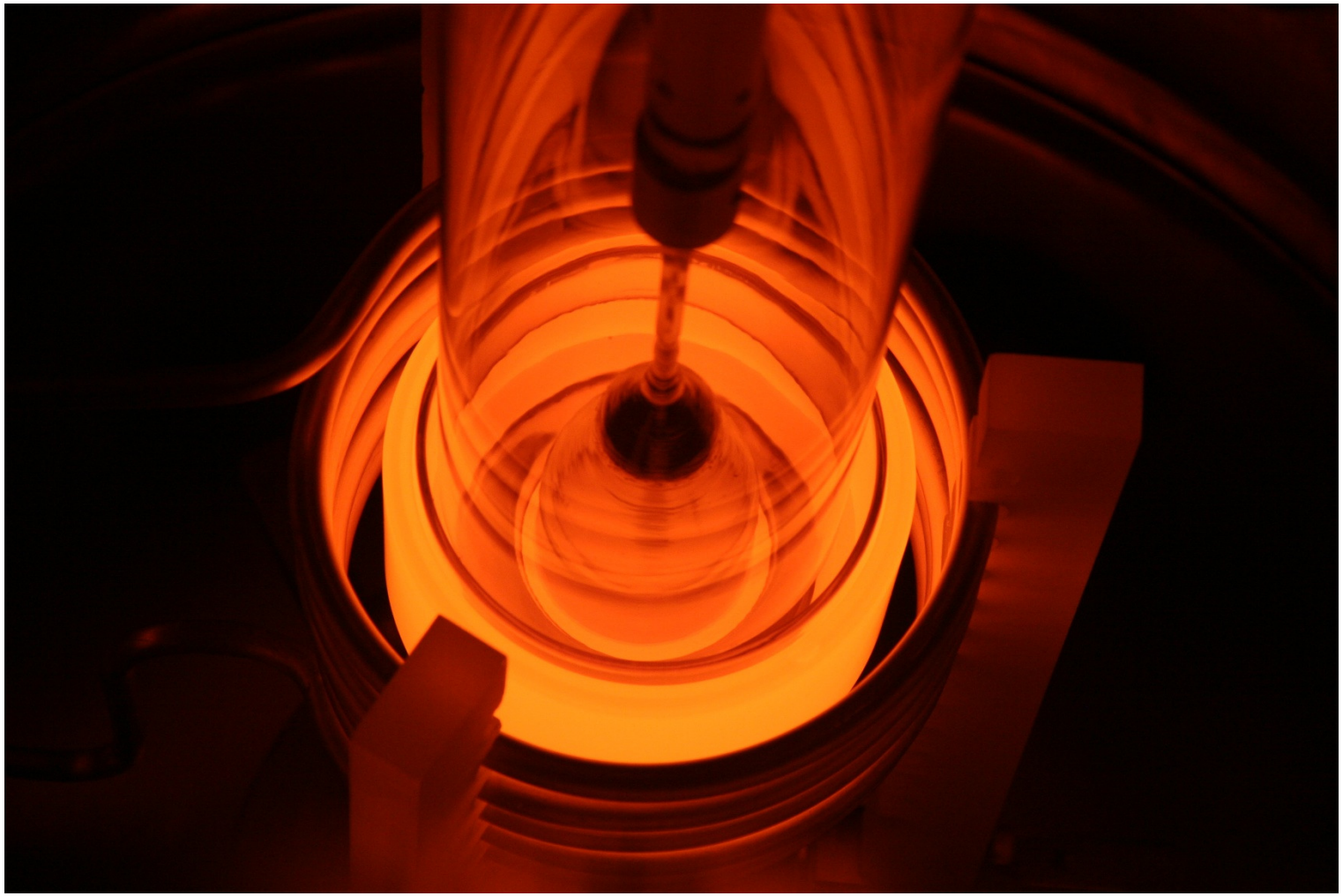
Machine design



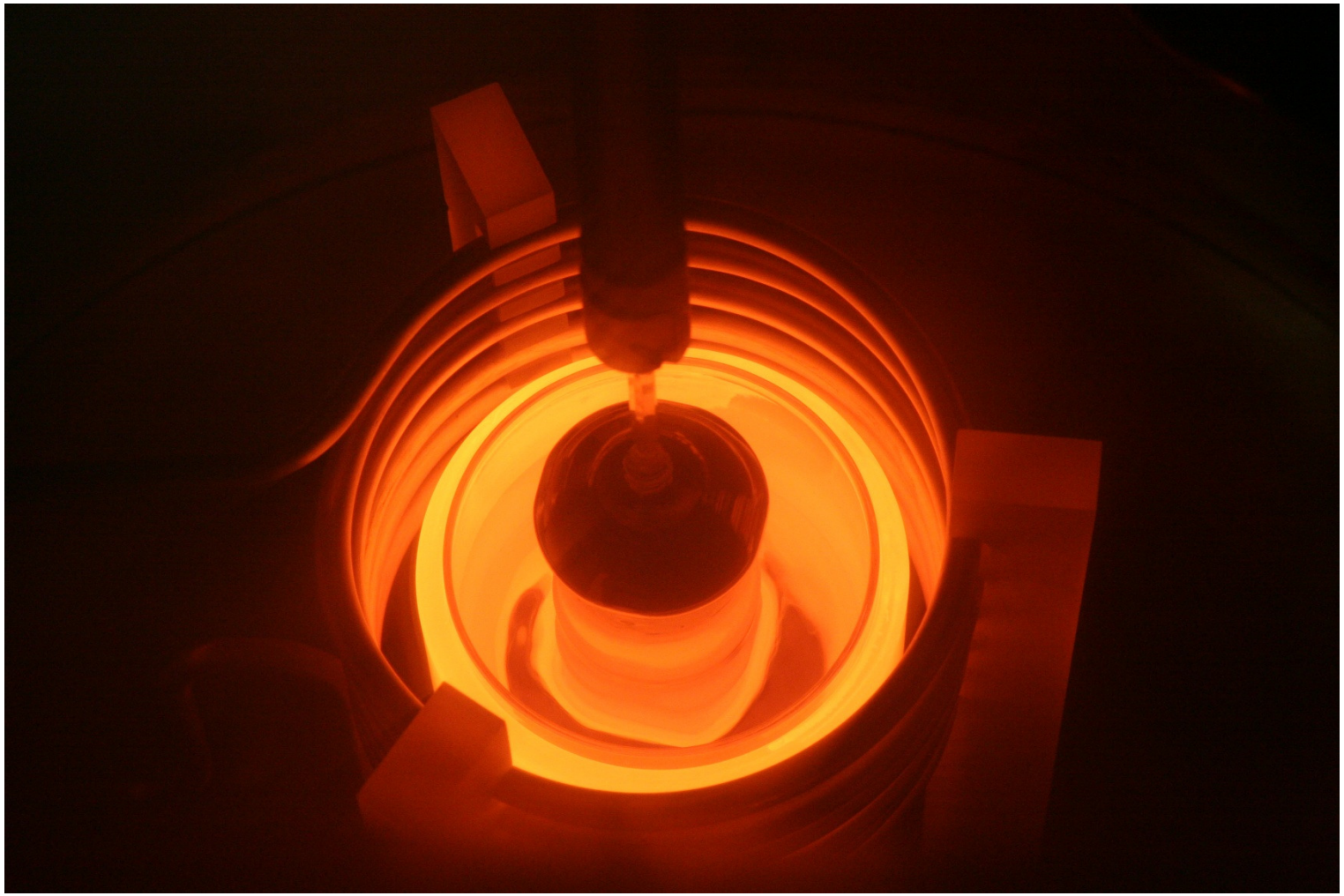
Seeding



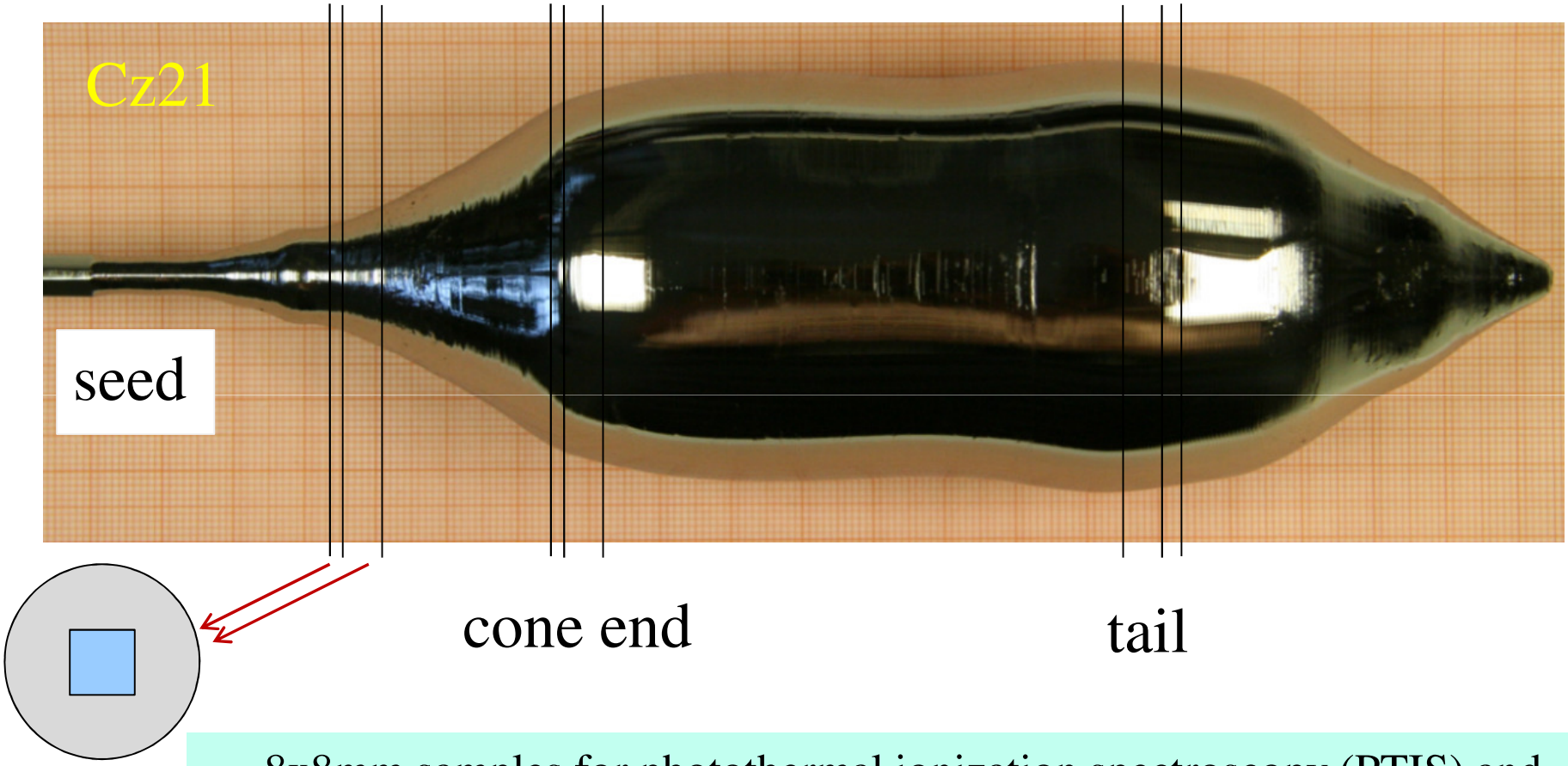
Cylindrical growth



End cone

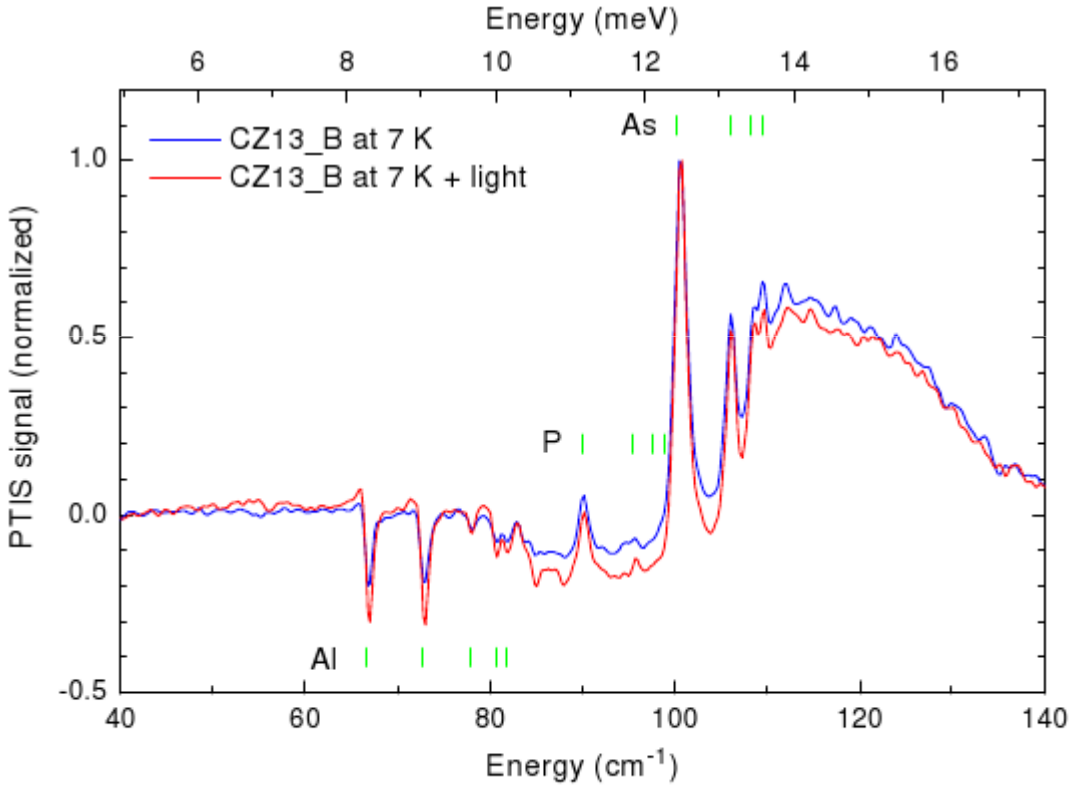


Sample crystal of Ø ≈ 50mm



8x8mm samples for photothermal ionization spectroscopy (PTIS) and Hall effect measurements; thickness 3.5mm and 1mm, respectively

First crystals with arsenic impurity



Cone end:

[As] >≈ 4×[P]; [P] ≈ [Al]

|

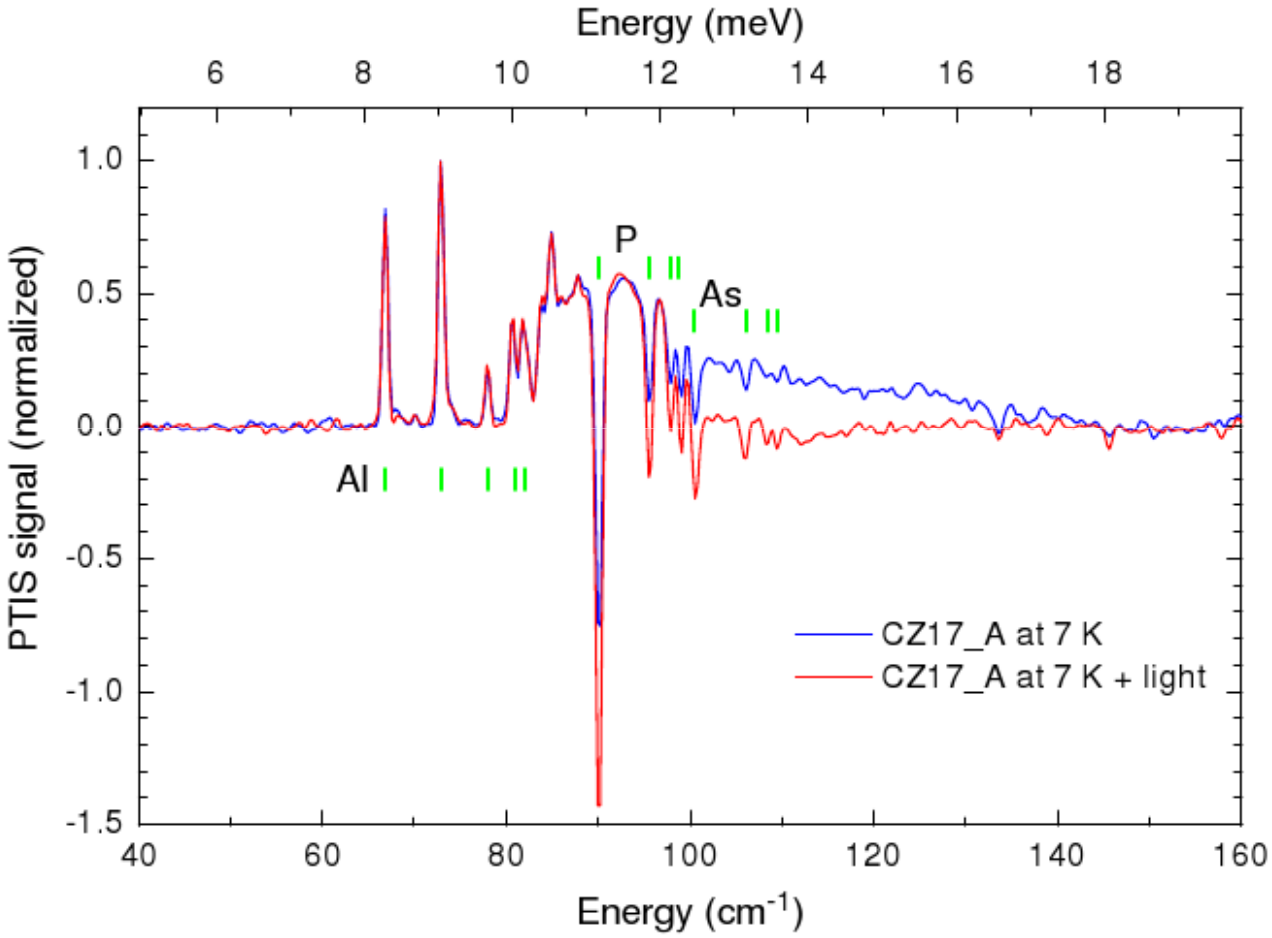
$|N_D-N_A|= -2.7 \times 10^{12} cm^{-3}$

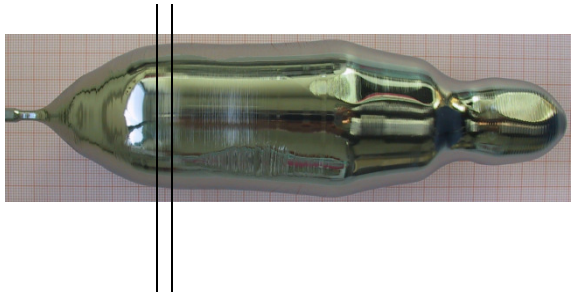
K. Irmscher and M. Pietsch from IKZ

“Mini-Cz” in FZ furnace

- Tube-like induction coil
  - 3MHz generator from FZ puller
- Coil also radiation shield
  - Reduced heat losses
- Less radiativ heating of the walls
  - Only melt, crucible and susceptor are hot

Crystal in FZ machine with “Mini-CZ”





Cone end:

$|N_D-N_A|=+1.9\times10^{11}cm^{-3}$

K. Irmscher and M. Pietsch from IKZ

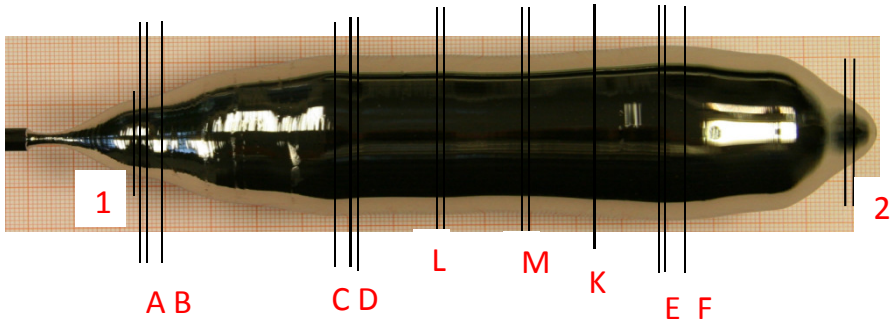
EKZ 2000 - refurbished

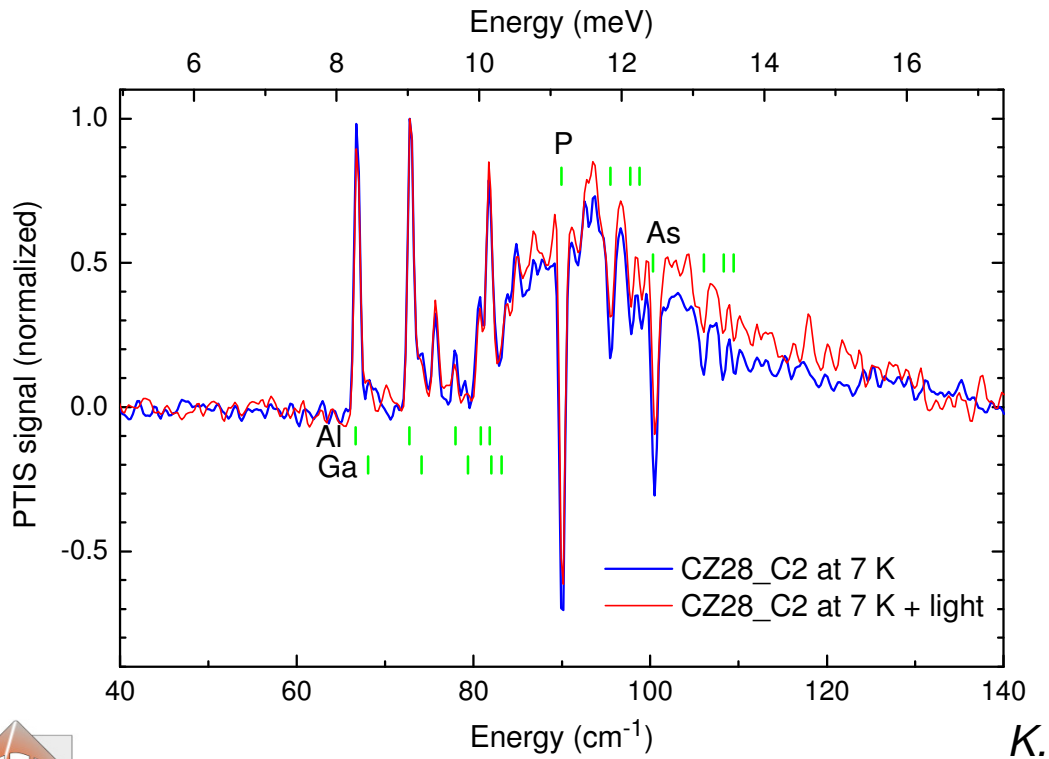
- **Modernized 2010**
  - Revised welding seams
  - Electrochemical polished inner surfaces
  - Housing for dust protection
  - 04.03.10: first crystal growth experiment

- **February 2011**
  - Inner quartz-housing for impurity shielding

Picture after modernization 2010

Ge-HP-28





Cone end (sample D):
$|N_D-N_A|=+3.9x10^{10}cm^{-3}$

Cylinder (sample M):
$|N_D-N_A|=+2.8x10^{8}cm^{-3}$

K. Irmscher and M. Pietsch from IKZ

Ge-HP-28: Electrical characterization

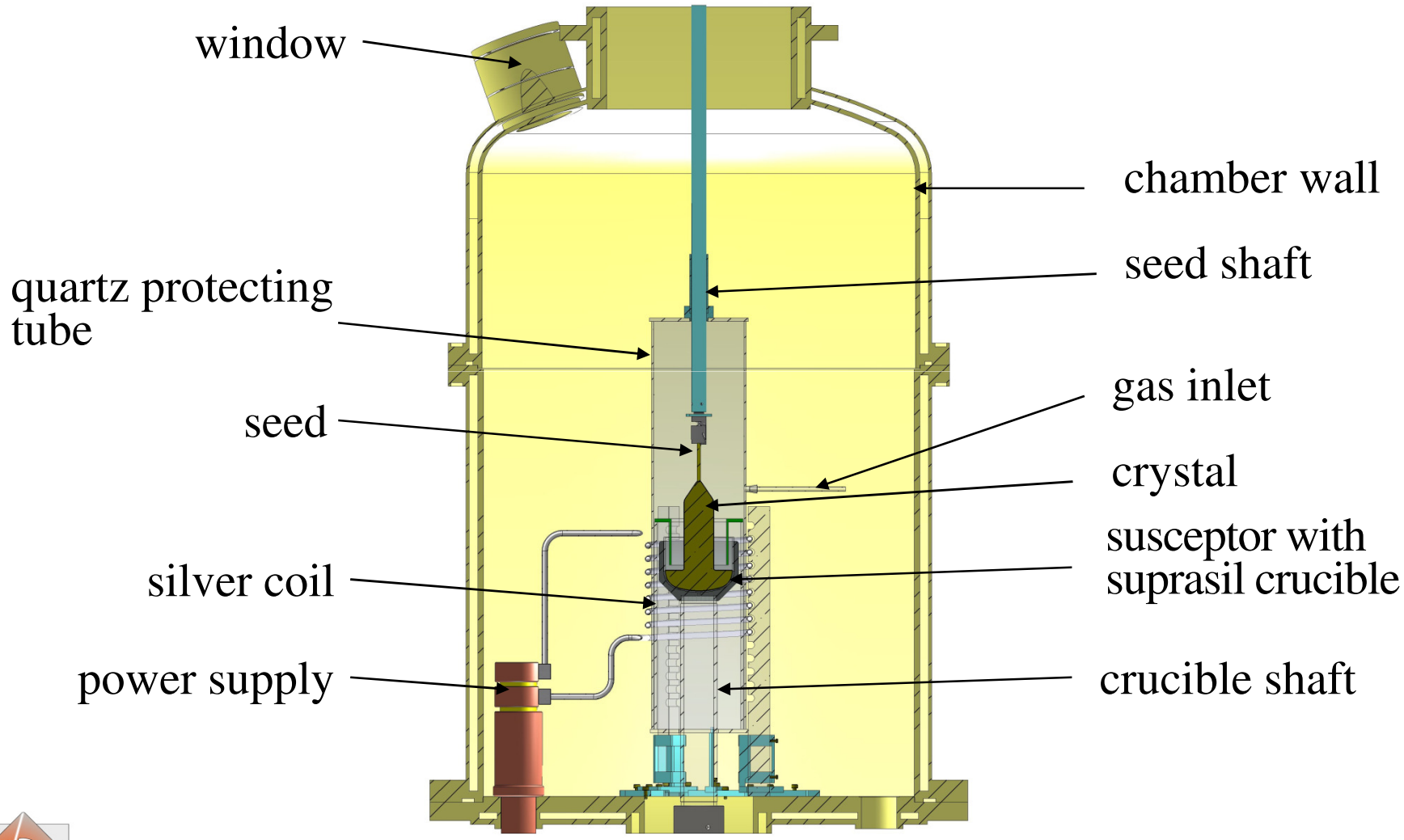
Conductivity and Hall effect results:

	Resistivity (Ωcm)		Carrier concentration (cm^{-3})		Mobility (cm^2/Vs)	
Temperature	RT	77 K	RT	77 K	RT	77 K
CZ28_1	32	1390	$-1.5x10^{14}$	$+1.1\times10^{11}$	1320	42900
CZ28_A	45	1370	$-1.5x10^{14}$	$+1.1\times10^{11}$	956	40900
CZ28_D2	26	3640	$-1.6x10^{14}$	$+3.9\times10^{10}$	1470	44300
CZ28_L2	50	8210	$-8.0x10^{13}$	$+1.7x10^{10}$	1530	44200
CZ28_M2	44	669000	$-9.1x10^{13}$	$+2.8\times10^{08}$	1570	33200
CZ28_K2	55	n.m. **	$-7.4x10^{13}$	n.m. **	1520	n.m. **
CZ28_E2	23	(6430)*	$-1.8x10^{14}$	$(+7.5x10^{11})$*	1470	(1290)*
CZ28_2	39	(283)*	$-7.9x10^{13}$	(-1.1×10^{13})*	2020	(1960)*

*Undefined temperature below 0°C; i.e. no measurement possible at 77 K.
** not measurable

K. Irmscher and M. Pietsch from IKZ

New inner quartz-housing



Next steps

- 2 experiments were made with new machine design: no efficient improvements of crystal purity were observed (imperfect gas flow system?)

Further improvements in machine design and growth conditions

- Exchange of the Mo- susceptor by a high purity graphite one, graphite can be cleaned from metals by hydroclorine-annealing!
- Crystal growth in H_2 atmosphere should be proved (security!)

Thank you for your attention!