Characterization of thermally induced shallow defects in HPGe

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- Aim of the study
- List of studied processes
- P diffusion by Spin-On-Doping ⟶ [V. Boldrini et al., Appl. Surf. Sci. 392 (2017)]
- Sb diffusion from remote source ⟶ [G. Maggioni et al., submitted to: Appl. Surf. Sci.]

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   - Role of active defects in Ge
   - How to measure active defects
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   - 4 wires resistance and Hall measurements
   - Sample preparation
4. **Results**
   - Sheet resistance @ low T
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5. **Empirical model for contamination dependence on annealing T and t**
   - Analysis results and discussion
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6. **Work in progress**

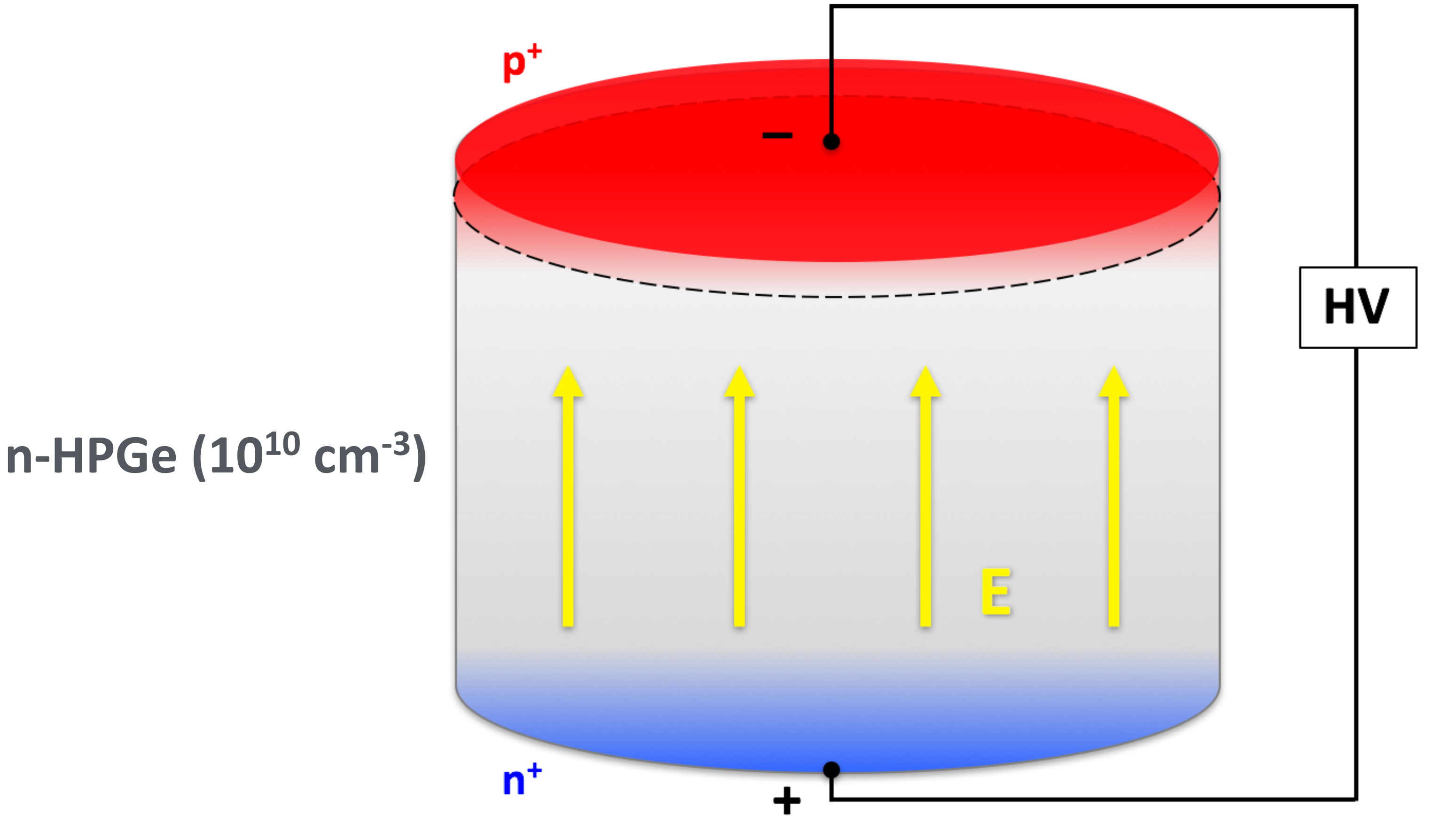
Do our fabrication processes contaminate?

Impurity diffusion inside semiconductors is a thermally activated process.

Thus, all doping processes that exploit thermal annealing treatments could introduce a high level of active defects inside HPGe.

Depletion region:

$$d = \sqrt{\frac{2\epsilon V}{en}}$$



List of studied processes

Samples come from HPGe wafers supplied by Umicore [$n_{growth} < 2x10^{10}$ cm^{-3}]:

✴ Reference samples:

- as cut n-type
- as cut p-type
- B ion implanted

✴ P diffusion by Spin-On-Doping

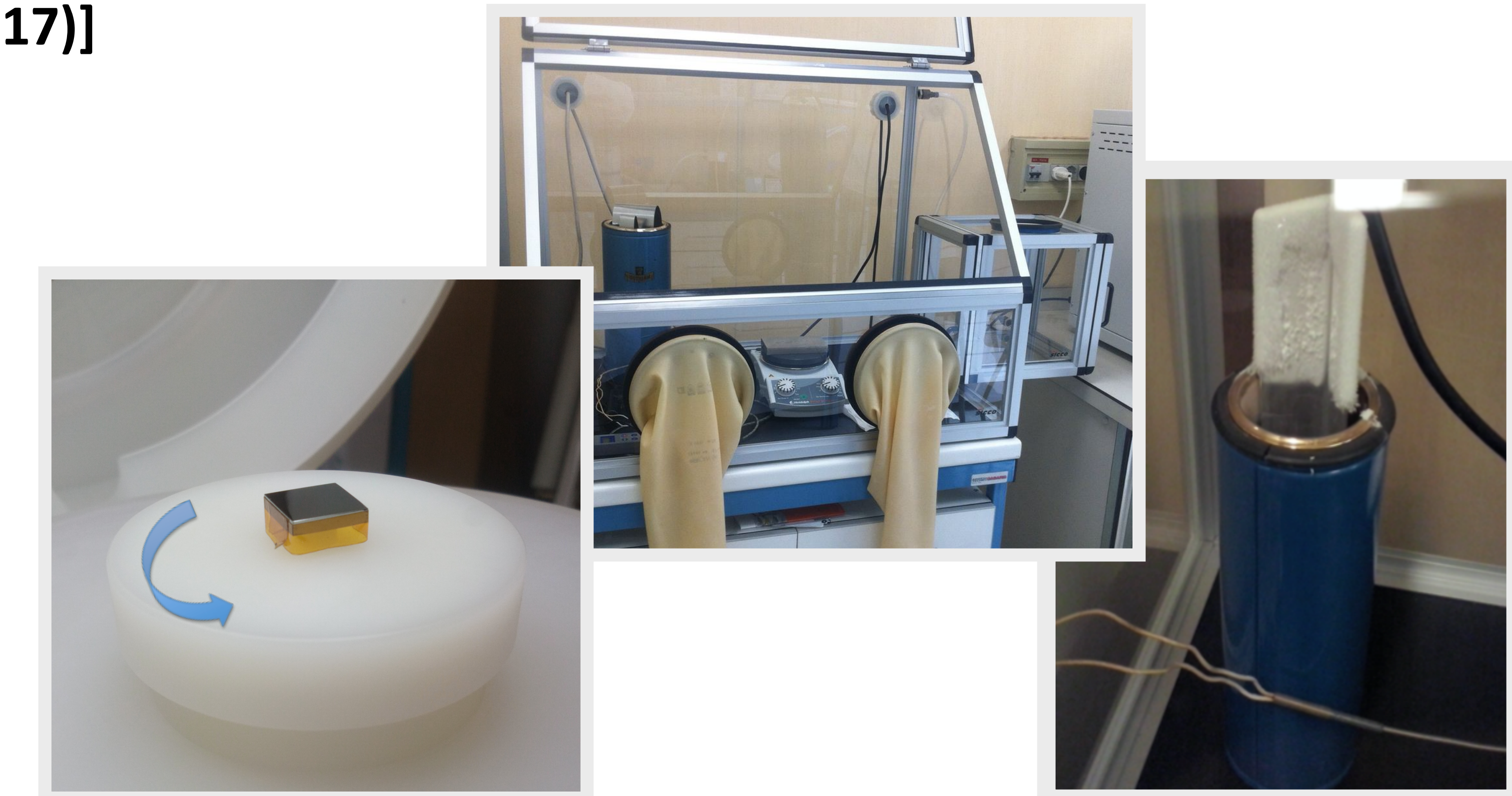
✴ Sb diffusion from a remote sputtered source

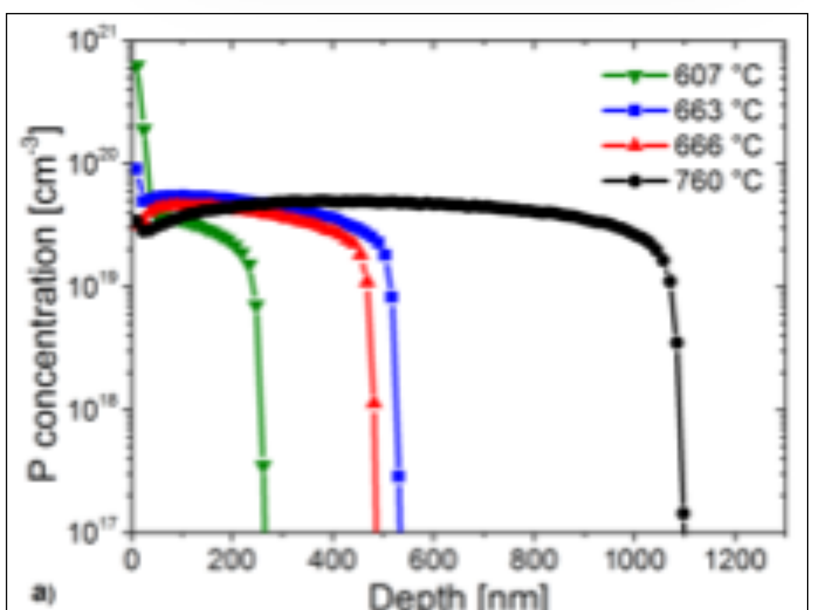
✴ Deposition of a SiO_2 protective coating

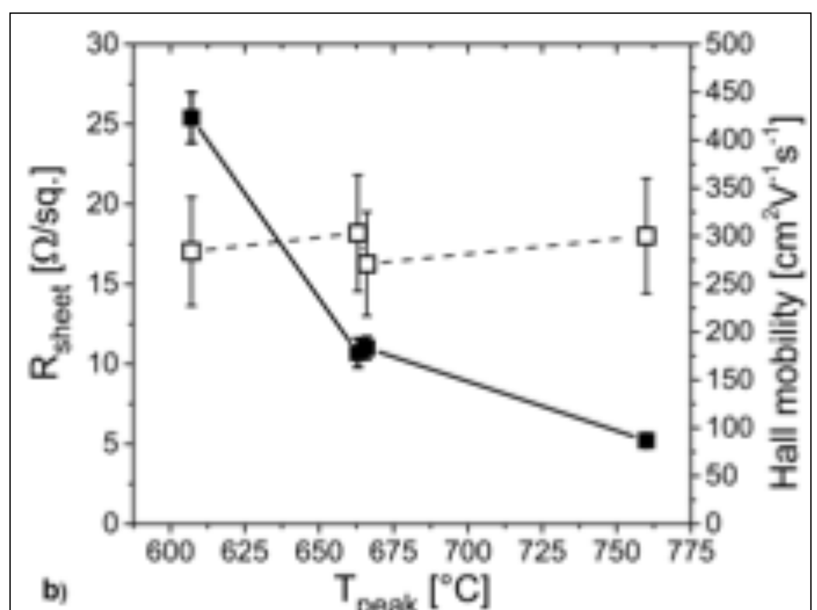
✴ High-T annealing treatments on as cut samples

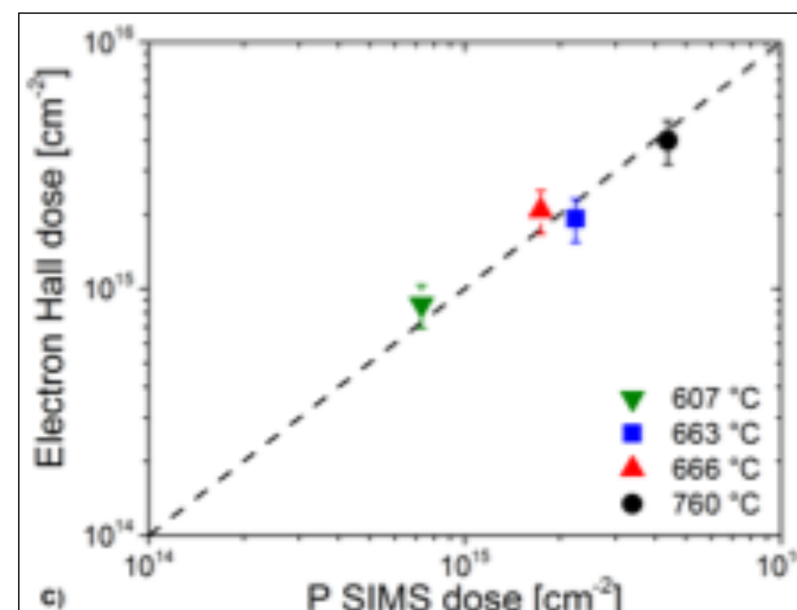
P diffusion from Spin-On-Doping source

[V. Boldrini et al., Appl. Surf. Sci. 392 (2017)]







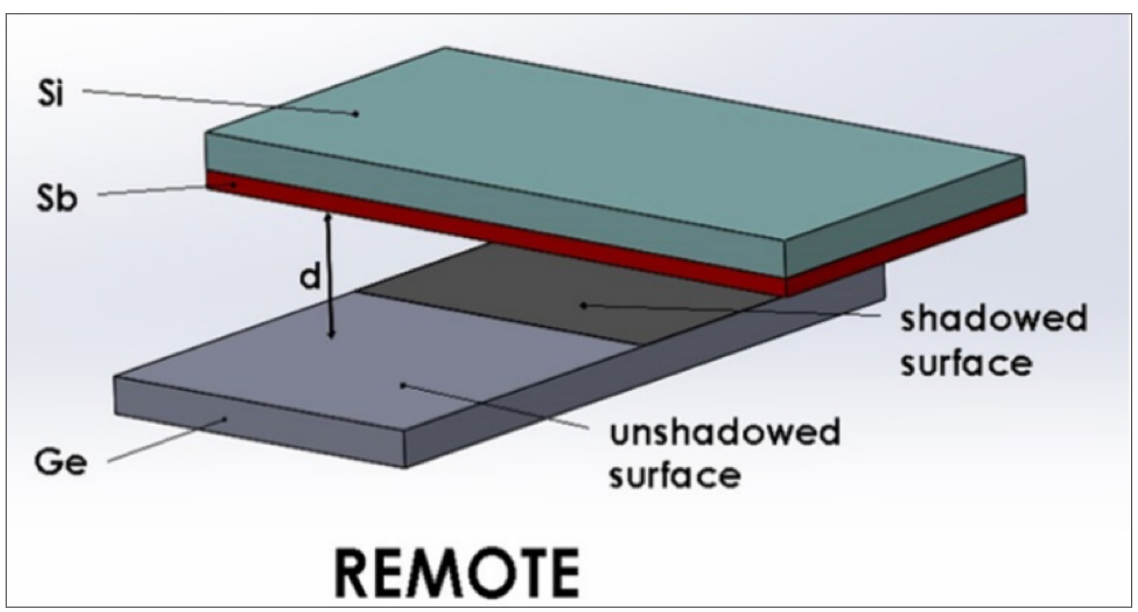


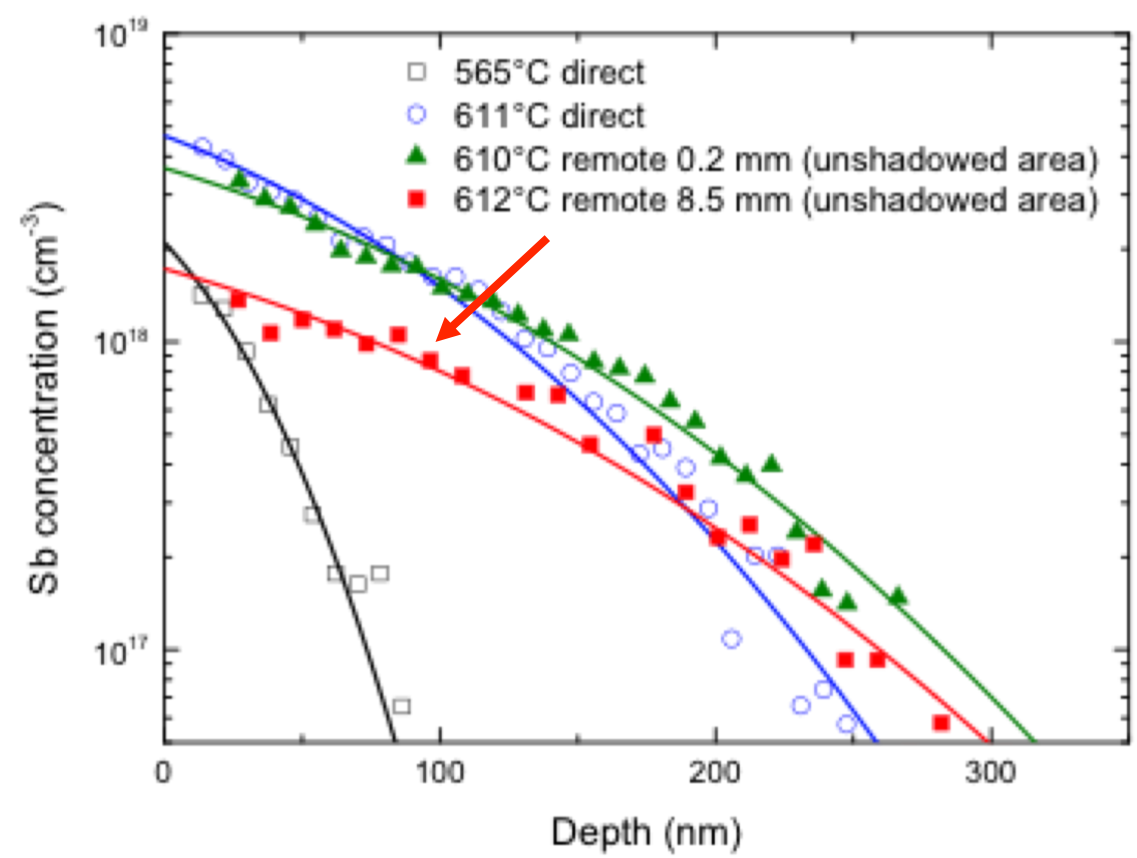
[G. Maggioni et al., submitted to: Appl. Surf. Sci. (sept. 2017)]

REMOTE SOURCE: Sputtering of 100 nm of Sb on a auxiliary piece of Si.

HPGe positioned at a distance of 8 mm.

Annealing treatment: @ 605 °C for 30 min in standard tube furnace.





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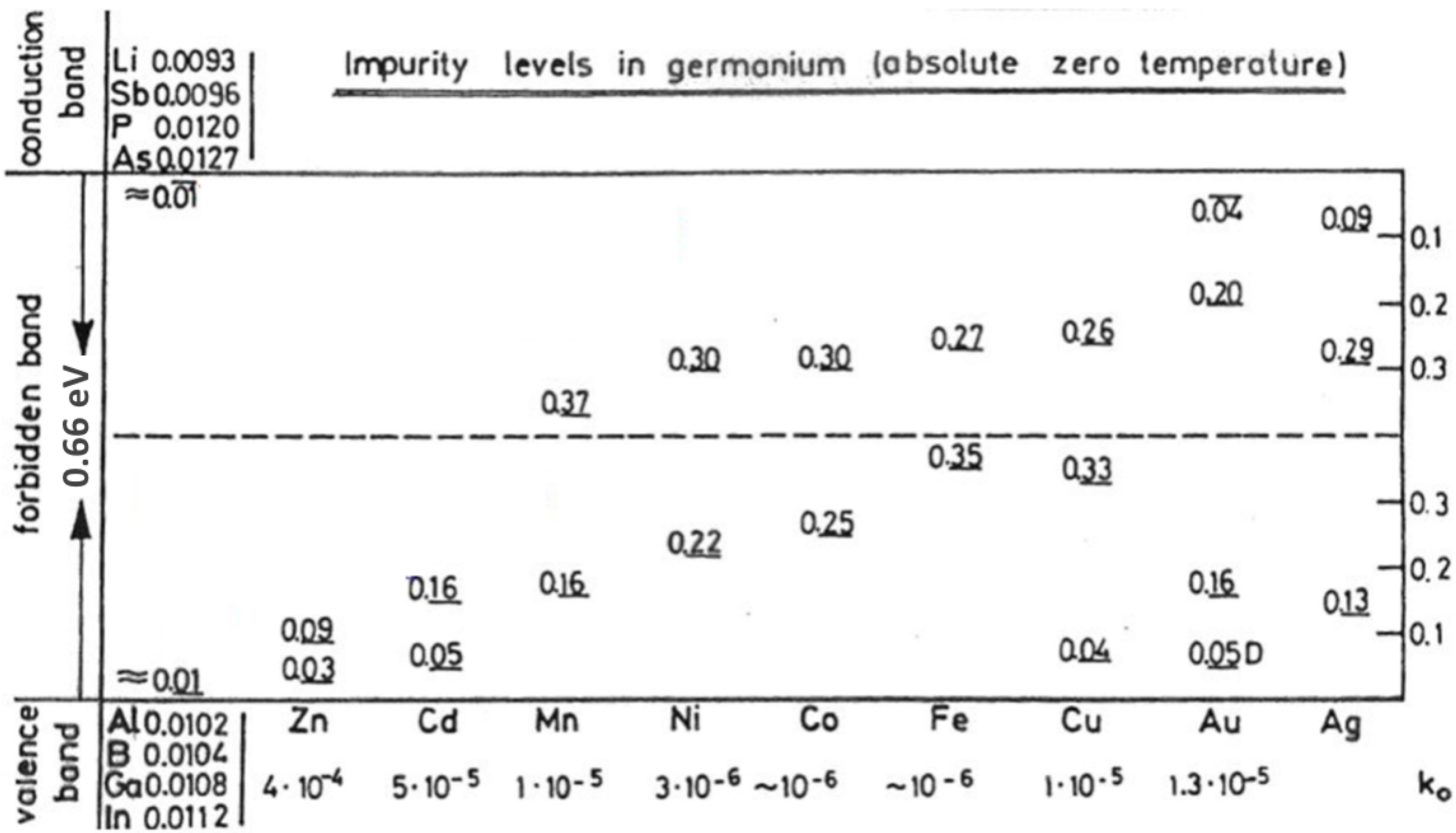
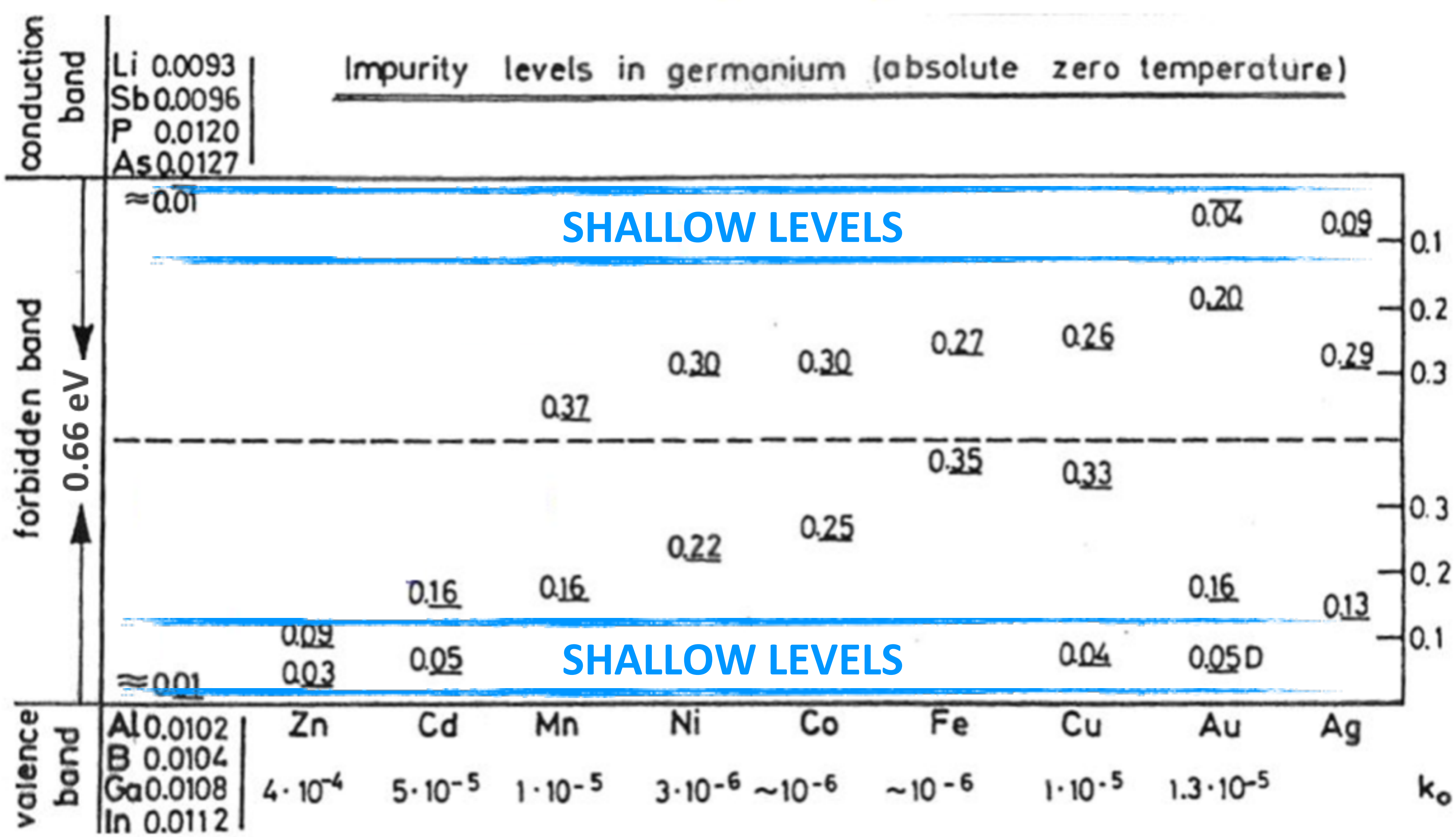
Electrical activation of defects



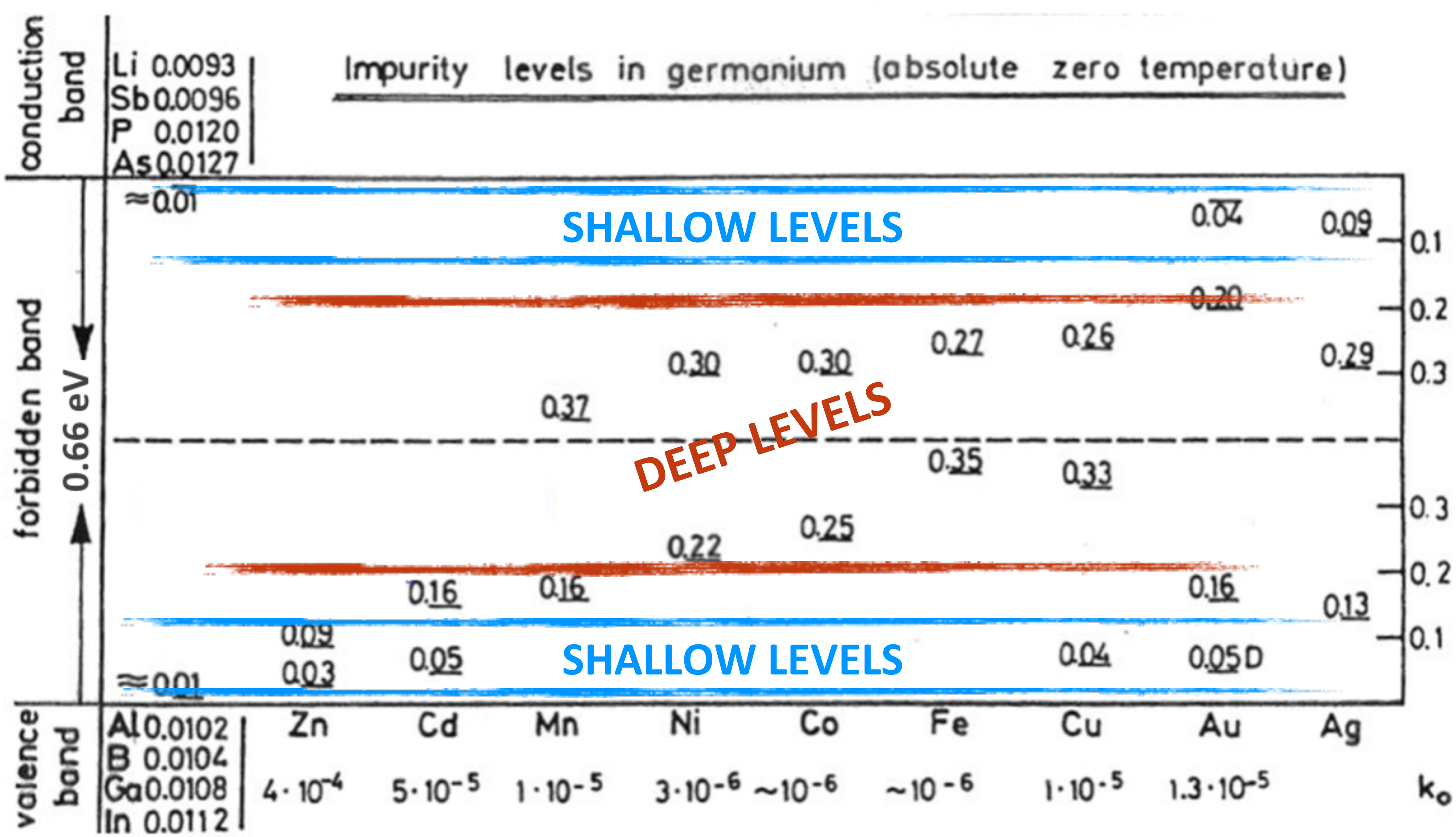
[Conwell, Proceedings of the IRE (1958)]

Electrical activation of defects



[Conwell, Proceedings of the IRE (1958)]

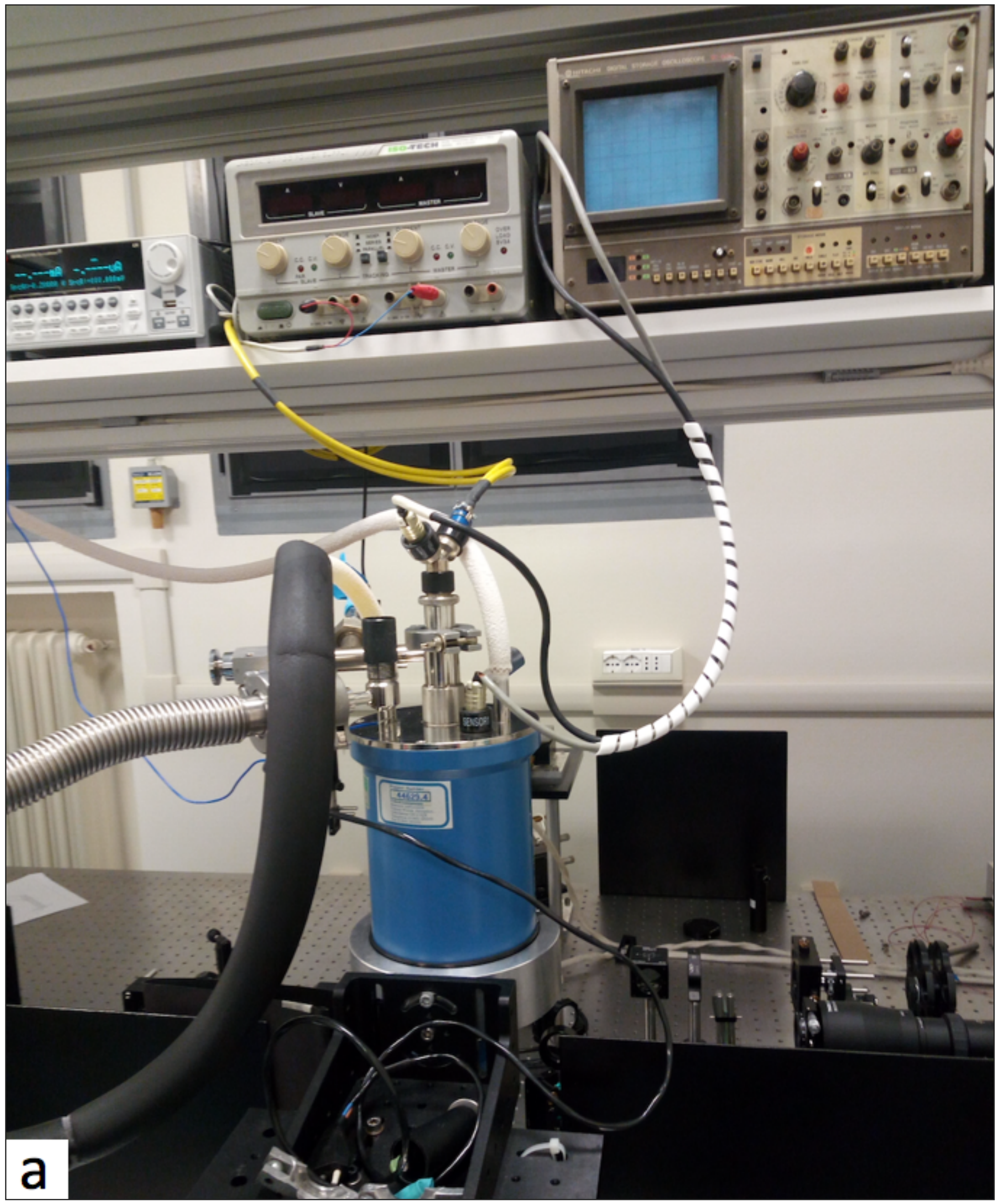
Electrical activation of defects

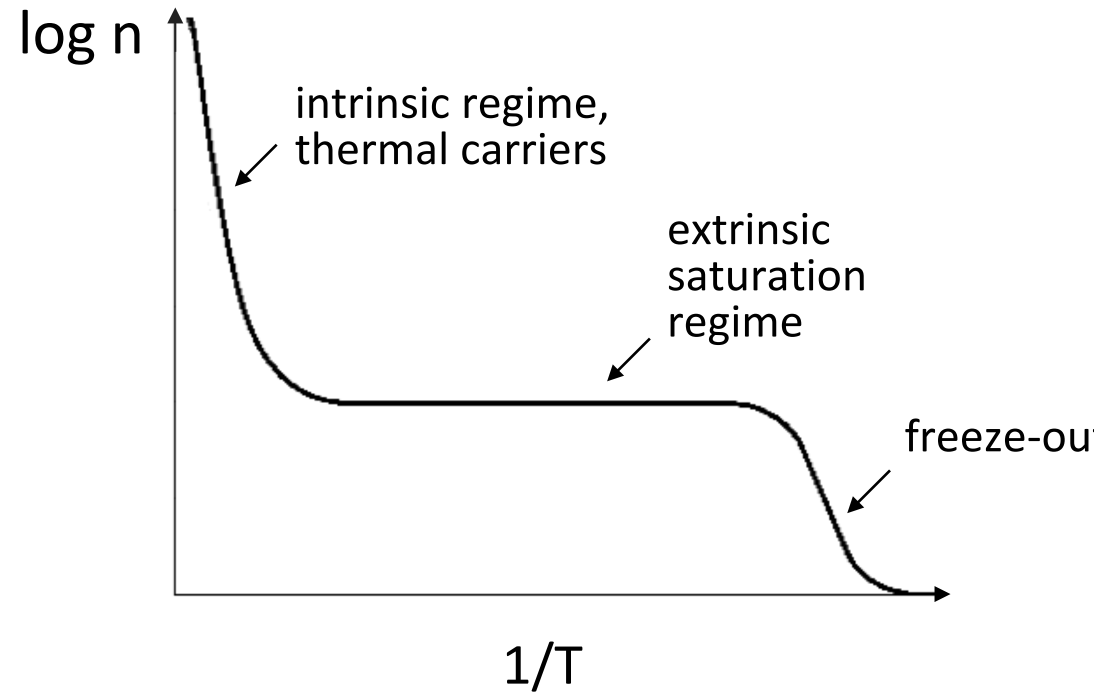


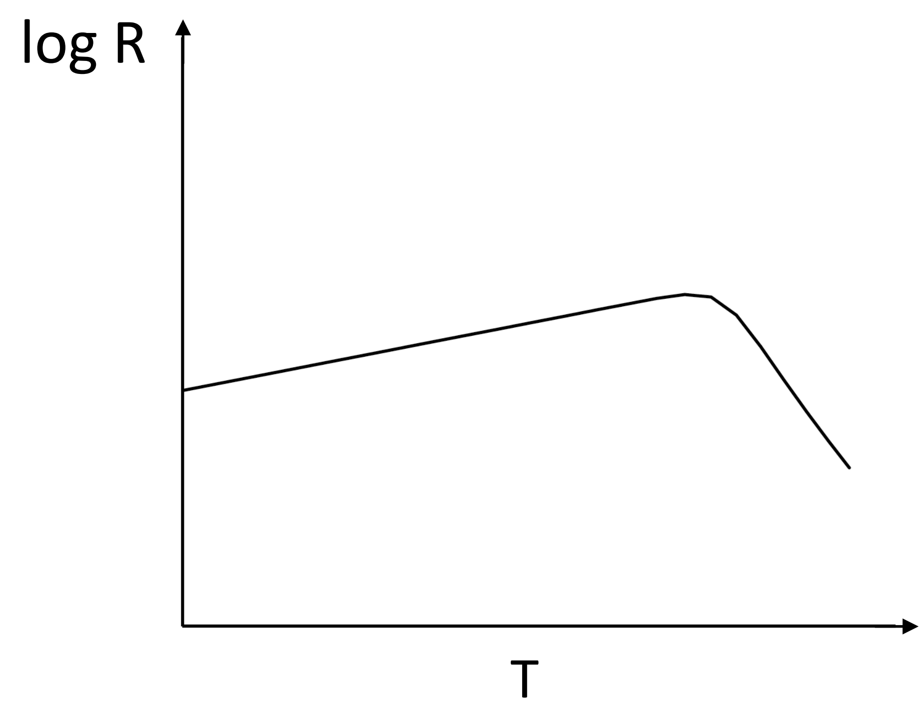
[Conwell, Proceedings of the IRE (1958)]

Carrier density curves at low temperature

Setup. Temperature range [120-300] K







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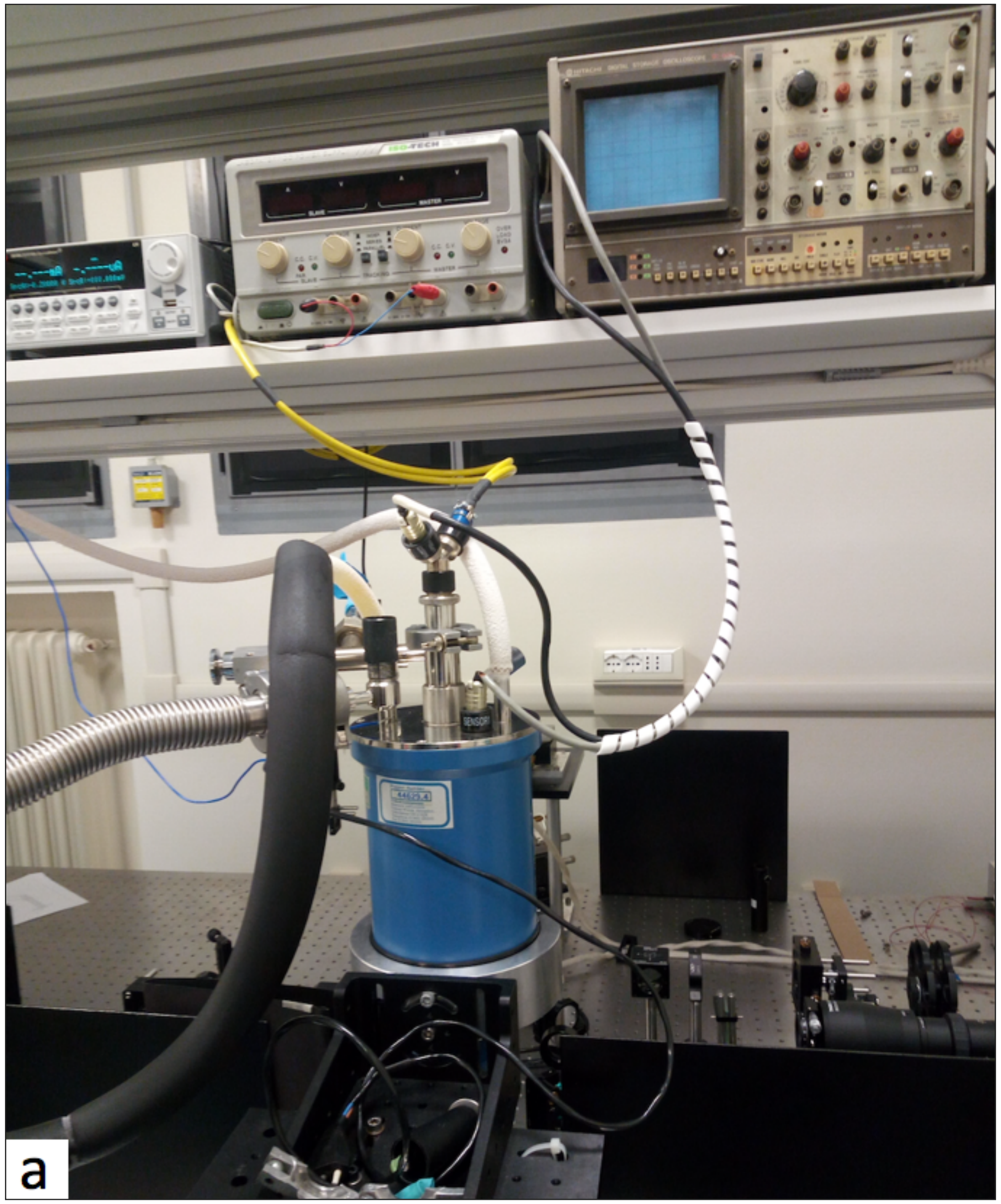

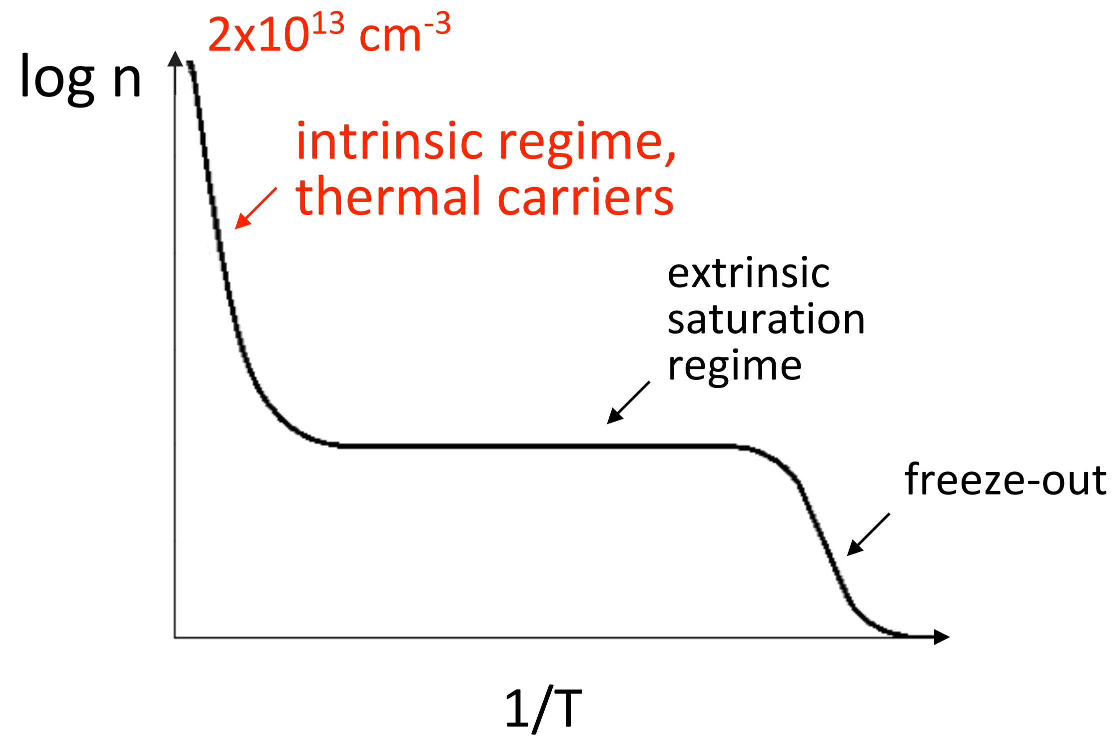

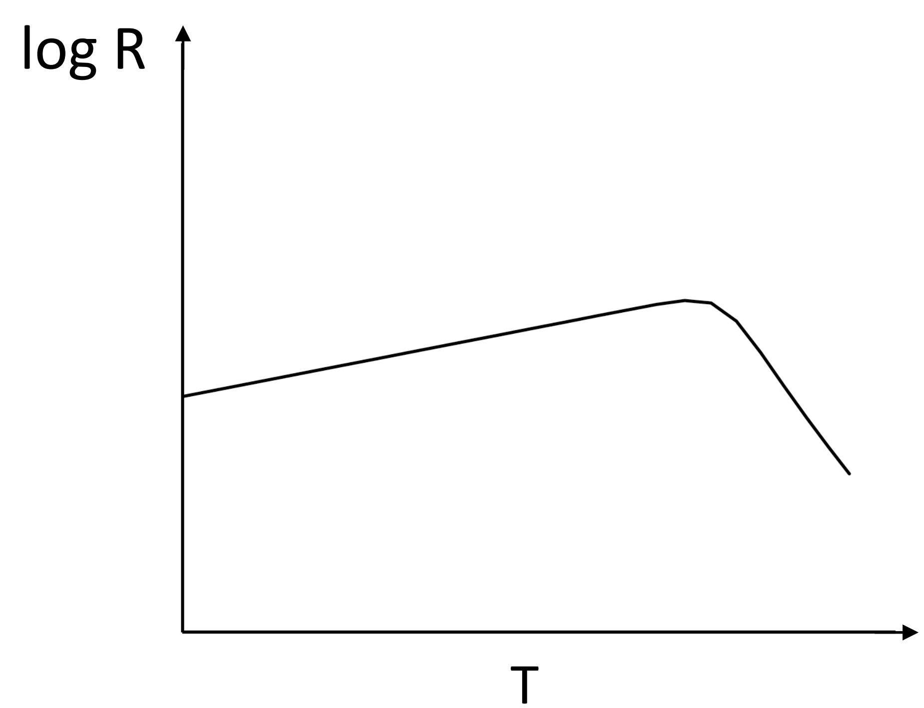

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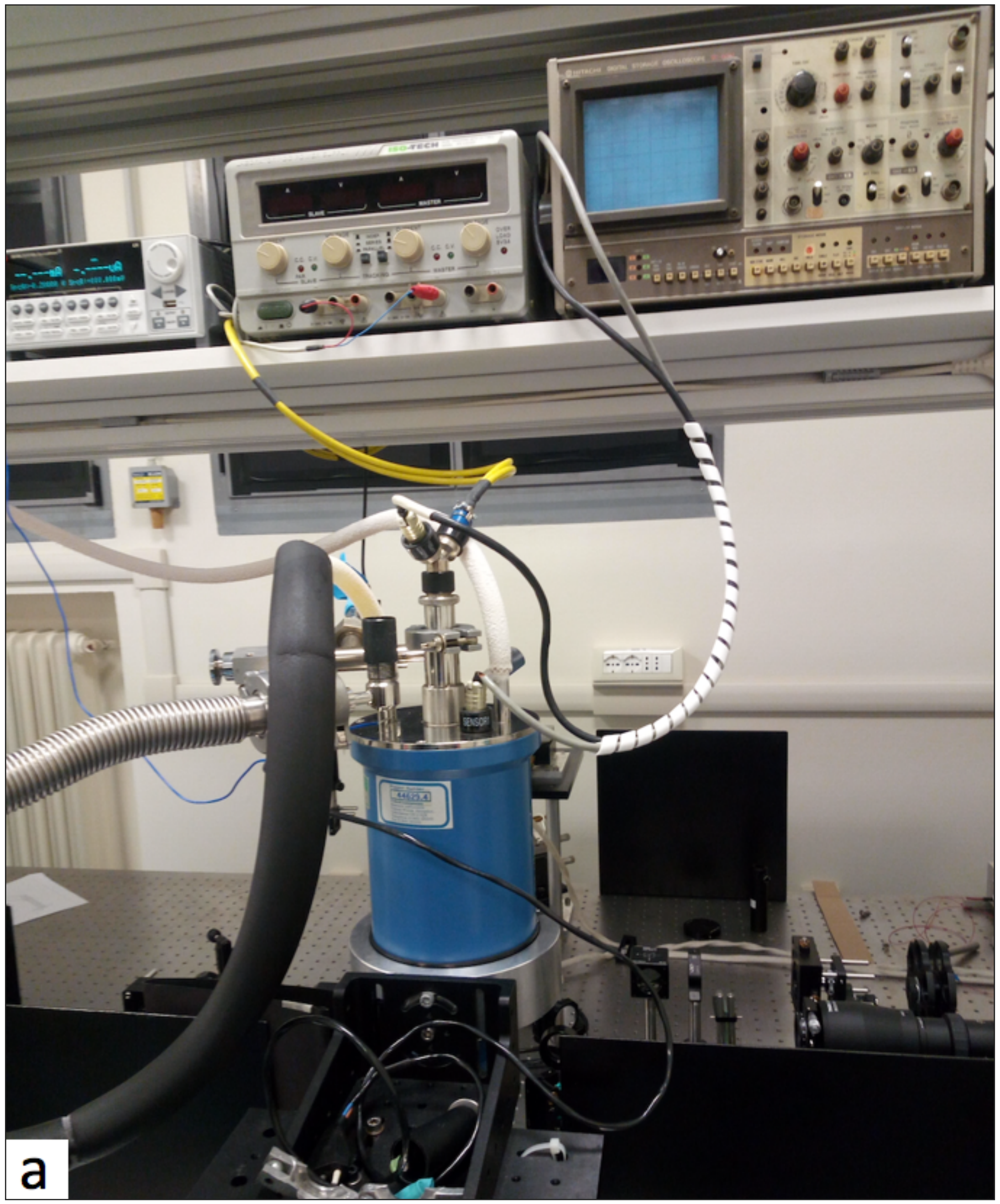

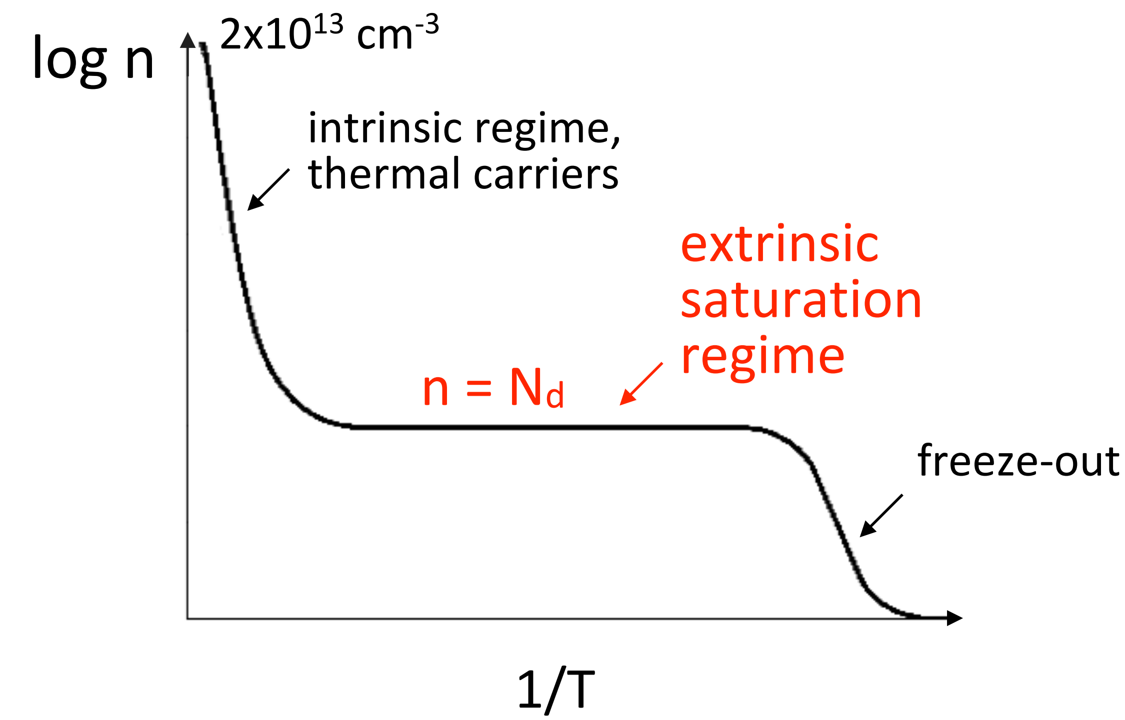

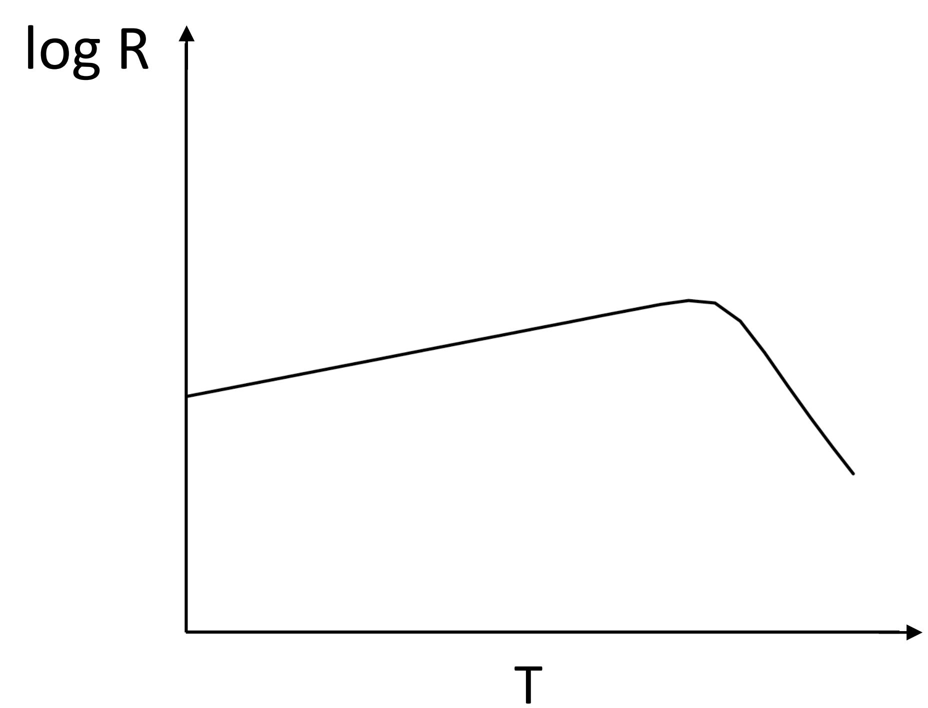

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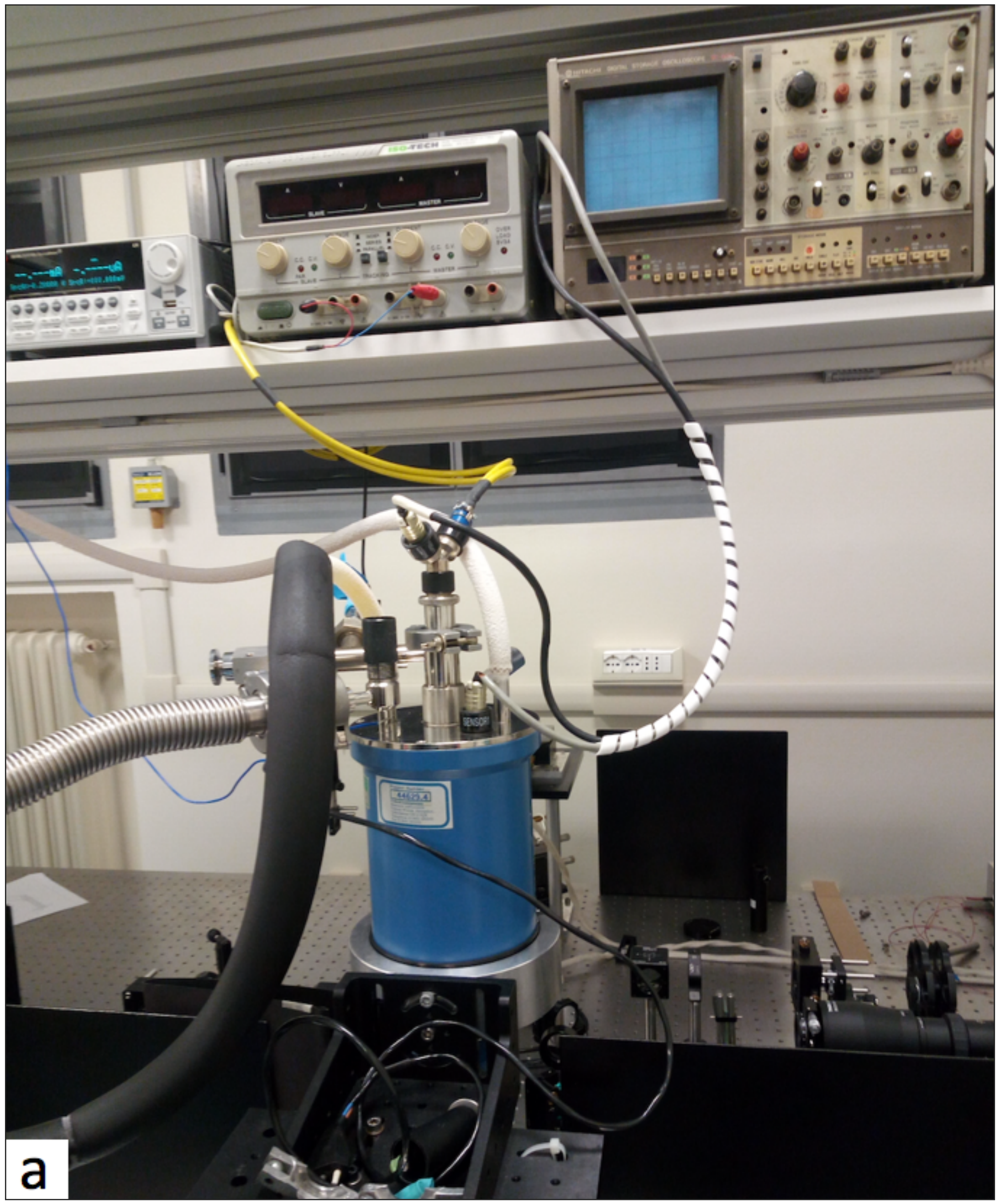

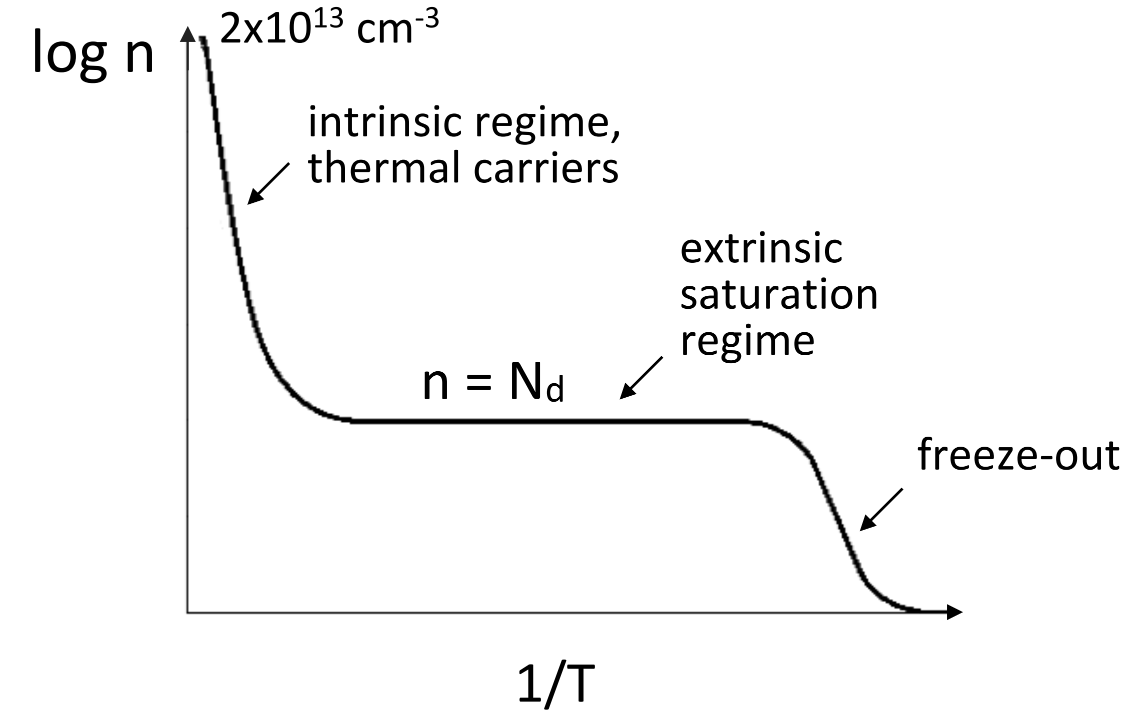

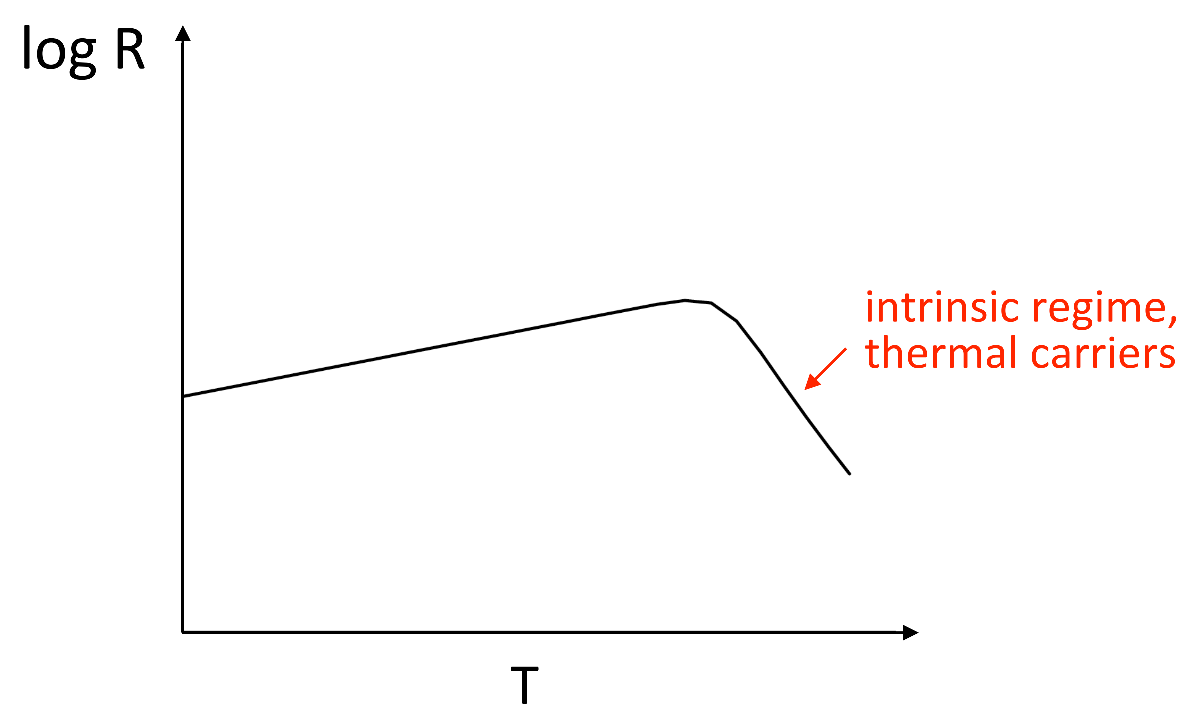

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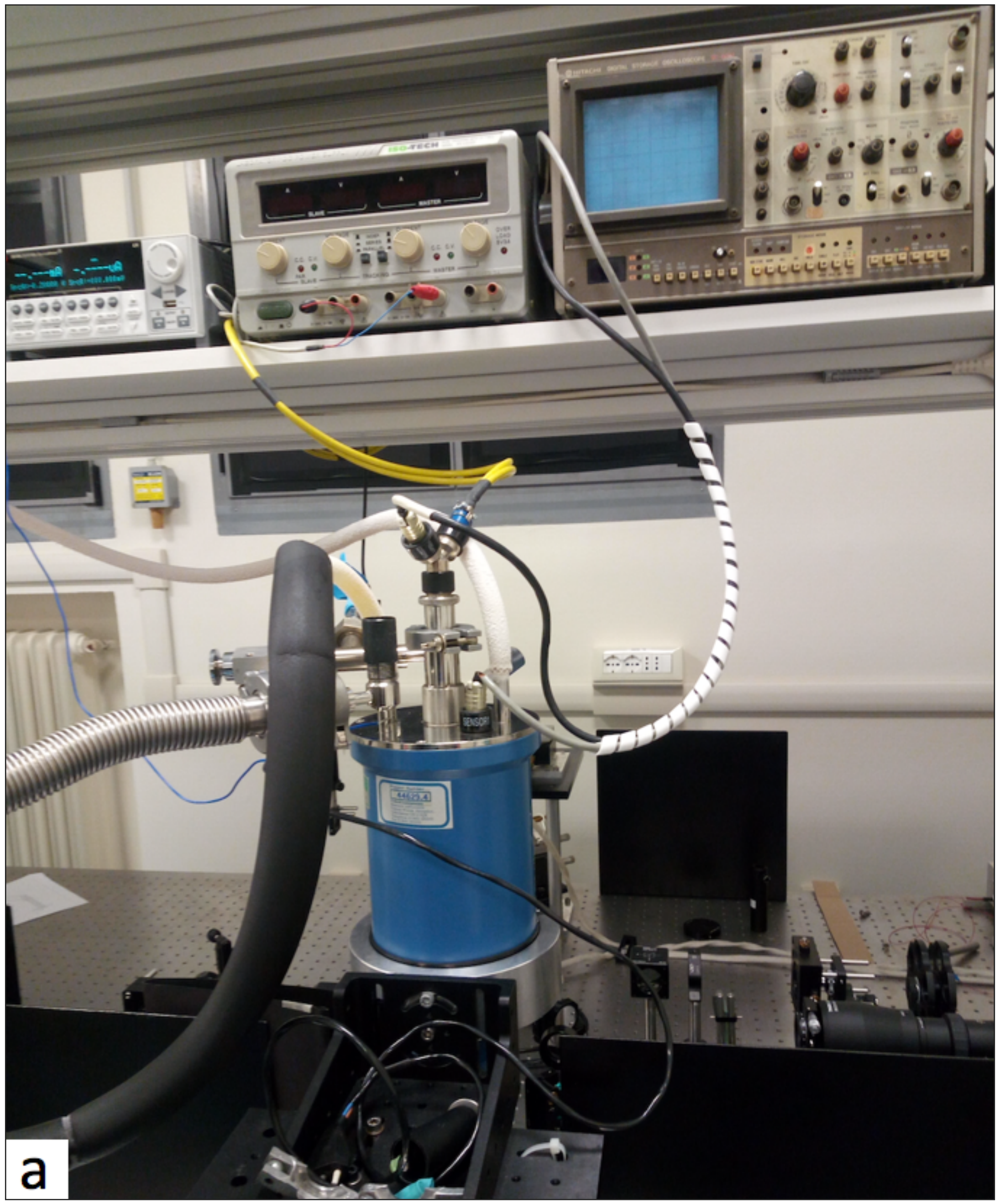

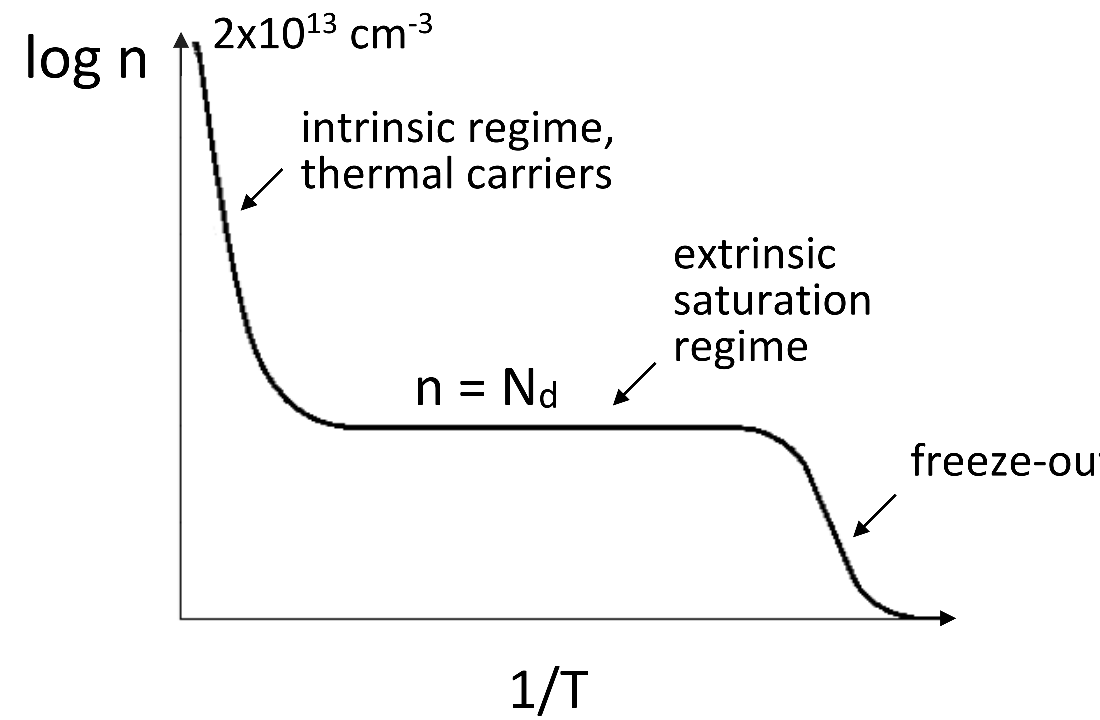

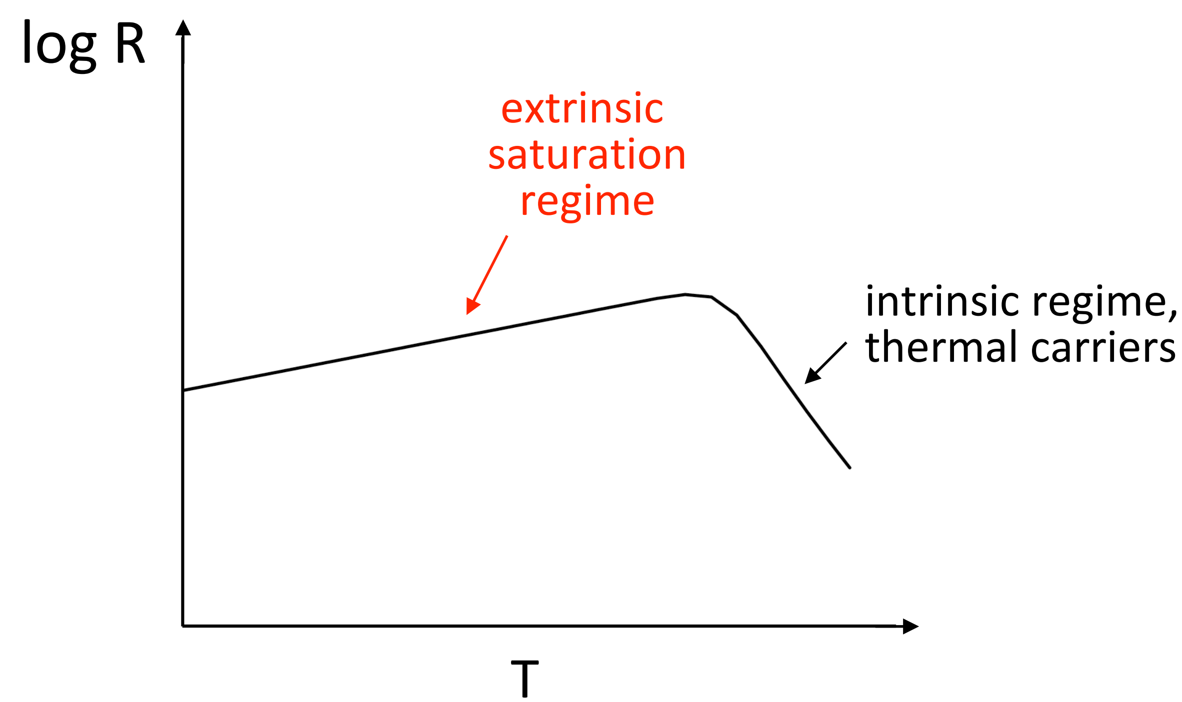

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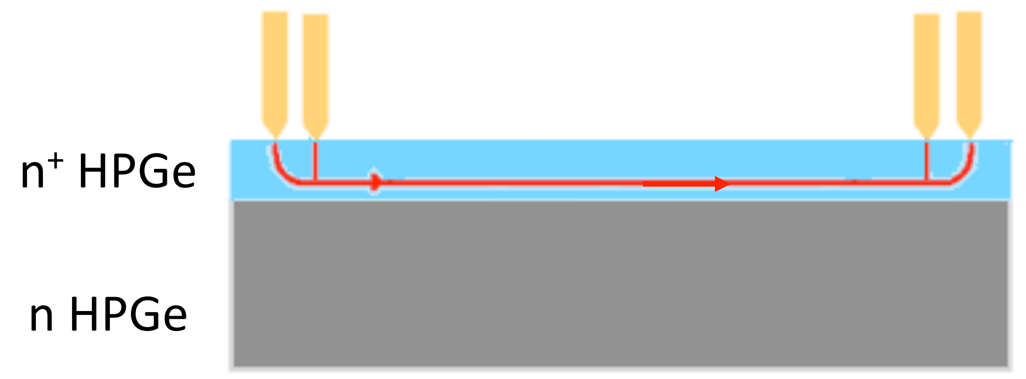
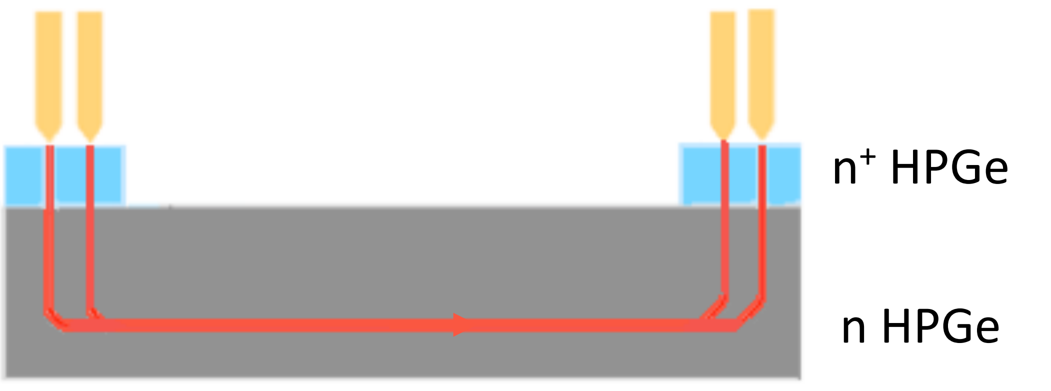
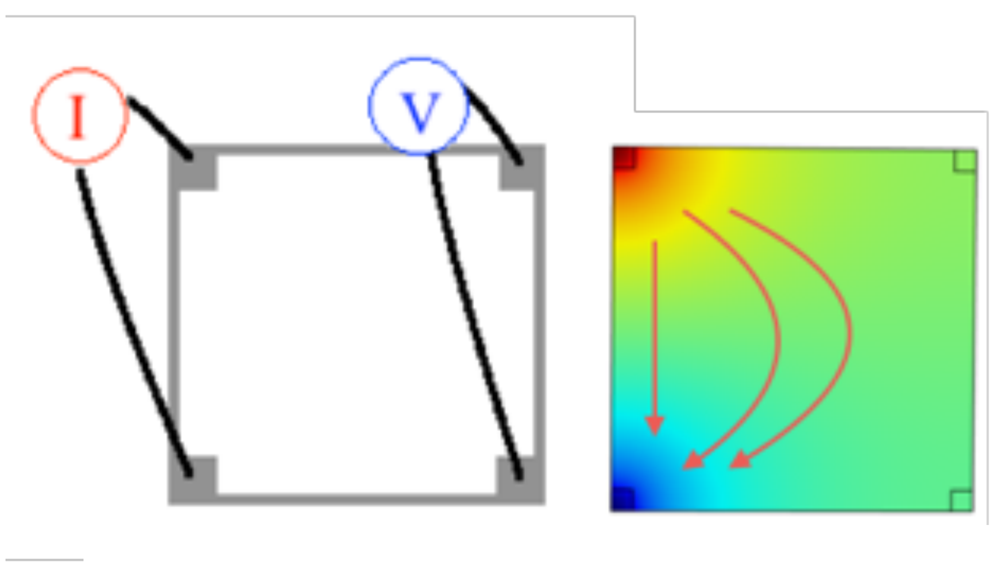
Four wires resistance and Hall measurement

Surface chemical etching in order to measure the electrical properties of bulk HPGe after processes:

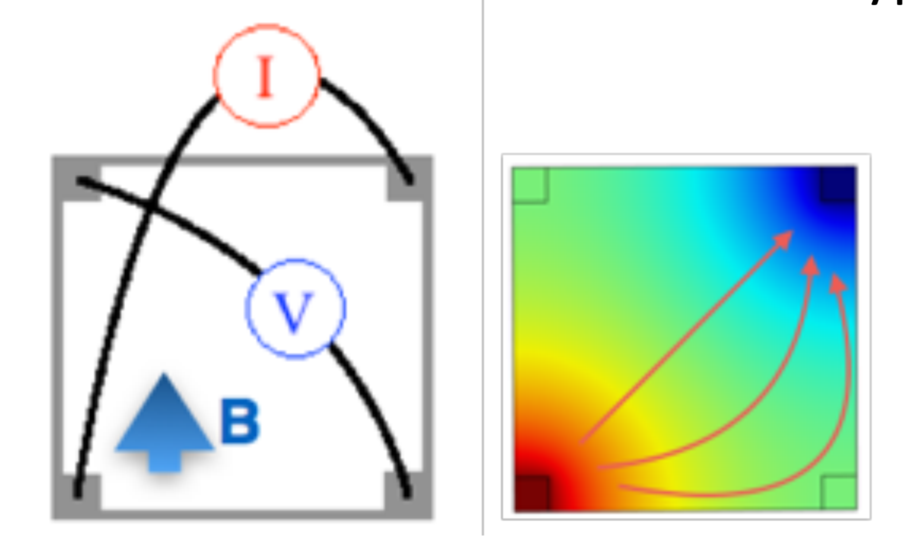




Van der Pauw method ⟶ R_{sheet}

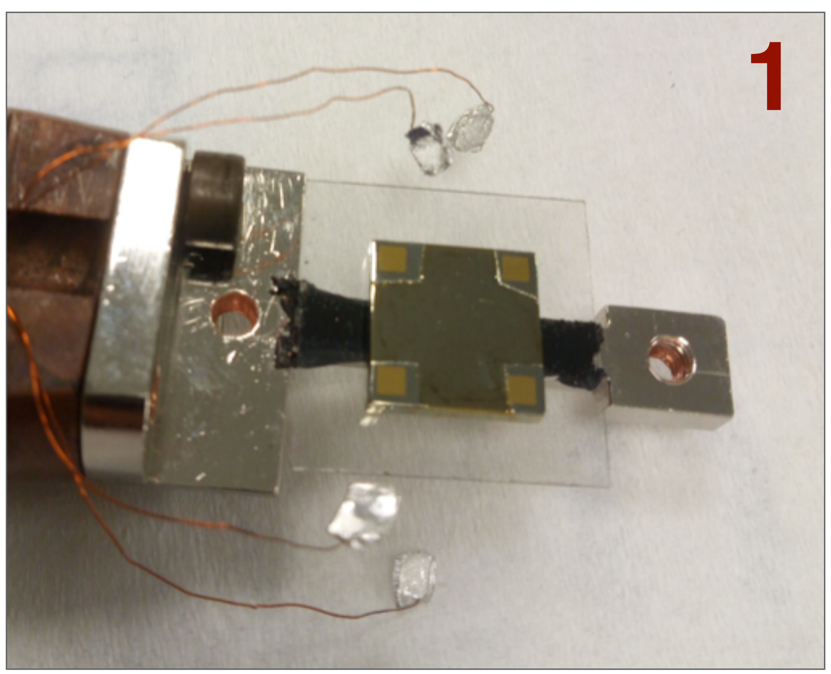


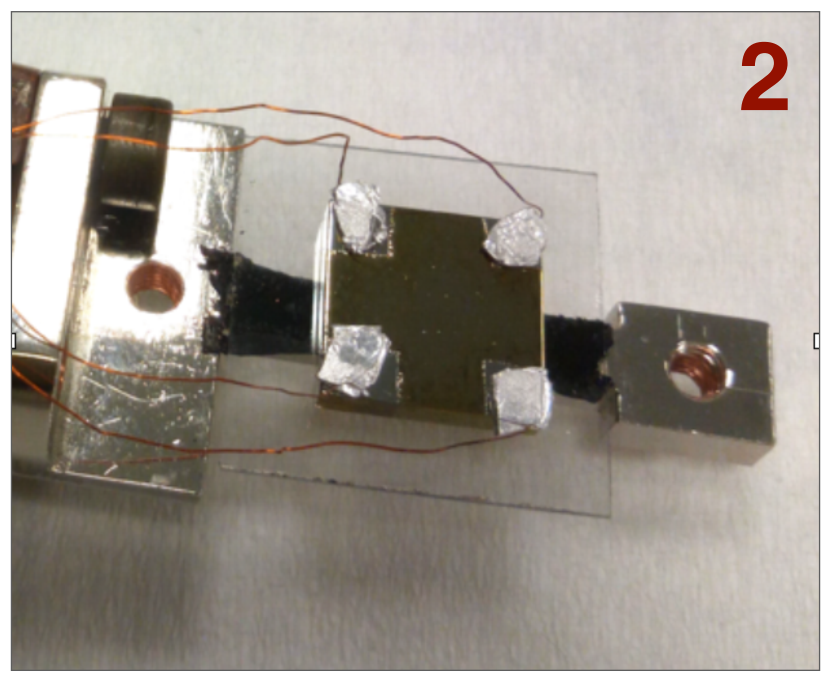
Hall-effect method ⟶ Carrier type

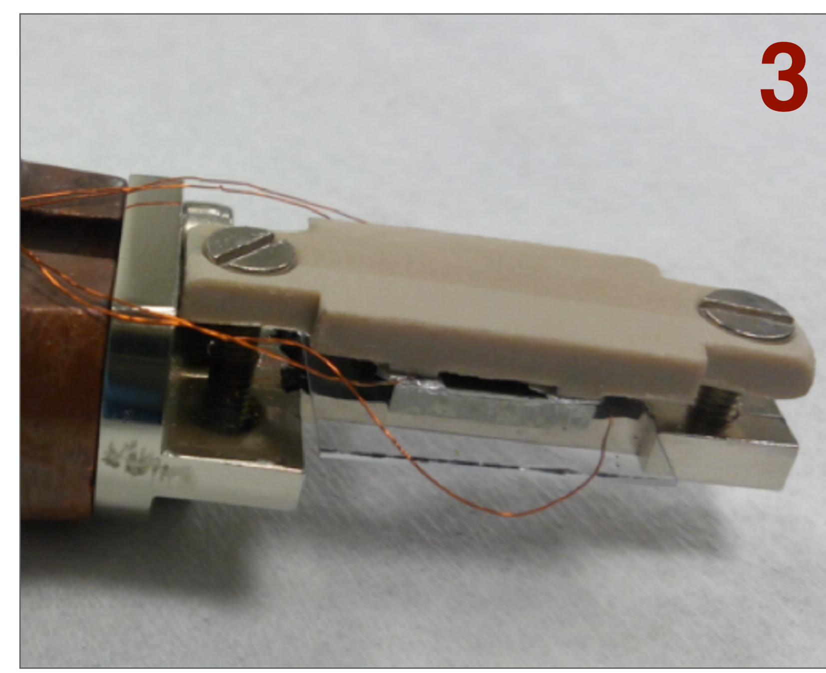


Sample preparation for electrical measurements

- 1 cm^2 area samples were cut from 2 mm thick HPGe wafer.
- 10 µm removal from front surface, by 3:1 HNO_3/HF chemical etching.
- CrAu square electrodes at corners.
- 4 wires pressed on CrAu with malleable In.







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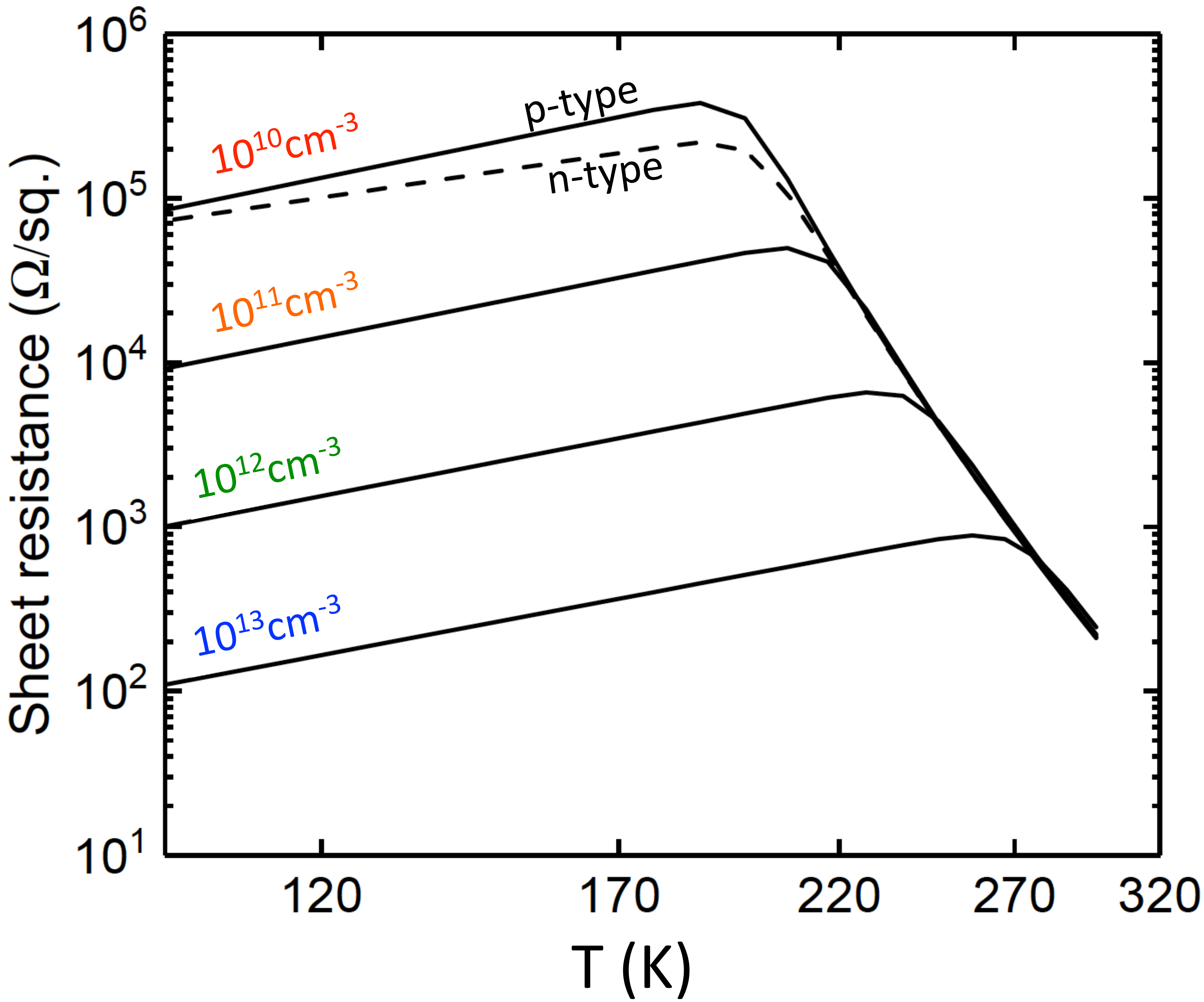
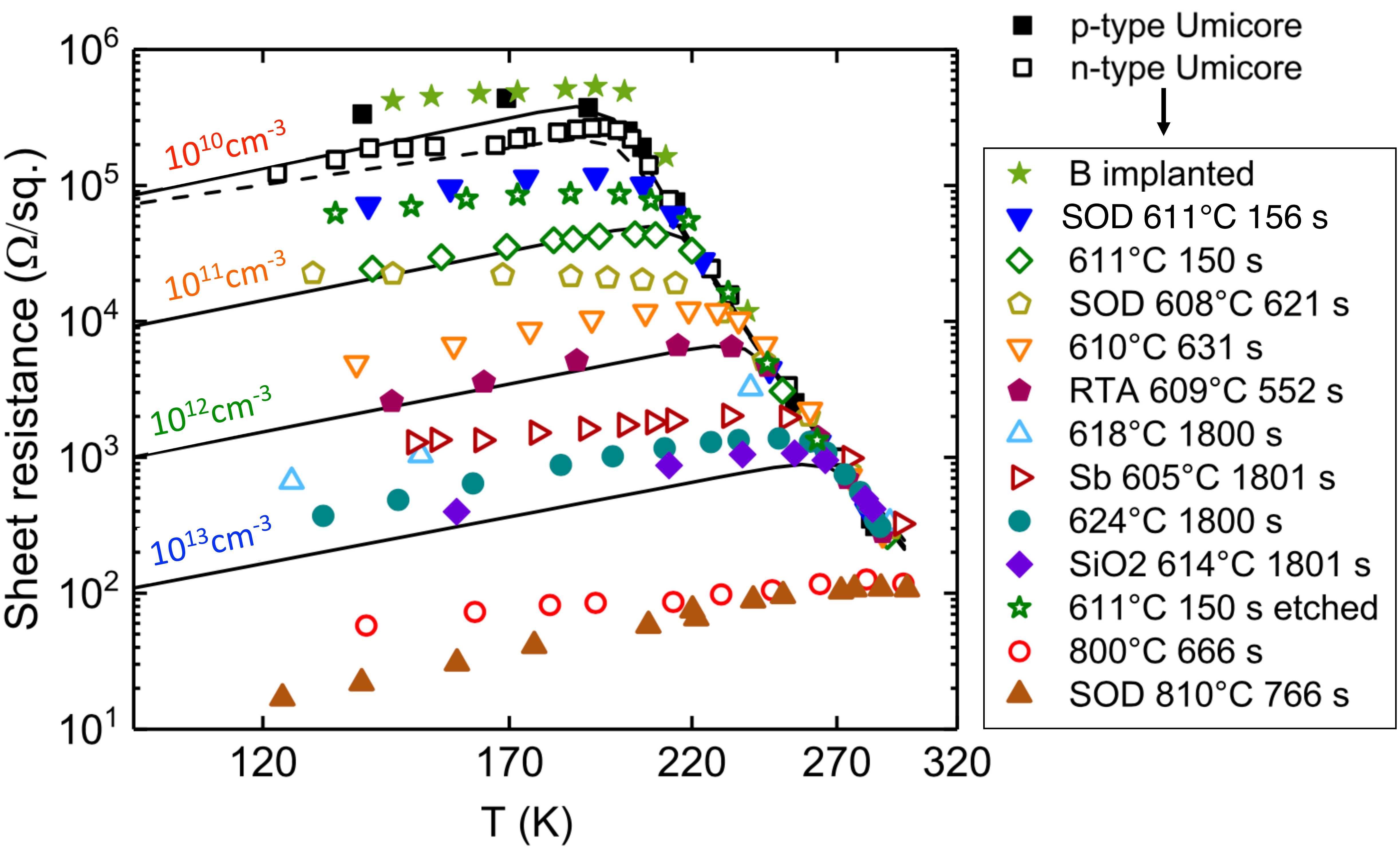
Theoretical sheet resistance curves

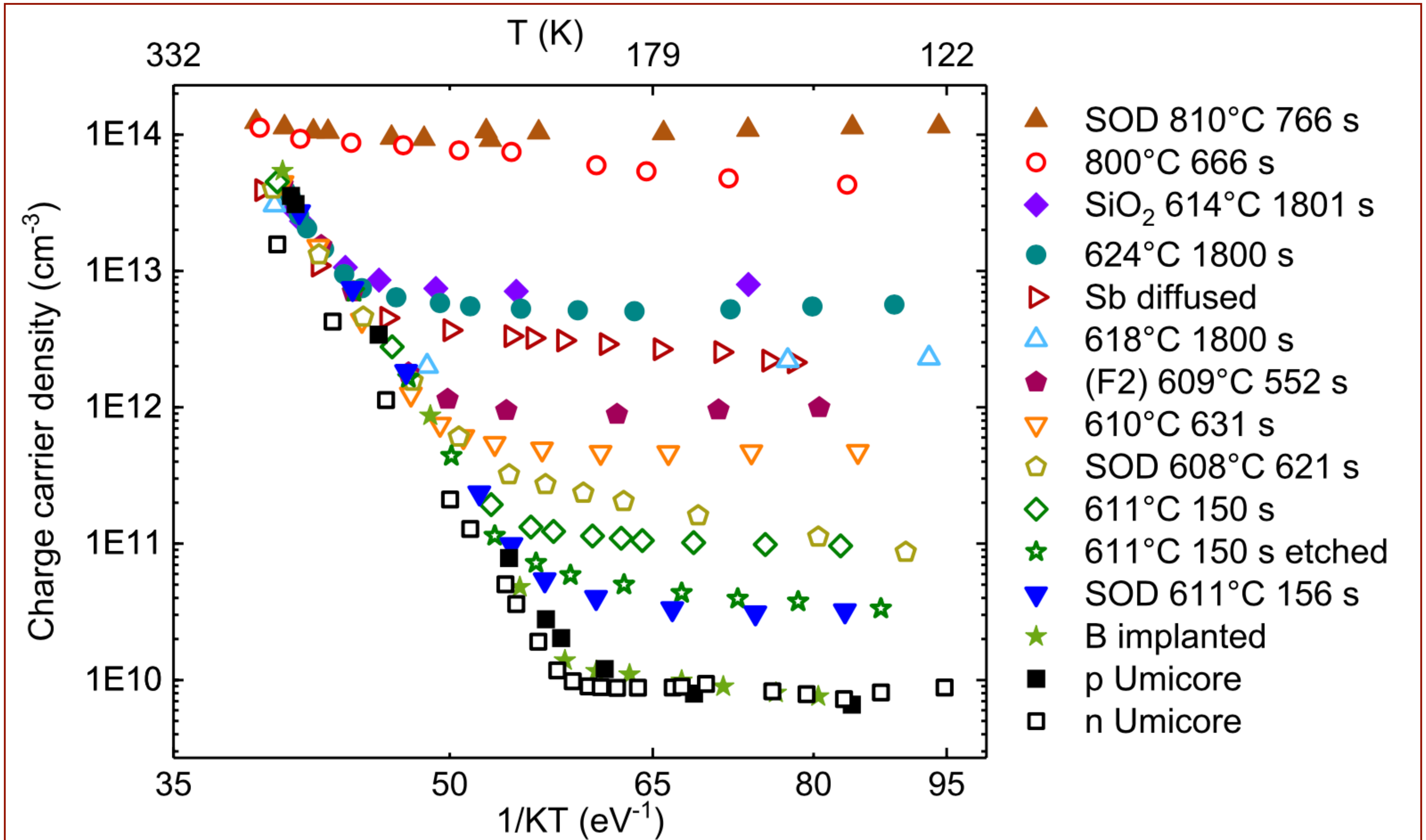


Sheet resistance measurements at low T



Calculation of carrier density

$$R_{sheet}(T) = \frac{1}{t(p(T)e\mu_h(T))}$$



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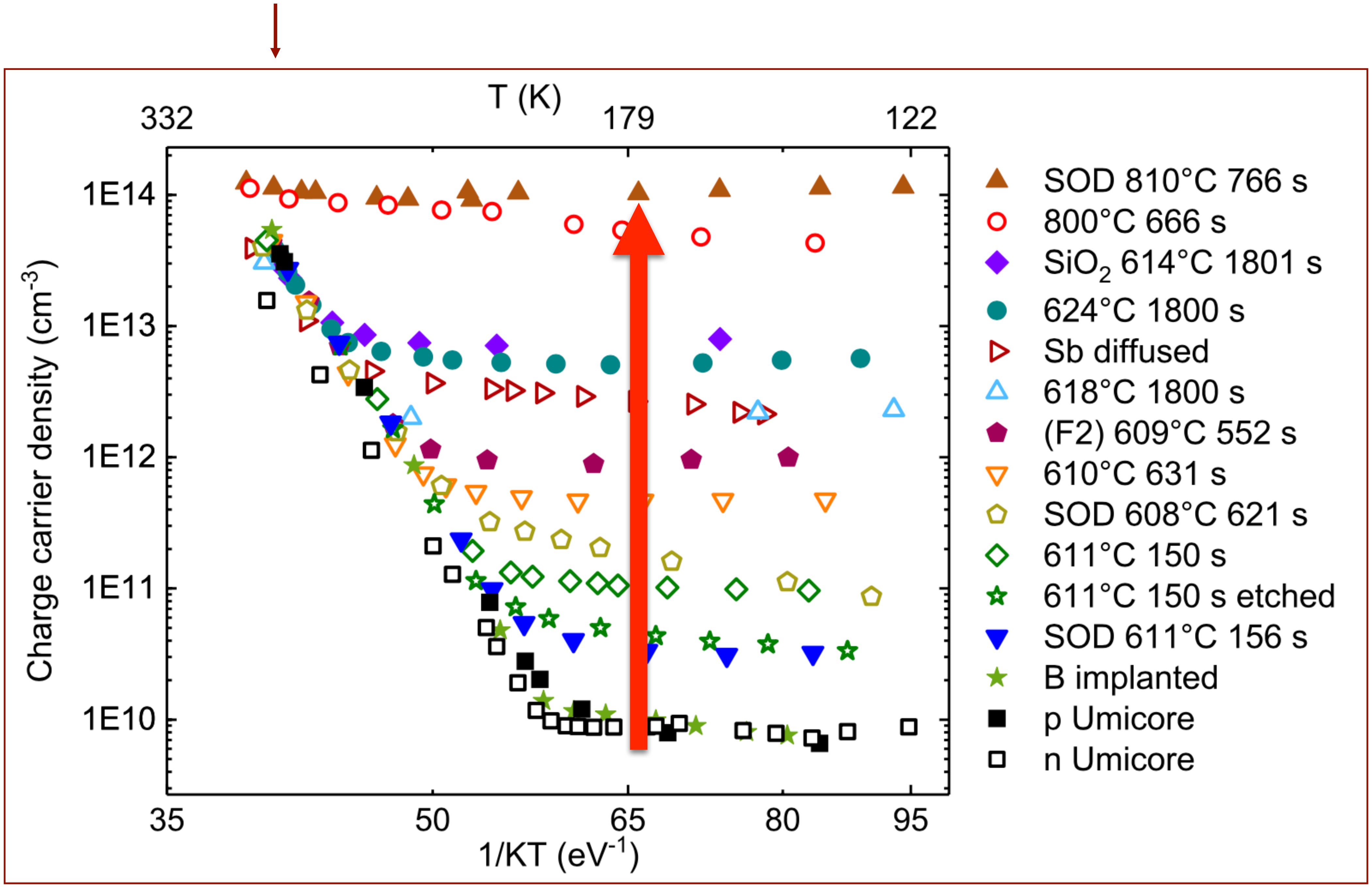

Carrier density and type after processes

HPGe type	Sample description	n_c (cm^{-3})	Type after process
n	n as-cut	$[8.7 \pm 0.7] \times 10^{9}$	**n**
p	p as-cut	$[8.9 \pm 2.9] \times 10^{9}$	**p**

p	B implanted	$[9.5 \pm 1.6] \times 10^{9}$	-
p	SOD 611 °C 156 s	$[3.4 \pm 0.4] \times 10^{10}$	p
n	SOD 608 °C 621 s	$[2.0 \pm 0.9] \times 10^{11}$	-
p	SOD 810 °C 766 s	$[1.1 \pm 0.1] \times 10^{14}$	-
n	Sb 605 °C 1801 s	$[3.0 \pm 0.7] \times 10^{12}$	-
p	SiO_2 614 °C 1801 s	$[7.8 \pm 0.7] \times 10^{12}$	-
n	611 °C 150 s	$[1.1 \pm 0.1] \times 10^{11}$	-
n	611 °C 150 s etch.	$[4.8 \pm 1.4] \times 10^{10}$	-
p	(F2) 609 °C 552 s	$[9.9 \pm 1.0] \times 10^{11}$	p
n	610 °C 631 s	$[5.0 \pm 0.5] \times 10^{11}$	p
n	618 °C 1800 s	$[2.2 \pm 0.2] \times 10^{12}$	p
p	624 °C 1800 s	$[5.7 \pm 0.8] \times 10^{12}$	-
n	800 °C 666 s	$[6.9 \pm 1.8] \times 10^{13}$	p

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An Arrhenius relation exists between contaminant density and temperature, with further dependence on time.

$$n_{eq} = n_0 exp\left(-\frac{E_{act}}{k_B T}\right) \qquad (1)$$

$$\frac{dn}{dt} = r(n_{eq} - n) \qquad (2)$$

$$(n_{eq} >> n)$$

$$\frac{dn}{dt} = r\, n_0\, exp\left(-\frac{E_{act}}{k_B T(t)}\right) \qquad (3)$$

$$n = r\, n_0 \int exp\left(-\frac{E_{act}}{k_B T(t)}\right) dt \qquad (4)$$

Empirical model: contamination density and thermal budget

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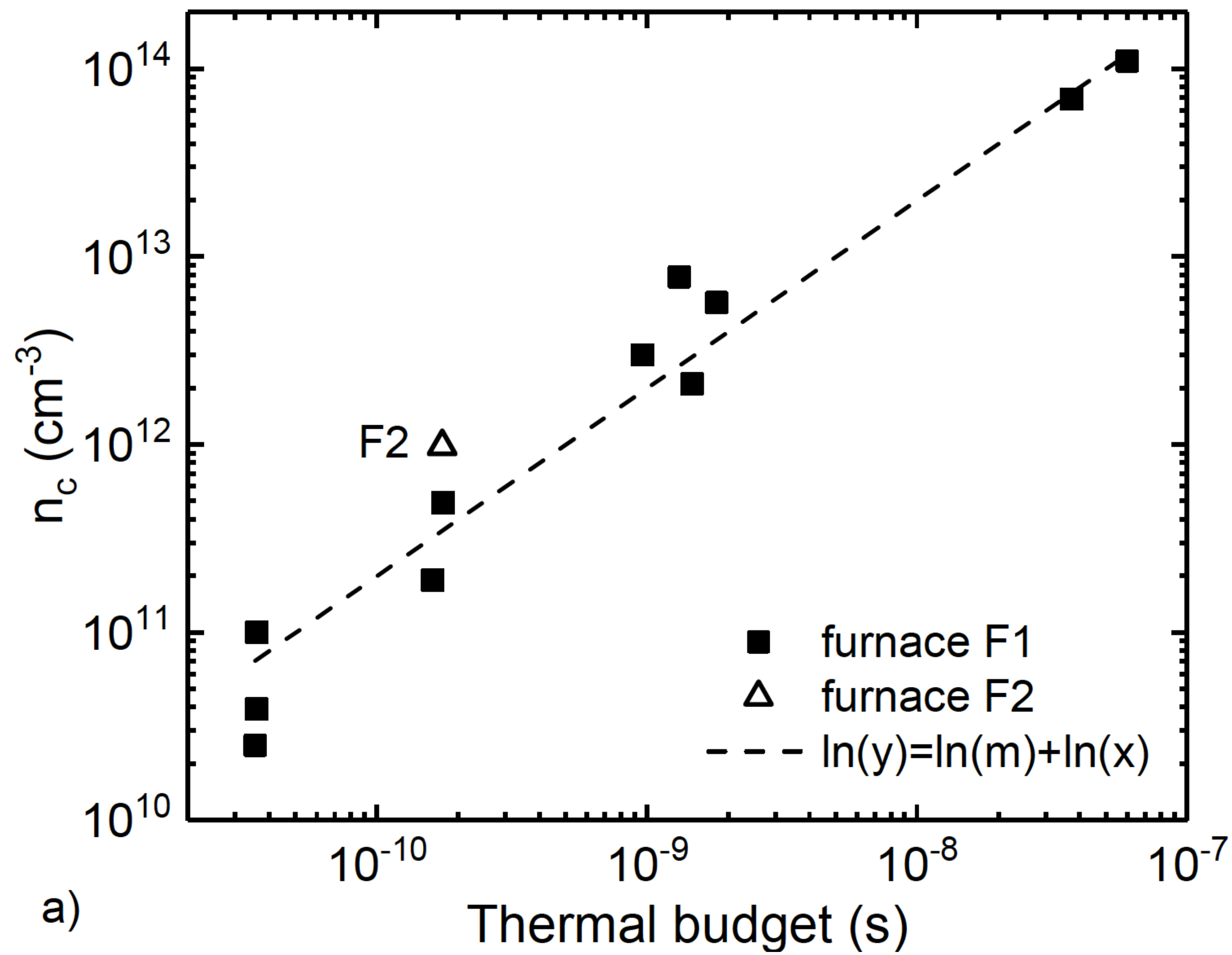
TB

$$n = r\, n_0 \int exp\left(-\frac{E_{act}}{k_B T(t)}\right) dt \qquad (4)$$

$$ln(n) = ln(r n_0) + ln(TB) \qquad (5)$$

Fit results

Best linear fit between $ln(n_c)$ and $ln(TB)$ is found through the minimization of the reduced chi squared.

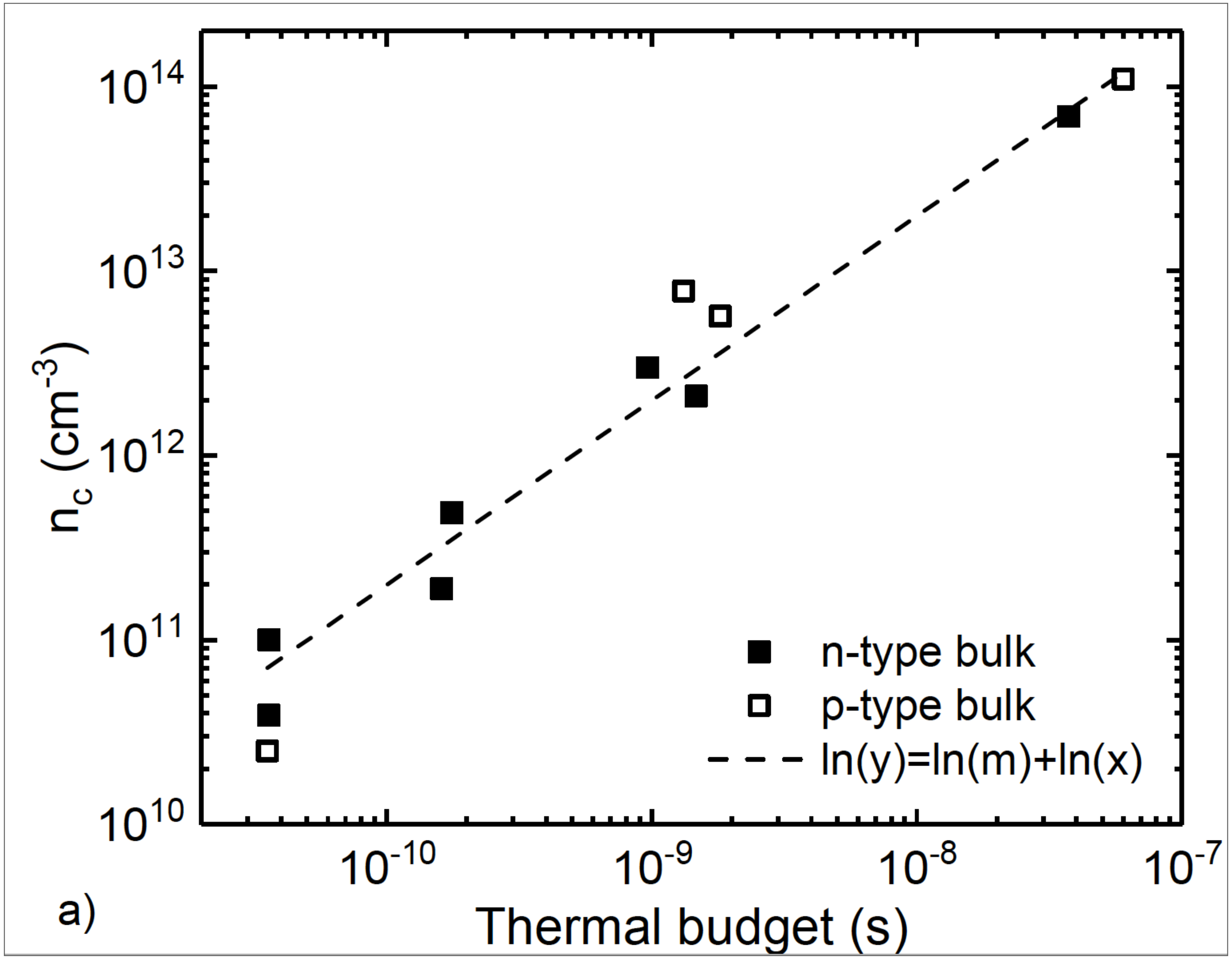


Best fit results:

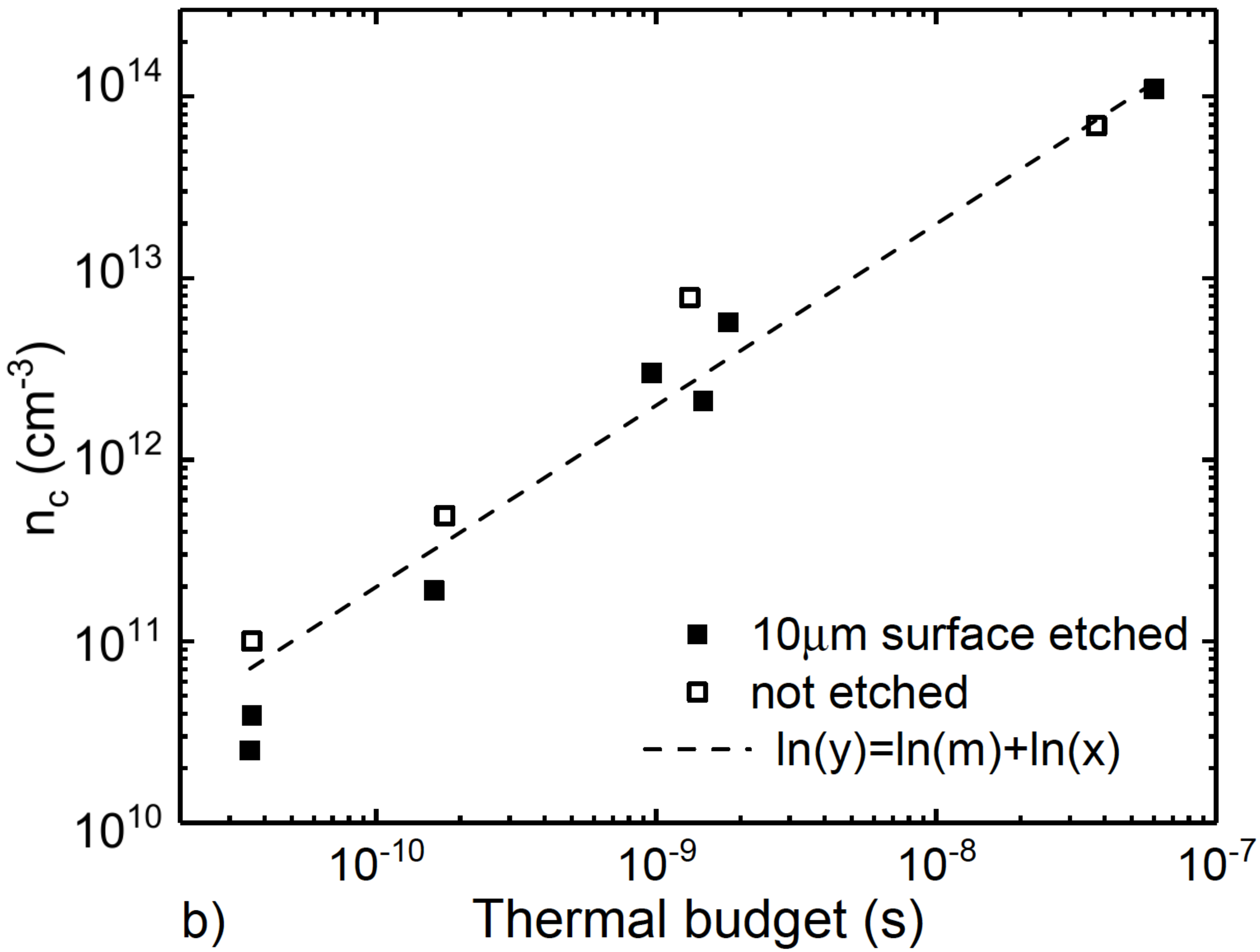
E_{act} = 2.1±0.1 eV

$r \cdot n_0$ = 2x10^{21} $cm^{-3}s^{-1}$

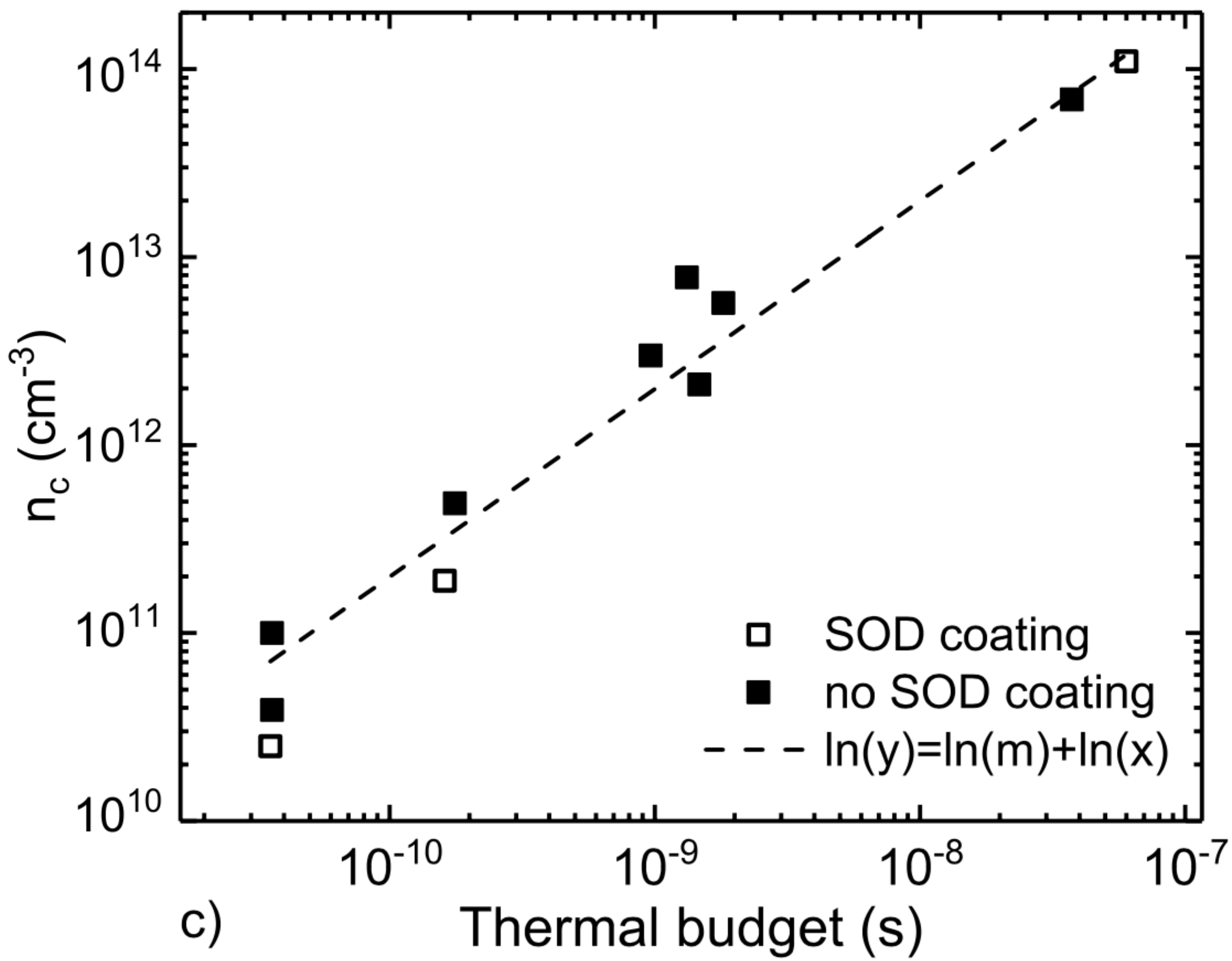
Comparison n-type/p-type



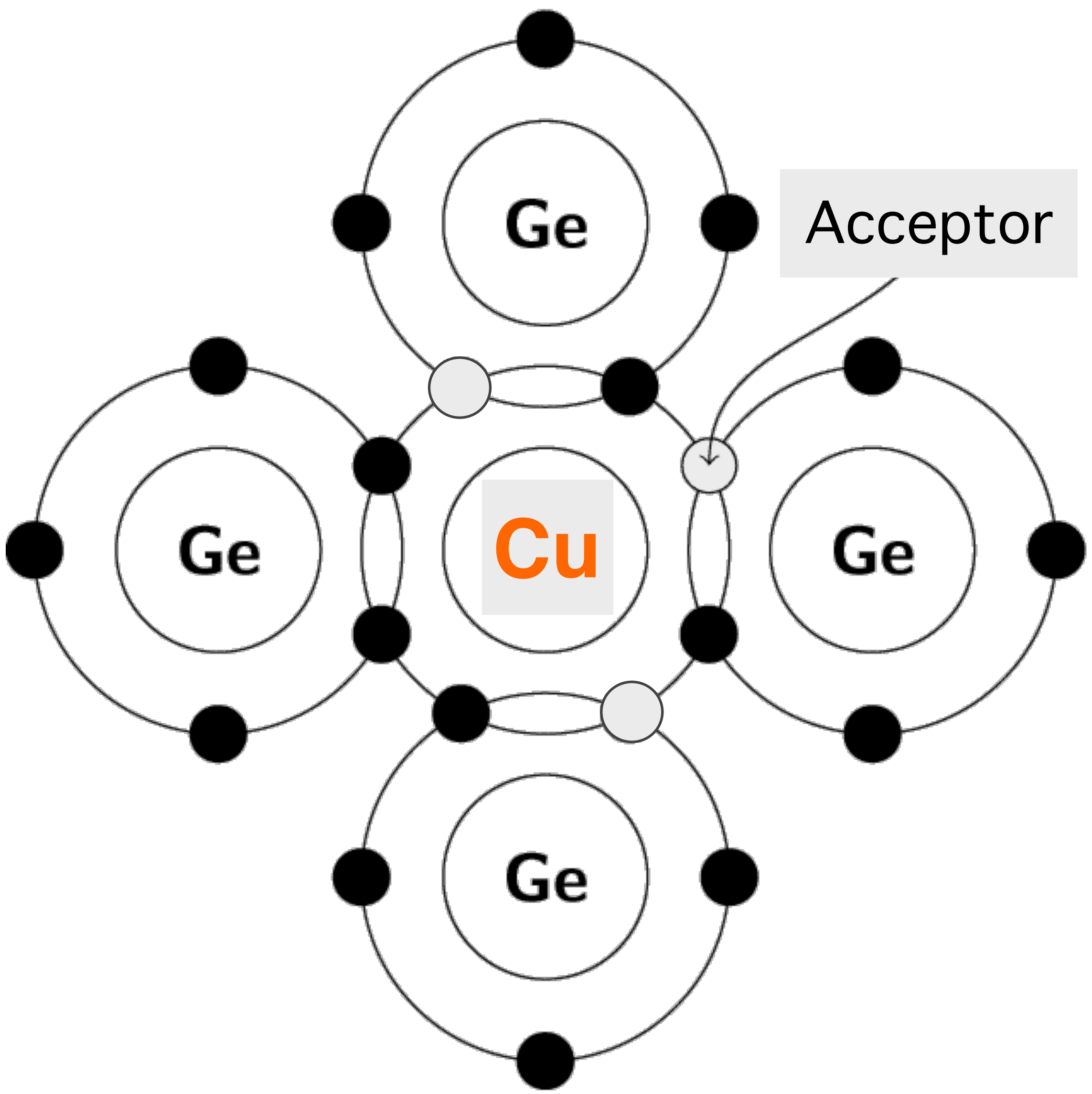
Comparison surface etched/non-etched



Comparison SOD coated/non-coated



What contaminant are we dealing with?



[Bracht, 2004]

In highly dislocated Ge, vacancies are provided by dislocations themselves, thus their density is fixed at thermal equilibrium C_V^{eq}.

Cu atoms diffuse through interstitials, very rapidly.

$$Cu_i + V \longleftrightarrow Cu_s$$

$$D_{Cu_s}^{eff} = 7.8 \cdot 10^{-4} exp\left(-\frac{0.084eV}{k_B T}\right)\ cm^2/s$$

$$C_{Cu_s}^{eq} = 3.44 \cdot 10^{23} exp\left(-\frac{1.56eV}{k_B T}\right)\ cm^{-3}$$

$$E_{act} = E_{act}(C_{Cu_s}^{eq}) + E_{act}(DC_{Cu_s}^{eff}) = 1.64eV$$

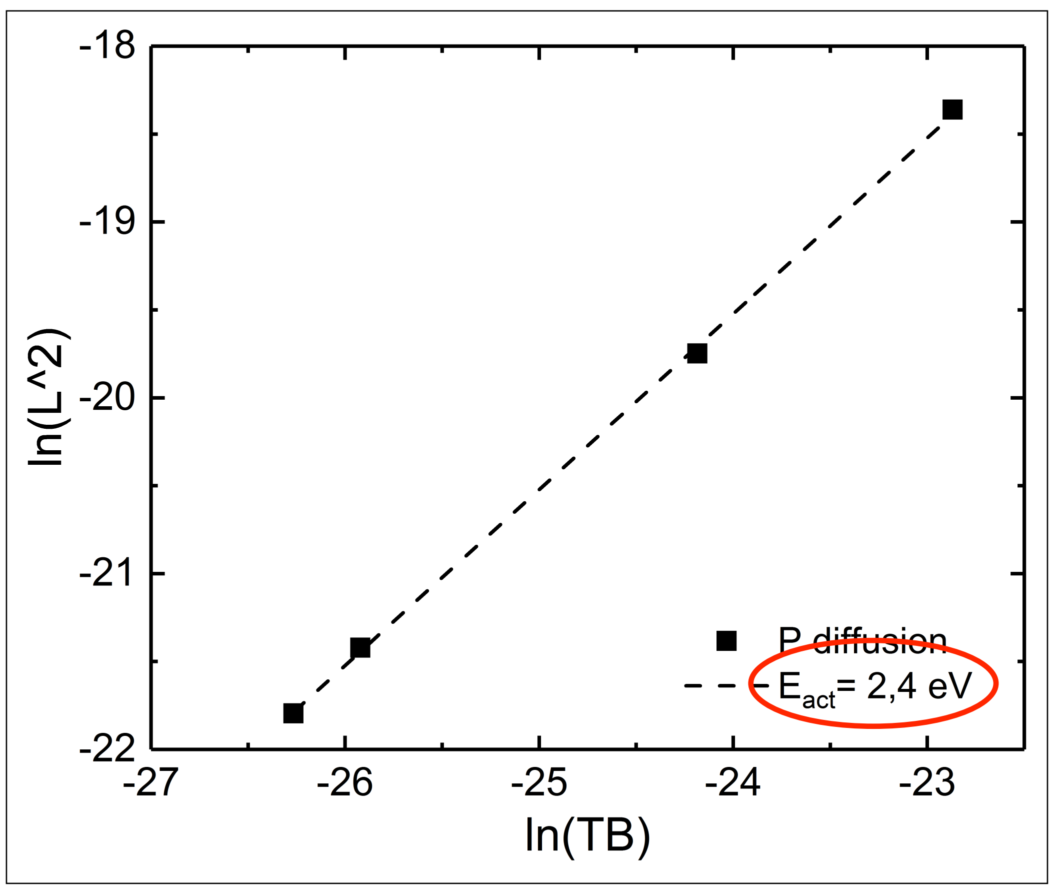
Diffusion length and thermal budget

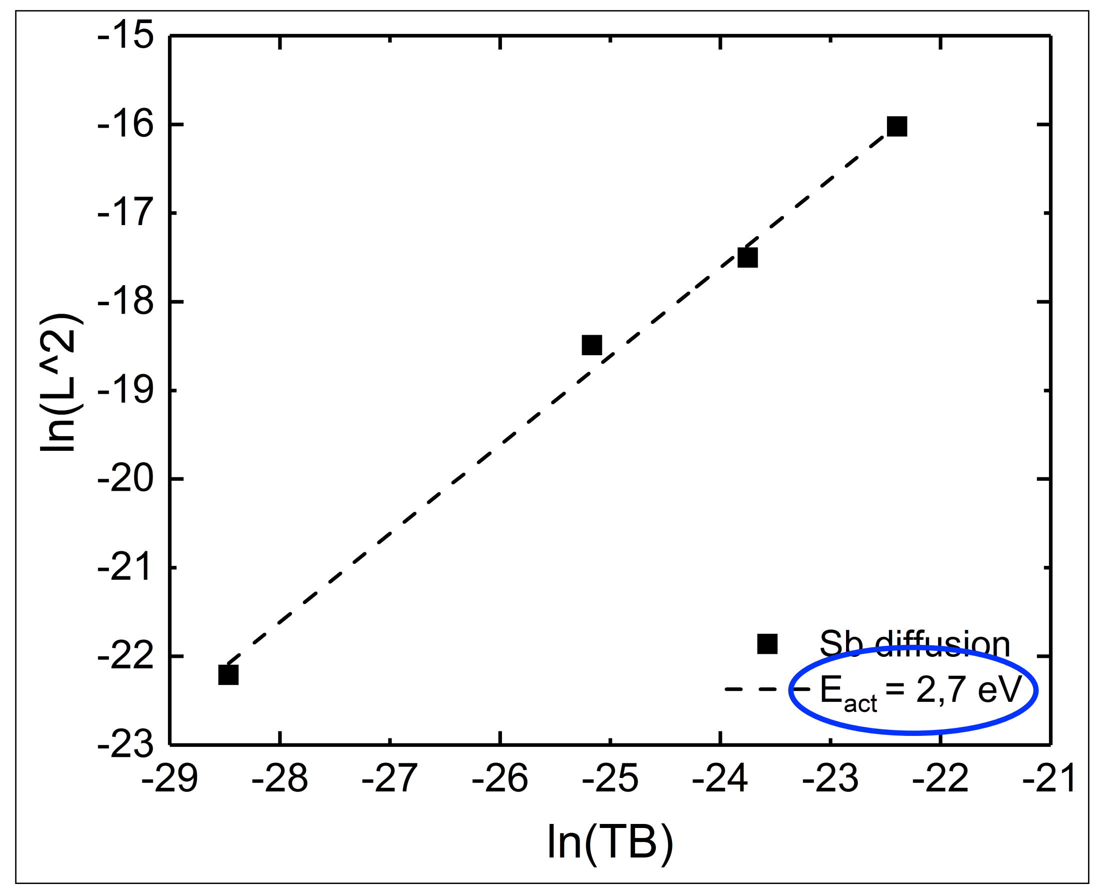
The dependence between diffusion coefficient and temperature is of Arrhenius type:

$$D = D_0 \; exp(-E_{act}/(k_B T))$$

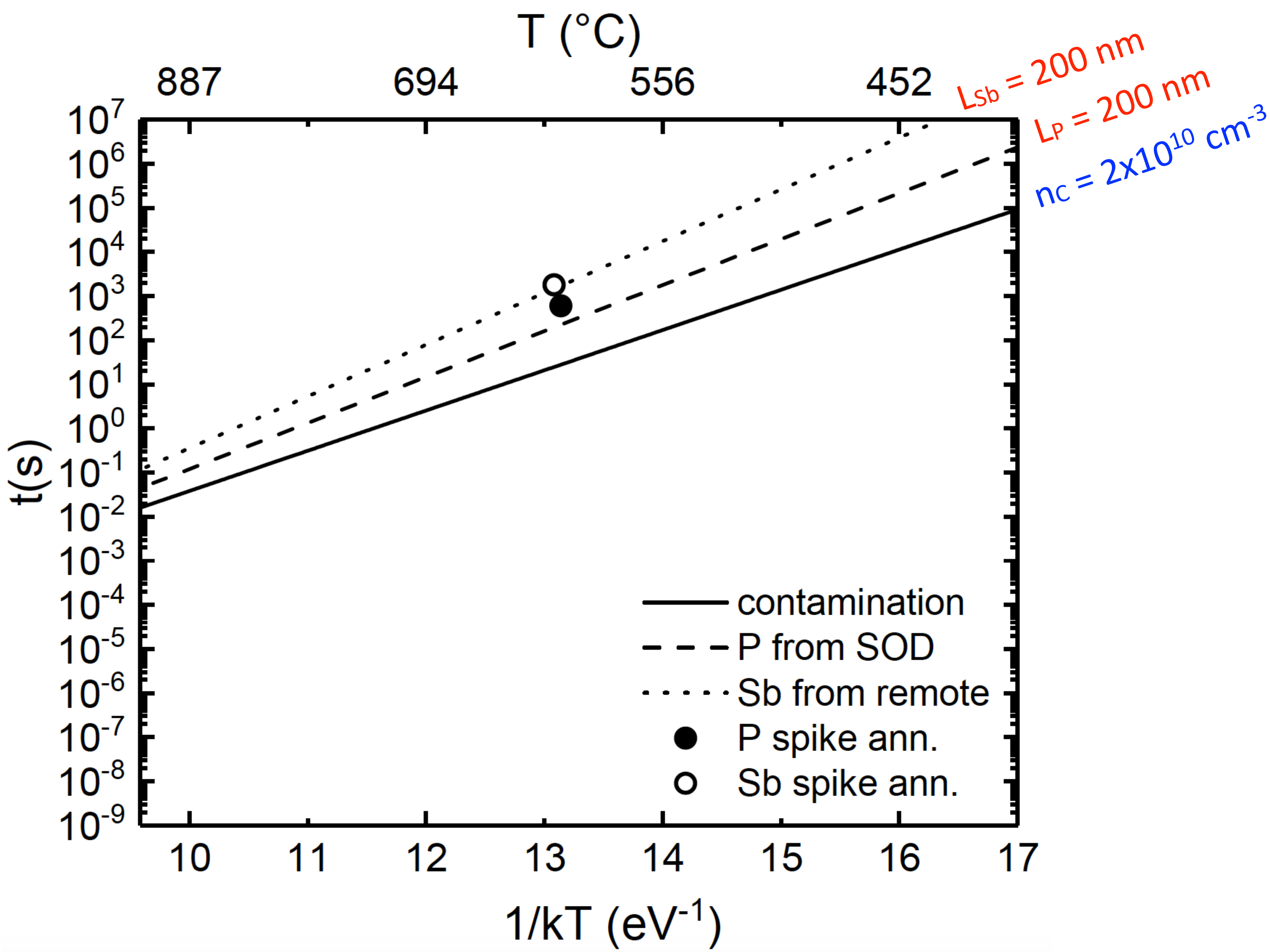
$$L^2 = D \cdot t$$

$$L^2 = L_0 \int exp(-E_{act}/k_B T(t))\,dt \longrightarrow ln(L^2) = ln(L_0) + ln(TB)$$

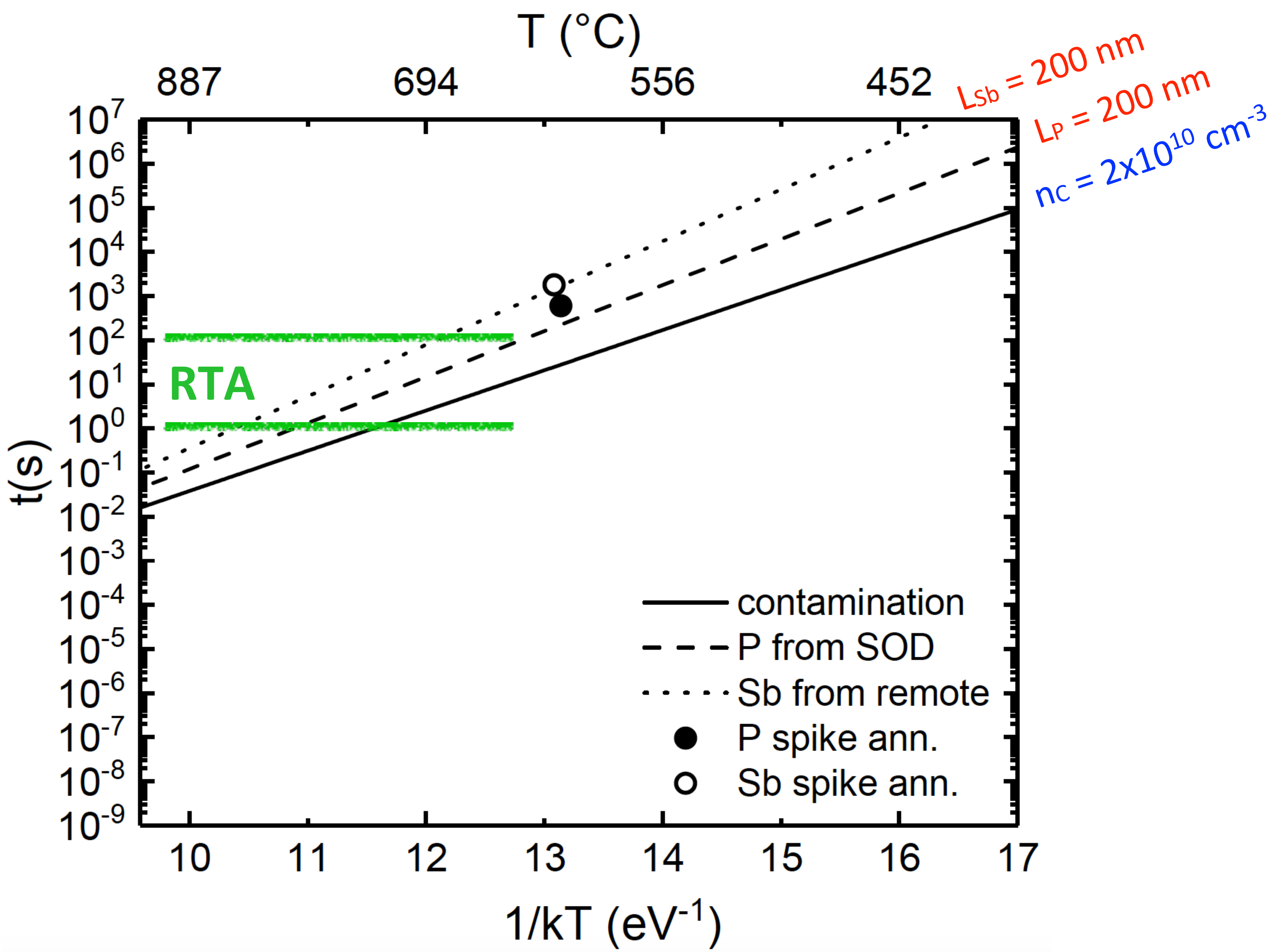




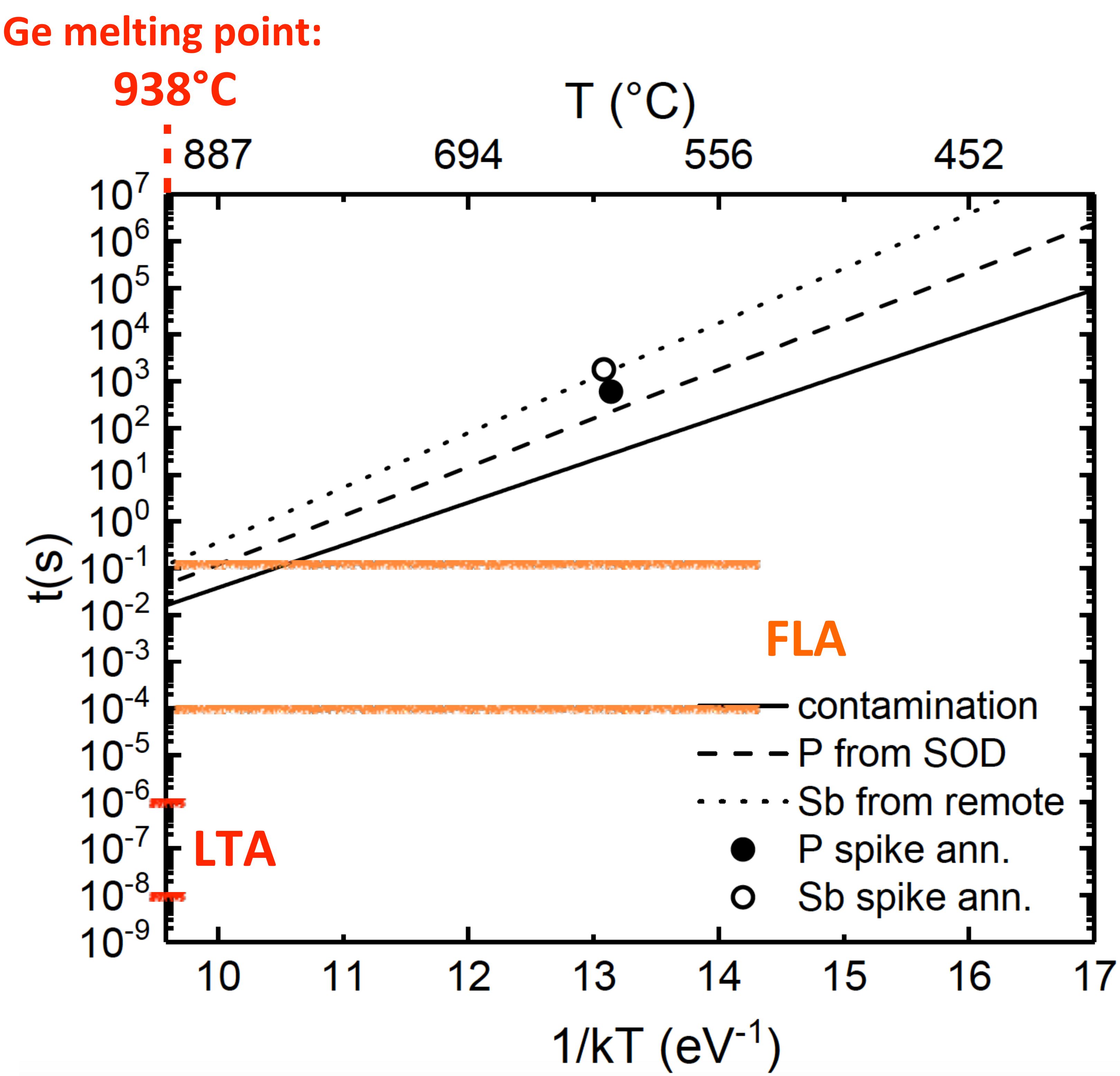
Thermal window for non-contaminant processes



Thermal window for non-contaminant processes



Thermal window for non-contaminant processes



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2. **Thermally-induced defects in HPGe**
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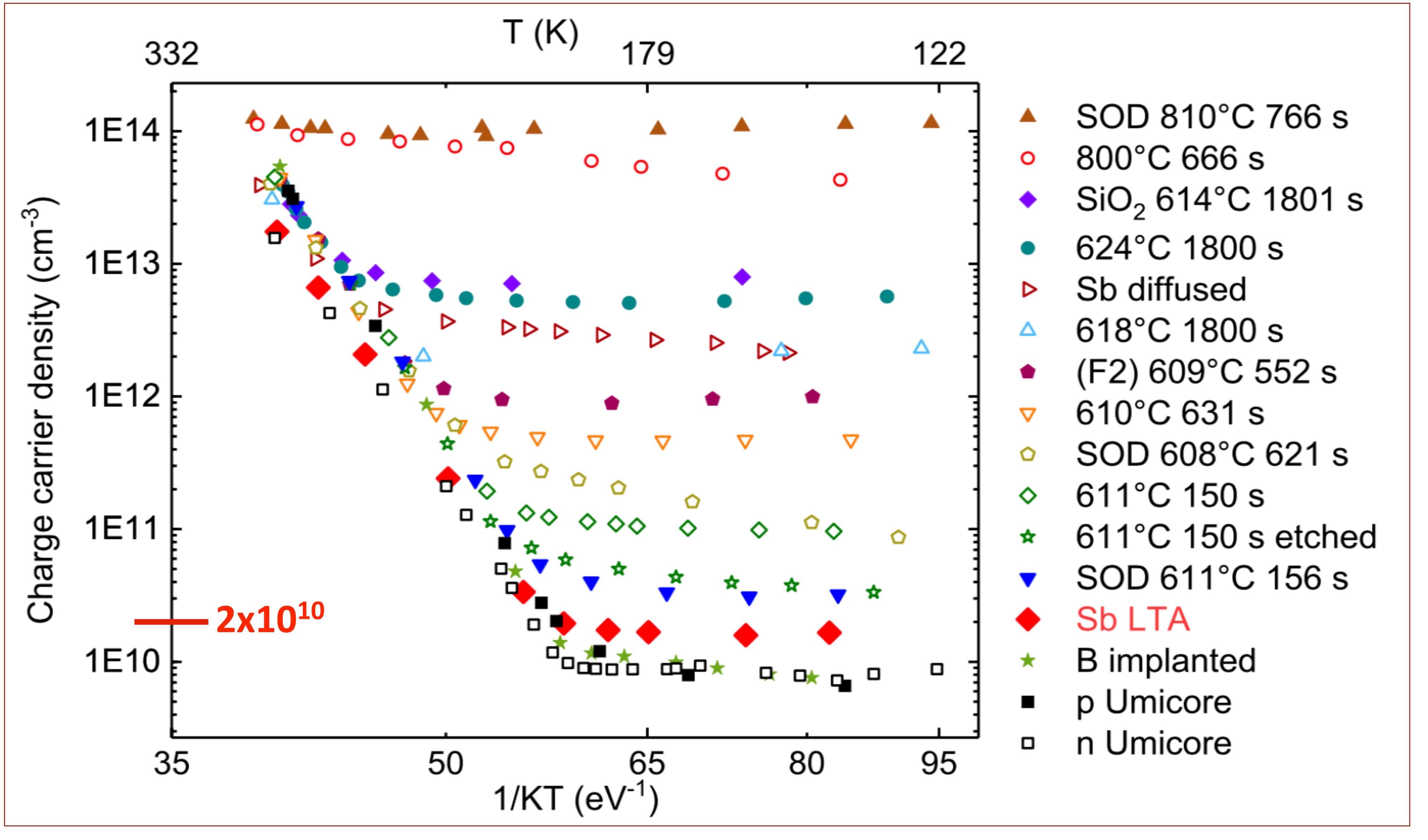
6. **Work in progress**

Work in progress

Work in Progress

Sb LTA sample:

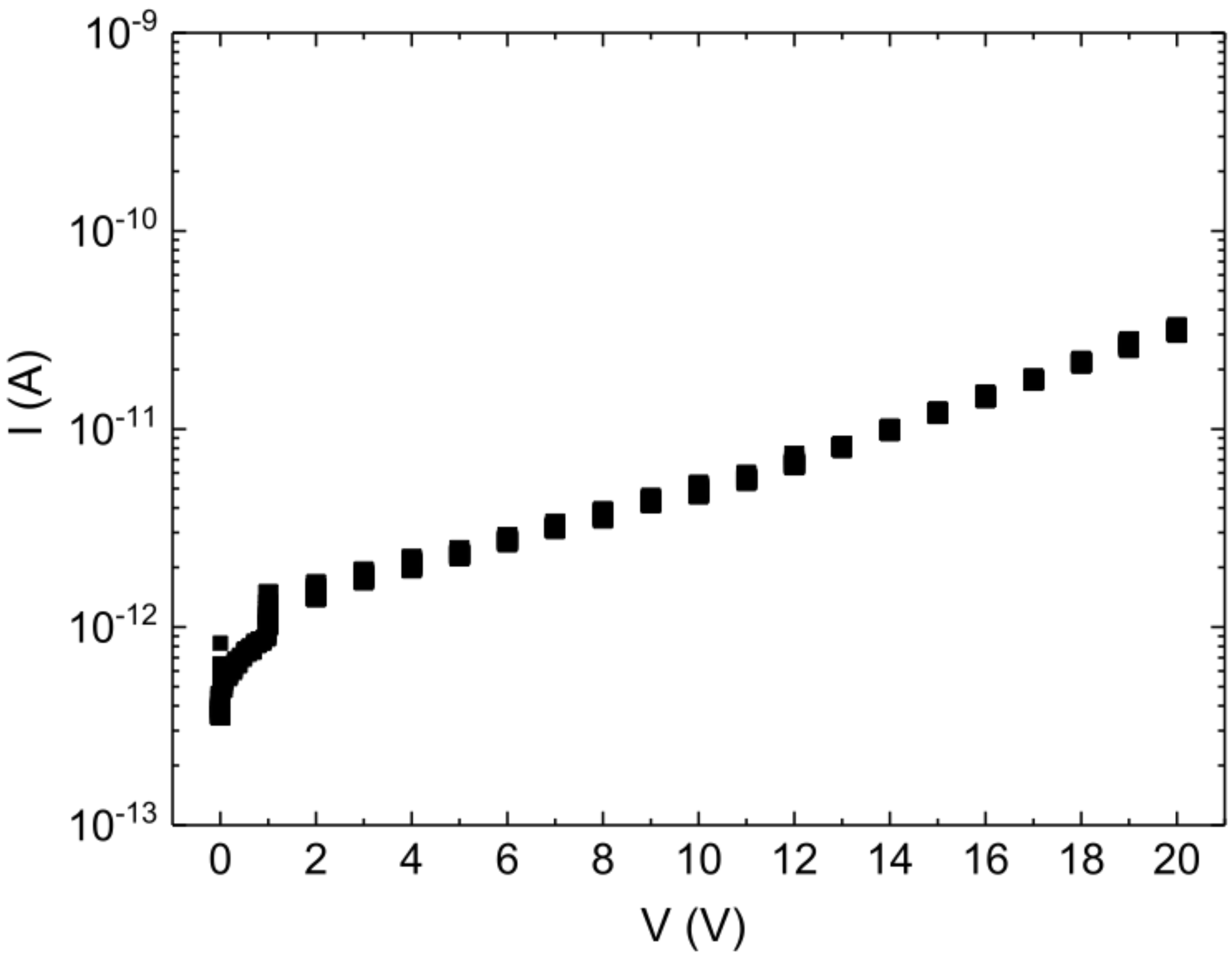
- 1 cm^2 area, 2 mm thick, p-HPGe
- 4 contacts at corners: 4 Sb sputtered spots + Laser Thermal Annealing (LTA)



I/V characteristic under reverse bias

Sb LTA DIODE PROTOTYPE:

- 1 cm^2 area, 2 mm thick, p-HPGe
- n contact: sputtered layer of Sb + Laser Thermal Annealing (LTA)
- p contact: B ion-implanted layer

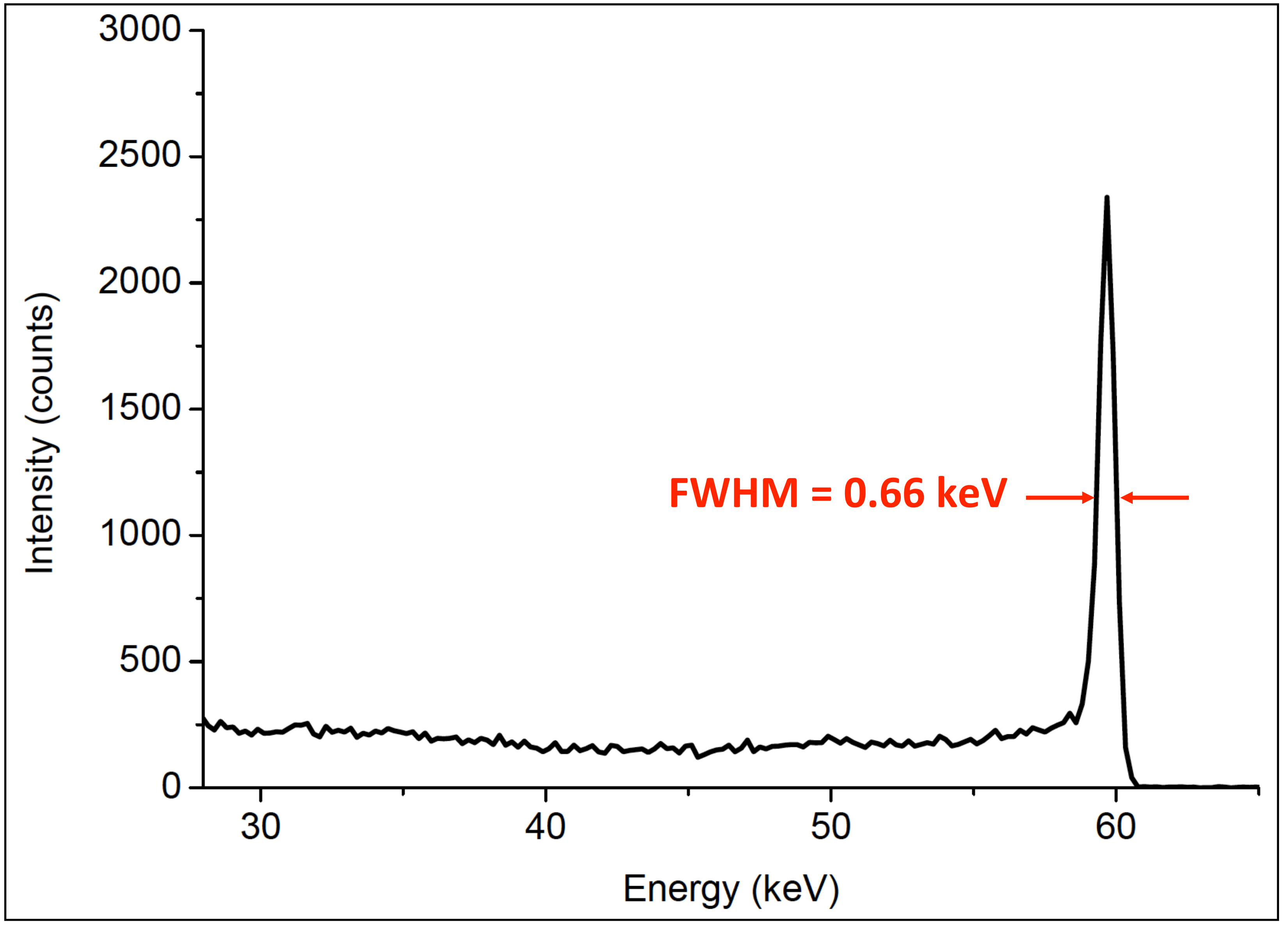


^{241}Am spectrum

Sb LTA DIODE PROTOTYPE:

- 1 cm^2 area, 2 mm thick, p-HPGe
- FWHM = 0.66 keV

Work in Progress



Conclusions

✓ We have demonstrated that high-T annealing introduce a concentration of Cu atoms coming from the external environment inside HPGe, varying with the applied thermal budget and usually higher than 10^{10} cm^{-3}.

✓ We have demonstrated that Cu atoms induce shallow acceptor levels inside HPGe, which would prevent a complete depletion of the detector volume.

✓ By analyzing the measured data through an empirical model, we have identified a window of allowed thermal budgets for which HPGe is not contaminated.

✓ Passing to laser thermal annealing technique, we have built a not contaminated small HPGe diode, that showed optimum resolution toward Am photopeak.

Thank you for your attention!