CERN

Journées thématiques du Réseau Semi-conducteurs IN2P3-IRFU. Méthodes de test orientées simulation, 10-11 June 2021, IP2I Lyon

RD50

Characterization of radiation induced defects in silicon devices

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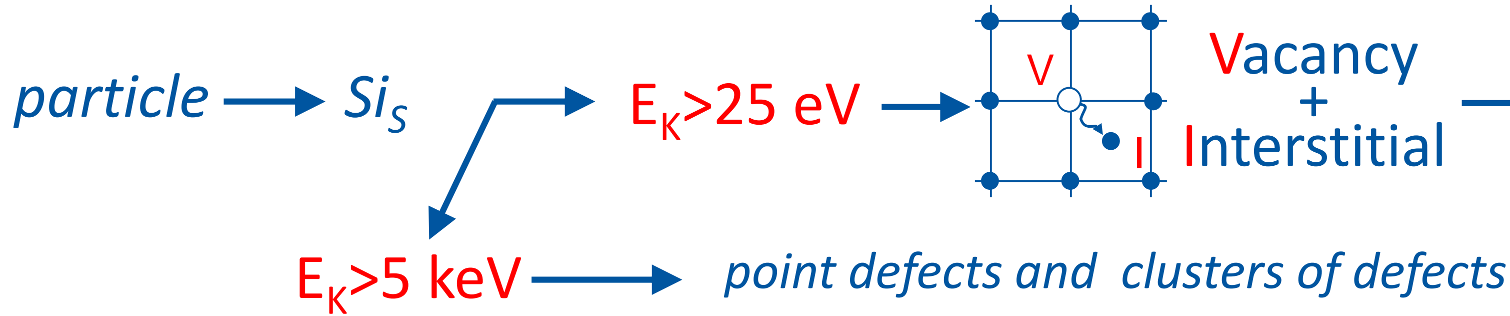
Outline:

- Introduction to radiation induced defects
- Characterization methods with examples
  - TSC, DLTS, PL, FTIR, EPR
- Microscopic Defects vs. Macroscopic Effects
  - Some results of the RD50 collaboration
- Summary

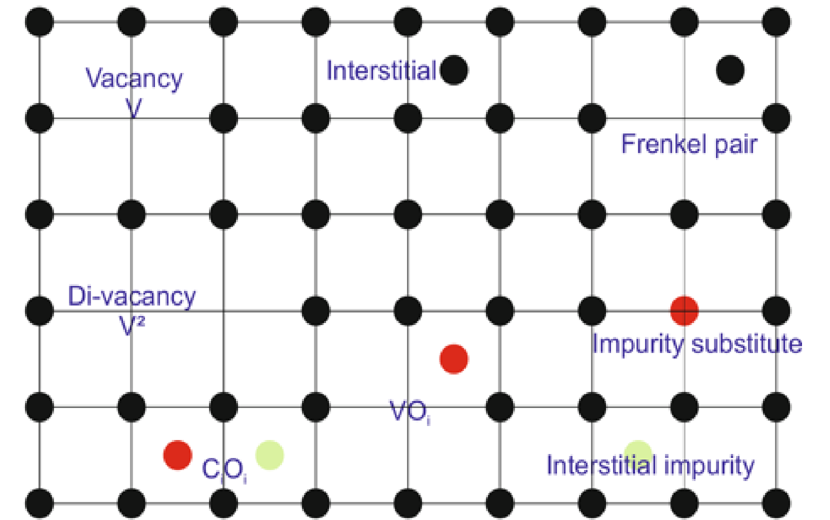
RD50

Radiation induced defects

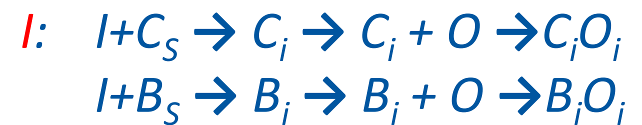
Displacement Damage



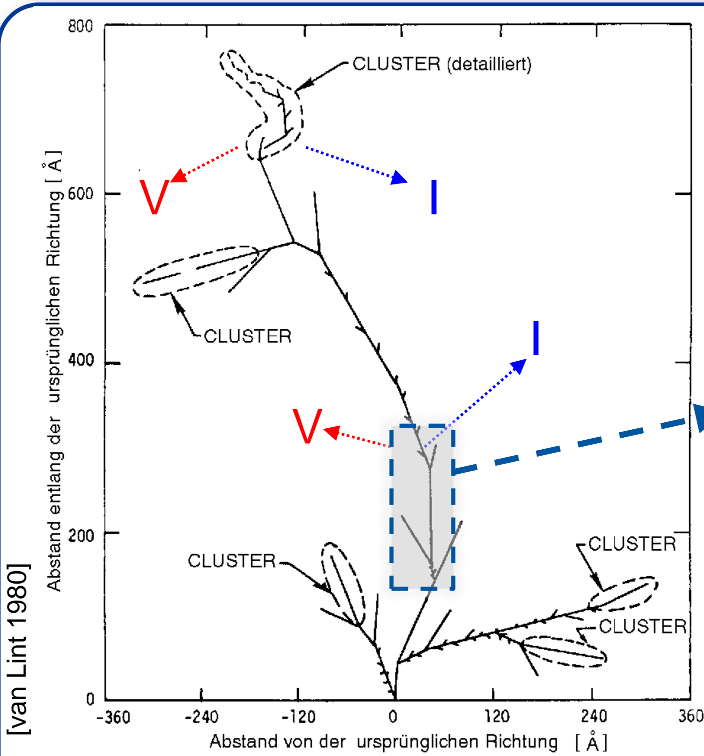
..... *a wide range of* ***point defects***

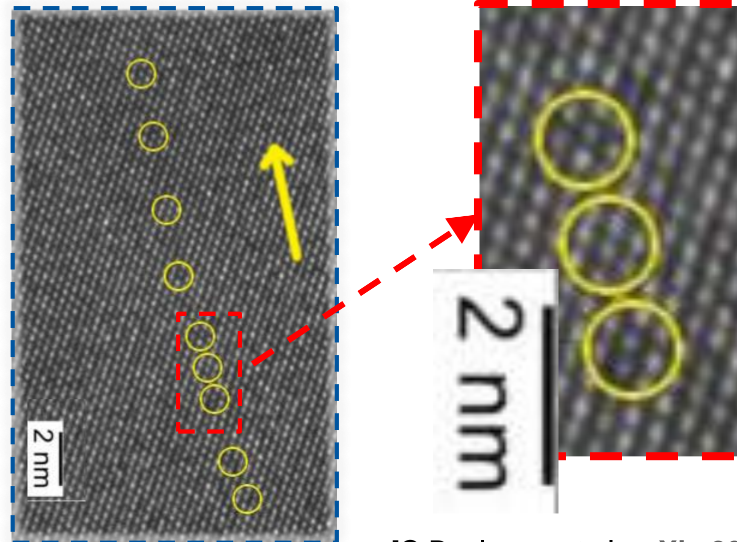


- example of point defect reactions:



... many more reactions!

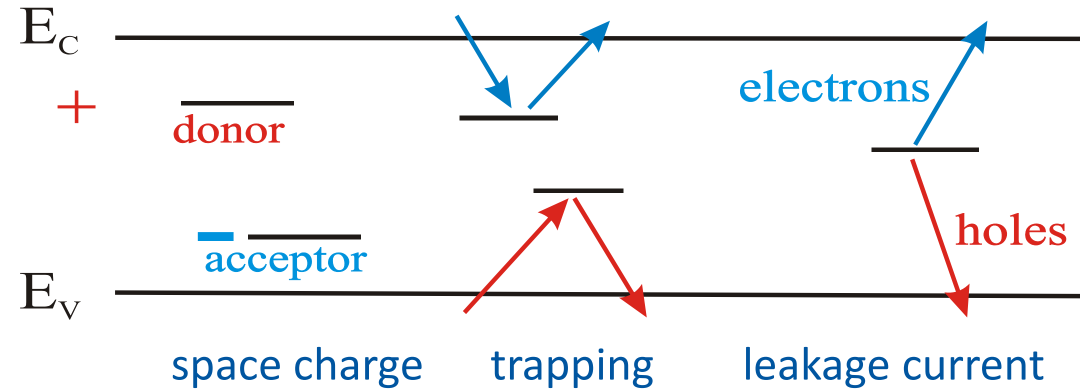




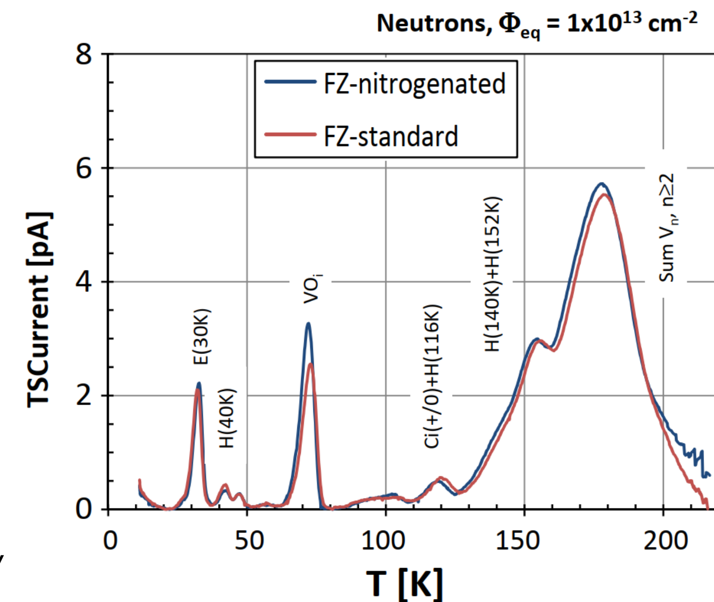
[C.Besleaga et al. arXiv 2021]

Defect Characterization

- Aim of defect studies:
  - **Identify defects** responsible for Change of N_{eff}, Change of E-Field, Trapping, Leakage Current
  - Understand if knowledge can be used to mitigate radiation damage (e.g. **defect engineering**)
  - Deliver input for device simulations to **predict detector performance** under various conditions

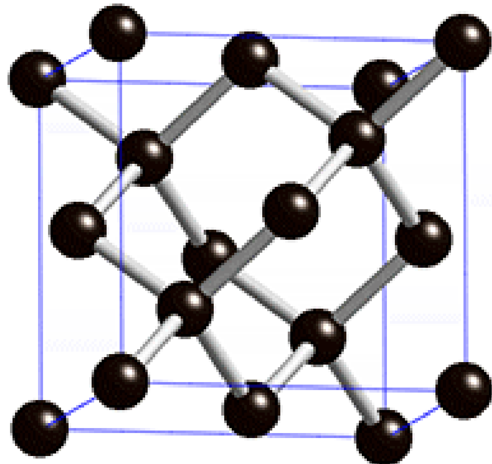


- Defect Characterization performed with various tools:
  - **DLTS** (Deep Level Transient Spectroscopy)
  - **TSC** (Thermally Stimulated Currents)
  - **PITS** (Photo Induced Transient Spectroscopy)
  - **FTIR** (Fourier Transform Infrared Spectroscopy)
  - **EPR** (Electron Paramagnetic Resonance)
  - **TCT** (Transient Current Technique)
  - **CV/IV** (Capacitance/Current-Voltage Measurement)
  - **MW-PC** (Microwave Probed Photo Conductivity)
  - **PC, PL, I-DLTS, TEM,… and simulations**
  - RD50: several hundred samples irradiated with protons, neutrons, electrons, ^{60}Co-γ



Silicon Point Defects: The V-O defect (A center)

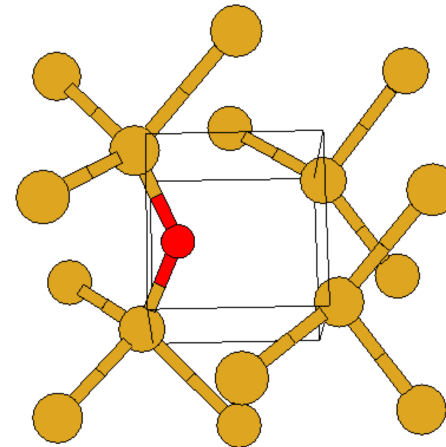
- **Silicon crystal**
  - diamond lattice structure
  - FCC: Face Centered Cubic



- **V-O defect (A centre)**

The dominant centres for vacancy capture in high purity silicon is the isolated oxygen interstitial O_i. Trapping of the vacancy results in the V-O centre, the so called A-centre.

Structure:



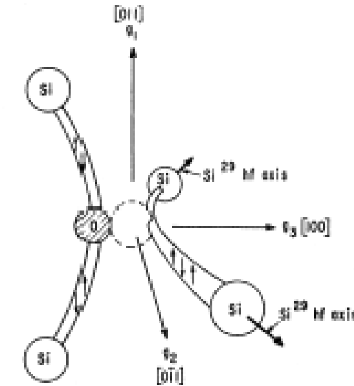
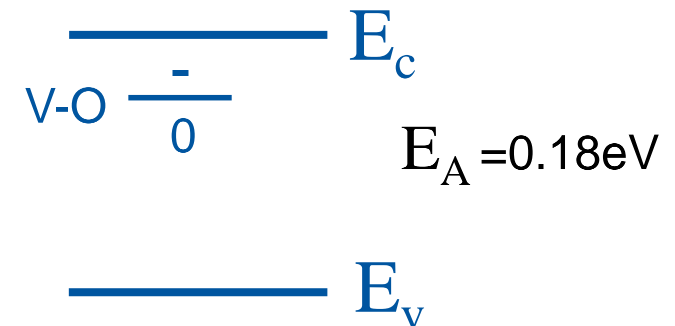


FIG. 5. Model of the *A* center as a substitutional oxygen atom. The oxygen atom bonds between two of the four neighboring silicon atoms, and the remaining two silicon atoms pull together to form a covalent bond. Spin resonance arises from an additional electron which is trapped in the Si–Si molecular bond as shown. The *g* tensor and hyperfine axes are indicated.

Watkins, Corbett: Phys.Rev.,121,4, (1961),1001

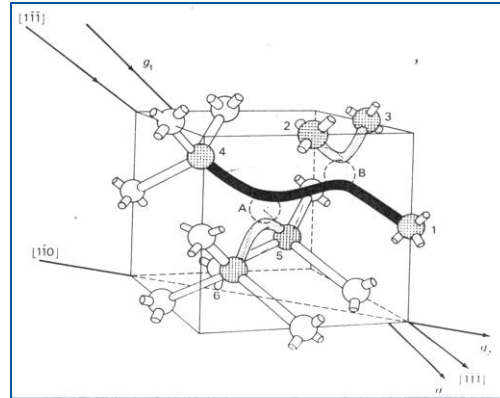
Electrical activity:
The A center has an acceptor level in the upper half of the band gap



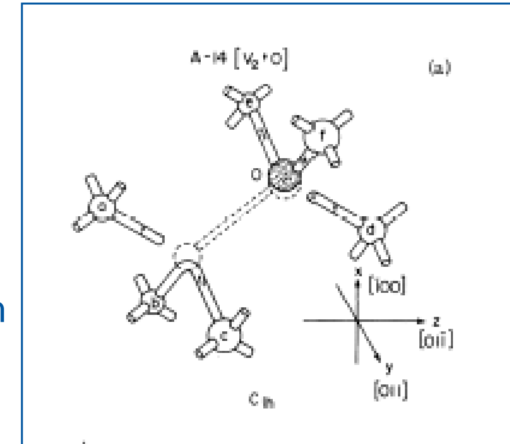
Silicon Point Defects: The Divacancy V_2

- **Point defects can involve more than one vacancy**
  - V_2, V_3, V_2O, V_2O_2 ….are well characterized defects

Divacancy V_2
two missing silicon atoms

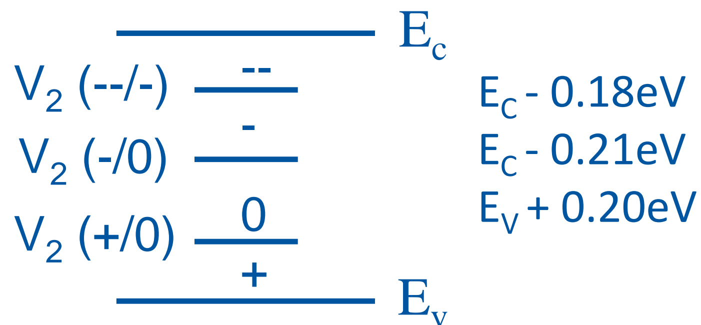


V_2O
two missing silicon atoms +one oxygen atom



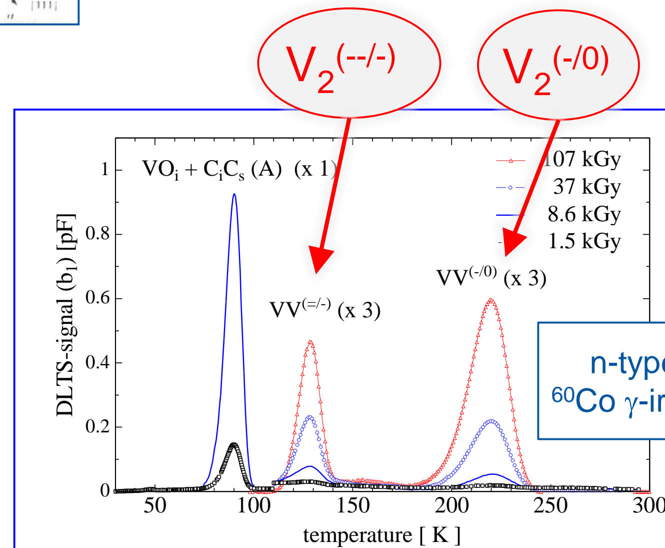
Watkins, Corbett: Phys.Rev.,121,4, (1961),1001

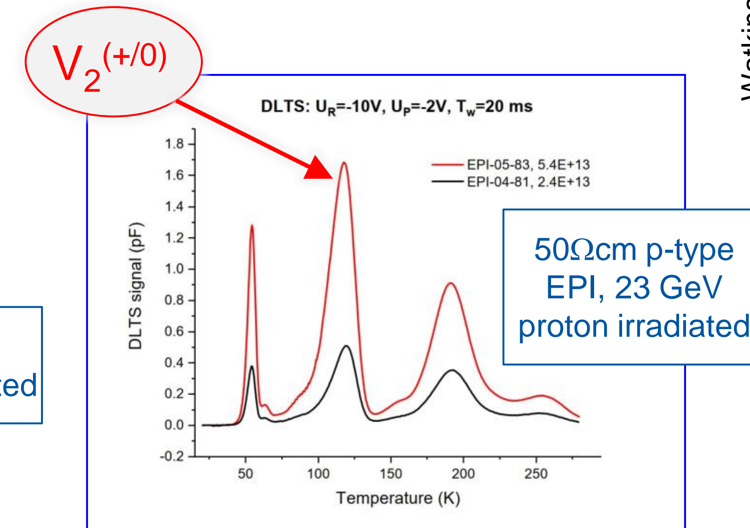
- Electrical activity of the V_2
  - The divacancy has has two acceptor levels in the upper half of the band gap and a donor level in the lower half of the band gap.



E_C - 0.18eV
E_C - 0.21eV
E_V + 0.20eV

$V_2^{(--/-)}$ $V_2^{(-/0)}$ $V_2^{(+/0)}$





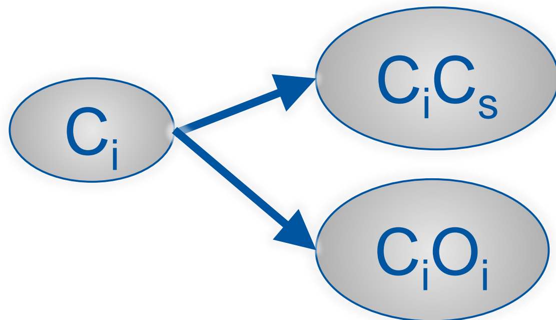
Defect Characterization Techniques

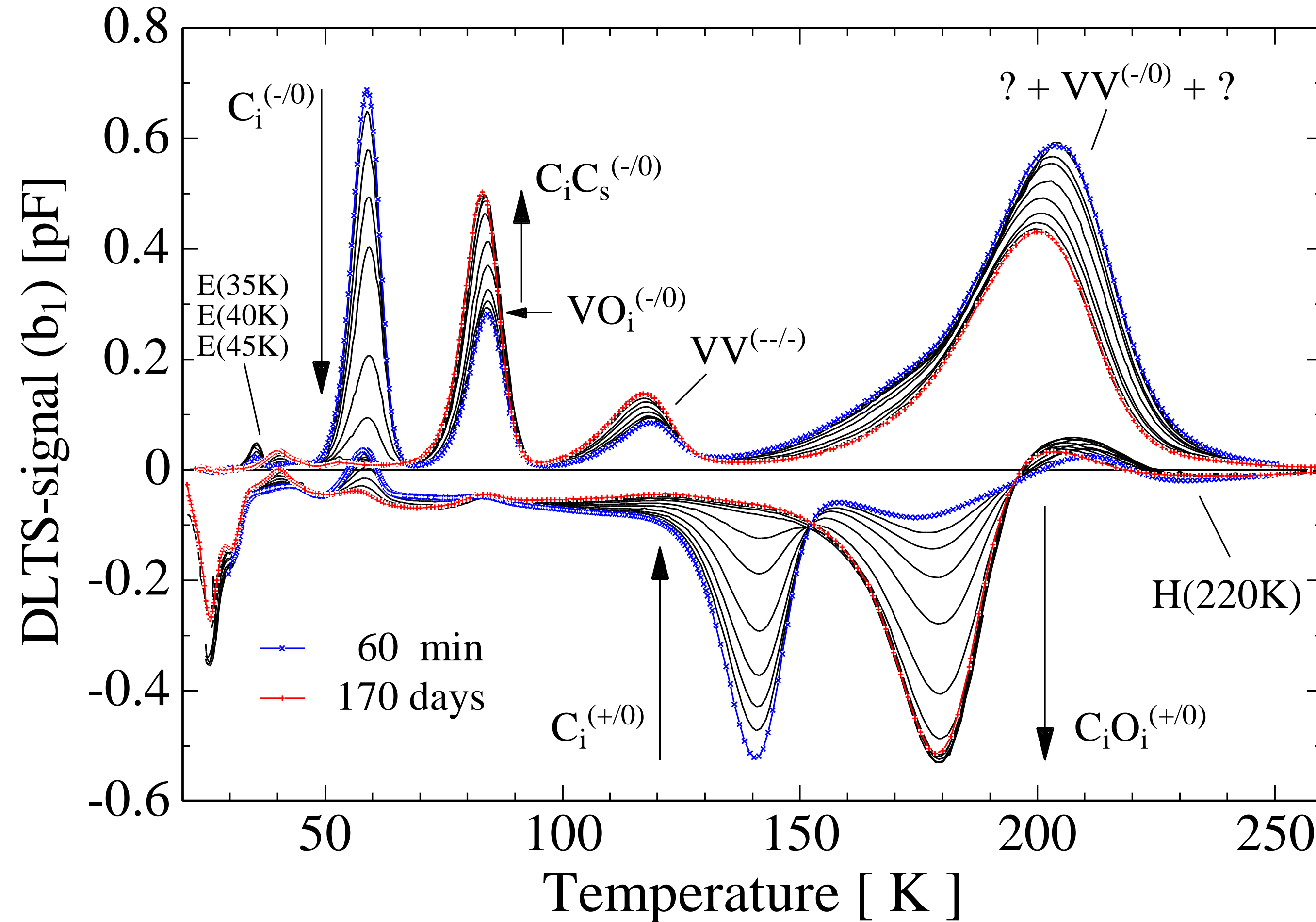
Deep Level Transient Spectroscopy

Example: n-type FZ silicon sensor irradiated with MeV neutrons

DLTS spectra
recorded during **annealing**
at room temperature

Carbon interstitial (C_i)
defect kinetics clearly visible:





- Introduction rates of main defects ≈ 1 cm^{-1}
- Introduction rate of negative space charge ≈ 0.05 cm^{-1}

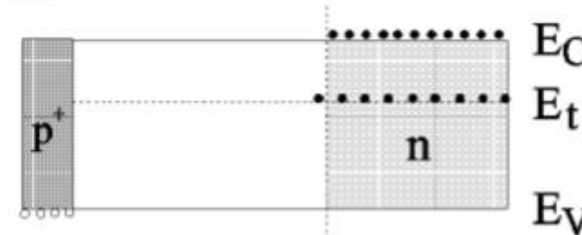
DLTS (Deep Level Transient Spectroscopy): Operation principle

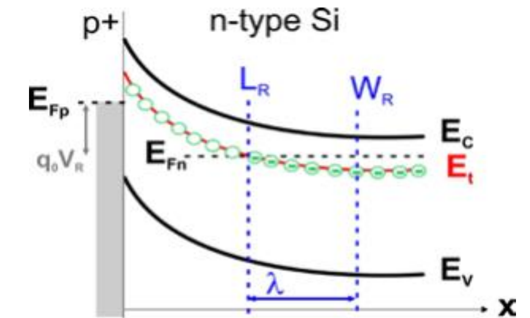
- *Assumption: Defect is an electron trap in n-type silicon, i.e. a majority carrier trap*

Measurement cycle at fixed T

[1] reverse bias V_R

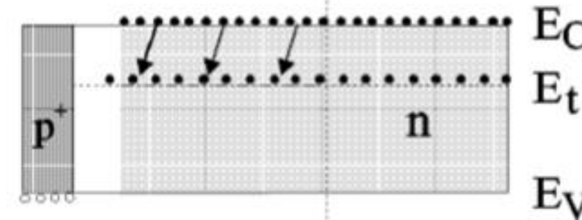
- junction under reverse bias
- defect states are not occupied

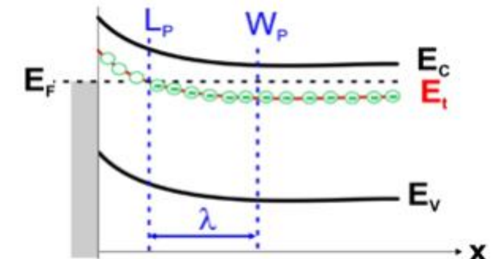




[2] injection pulse V_P

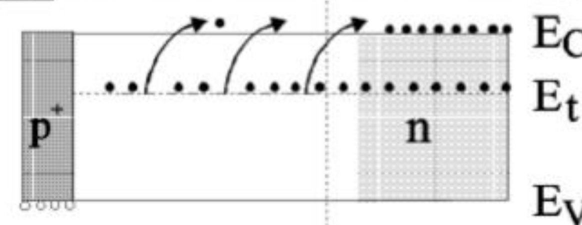
- reduction of reverse bias
- injection of majority carriers
- occupation of defect levels

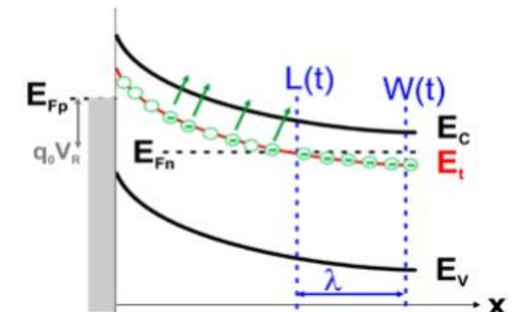




[3] reverse bias V_R

- junction under reverse bias
- thermal emission of carriers
  - expansion of depletion zone
  - decrease of capacitance





DLTS (Deep Level Transient Spectroscopy): Operation principle

Measurement cycle at fixed T

[1] reverse bias V_R
- junction under reverse bias
- defect states are not occupied

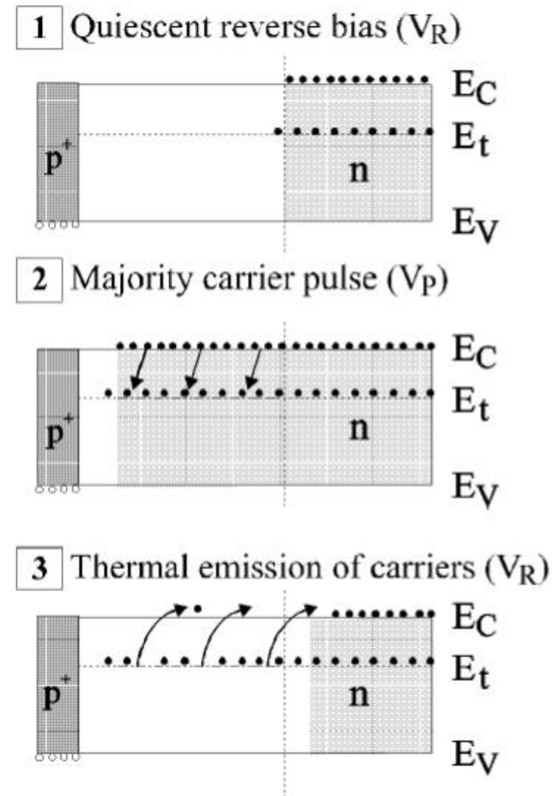
[2] injection pulse V_P
- (a) reduction of reverse bias
  - injection of majority carriers

or

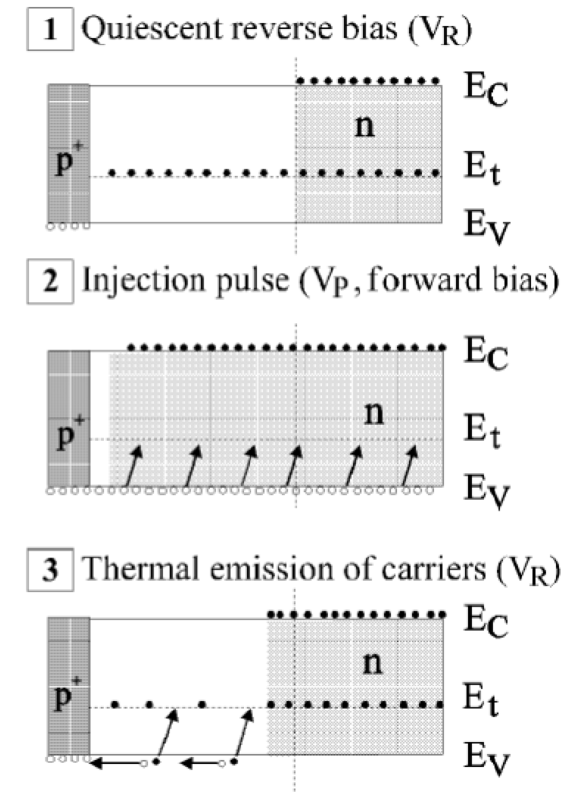
- (b) forward bias
  - injection of minority &majority carriers

[3] reverse bias V_R
- junction under reverse bias
- thermal emission of carriers
  - expansion of depletion zone
  - change of capacitance

(a) majority carrier injection
(electrons in n-type silicon)
electron trap



(b) high injection ($n \approx p$)
(electrons and holes injected)
hole trap with $c_p >> c_n$



DLTS (Deep Level Transient Spectroscopy): Operation principle

[3] reverse bias V_R

- junction under reverse bias
- thermal emission of carriers
  - expansion of depletion zone
  - change of capacitance

Change of capacitance:

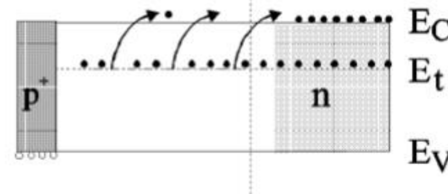
- trapped carriers change space charge (N_{eff})
- space charge determines depletion depth w
- depletion depth determines the capacitance

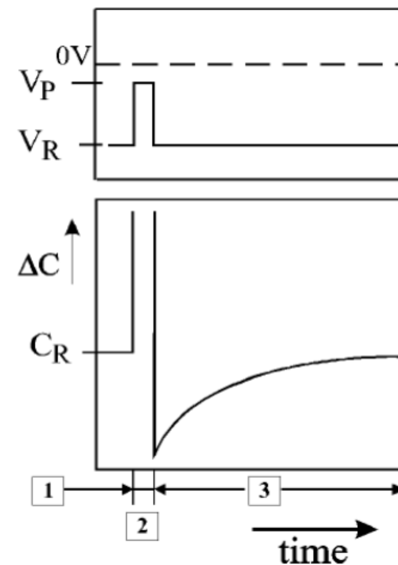
$$C(w) = \epsilon_0 \epsilon_{Si} \frac{A}{w(V)} = A \sqrt{\frac{\epsilon_0 \epsilon_{Si} q_0 |N_{eff}|}{2(V + V_{bi})}}$$

C(t=0) < C_R : trapping of majority carriers
C(t=0) > C_R : trapping of minority carriers

sign of ΔC allows to differentiate between electron and hole traps

(a) majority carrier injection
(electrons in n-type silicon)
electron trap

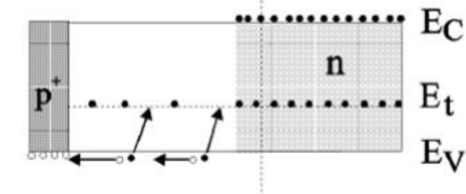


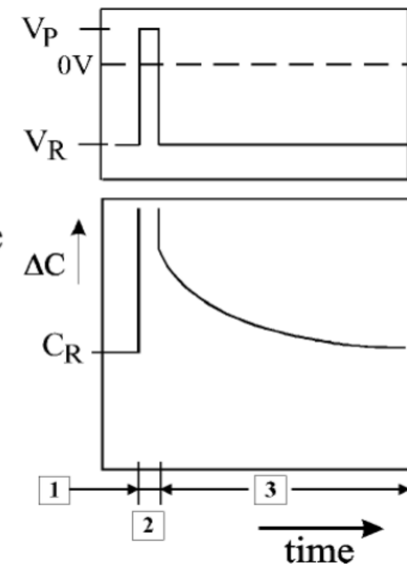


Bias pulse

Capacitance transient

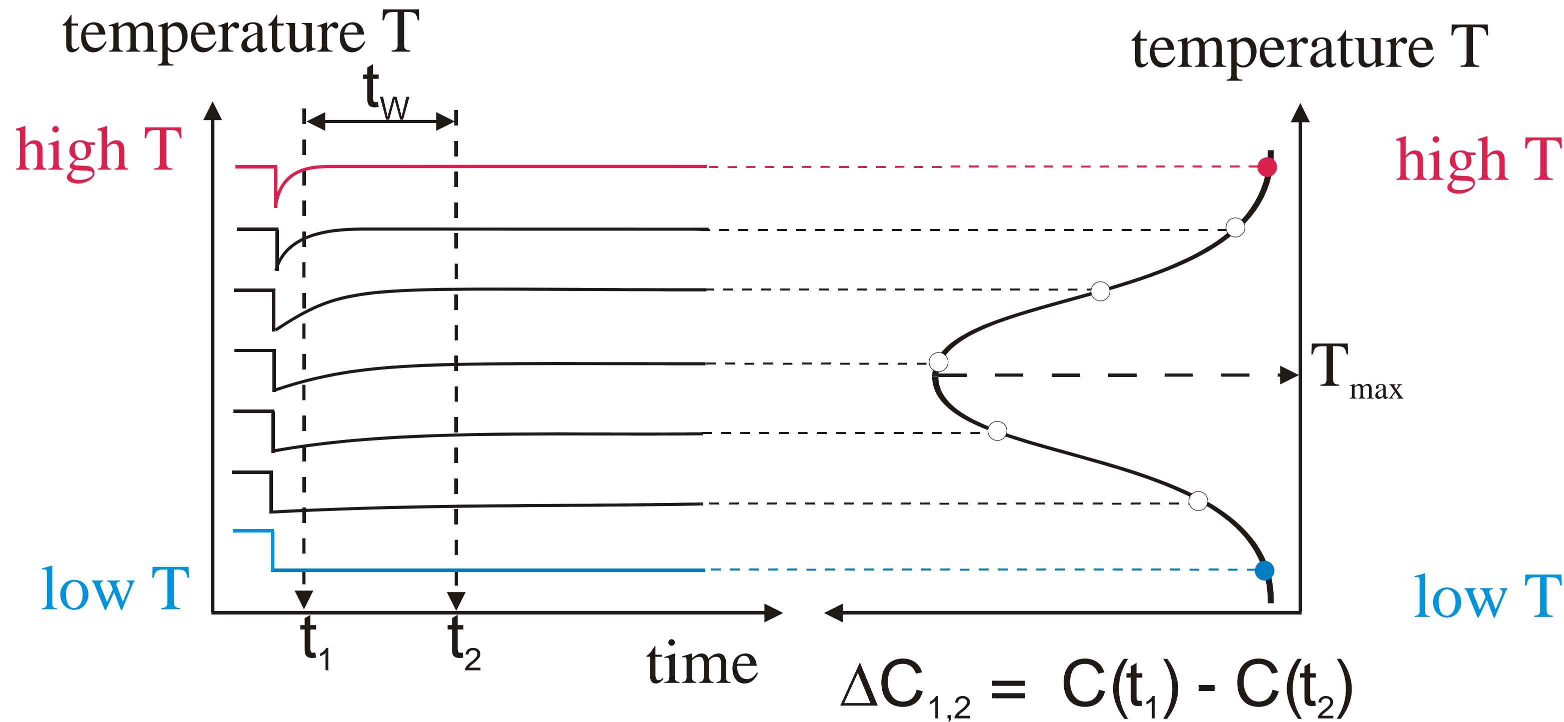
(b) high injection (n ≈ p)
(electrons and holes injected)
hole trap with c_p >> c_n

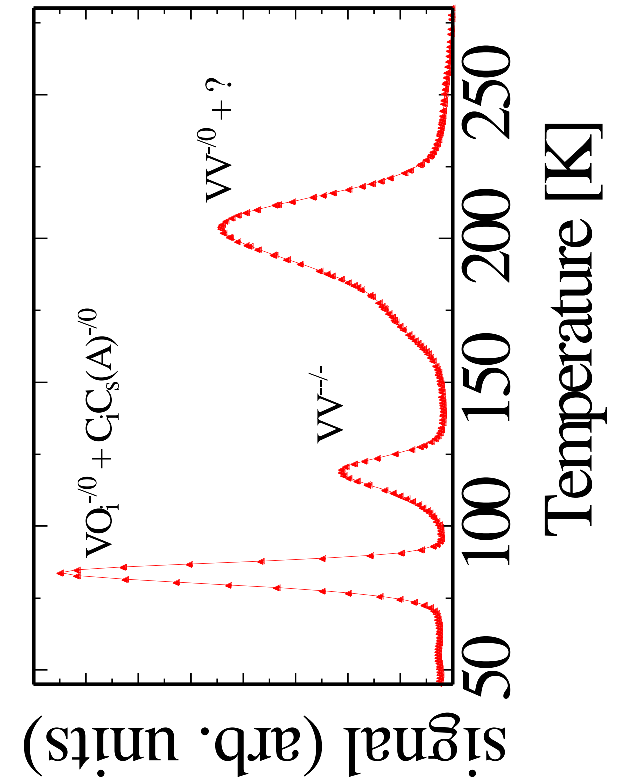




DLTS (Deep Level Transient Spectroscopy): Spectrum

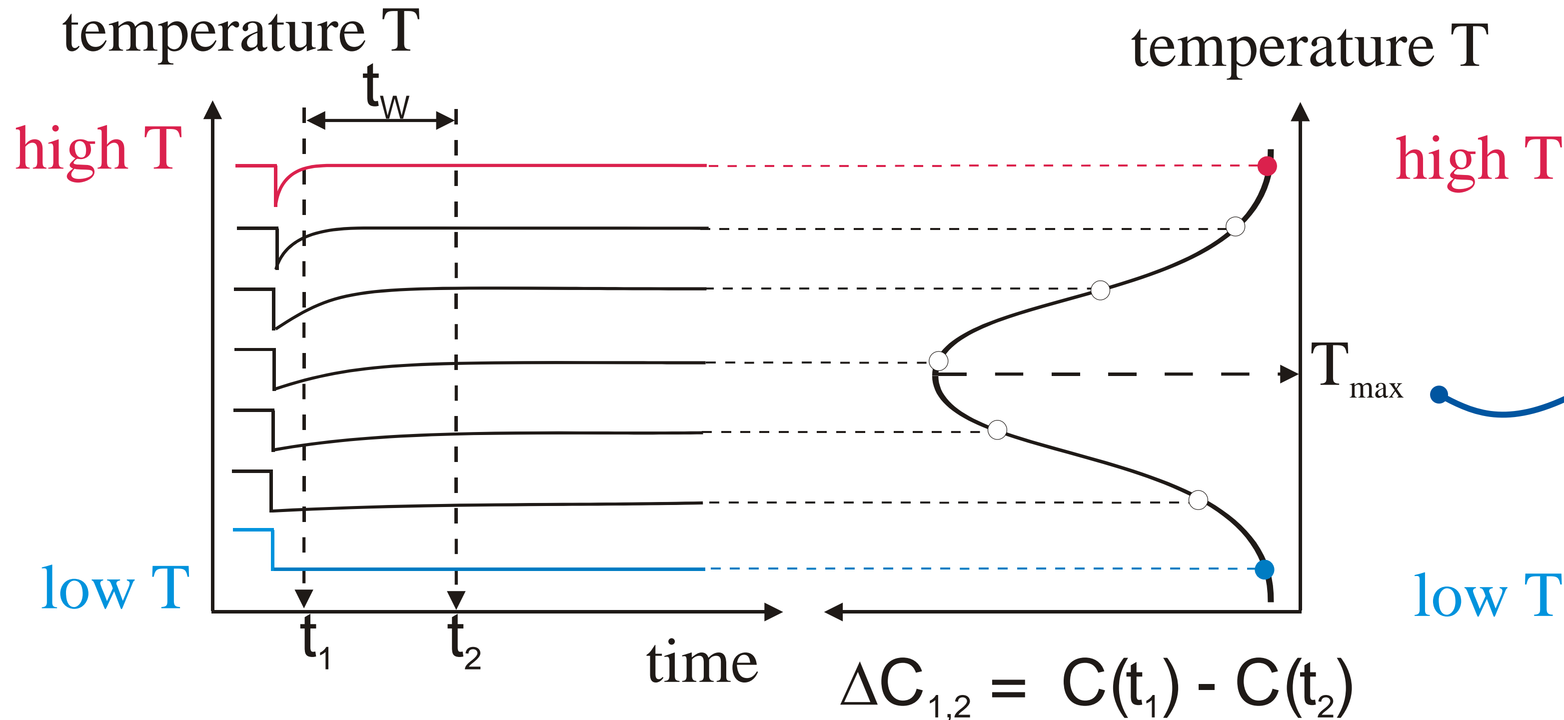
- **DLTS spectrum** is obtained from transients measured at different temperatures





DLTS (Deep Level Transient Spectroscopy): Transient Analyses

- **DLTS spectrum**



$\Delta C_{1,2} = C(t_1) - C(t_2)$

emission time constant at peak maximum temperature can be extracted from time window parameters

$$\tau_e(T_{max}) = \frac{t_2 - t_1}{ln\left(\frac{t_1}{t_2}\right)}$$

perform several measurements with different time windows to gain a set of $\tau_e(T_i)$, T_i pairs and use them to construct an **Arrhenius Plot**

Transient Analyses: Defect parameters from Arrhenius Plot

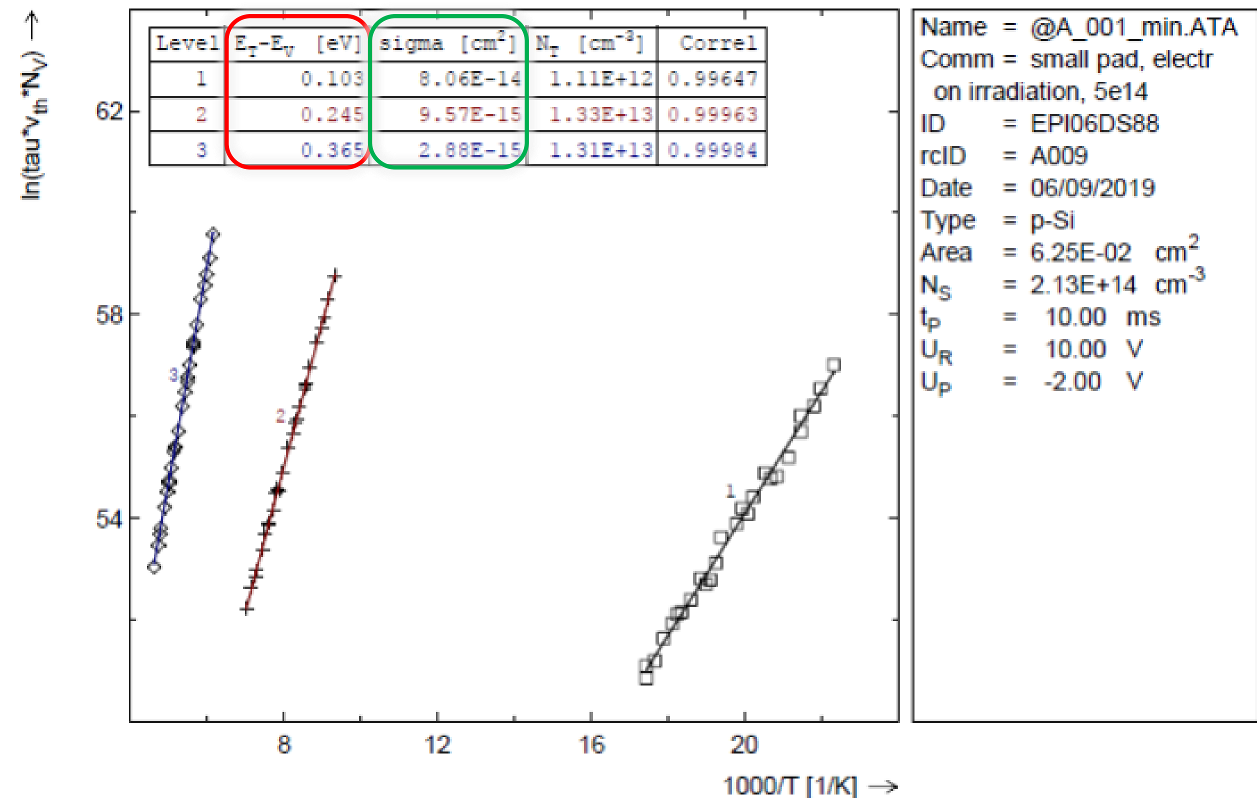
Emission rate

$$e_{n,p} = \frac{1}{\tau_e} = \sigma_{n,p} \cdot v_{th,n,p} \cdot N_{C,V} \cdot exp\left(-\frac{E_a}{k_B T}\right)$$

Arrhenius Plot

$$ln(\tau_e v_{th,n,p} N_{C,V}) = -ln(\sigma_{n,p}) + \frac{E_a}{k_B T}$$

e_n	emission rate electrons
τ_e	emission time constant
c_n	capture coefficient electrons ($c_n = \sigma_n v_{th,n}$)
σ_n	capture cross section for electrons
v_{th}	thermal velocity
N_C	density of states in conduction band
k_B	Boltzmann constant



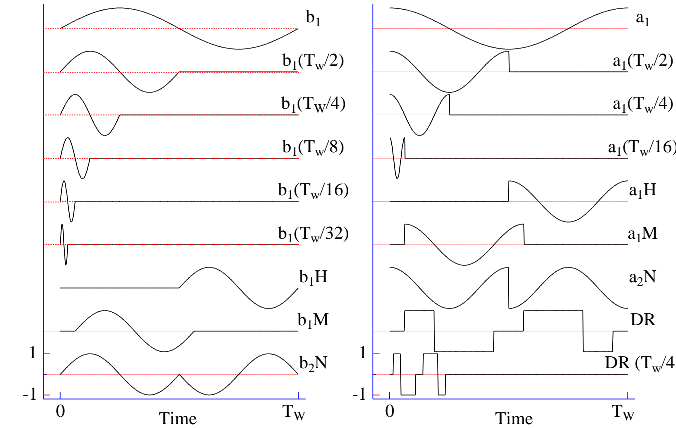
Level	E_T-E_V [eV]	sigma [cm^2]	N_T [cm^{-3}]	Correl
1	0.103	8.06E-14	1.11E+12	0.99647
2	0.245	9.57E-15	1.33E+13	0.99963
3	0.365	2.88E-15	1.31E+13	0.99984

Defect parameters extracted from Arrhenius plot

- slope: activation energy E_a
- intercept: capture cross section σ

DLTS (Deep Level Transient Spectroscopy): Transient Analyses

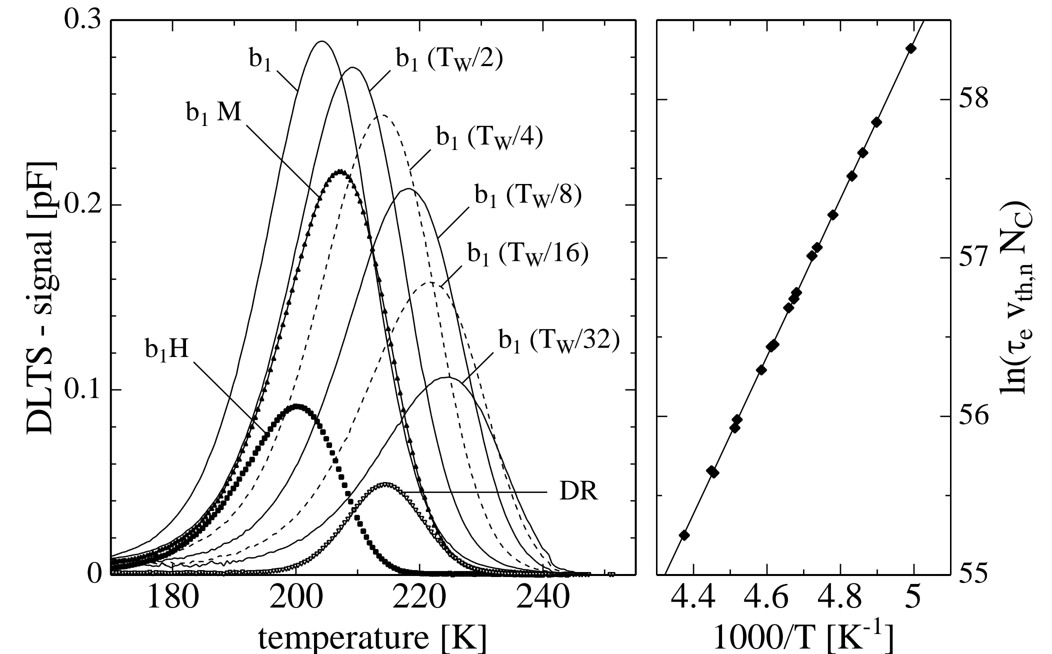
- **Various signal processing techniques**
  - **Analog signal processing**
    - double boxcar integrator
    - lock-in amplifier, analog correlator, ….
  - **Digital signal processing**
    - Fast-Fourier Transformation FFT
    - Laplace Transformation
    - Various correlator functions
    - ..



correlator functions

Example used at CERN:

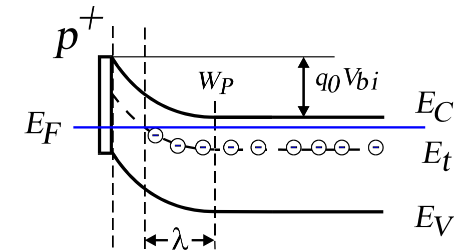
- Folding transient with 28 different correlator functions
- maxima analysis of a single DLTS scan (one time window) leads to 28 value pairs (τ_e,T) which can be used for an Arrhenius plot



DLTS: Determination of Defect Concentration

- Band bending diagrams for deep acceptor:
  - [2] during filling pulse
  - [3] during transient phase

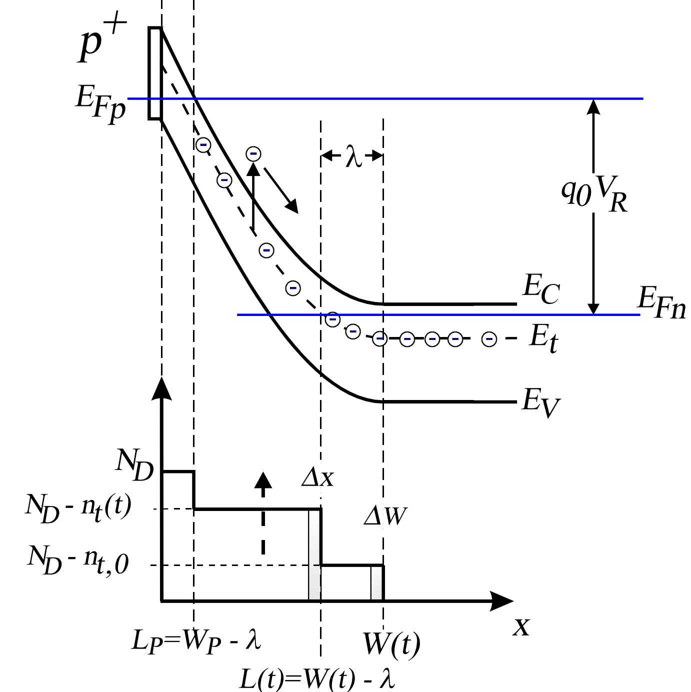
Filling pulse: $V_P = 0V$ ([2])



- Transition region λ:

$$\lambda = \sqrt{\frac{2\epsilon\epsilon_0(E_F - E_t)}{q_0^2 N_D}}$$

Transient: Reverse bias V_R ([3])



- Defect concentration N_t
  - Amplitude of the C-transient $\Delta C_0 \sim N_t$

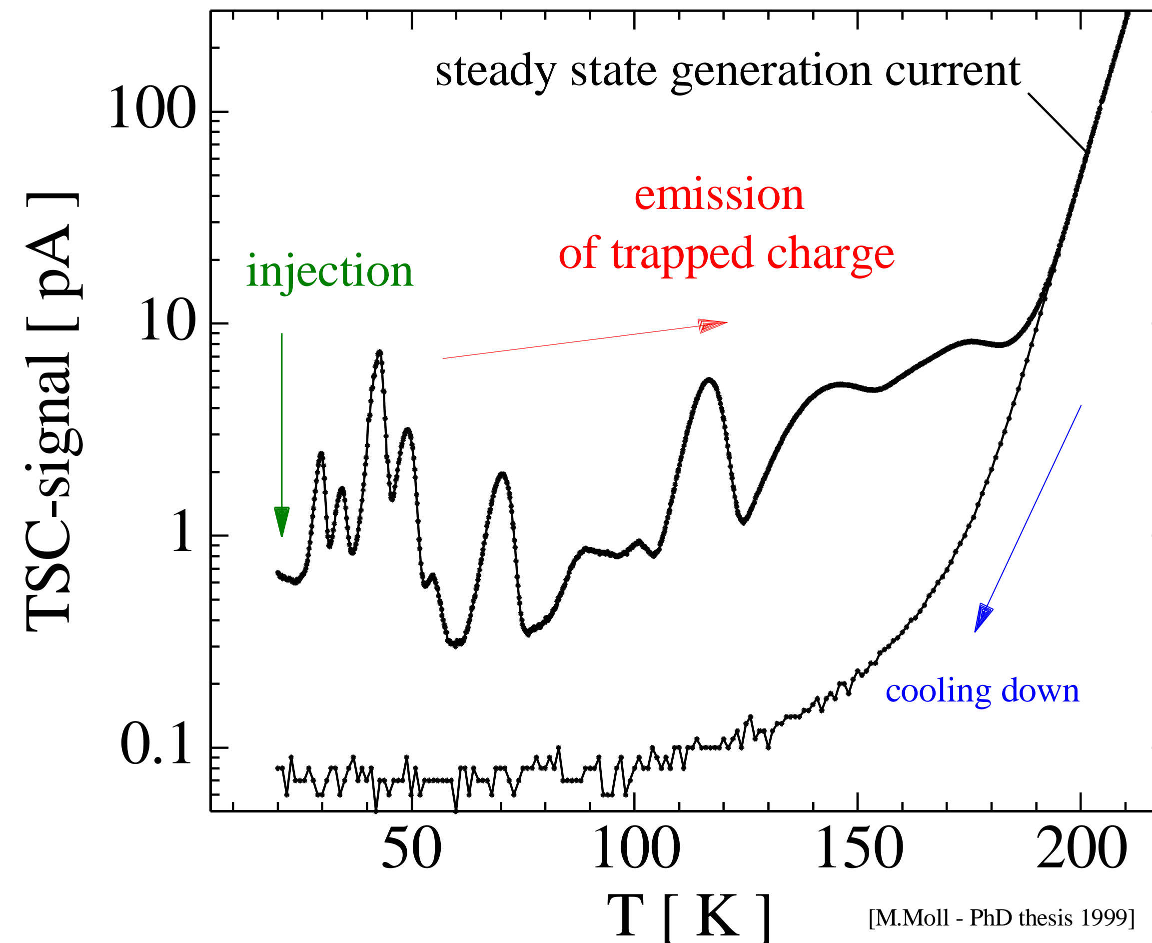
$$N_t = -2N_D \frac{\Delta C_0}{C_R}\left[1-\left(\frac{C_R}{C_p}\right)^2 - \frac{2\lambda C_R}{\varepsilon\varepsilon_0 A}\left(1-\frac{C_R}{C_p}\right)\right]^{-1}$$

  - For $\lambda \ll W_R$ simplifies to:

$$N_t \approx 2N_D \frac{|\Delta C_0|}{C_R}$$

TSC: Thermally Stimulated Current

- Measurement cycle
  - **(1) Cooling**
    - under bias or without
  - **(2) Filling (charge injection)**
    - Forward bias, zero bias, optical filling
  - **(3) Current measurement**
    - Measure current while ramping up the temperature, discharging of traps results in current peaks
- Analyses
  - **Peak heights** or integral over peak
    - → Defect concentration
  - **Peak position** gives indication for E_a and cross section σ
    - more precise: fit to spectrum and/or delayed heating measurement (see next slide)



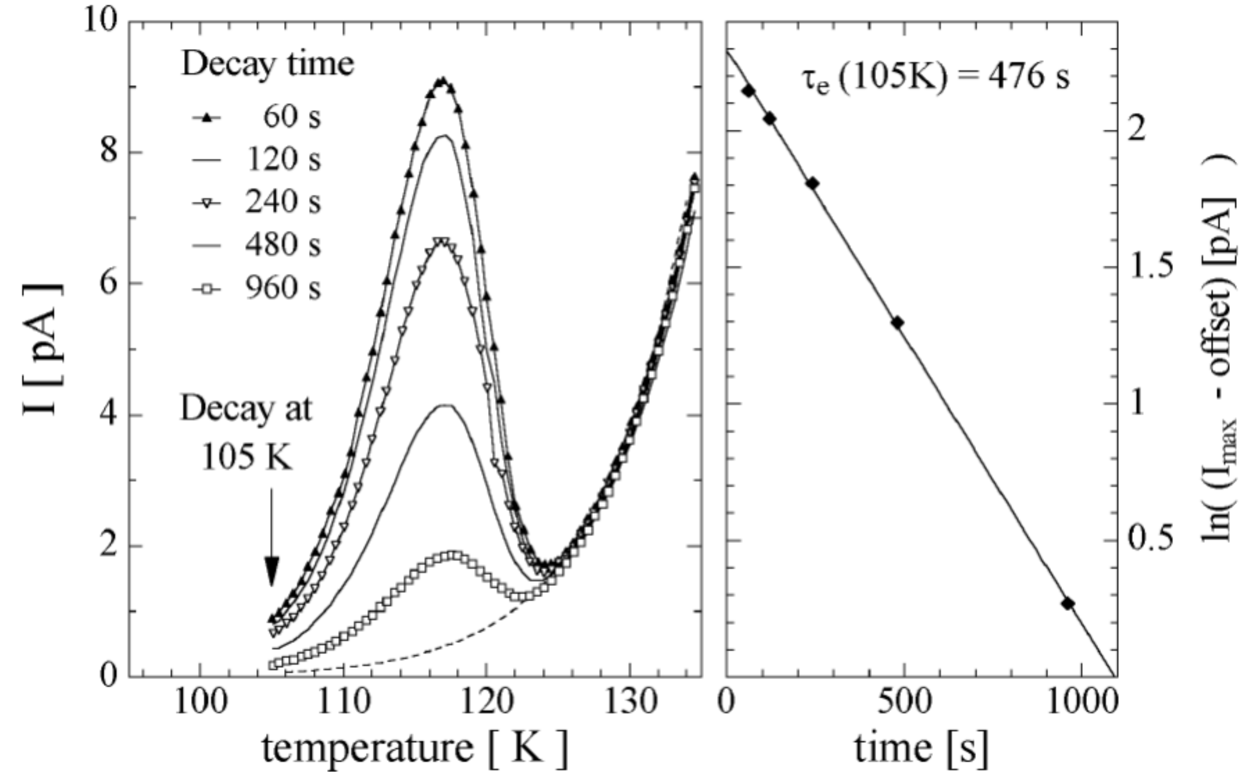
Current

$$I_{TSC}(T) = \frac{1}{2}\, q_0 A w(T) N_t \cdot e_n(T) \cdot exp\left(-\frac{1}{\beta}\int_{T_0}^{T} e_n(T) dT\right)$$

Note: hole and electron traps give same polarity signal

TSC: Delayed heating method

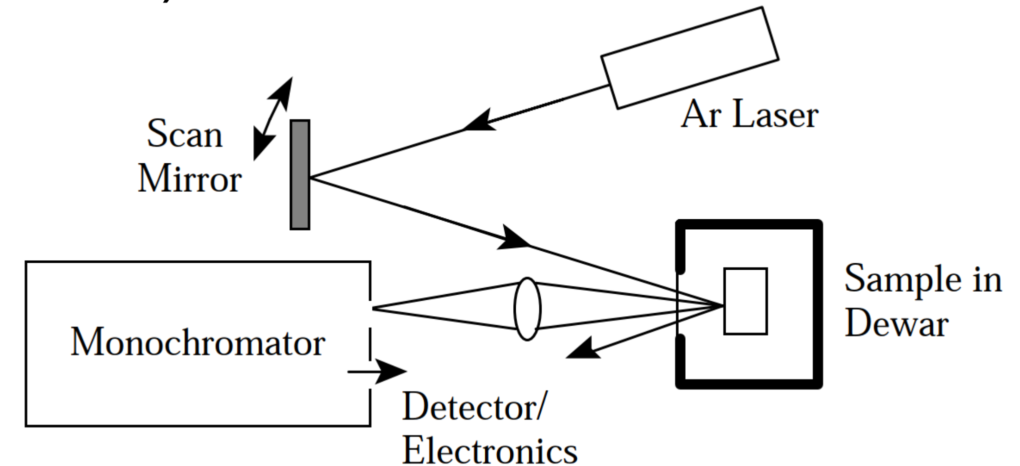
- "Delayed heating" or "Thermal cleaning"
  - Measuring the TSC peak several times with different delay times between the end of filling at T_0 and the start of heating
  - **Increasing delay time:** more charge carriers already emitted at T_0 → TSC peak decreases
  - **Plot** logarithm of the peak amplitude against delay time → slope: emission time constant $\tau_e(T_0)$
  - Repeat the delay measurements at different T_0
    → Arrhenius-plot to determine E_a and σ

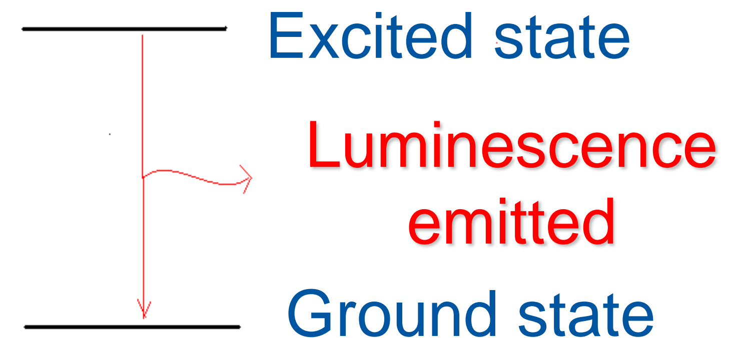


Photoluminescence (PL) [1/2]

Excitation of the sample with light ('photo') and re-emission of part of the absorbed energy as light ('luminescence')

- Excitation is usually by a laser, for convenience of directed beam, with beam power of 100's of mW.
- Green laser light is absorbed by the crystal, exciting an electron from the valence band to the conduction band, with a penetration depth of 1/e = 1 μm.
- Electron-hole pairs (excitons) are created with a lifetime of 10's of ms in pure Si.
  - Excitons are captured by impurities, exiting the impurity.

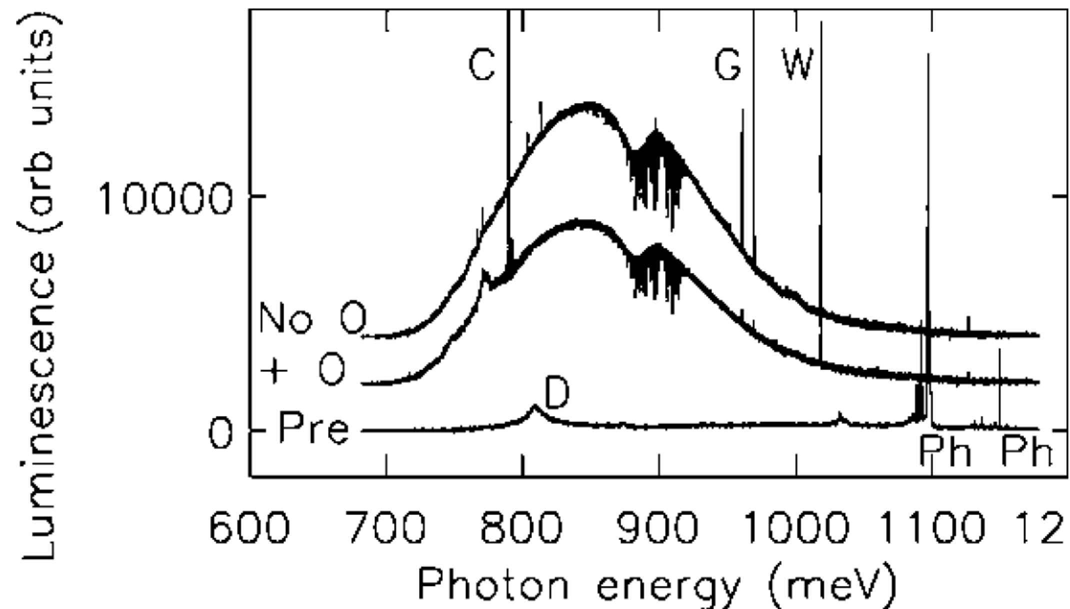




Photoluminescence (PL) [2/2]

- **What is observed?**
  - Only (usually) neutral centres (not charged)
  - Required concentrations for detection are over 10^{11} cm^{-3}; best 10^{14} to 10^{16} cm^{-3}
  - Very sharp lines with typical energy resolution of 0.1 meV
  - PL is not a quantitative method: PL not necessarily proportional to the concentration of defect

- **Example:**
  - Standard FZ silicon (No O)
  - Oxygenated FZ silicon (+O)
  - irradiated with 23 GeV protons
    - fluence 10^{16} cm^{-2}



C-line: C_iO_i
G-line: C_iC_s
W-line: Si_i related

Phosphorus
(before irradiation)

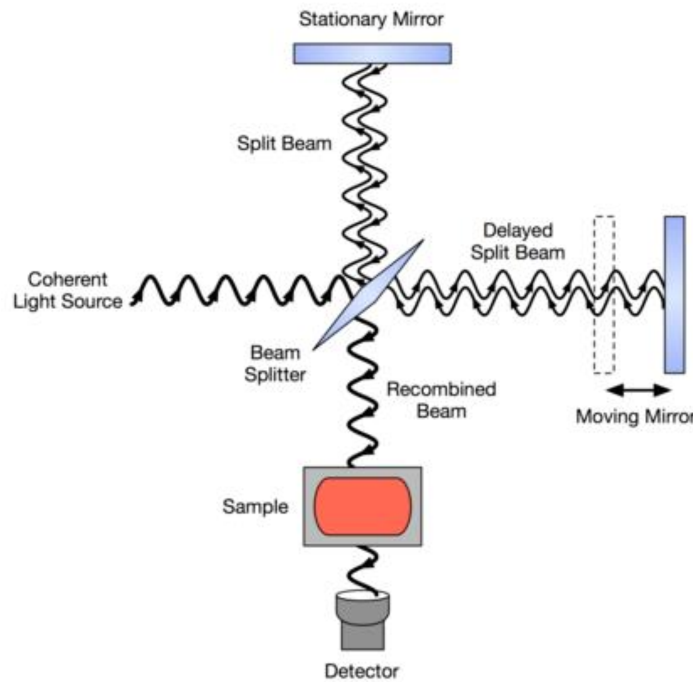
Infrared Spectroscopy (FTIR) [1/3]

- Measurement of the light transmitted through a sample thickness d with polished parallel surfaces

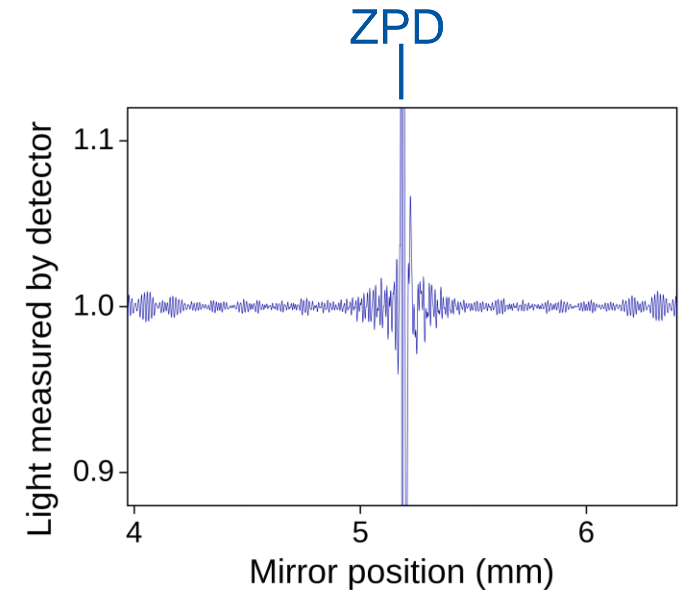
$$I = I_0 \frac{(1-R)^2 \exp(-\alpha d)}{1 - R^2 \exp(-2\alpha d)}$$

I_0 light intensity incident on the sample
d thickness of the sample
α frequency dependent absorption coefficient
R reflectivity

$$R \approx \left[\frac{n-1}{n+1}\right]^2 \approx 0.3 \; in\; the\; mid\; infrared$$



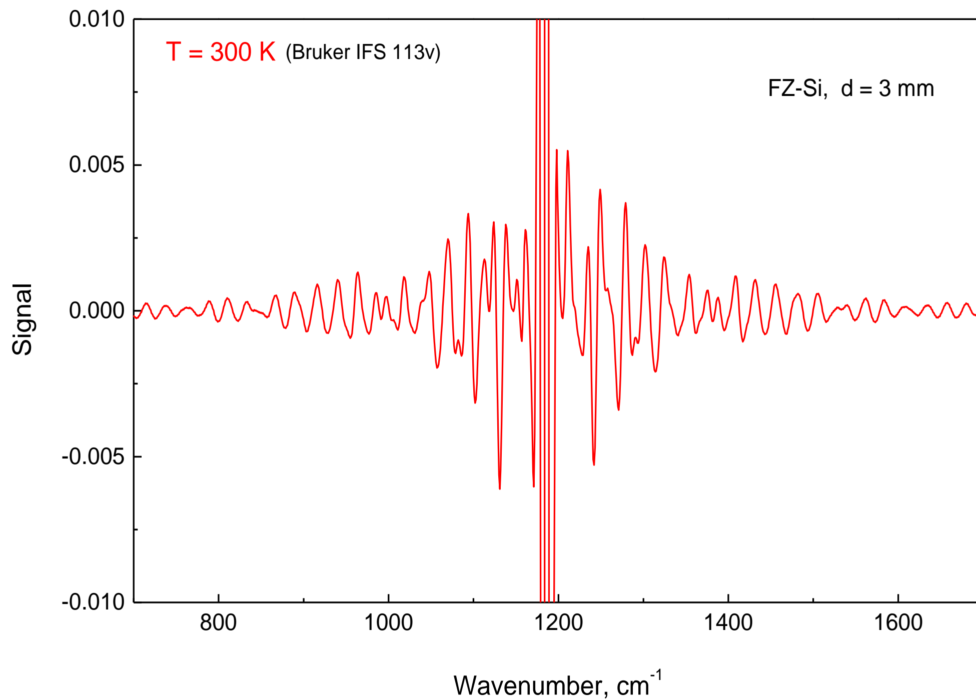
Michelson Interferometer

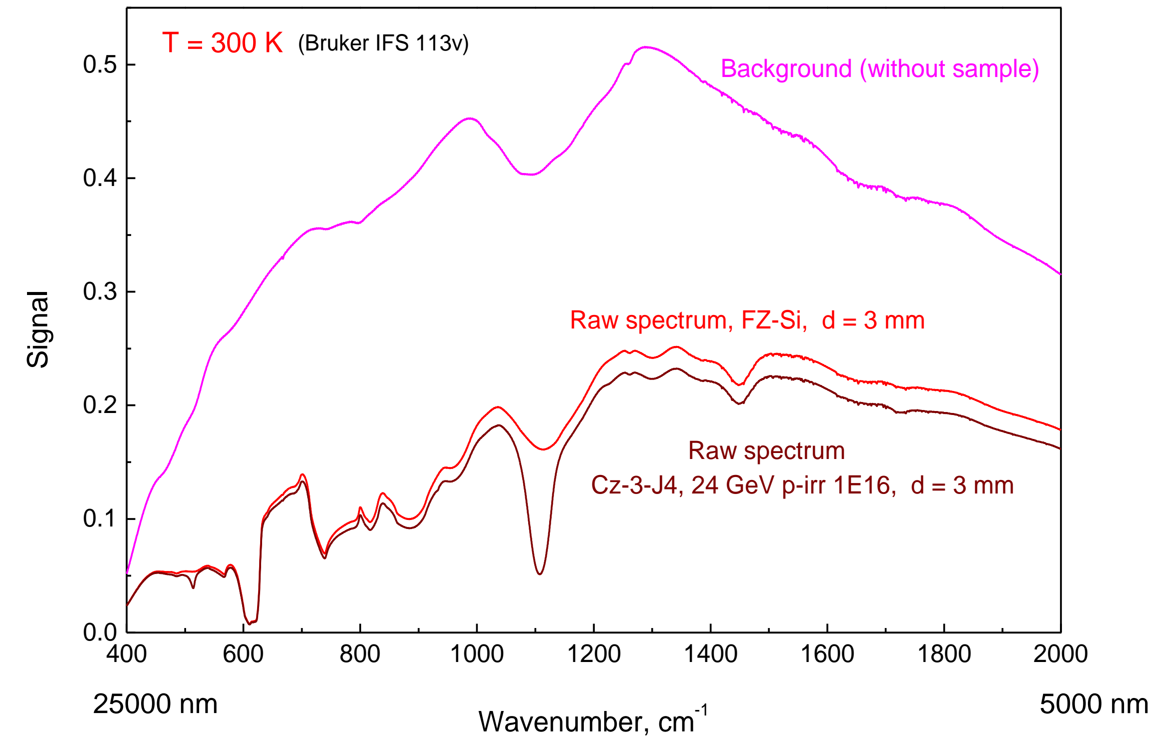


Interferogram

Infrared Spectroscopy (FTIR) [2/3]

- FTIR – Fourier Transform Infrared Spectroscopy





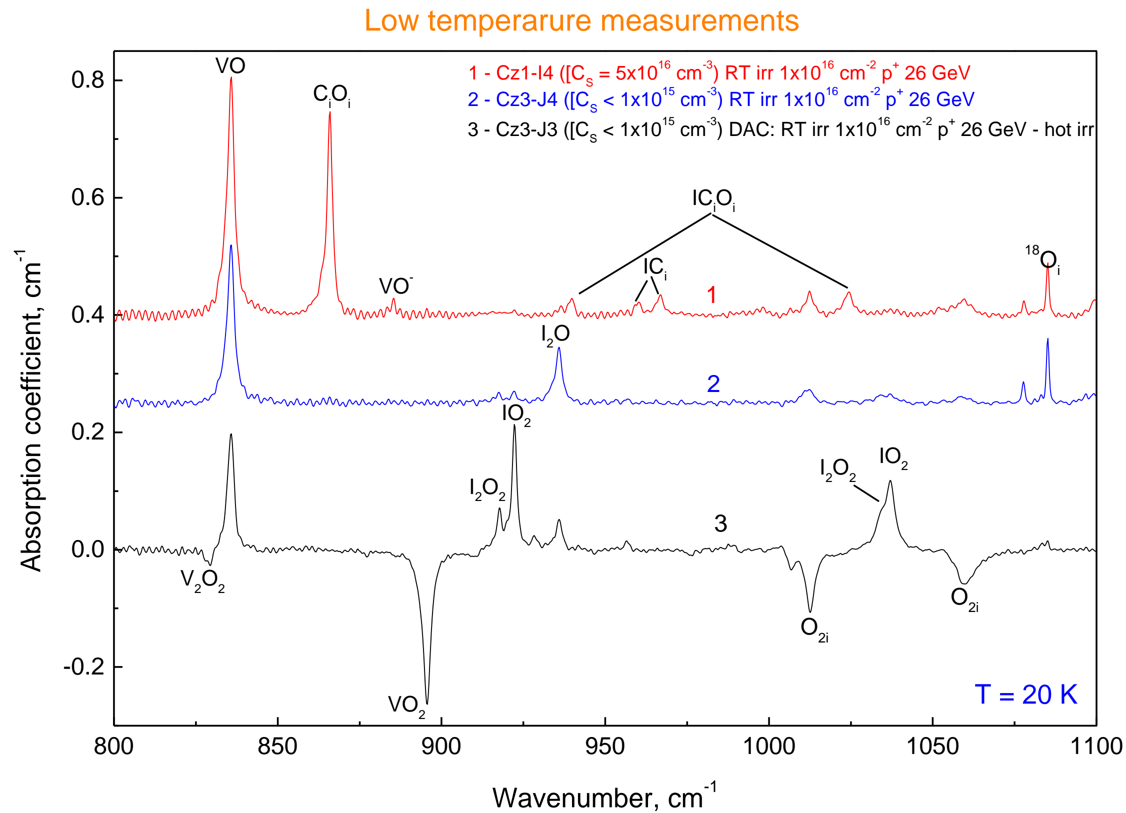
Interferogram

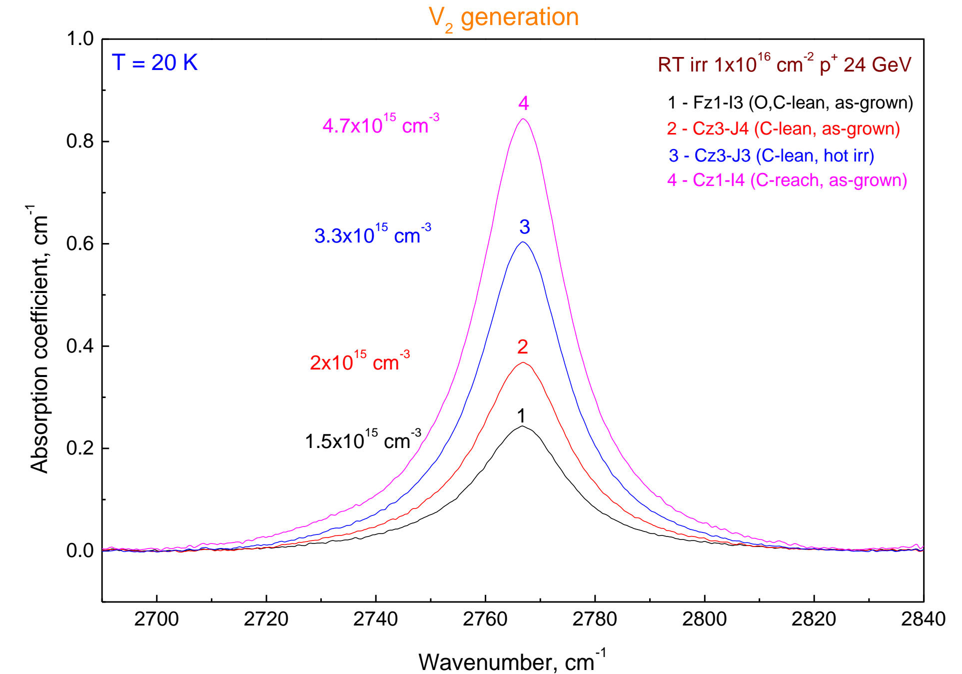
Fourier Transformation

Spectrum

Infrared Spectroscopy (FTIR) [3/3]

- Measurements at low temperature (20K)

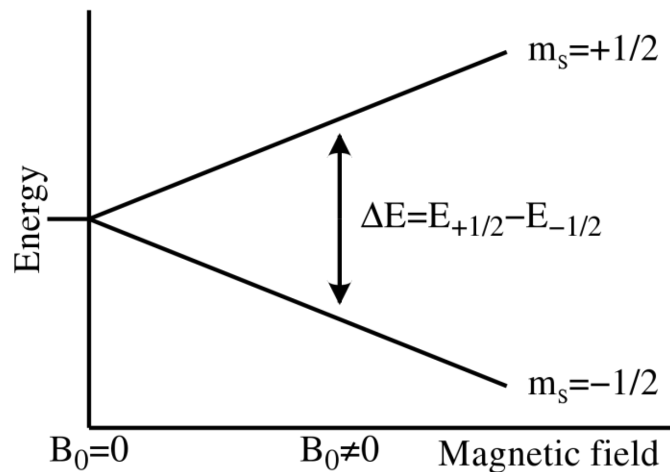




- Detection limits depend on
  - measurement temperature (LT or RT)
  - Sharpness of the lines
  - Wavenumber position
- Detection limits normally in the range
  - 5×10^{13} to 10^{15} cm^{-3}

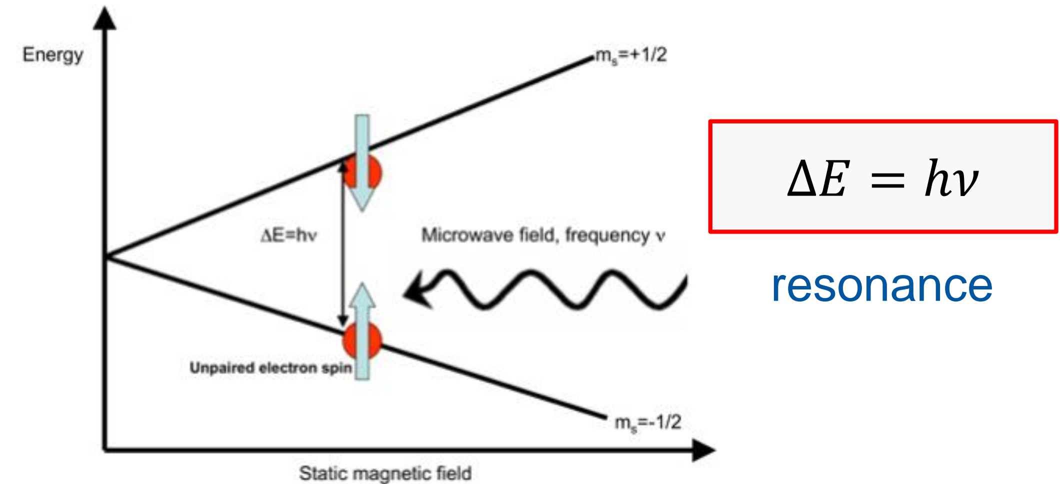
EPR:Electron Paramagnetic Resonance [1/3]

- **EPR** is the best experimental technique for determining the structure of irradiation induced paramagnetic point defects in semiconductors.
- **EPR** spectroscopy deals with the interaction of the electron spins with external magnetic fields
  - **EPR = Zeeman spectroscopy:** The effect of splitting a spectral line into several components in the presence of a static magnetic field (B ≠ 0) of defects with unpaired electron states (S = 1/2, 1, …).



$$\Delta E = g_e \mu_B B_0$$

splitting of energy levels



$$\Delta E = h\nu$$

resonance

- The condition of resonance: $h\nu = \Delta E = g_e \mu_B B_0$, $g_e = 2.0023$ for free electron
  - $\nu \rightarrow$ 9.5 – 34 GHz, 95 GHz, …
  - Sensitivity: 2 x 10^{10} spins/Gauss (~ 1 ppb)

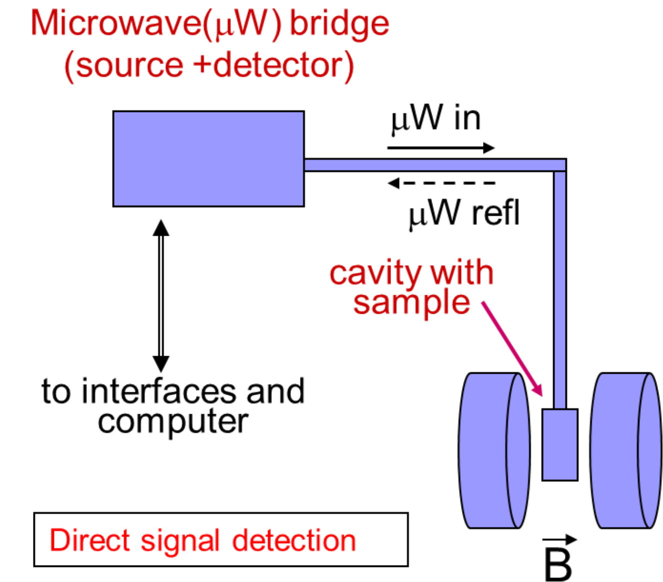
g_e electron's g-factor
$g_e = 2.0023$ (free electron)
μ_B Bohr magneton

$$\mu_B = \frac{e\hbar}{2mc}$$

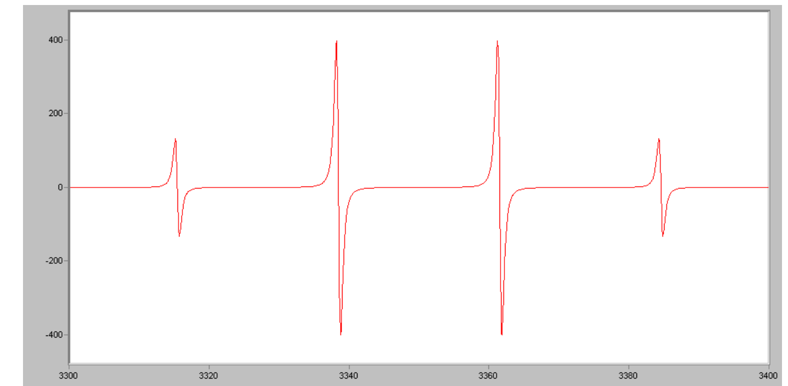
1 Gauss = 10^{-4} Tesla

EPR:Electron Paramagnetic Resonance [2/3]

- **An unpaired electron can gain or lose angular momentum**
  - change the g-factor value through **spin-orbit coupling**
  - **information about the nature of the atomic/molecular orbital containing the unpaired electron - defect 's electronic structure**
  - The magnetic moment of a nucleus (I≠0) affects any unpaired electrons associated with that atom.
    - **hyperfine splitting** of the EPR resonance signal into doublets, triplets …..
    - further surrounding nuclei lead to super hyperfine splitting
  - Also the nuclear quadrupole moment impacts on the measured signal



- The g-factor and hyperfine coupling in an atom/ molecule may not be the same for all orientations of the unpaired electron in external magnetic field
  - spectra anisotropy depending on the electronic structure of the atom/molecule
  - → reflects the local structure

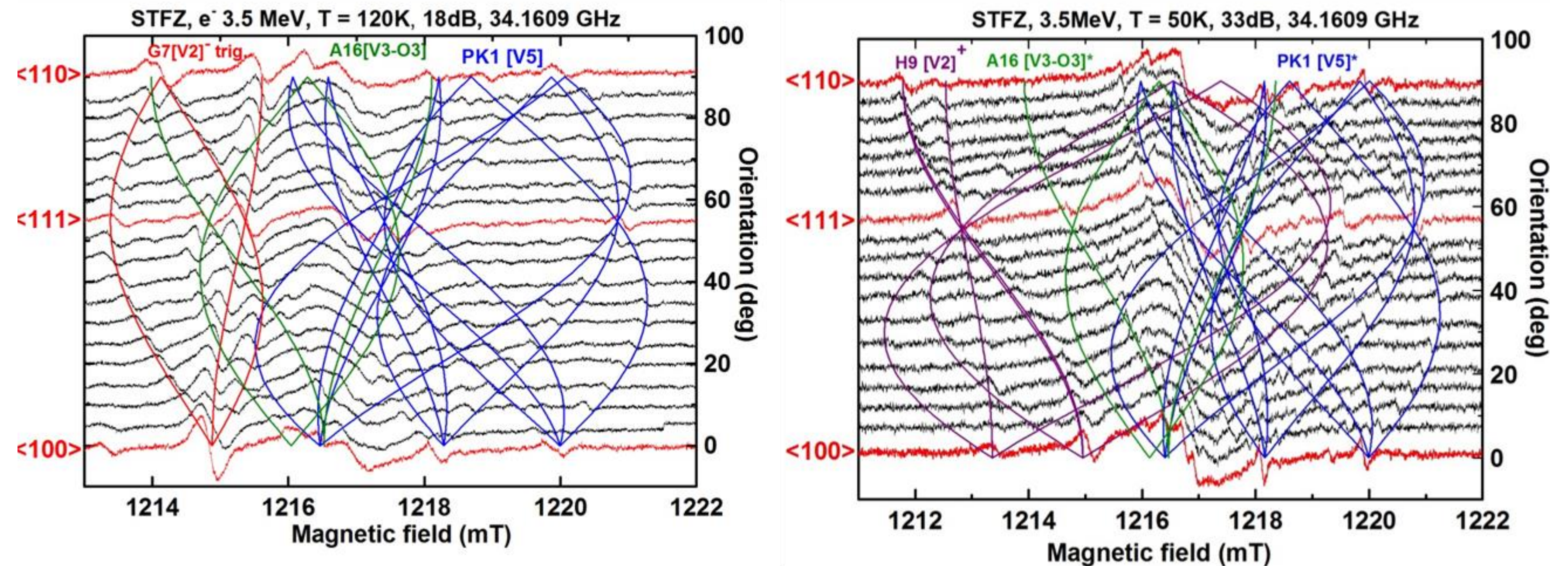


EPR spectrum of CH_3 radical

EPR:Electron Paramagnetic Resonance [3/3]

- **Example: EPR results after irrad. with 3.5 MeV electrons, Φ=10^{17} cm^{-2}**

Variation of ESR spectra with measuring temperature (120K) and (50 K).



- G7[**V2**]$^-$ (trig.)
- PK1 [**V5]*** (triclinic) vacancy aggregate seen at 120K.
- A16[**V3-O3**] (ortorh.) .

- G7[V2]$^-$ is not observed (ESR signal saturates) at T = 50K.
- H9 [V2]+ (monocl.) observed at T =50 K.
- A16[V3-O3]*
- PK1[V5]*

} -observed at low μW power (33 dB).

Radiation Damage

RD50

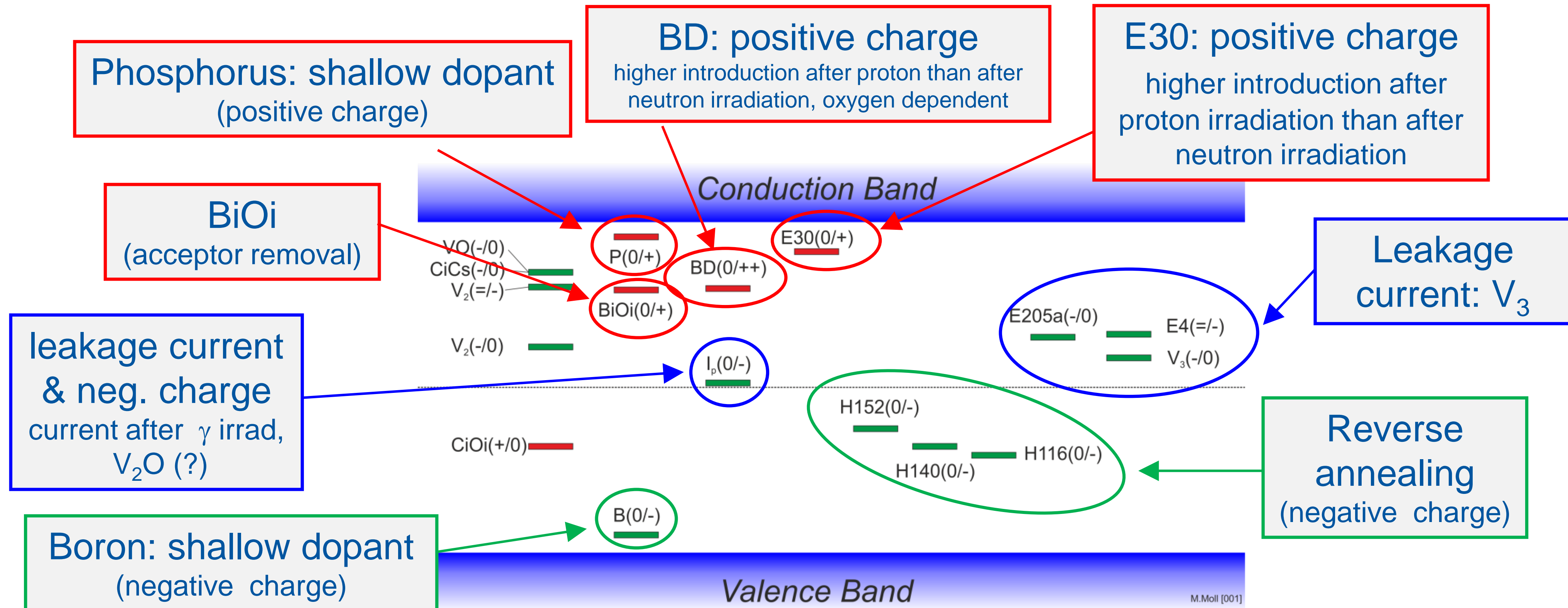
Microscopic Defects
vs.
Macroscopic Device Properties

Some selected examples with clear correlations between defects and device degradation:

- Leakage current after hadron irradiation (short term annealing)
- Space charge changes during reverse annealing (long term annealing)
- Oxygen enriched silicon: proton vs. neutron damage
- Acceptor removal effect in LGAD sensors

Radiation induced defects with impact on device performance

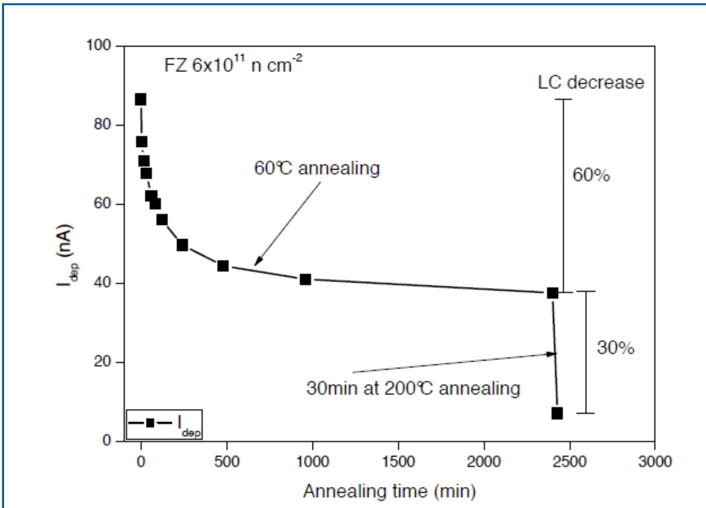
RD50 map of most relevant defects for device performance near room temperature:



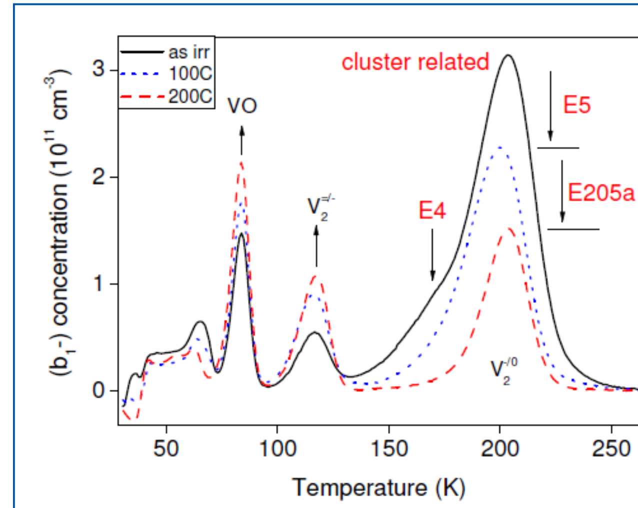
- Trapping: Indications that E205a and H152K are important (further work needed)
- Converging on consistent set of defects observed after p, π, n, γ and e irradiation.
- Defect introduction rates are depending on particle type and energy, and some on material!

Example: Defects with impact on leakage current

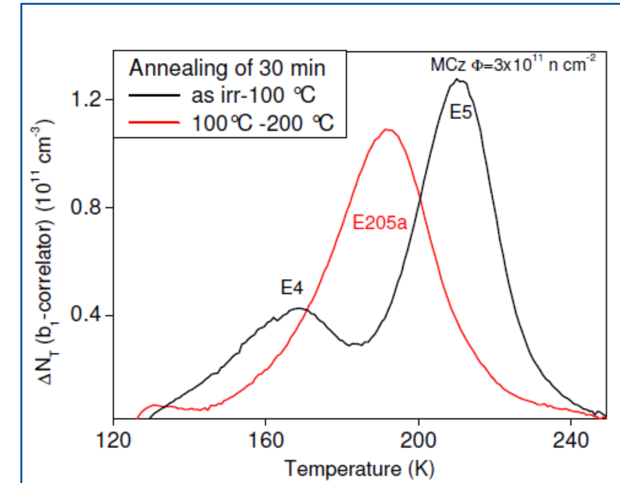
- Macroscopic observation: Leakage current build-up following NIEL (for hadrons)
  - Leakage current scaling (almost) with NIEL and independent of silicon material (not for gammas!)
  - Leakage current is annealing in time and with temperature.
- Example: Annealing study on a FZ sample ($6x10^{11}$ n/cm^2)



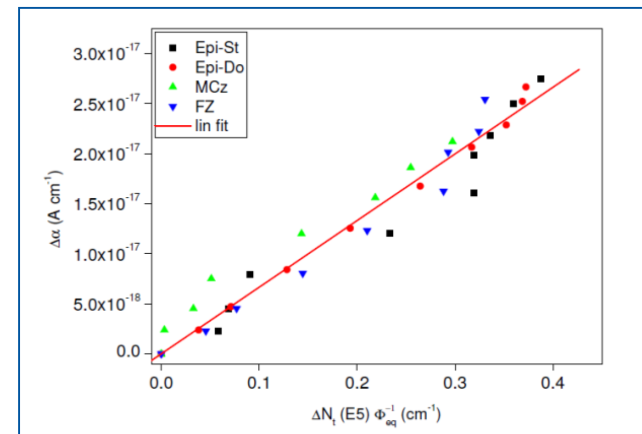
Leakage Current



DLTS spectra



DLTS difference spectra (disappearing peaks)



Decrease in defect concentration

$\Delta N_t(E5)$ vs. $\Delta\alpha$ Correlation found for many materials after neutron irradiation

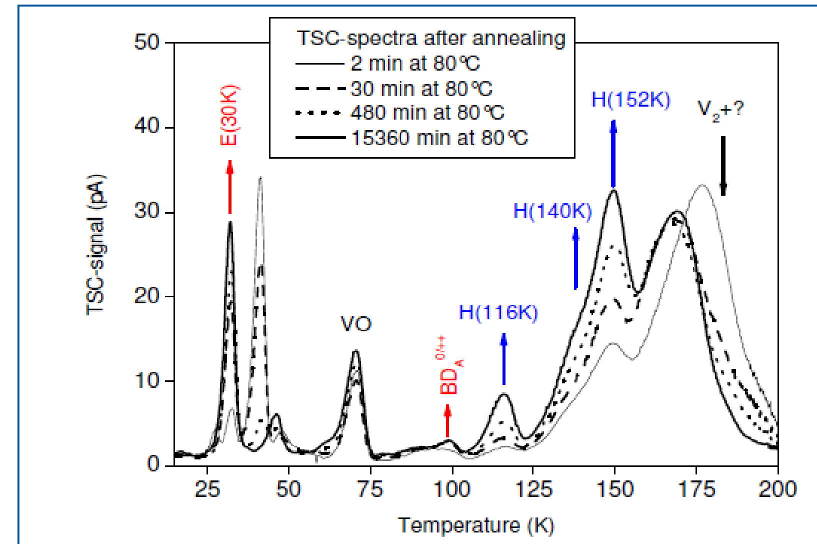
- Microscopic observation:
  - The defects E4/E5 (annealing at 60°C) and E205a (annealing at 200°C) are contributing to the leakage current with 60% and 30 % respectively.

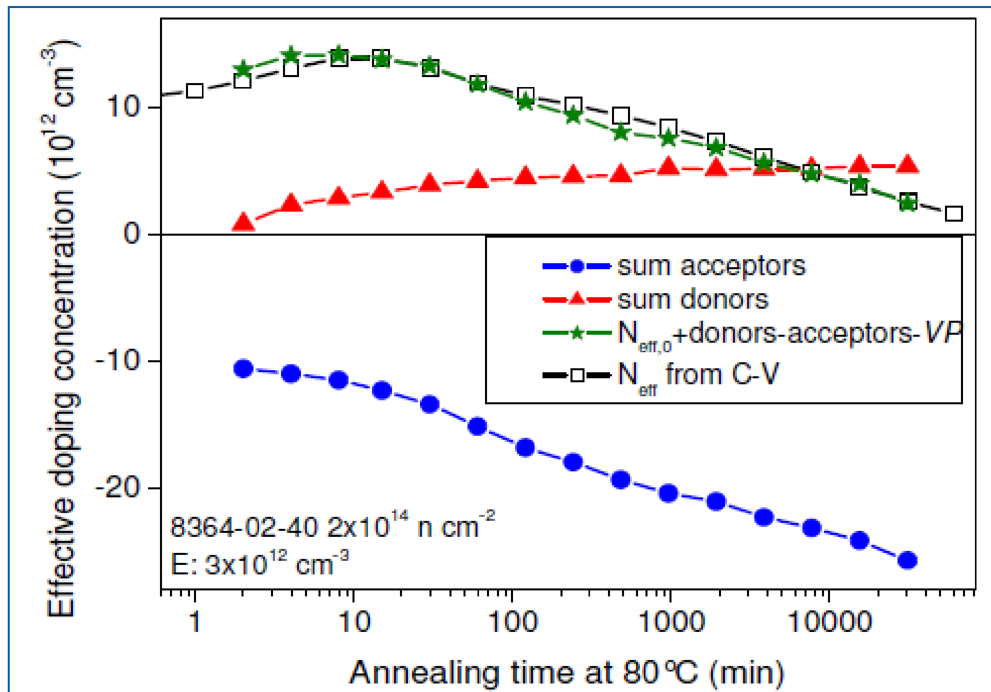
[A.Junkes, PhD thesis 2011 & Vertex 2011 Proceedings]

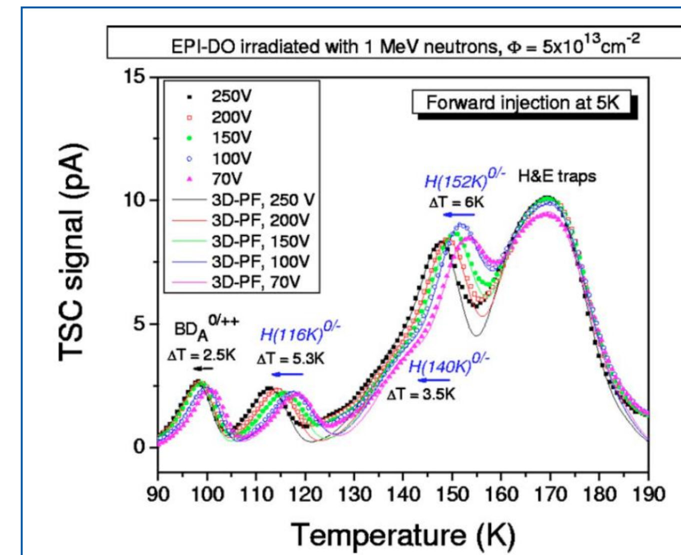
Defects: Impact on N_{eff} (reverse annealing)

- Macroscopic observation:
  - Irradiated silicon sensor show “reverse annealing” (negative space charge increasing with time)
  - **Example:** Neutron irradiated epitaxial silicon
    - Identification of hole traps that grow with reverse annealing and are deep acceptors (labelled: H(116K),H(140K),H(152K))
    - Absolute correlation of defect concentration to increase of |Neff| (reverse annealing)

$2x10^{14}$ n/cm², Epi-St 75µm



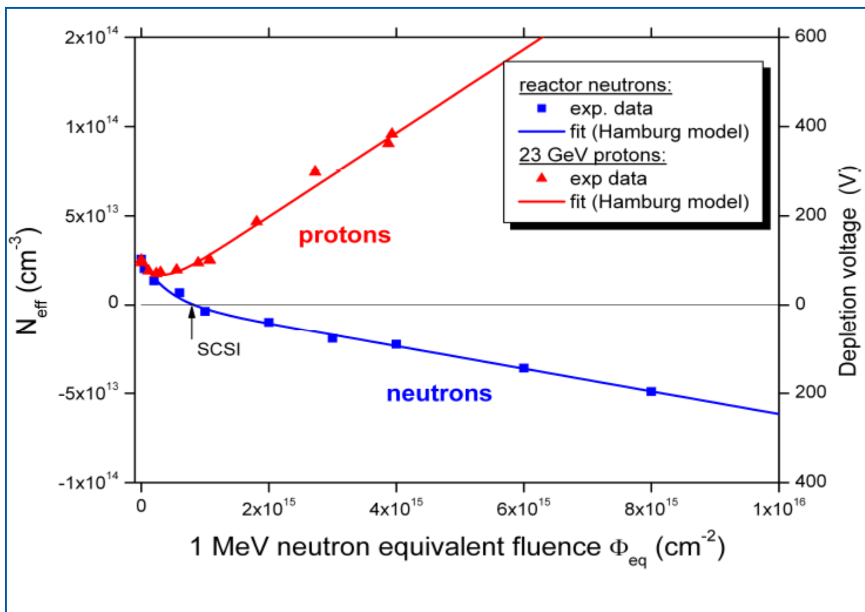


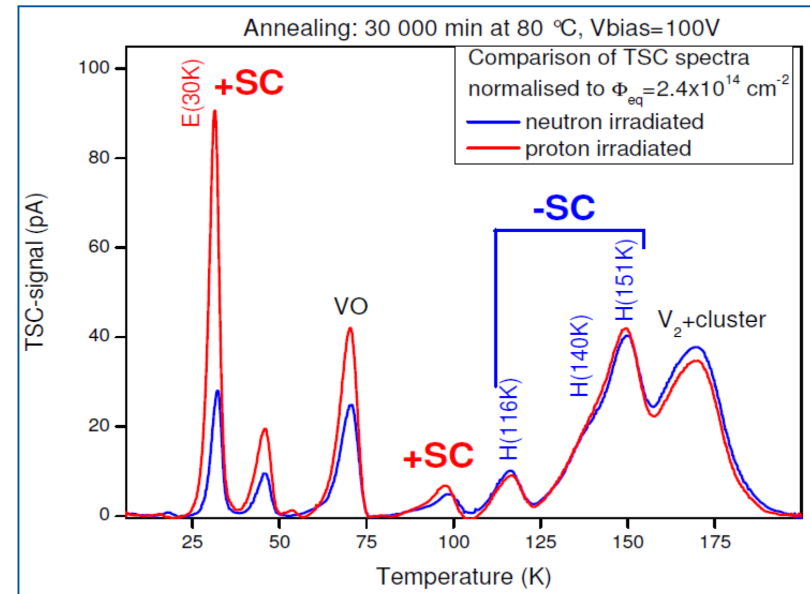


[A.Junkes PoS(Vertex 2011) 035; I.Pintilie, APL 92,024101 (2008); Junkes PhD thesis]

Defects: Impact on N_{eff} (particle type)

- Macroscopic observation: Dependence on particle type (protons vs. neutrons)
  - In several oxygen rich silicon materials neutron irradiation leads to build-up of net negative space charge ("type inversion") while charged hadron irradiation leads to build up of net positive space charge.
  - Note: Violation of NIEL (Non Ionizing Energy Loss) Hypothesis!
- Example: Epi silicon (EPI-DO, 72μm,170Ωcm) irradiated with 23 GeV protons or reactor neutrons





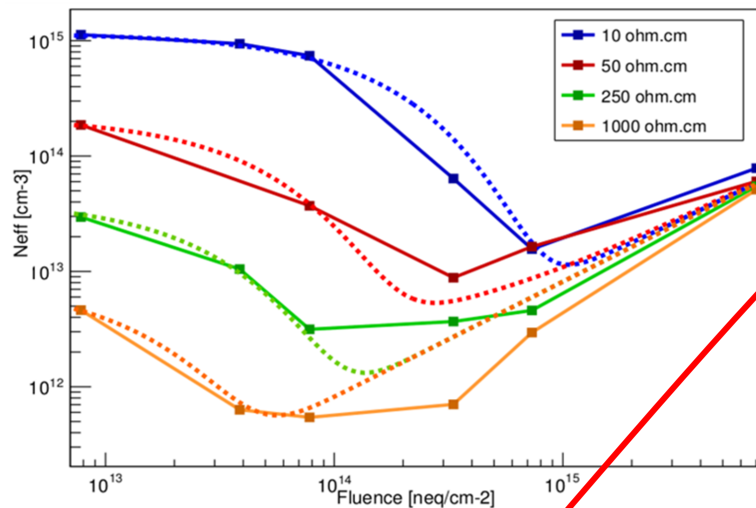
- Microscopic observation
  - The Donor E(30K) is introducing the additional positive space charge after proton irradiation
  - Defects related to build-up of negative space charge not influenced (follow the NIEL scaling)

[I.Pintilie et al., NIMA 611 (2009)52-68]

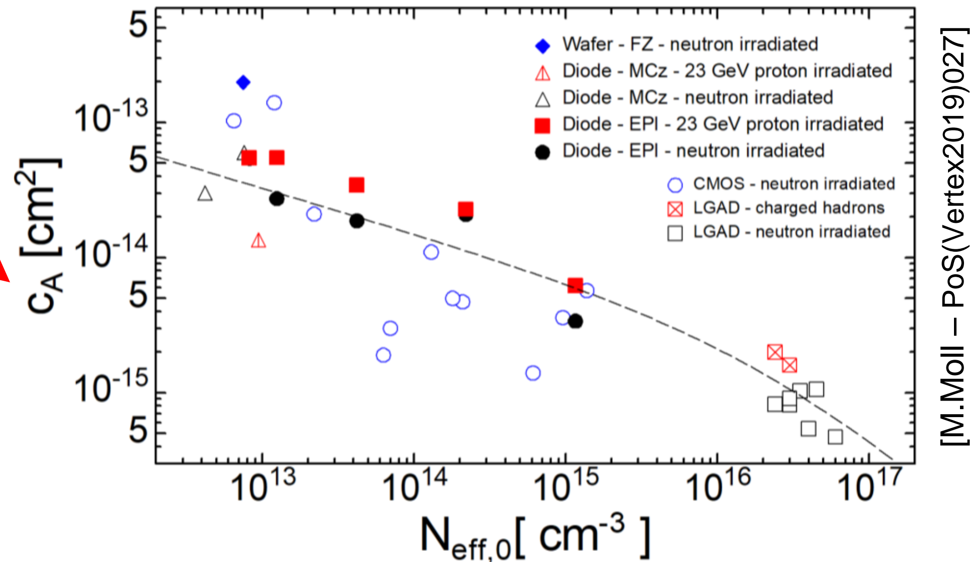
RD50: Dedicated acceptor removal studies

- **Acceptor removal:** Radiation induced de-activation of acceptors (p-type doping, Boron)
- Impact:
  - Change of silicon conductivity; Change of sensor depletion voltage and/or active volume
  - **Loss of gain in LGAD sensors,** sets radiation harness limits for timing detectors (ETL, HGTD)
- Macroscopic studies:

Example: 23 GeV proton irradiated epi diodes



$$N_{eff}(\Phi) = N_{B0}\exp(-c_A\Phi) + g\cdot\Phi$$

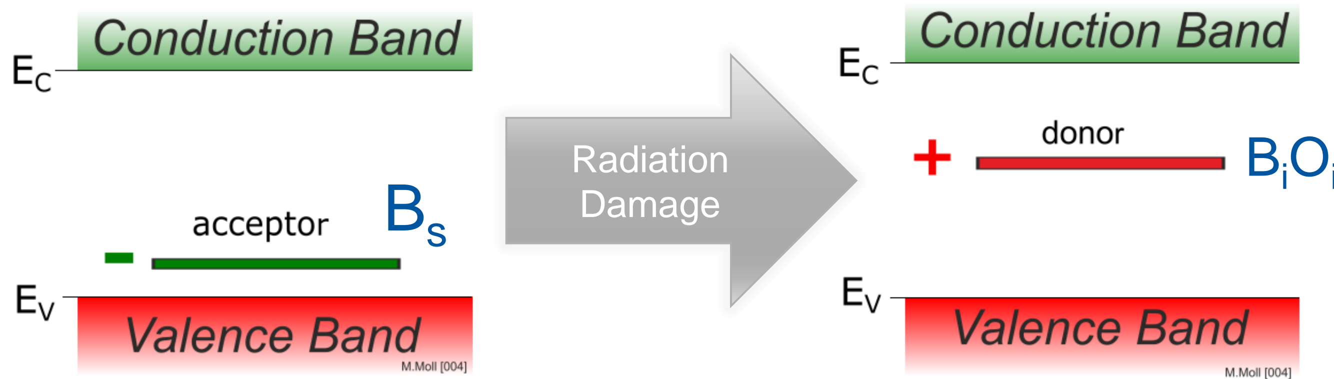


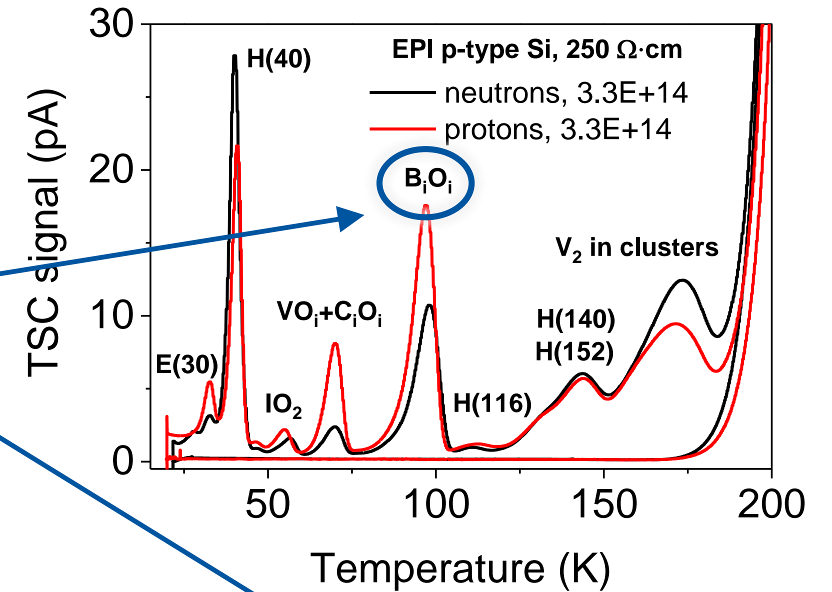
- Acceptor removal coefficients obtained on a wide range of sensor types
  - pin diodes (epi, FZ, MCZ, …), LGAD detectors, CMOS sensors
  - after charged hadron irradiation (red) and neutron irradiation (black/blue)

- **Parameterization of acceptor removal established within RD50**
  - covering the range [B]=10^{12} to 10^{18} cm^{-3} (10 kΩcm to 5 mΩcm) i.e. damage predictions can be done

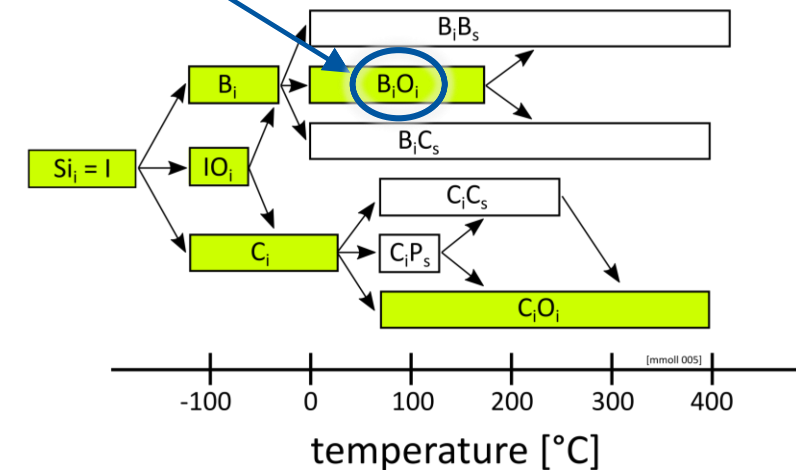
Defect studies: Acceptor Removal

- Microscopic origin:
  - Formation of defects containing Boron that no longer acts as shallow dopant





- Status
  - Large amount of data (Wafers, Detectors, CMOS, LGAD)
  - Acceptor removal is parametrized over 6 orders of magnitude in resistivity
    - Damage predictions are possible
  - Defect engineering (with Carbon) works but microscopic understanding needs more work!
    - Measured defect concentrations do not fully explain the macroscopic observations.



Summary

Technique	Based on	Defect parameters	Limitations
C-DLTS Deep Level Transient Spectroscopy	Charge capture/emission -Capacitance transients	E_t, $\sigma_{n,p}$, N_t	low density of bulk or interface defects ($<N_d/3$) Chemical nature (indirect)
TSC Thermally Stimulated Current	Charge capture/emission Current –free charged carriers	E_t, $\sigma_{n,p}$, N_t	high density of bulk defects (up to 1000 N_d) Chemical nature (indirect)
TDRC Thermally Dielectric Relaxation Current	Charge capture/emission Displacement Current	E_t, $\sigma_{n,p}$, N_t	high density of interface states Chemical nature (indirect)
PL –Photoluminescence	Photon Absorption followed by Photon Emission	PL bands; (E_t, τ)	Only for radiative bulk recombination centers Chemical nature (indirect)
FTIR - Infrared Spectroscopy	Absorption of IR energy on molecules vibrational modes	N_t (acc. 20-30%), Defect structure	-Large density of defects ($> 10^{15}$ cm^{-3})
EPR Electron Paramagnetic Resonance	Zeeman effect and Spins resonance	Chemical nature and vicinity N_t	Large density of defects ($> 10^{16}$ cm^{-3}) Only paramagnetic centers
High Resolution Transmission Electron Microscopy	Electron microscopy	structure and chemical composition	Large density of defects - clusters electron beam damage of the sample during the observations

…there are many more methods (see references).

- Most powerful is the combination of various methods in combination with annealing experiments and variation of materials (e.g. doping levels, impurities, isotope doping; exposure to gammas, electrons, protons, neutrons.
- **RD50** has successfully identified several radiation induced defects responsible for device degradation effects (e.g. leakage current and effective doping of silicon sensors, impact of oxygen).
- Hot topic today: Acceptor removal …. ongoing work

References and Acknowledgements

Material taken from:

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