

1st Workshop on QA Issues in Silicon Detectors, CERN, 17.-18.5.2001

Radiation Damage and Annealing in View of QA Aspects

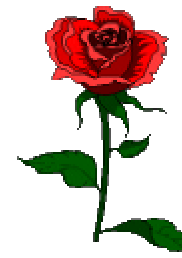
Michael Moll

CERN EP/TA1-SD
<http://www.cern.ch/ssd>

Many of the shown results were achieved by the former
ROSE Collaboration (CERN RD48)

**ROSE - Research and Development
on Silicon Detectors for Future Experiments**

<http://www.cern.ch/rd48>



Motivation

◆ Motivation for Silicon Detector QA

- **Ensure:**
 - operability of detectors for 10 years in the LHC-environment

◆ QA-sub-issue: Radiation damage in the silicon bulk

- **Ensure:**
 - that the radiation induced changes of the silicon bulk material
 - a) do not prohibit a 10 year operation of the detectors at LHC
 - b) do only as little harm to the detector operability as possible
(However, take into account costs and feasibility!)
- **Questions:**
 - Can the material be modified in order to make it radiation harder ?
 - Which is the best operational temperature ? \Rightarrow Annealing of damage ?
 - To which extend can we predict the damage in the “LHC-environment”?
 - How should a radiation test look like ? Where are the problems ?

Outline

◆ Radiation induced changes in silicon detector properties

- Increase of leakage current
- Change of effective doping concentration (depletion voltage) **▶ most problematic !**
- Decrease of charge collection efficiency

◆ Predictability of radiation damage

- Particle dependence of radiation damage (NIEL Violations)
- Material dependence of radiation damage (impurities, resistivity)
⇒ Material modifications **▶ Radiation hardening !**
- Examples for unexpected results after irradiation tests

◆ Annealing of radiation damage

- Theory and parameterisation (material dependence)
⇒ Different experiments do different annealing cycles after irradiation tests,
to which extent are they comparable ?

◆ Damage projections for LHC operation

◆ Summary

Primary Damage

◆ PKA - Primary Knock on Atom

◆ Simulation

(Fig.: van Lint 1980)

- 50 KeV PKA
(average recoil energy for PKA produced by 1 MeV neutrons)

◆ Displacement threshold in Silicon:

- Single lattice atom (Frenkel pair):
 $E_d \gg 25 \text{ eV}$
- Defect cluster
 $E_C \gg 5 \text{ keV}$

◆ Neutrons (elastic scattering)

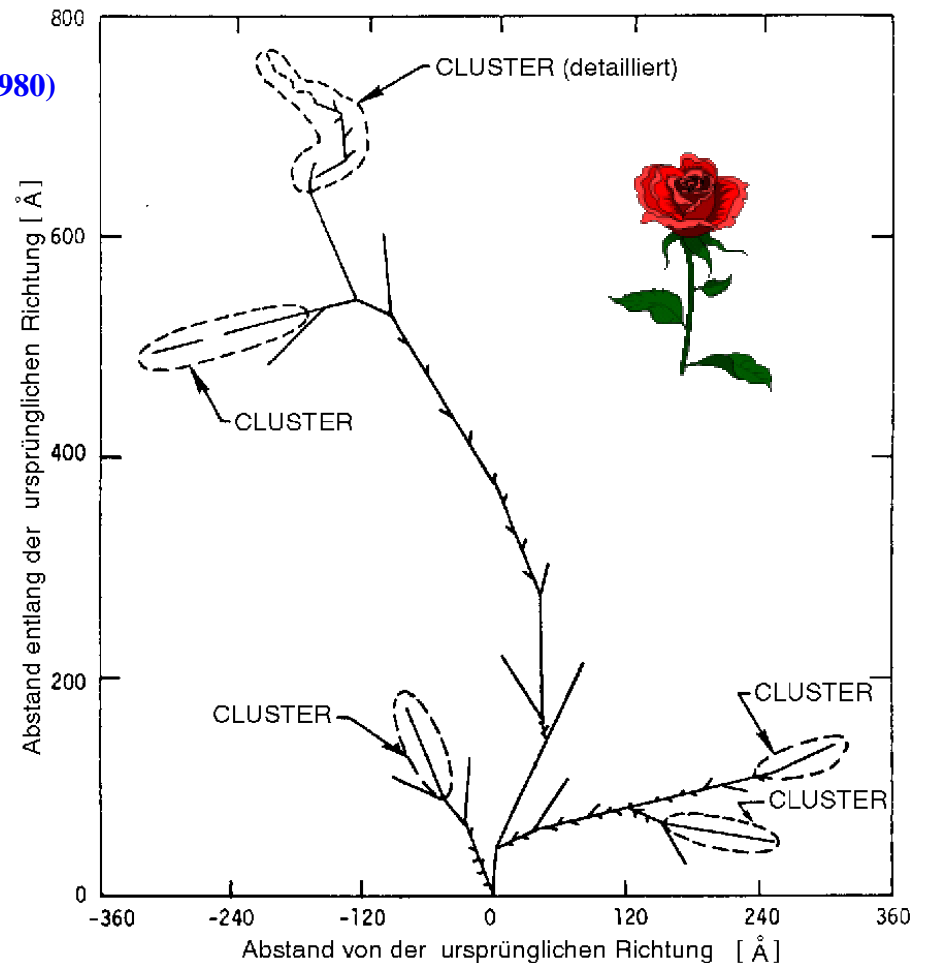
- $E_n > 185 \text{ eV}$ for single displacement
- $E_n > 35 \text{ keV}$ for cluster

◆ Electrons

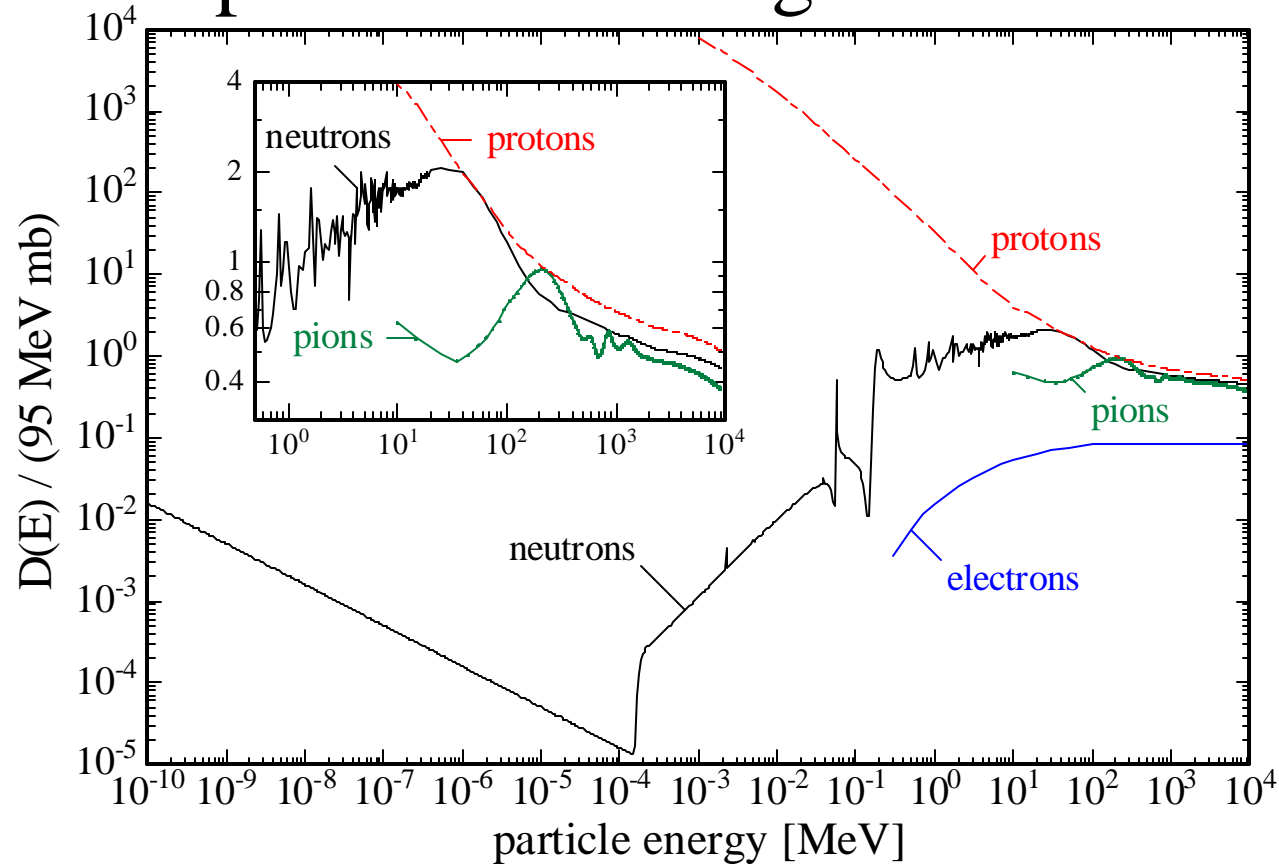
- $E_e > 255 \text{ keV}$ for single displacement
- $E_e > 8 \text{ MeV}$ for cluster

◆ ^{60}Co -gammas

- Compton Electrons with max. $E_g \gg 1 \text{ MeV}$ (no cluster production)



Displacement damage functions

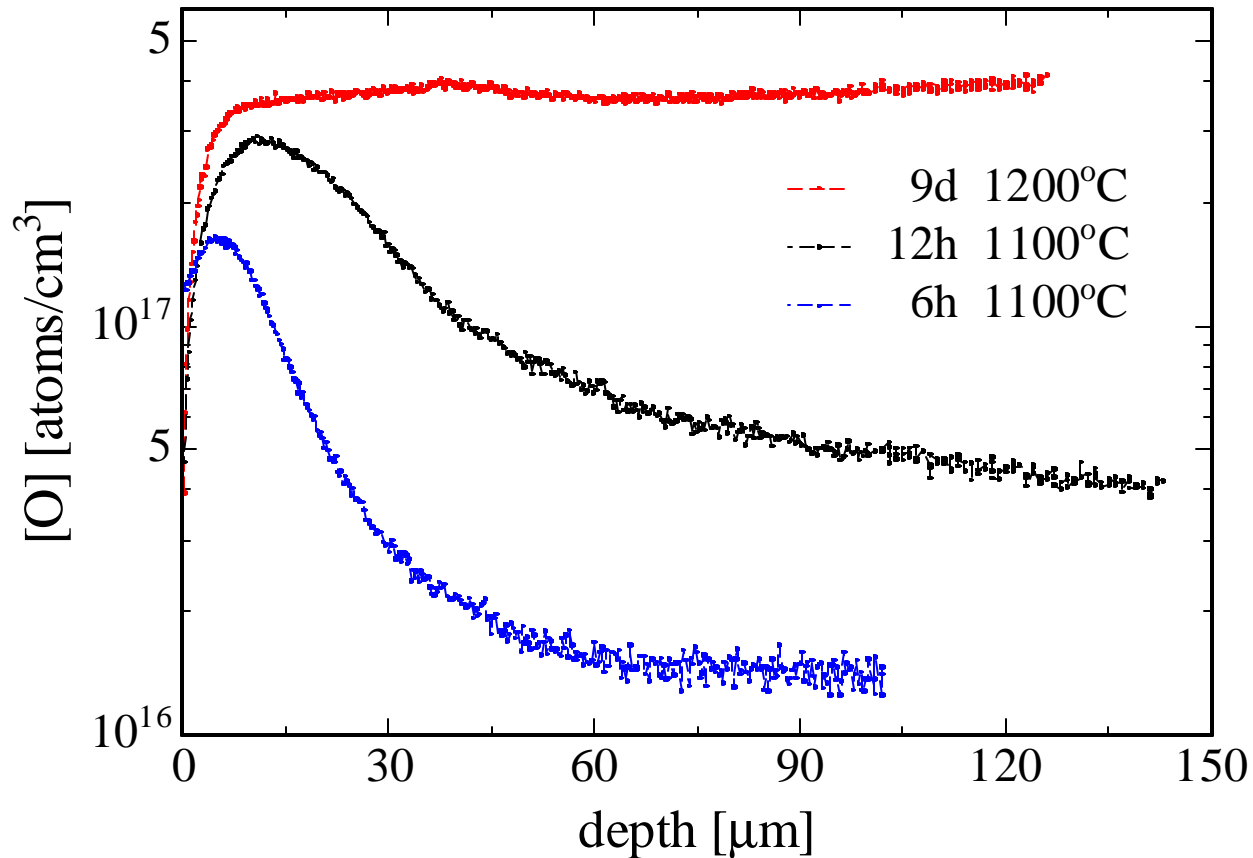


◆ **NIEL - Non Ionizing Energy Loss**

◆ **NIEL - Hypothesis:**

- Damage parameters scale with the NIEL
- ⇒ **Be careful, does not hold for all particles / damage parameters !**

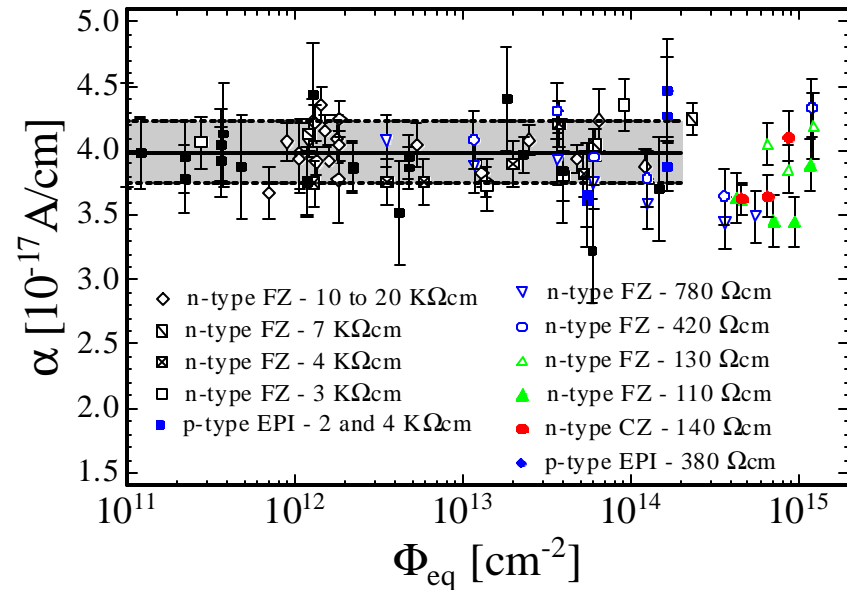
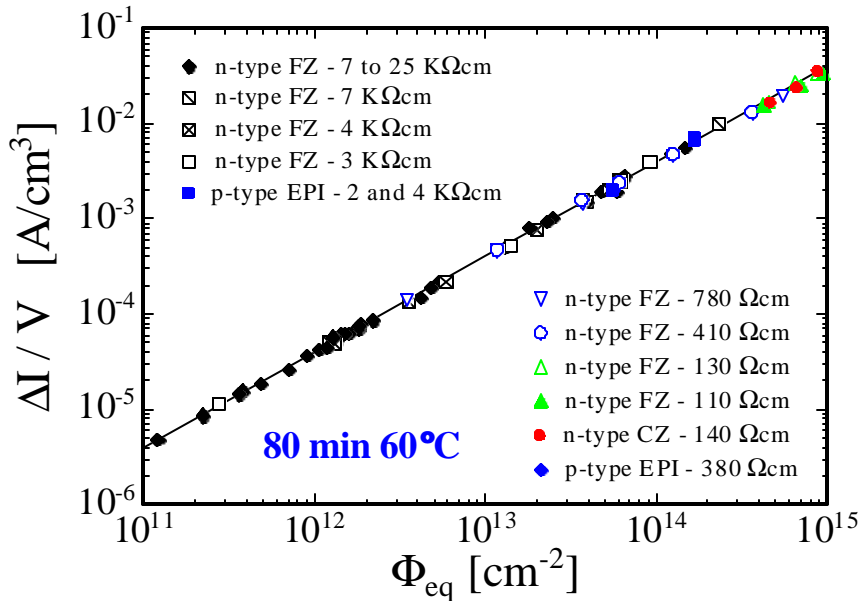
ROSE Collaboration -- Oxygen diffusion



◆ DOFZ - Diffusion Oxygenated Float Zone

- Profiles measured by SIMS (Secondary Ion Emission Spectroscopy)

Increase of Leakage Current



- ◆ Increase of leakage current independent of:
 - Conduction type (p or n), resistivity
 - oxygen and carbon content
 - crystal orientation $\langle 111 \rangle$, $\langle 100 \rangle$

- ◆ Temperature dependence:

$$I \propto \exp\left(-\frac{E_g}{2k_B T}\right)$$

cooling strongly reduces current

- ◆ Damage parameter **a**

- definition:

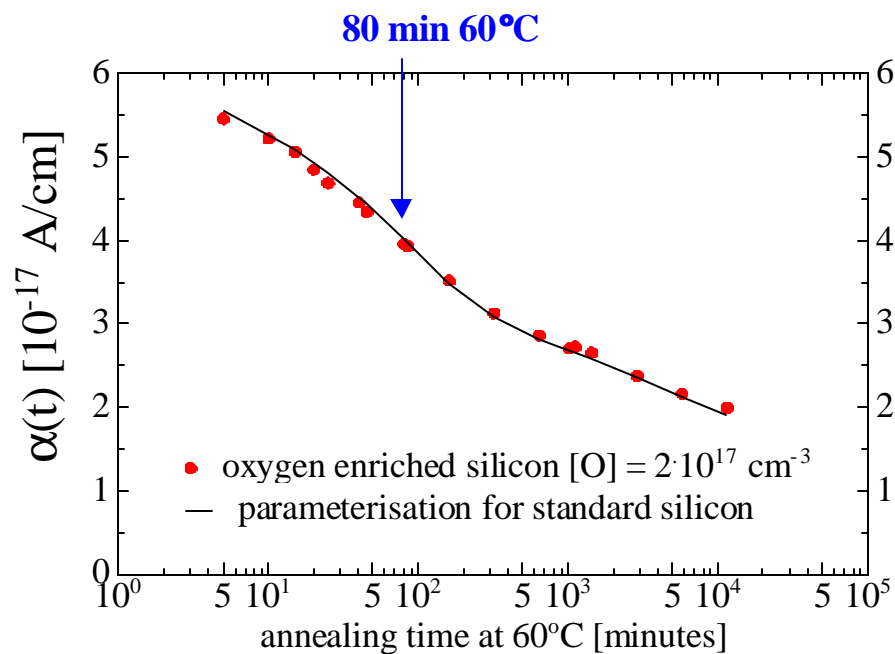
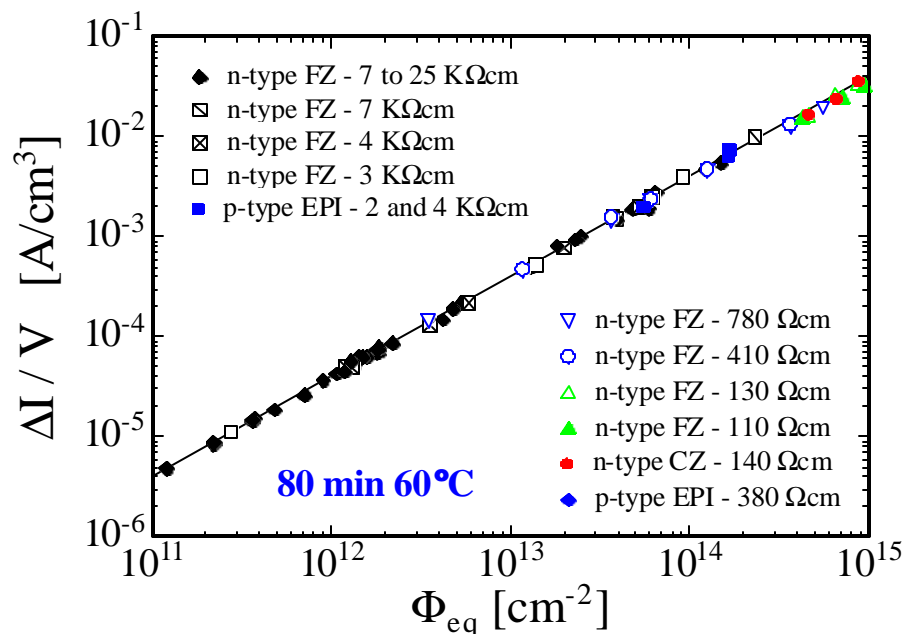
$$a = \frac{\Delta I}{V \cdot \Phi_{eq}}$$

- measured after 80min at 60C:

$$\mathbf{a}_{80/60} = (3.99 \pm 0.03) 10^{-17} \text{ A/cm}$$

- used for fluence (NIEL) calibration

Leakage Current Annealing - Oxygenated Silicon



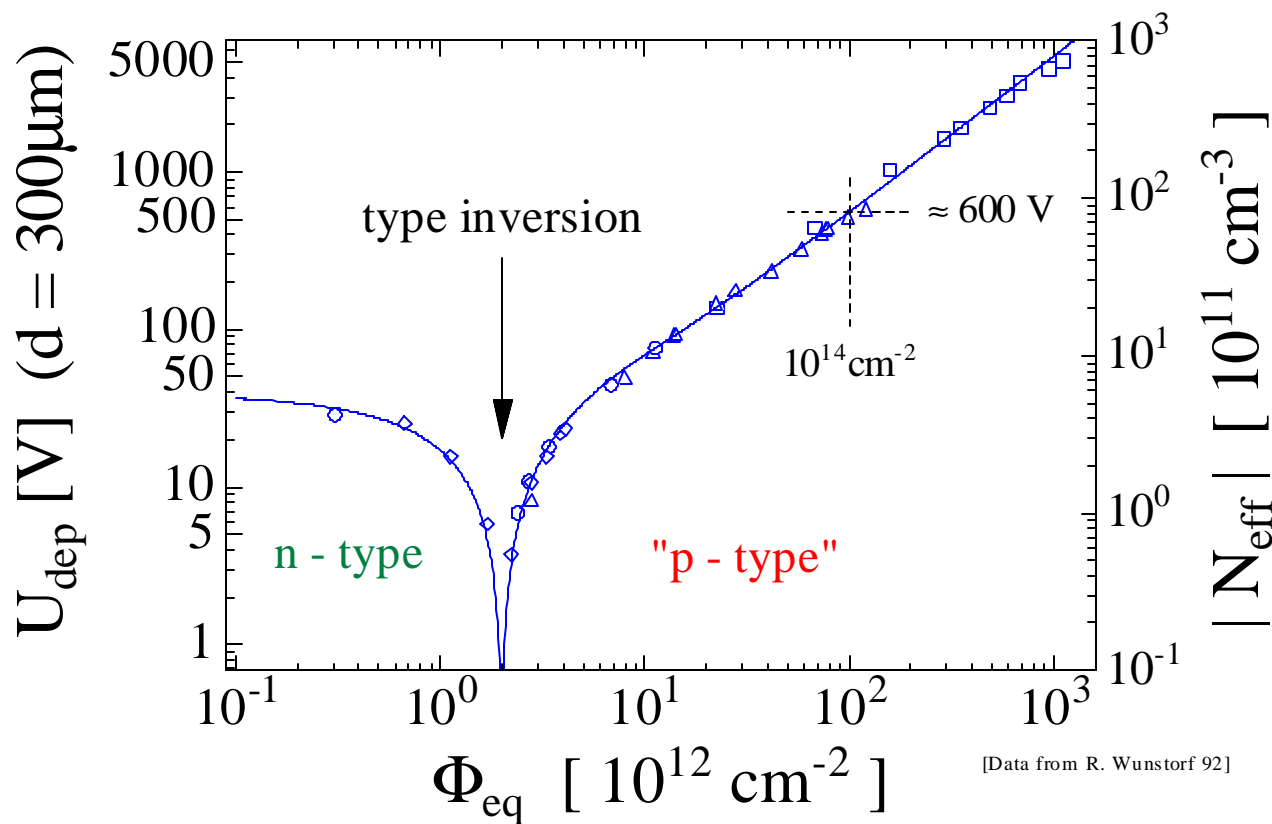
◆ Oxygenated and Standard Silicon show same annealing

- Annealing does not depend on the impurity content of the material

N_{eff} - effective doping concentration

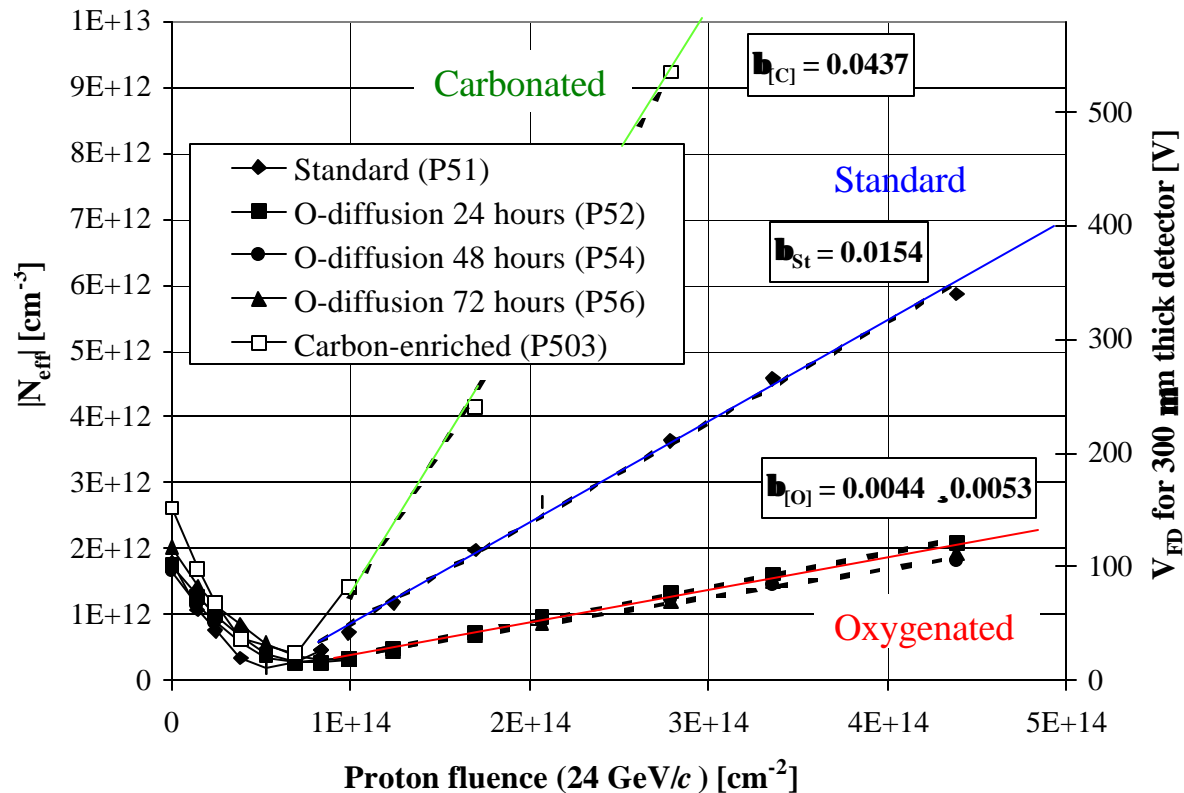
$$|N_{\text{eff}}| \propto \frac{V_{\text{dep}}}{d^2}$$

- N_{eff} positive - n-type silicon (e.g. Phosphorus doped - Donor)
- N_{eff} negative - p-type silicon (e.g. Boron doped - Acceptor)
- $|N_{\text{eff}}|$ proportional to depletion voltage and $1/(\text{device thickness})^2$



Influence of Carbon and Oxygen concentration

24 GeV/c proton irradiation

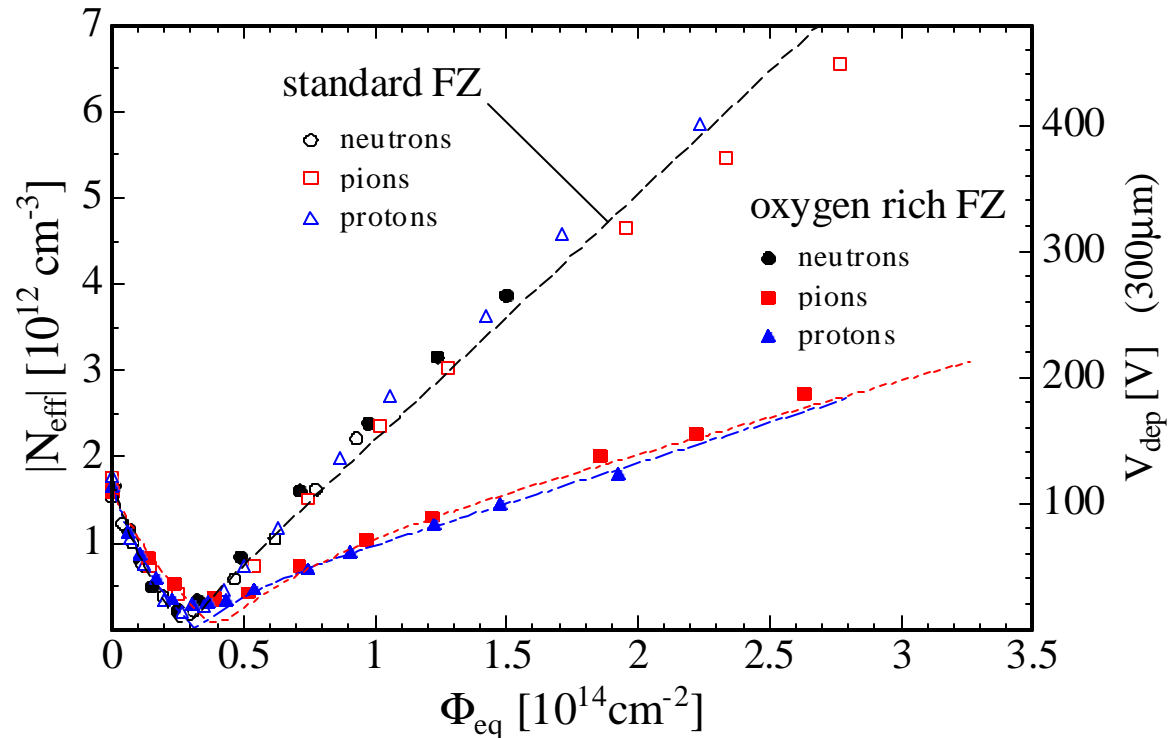


Compared to standard silicon:

- ◆ High Carbon **P** less radiation tolerant
- ◆ High Oxygen **P** more radiation tolerant

Oxygen and standard silicon - Particle dependence -

23 GeV protons - 192 MeV pions - reactor neutrons



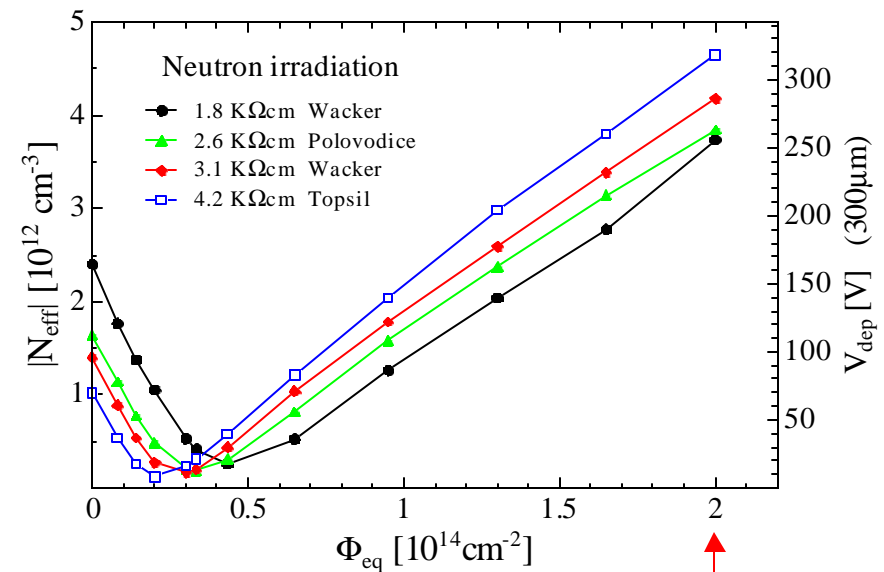
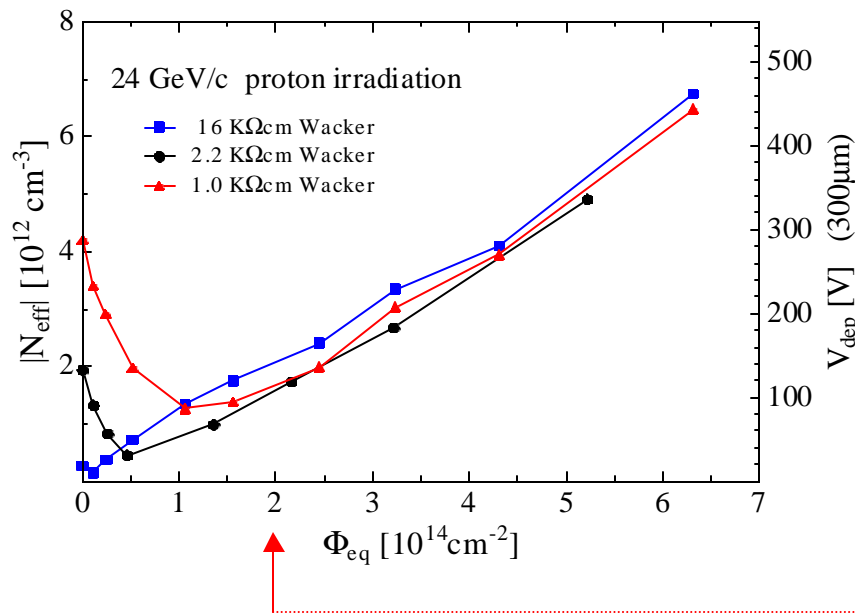
- ◆ Strong improvement for pions and protons
- ◆ Almost no improvement for neutrons

Influence of initial resistivity - particle dependence -

24 GeV/c proton irradiation



Reactor neutron irradiation



(Neutron irradiation with higher Φ_{eq} needed for conclusive comparison)

- ◆ Measured in “CERN-scenario”
(irradiation - 4min/80°C - measurement - irradiation - 4min/80°C -)
- ◆ “Complete donor removal” after 24 GeV/c proton irradiation
- ◆ Low resistivity material is beneficial for neutron irradiation

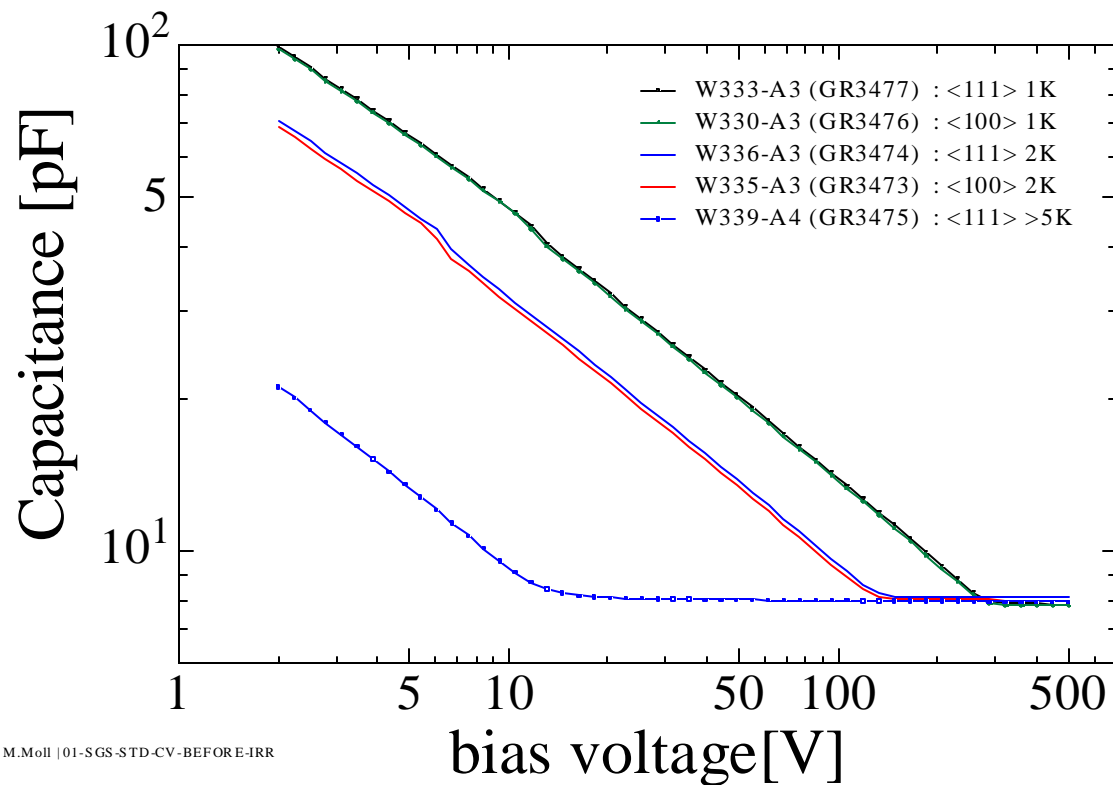
A recent experiment showing strong variations in standard silicon

- ◆ **Wacker silicon**
- ◆ **Different orientations: $\langle 111 \rangle$ and $\langle 100 \rangle$**
- ◆ **Different resistivities: 1, 2 and 15 $\text{K}\Omega\text{cm}$**
- ◆ **Diode producer: ST Microelectronics - ROSE mask**
- ◆ **Two batches:**
 - 1.) No oxygen enrichment \Rightarrow Standard diodes
 - 2.) Oxygen enrichment \Rightarrow Oxygenated diodes
(30h or 60h at 1200°C)
- ◆ **Irradiations:**
 - 24 GeV/c protons (CERN PS)
 - Reactor Neutrons (Ljubljana)

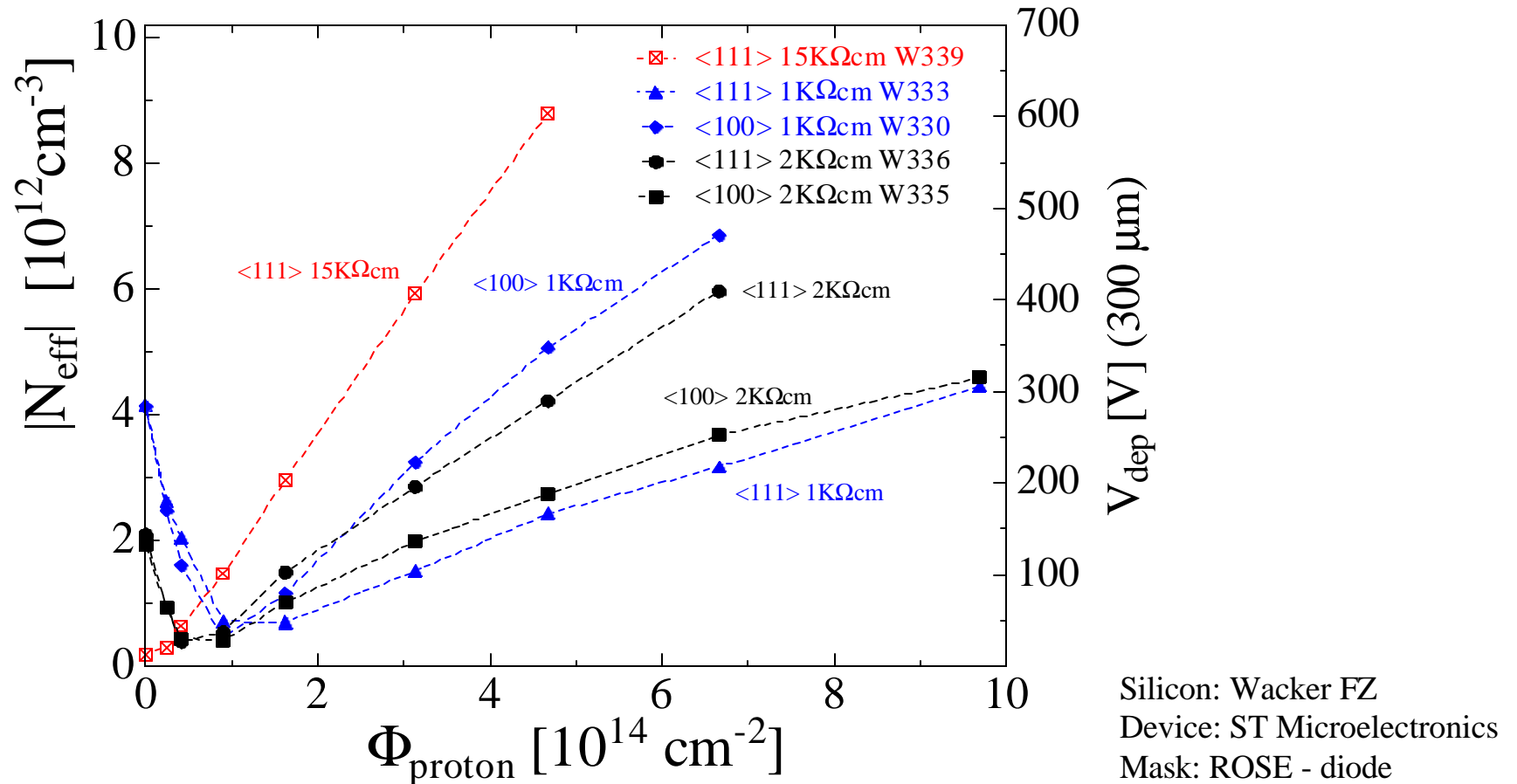
ST Microelectronics - standard diodes

- ◆ Different orientations $\langle 111 \rangle$ and $\langle 100 \rangle$ and resistivities
- ◆ CV measurements before irradiation

Standard silicon : ST - Microelectronics test structures before irradiation

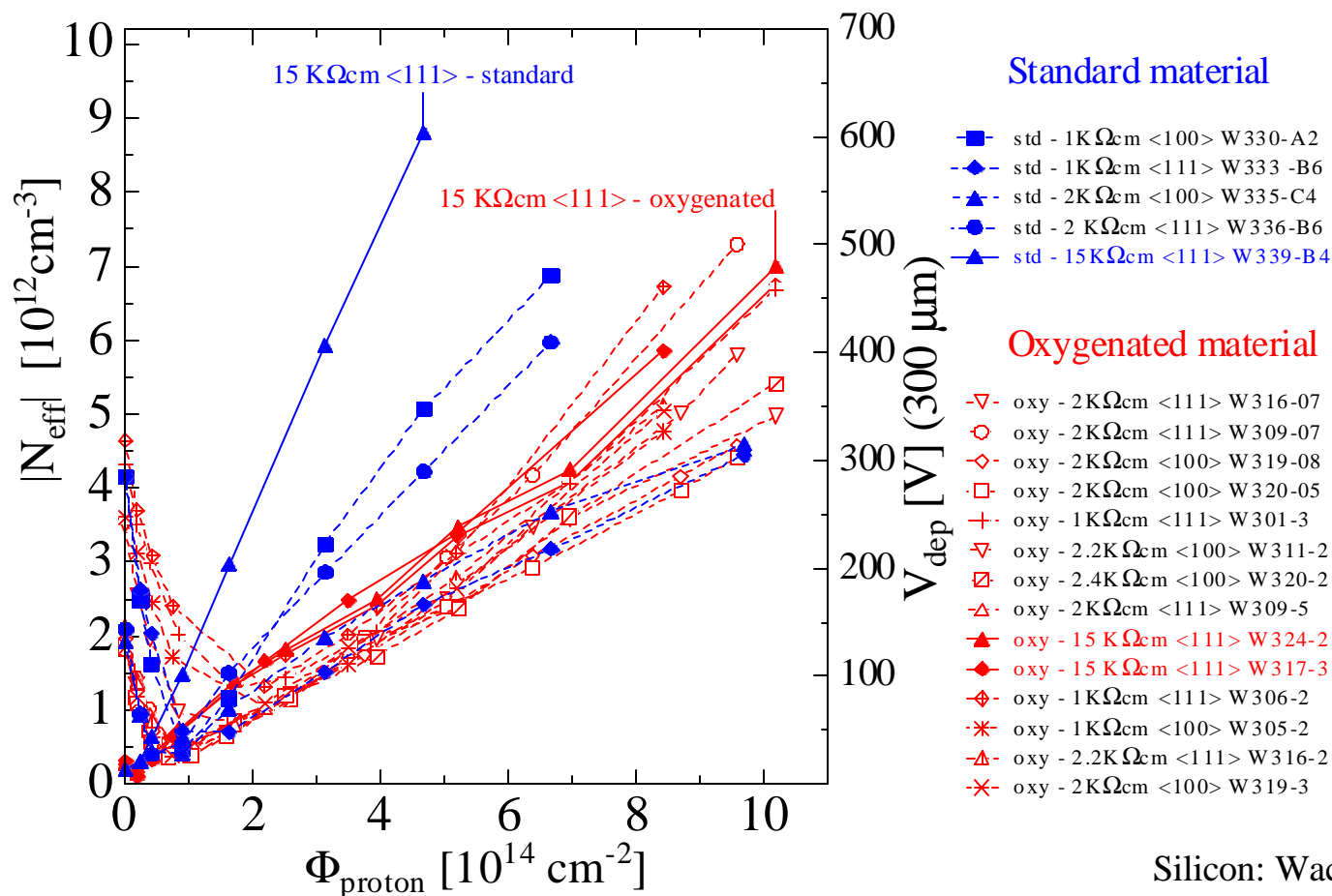


Warning: Variation of “standard material”



- ◆ Strong variation of standard silicon
- ◆ Seems not to depend on resistivity / crystal orientation

Comparison: Standard and oxygenated silicon

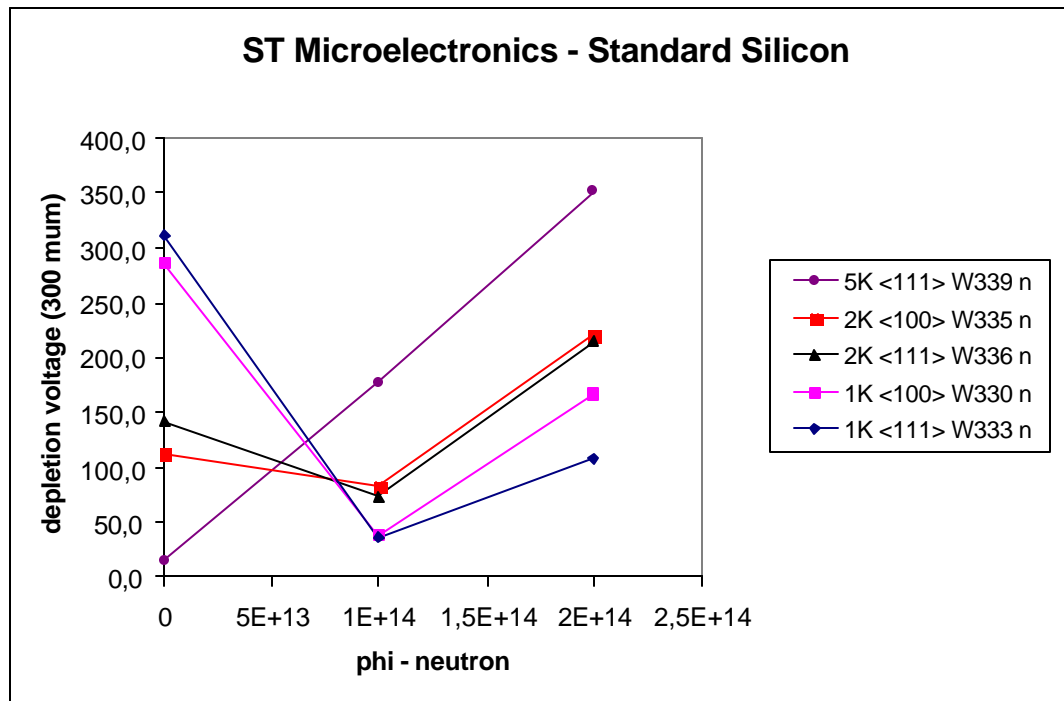


Silicon: Wacker FZ std/oxy
 Device: ST Microelectronics
 Mask: ROSE - diode

- ◆ **Strong variation of standard silicon**
- ◆ **After oxygenation (same material) only small variation**

Neutron irradiation of the standard diodes

- ◆ Neutron irradiation (Ljubljana) + 4min annealing at 80°C



- ◆ Strong dependence on the initial resistivity is observed
- ◆ However, no strong fluctuation in the beta-parameter

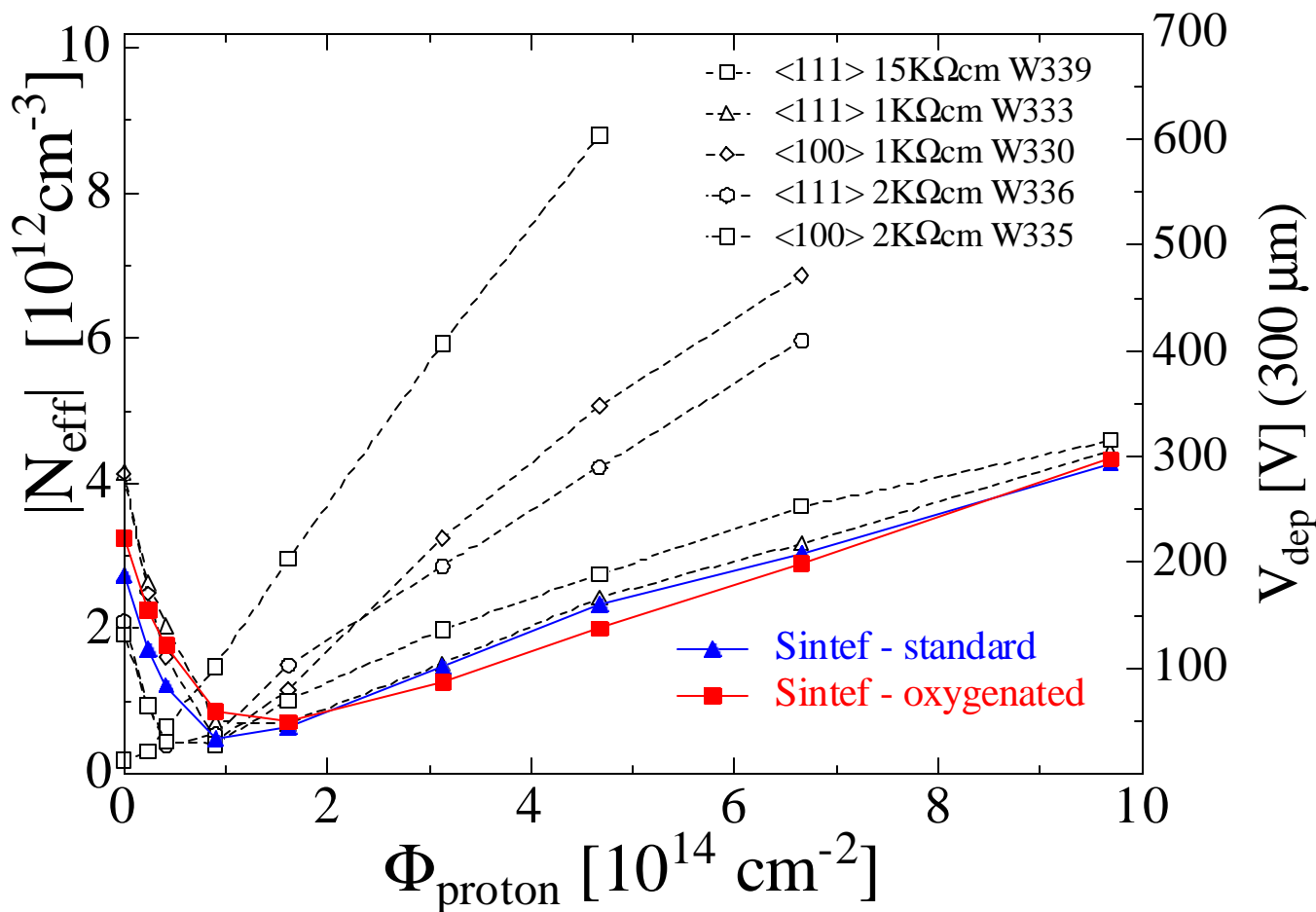
Another experiment: Sintef diodes

Material / Diodes / Irradiations

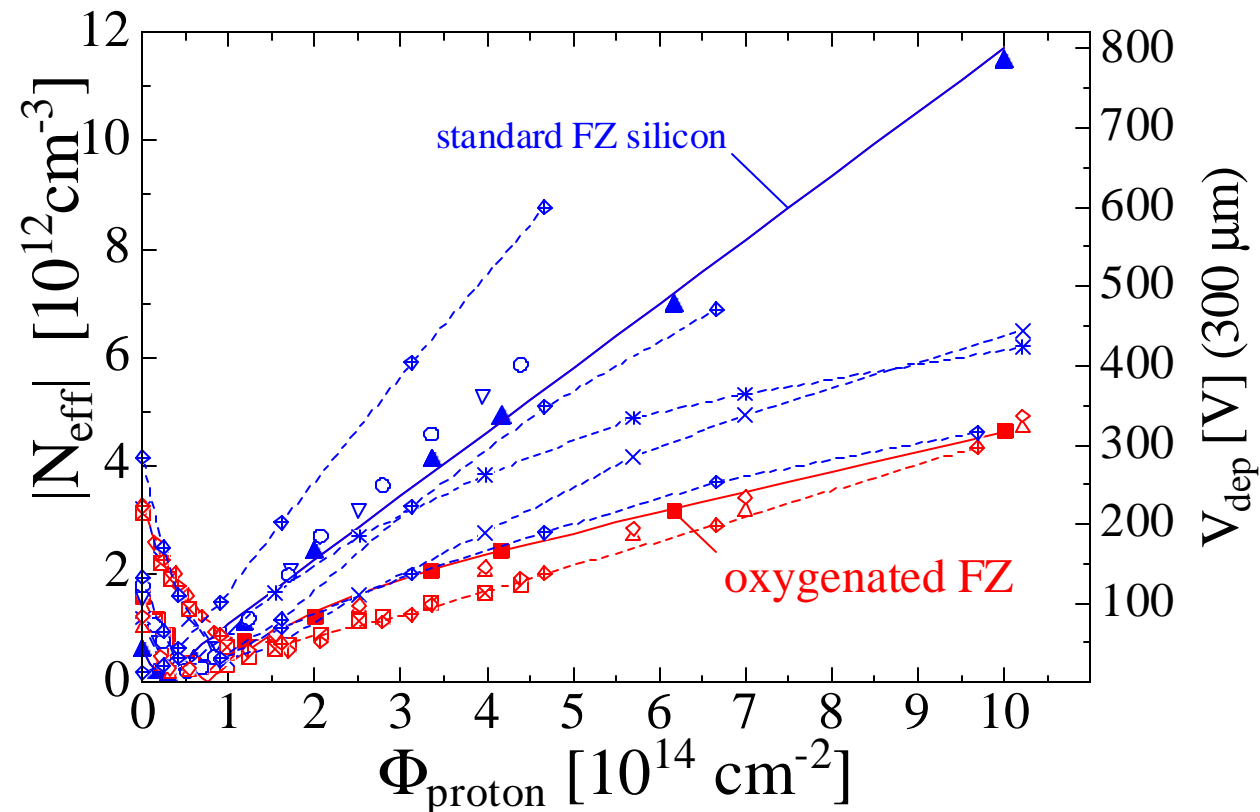
- ◆ **Topsil silicon**
- ◆ **Orientations: $\langle 100 \rangle$**
- ◆ **Resistivity: $1\text{K}\Omega\text{cm}$**
- ◆ **Diode producer: Sintef - ROSE mask**
- ◆ **Two batches:**
 - 1.) No oxygen enrichment \Rightarrow Standard diodes
 - 2.) Oxygen enrichment \Rightarrow Oxygenated diodes
(80h at 1150°C)
- ◆ **Irradiation: CERN PS $24\text{GeV}/c$ protons**

Sintef - standard and oxygenated diodes

- ◆ No difference between oxygenated and standard diode ?



Warning: Variation of “standard material”



- ◆ Strong variation of standard silicon
- ◆ Reproducible results for oxygenated silicon

Annealing of Radiation Damage

- **Temperature dependence**

- **Material dependence**

Primary Damage

◆ Two basic defects

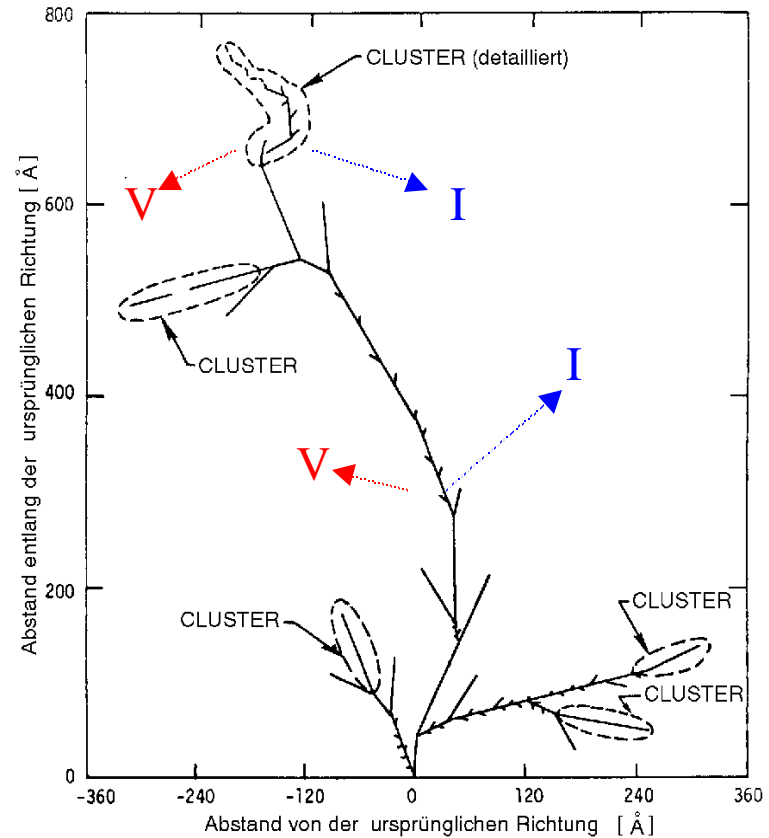
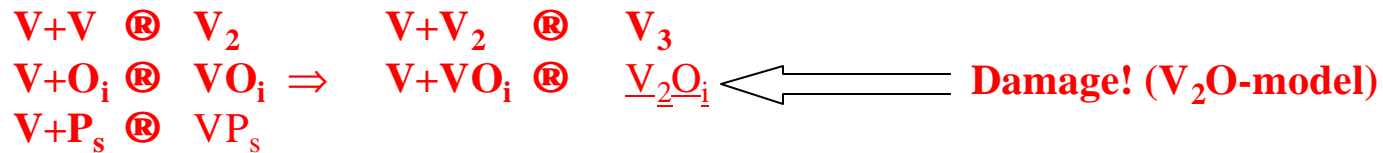
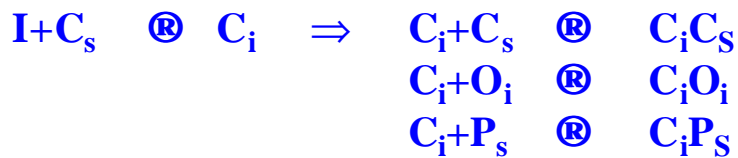
I - Silicon Interstitial V - Vacancy

◆ Primary defect generation

I, higher order I (?)
 V, V₂, higher order V (?)
P CLUSTER (?)

◆ Secondary defect generation

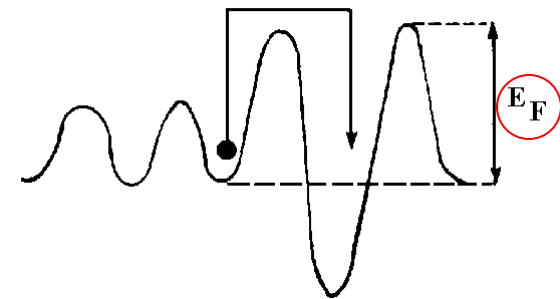
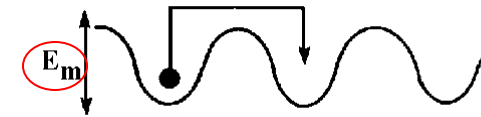
Main impurities in silicon: Carbon (C_s)
 Oxygen (O_i)



Annealing mechanisms

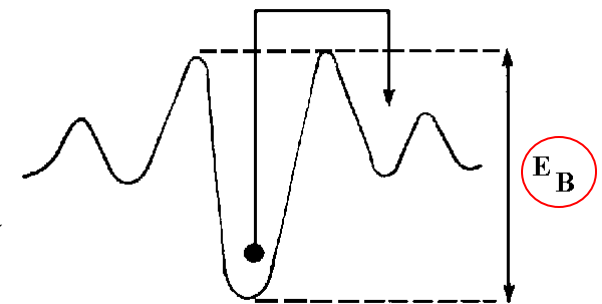
◆ Migration and complex formation

- **Defects** become mobile at certain temperature and **migrate** through the silicon lattice
e.g. Vacancies (V) between 70 and 200 K
(depending on their charge state).
- **Migrating defects are gettered** at sinks, recombine with their counterparts or form new defects (complex) by association with identical or other types of defects
e.g. $V + O_i \rightarrow VO_i$.



◆ Dissociation

- **A complex dissociates** into its components if the lattice vibrational energy is sufficient to overcome the binding energy. At least one of the constituents migrates through the lattice until it forms another defect or disappears into a sink e.g. at $\sim 350^\circ\text{C}$: $VO_i \rightarrow V + O_i$.



⇒ **All mechanisms need to overcome a certain energetic barrier E_A**

E_m, E_F, E_B
= activation energies (E_A)

Rate of Reaction - Example I

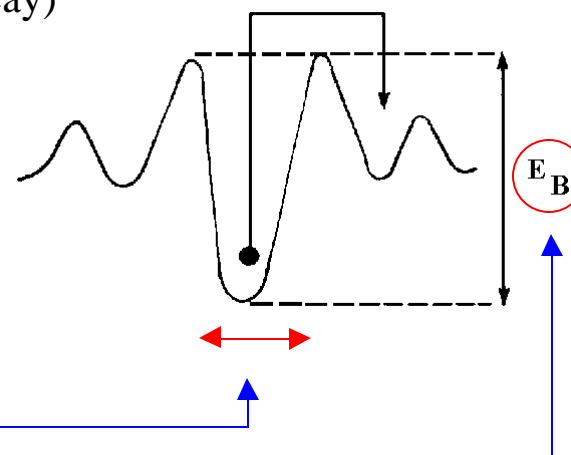
◆ Defect dissociation (e.g. at ~350°C : $\text{VO}_i \rightleftharpoons \text{V} + \text{O}_i$)

- Simple description as **1st order process** (like radioactive decay)

$$\frac{d N_X}{dt} = -k N_X$$

$N_X =$ defect concentration

$k =$ rate constant



- Rate constant k** is given by the **Arrhenius relation**

$$k = k_0 \exp\left(-\frac{E_A}{k_B \cdot T}\right)$$

$k_0 =$ frequency factor

$E_A =$ activation energy

$k_B =$ Boltzmann constant (8.6×10^{-5} eV/K)

- Frequency factor k_0** lies in the order of the most abundant phonon frequency

$$\approx k_B T / h = 2.1 \cdot 10^{10} \times T[\text{K}] \text{ s}^{-1}$$

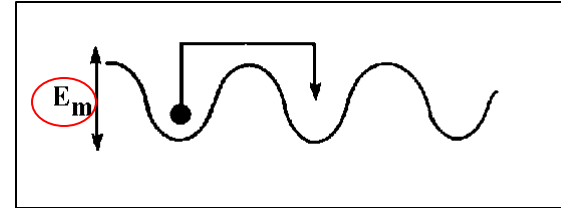
$$\approx 10^{13} \text{ s}^{-1} \text{ (at 300K)}$$

Ref.: [Corbett 1966]

“ attempt-to-escape frequency”

Rate of Reaction - Example II

◆ Diffusion limited processes



- Diffusion limited reaction of two defects X and Y

$$\boxed{-\frac{d N_X}{dt} = -\frac{d N_Y}{dt} = 4\pi R D N_X N_Y}$$

N_X = concentration defect X
 N_Y = concentration defect Y
 D = Diffusivity
 R = capture radius

- Diffusion constant D_0 is given by the Arrhenius relation

$$\boxed{D = D_0 \exp\left(-\frac{E_A}{k_B \cdot T}\right)}$$

D_0 = diffusion constant
 E_A = activation energy
 k_B = Boltzmann constant (8.6×10^{-5} eV/K)

- Special case: $N_X \ll N_Y$ e.g. $V + O_i \rightarrow VO_i$ with $[V] \ll [O_i]$

• similar kinetics as for simple 1st order process (Example I)

$$\boxed{-\frac{d N_X}{dt} = (4\pi R D N_Y) N_X}$$

•

$$\boxed{-\frac{d N_X}{dt} = k N_X}$$

$$4\pi R D_0 N_Y$$

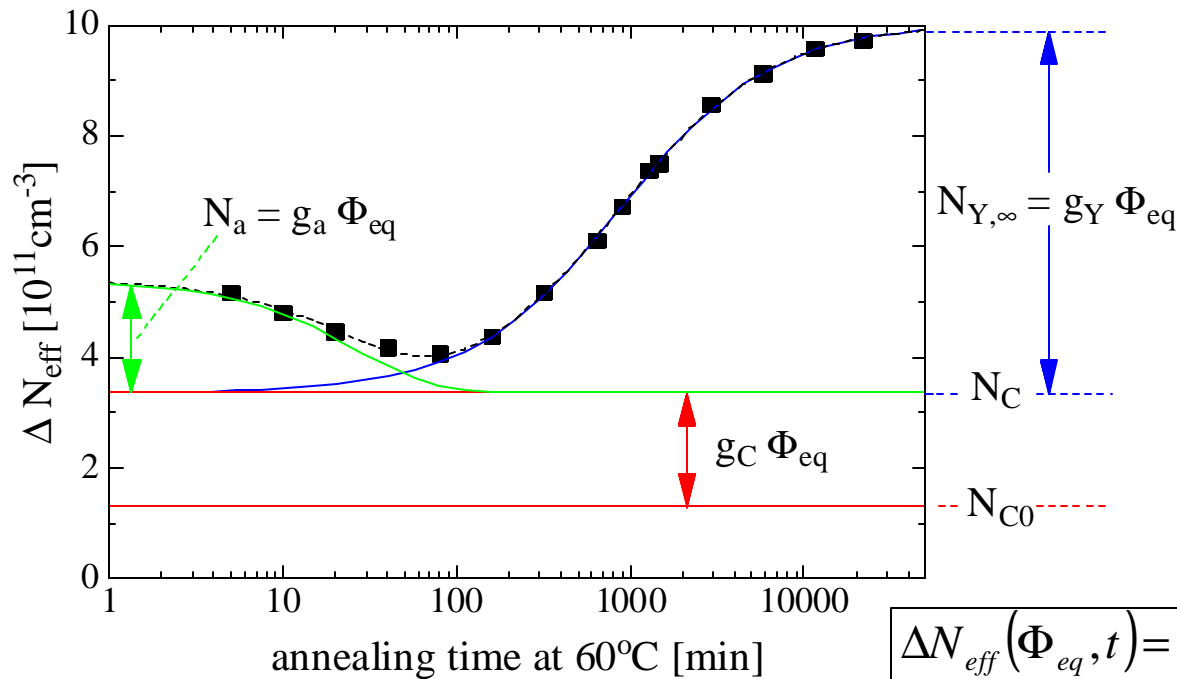
•

$$k_0$$

Annealing behavior of N_{eff} - Hamburg model -

$$\Delta N_{\text{eff}}(\Phi_{\text{eq}}, t) = N_{\text{eff}0} - N_{\text{eff}}(\Phi_{\text{eq}}, t)$$

ΔN_{eff} = Change of N_{eff} with respect to $N_{\text{eff}0}$ (value before irradiation)



long term reverse annealing:

$$N_Y = N_{Y,\infty} \cdot \left(1 - \frac{1}{1+t/t_y} \right)$$

second order parameterization (with $N_{y,\infty} = g_y \times \Phi_{\text{eq}}$). gives best fit

But:

τ_y independent of Φ_{eq}
P underlying defect reaction based on **first order** process!

$$\Delta N_{\text{eff}}(\Phi_{\text{eq}}, t) = \underbrace{N_a(\Phi_{\text{eq}}, t)}_{\text{green}} + \underbrace{N_C(\Phi_{\text{eq}})}_{\text{red}} + \underbrace{N_Y(\Phi_{\text{eq}}, t)}_{\text{blue}}$$

short term annealing:

$$N_a = \Phi_{\text{eq}} \times \sum_i g_{ai} \times \exp\left(-t/t_i\right)$$

first order decay of acceptors introduced proportional to Φ_{eq} during irradiation

stable damage:

$$N_C = N_{C0} \cdot (1 - \exp(-c \cdot \Phi_{\text{eq}})) + g_C \cdot \Phi_{\text{eq}}$$

incomplete „donor removal“
 + introduction of stable acceptors

Short Term Annealing - Temperature dependence

♦ Measurement of $N_a(t)$ at different temperatures

$$N_a(t) \propto \exp\left(-\frac{t}{\tau_a}\right)$$

- Extraction of time constants $\tau_a(T)$

$$\tau_a(T) = \frac{1}{k_{0a}} \cdot \exp\left(\frac{E_{Aa}}{k_B T}\right)$$

⇒

Arrhenius plot

⇒

$$\ln(\tau_a) = -\ln(k_{0a}) + \frac{E_{Aa}}{k_B T}$$

♦ activation energy :

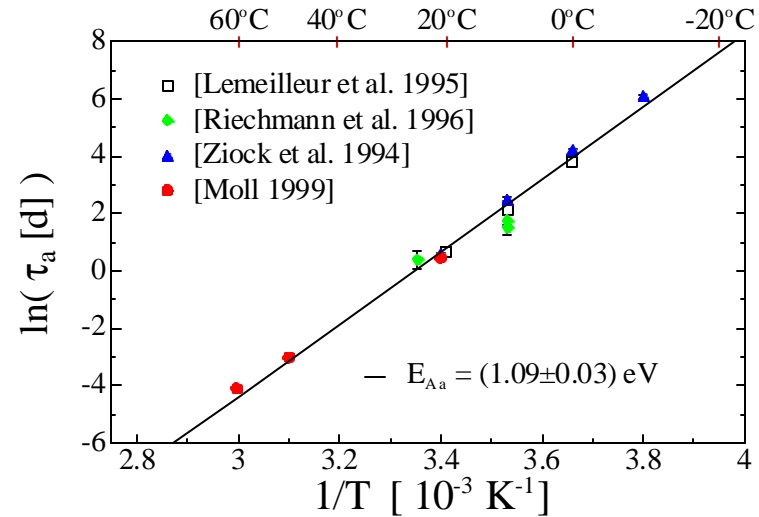
$$E_{Aa} = (1.09 \pm 0.03) \text{ eV}$$

♦ frequency factor :

$$k_{0a} = 2.4 \times 10^{13} \{1.6 \dots 3.6 \times 10^{13}\} \text{ s}^{-1}$$

⇒ interpretation: decay of defects
(k_0 close to most abundant phonon frequency)

⇒ prediction:
time constants for other temperatures



T	-10°C	-7°C	0°C	10°C	20°C	40°C	60°C	80°C
τ_a	306 d	180 d	53 d	10 d	55 h	4 h	19 min	2 min
accel	1/134	1/78	1/23	1/5	1	16	174	1490

Reverse Annealing - Temperature dependence

◆ Measurement of $N_Y(t)$ at different temperatures

$$N_Y(t) \propto \left(1 - \frac{1}{1 + t/t_Y} \right) \quad \mathbf{P} \quad t_Y(T) = \frac{1}{k_{0Y}} \cdot \exp(E_{AY}/k_B T) \quad \mathbf{P} \quad \ln(t_Y) = -\ln(k_{0Y}) + E_{AY}/k_B T$$

◆ activation energy :

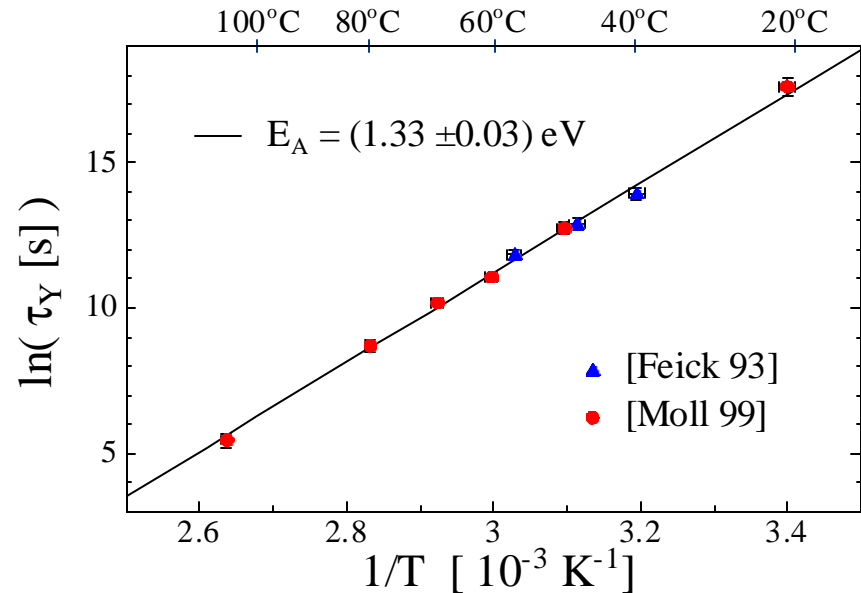
$$E_{Aa} = (1.33 \pm 0.03) \text{ eV}$$

◆ frequency factor :

$$k_{0Y} = 1.5 \times 10^{15} \{ 4 \dots 34 \times 10^{14} \} \text{ s}^{-1}$$

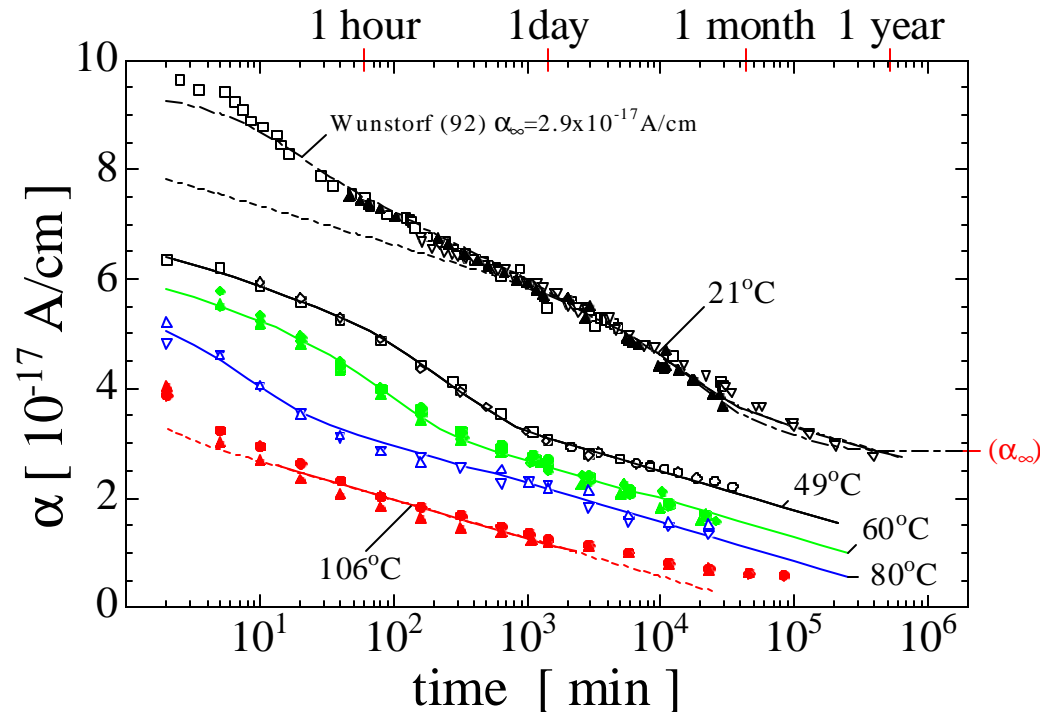
⇒ interpretation: decay of defects
(k_0 close to most abundant phonon frequency)

⇒ prediction:
time constants for other temperatures



T	-10°C	0°C	10°C	20°C	40°C	60°C	80°C
t_Y	516 y	61 y	8 y	475d	17d	1260 min	92 min
accel	1/396	1/47	1/6	1	29	544	7430

Annealing of leakage current



◆ **Annealing at RT (21°C):**

- good agreement with previous parameterization (Wunstorf 92, parameters for non inverted detectors)

◆ **Annealing at higher temperature (long term at RT):**

- new parameterization:
$$a(T, t) = a_1 \cdot \exp(-t/t_1(T)) + (a_0 - b \cdot \ln(q(T) \cdot t/t_0))$$
- **exponential term:** activation energy: $E_A = 1,11\text{eV}$, $\nu = 1.2 \times 10^{13} \text{ s}^{-1}$
correlated with defect at $E_C - 0.46\text{eV}$ (DLTS)
- **logarithmic term:** acceleration factor $\theta(T) \propto \exp(1.3 \text{ eV} / k_B T) \Rightarrow$ **no saturation value ! (no a_∞)**

Leakage Current Annealing - Short term component

♦ Measurement of $\mathbf{a(t)}$ at different temperatures

$$\mathbf{a(T, t) = a_1 \cdot \exp(-t / \mathbf{t_1(T)}) + (a_0 - b \cdot \ln(q(T) \cdot t / t_0))}$$

- Extraction of time constants $\tau_1(T)$ \Rightarrow

$$\mathbf{t_1(T) = 1/k_{0I} \cdot \exp(E_I/k_B T)}$$

Arrhenius plot

$$\ln(\mathbf{t_1}) = -\ln(k_{0I}) + E_I/k_B T$$

♦ activation energy :

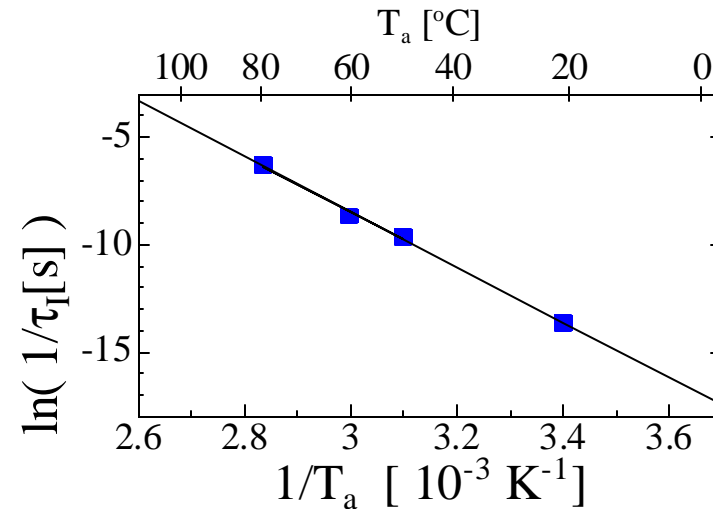
$$E_I = (1.11 \pm 0.05) \text{ eV}$$

♦ frequency factor :

$$k_{0a} = 1.2 \times 10^{13} \{ 2 \dots 63 \times 10^{12} \} \text{ s}^{-1}$$

\Rightarrow interpretation: decay of defects
(k_0 close to most abundant phonon frequency)

\Rightarrow prediction:
time constants for other temperatures



T	-10°C	-7°C	0°C	10°C	20°C	40°C	60°C	80°C
$\mathbf{t_1}$	1748 d	1007 d	291 d	55 d	12 d	17 h	86 min	10 min
accel	1/150	1/86	1/25	1/5	1	17	196	1746

Leakage Current Annealing - Logarithmic component

◆ **Measurement of $\mathbf{a(t)}$ at different temperatures**

$$\mathbf{a(T, t) = a_1 \cdot \exp(-t/t_I(T)) + (a_0 - b \cdot \ln(q(T) \cdot t/t_0))}$$

- Time and temperature dependence can be parameterized.
However, physical mechanism of annealing is not understood !!!
- Parameterization under the assumption that activation of underlying mechanism can also be described by an Arrhenius relation:

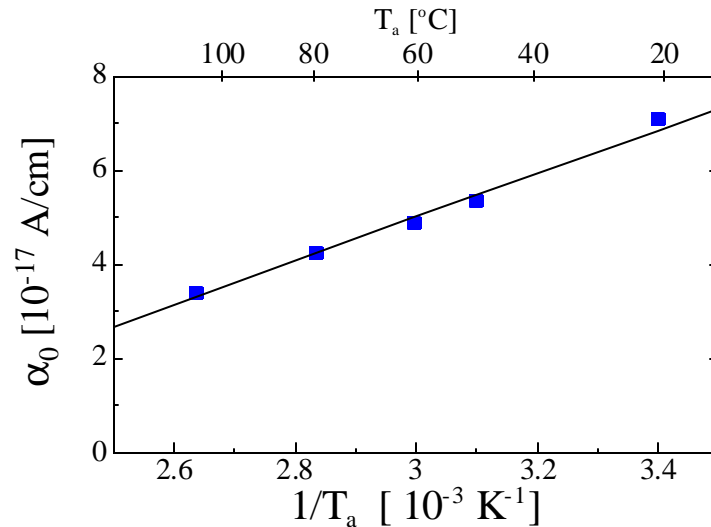
$$\Theta(T_a) = \exp\left(\frac{E_I^*}{k_B} \left(\frac{1}{T_a} - \frac{1}{T_{ref}}\right)\right)$$

◆ **activation energy :**

$$E_I = (1.30 \pm 0.14) \text{ eV}$$

⇒ **prediction:**

scaling with temperature relative to reference temperature of 20°C



T	-10°C	-7°C	0°C	10°C	20°C	40°C	60°C	80°C
accel	1/353	1/185	1/43	1/6	1	27	482	6268

Comparison

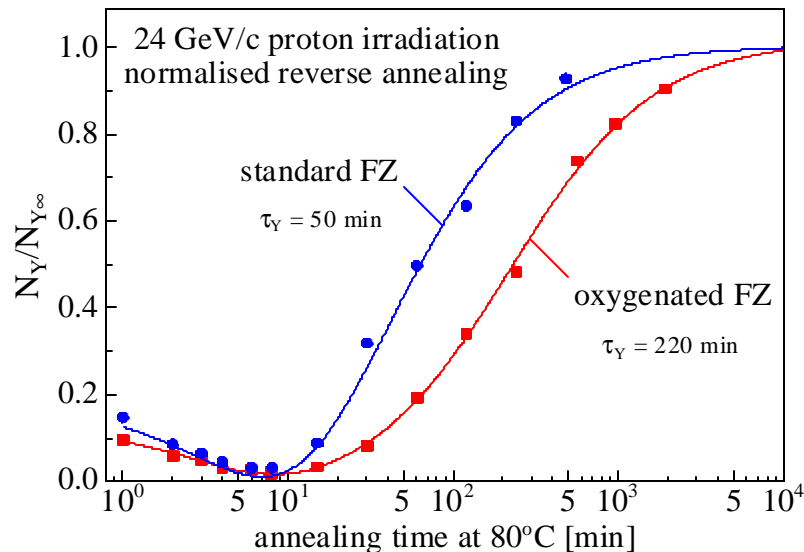
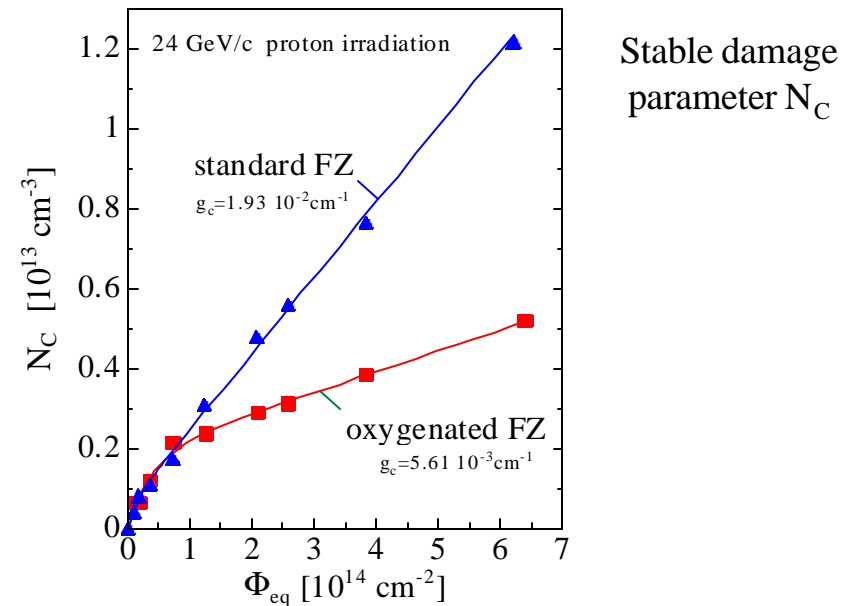
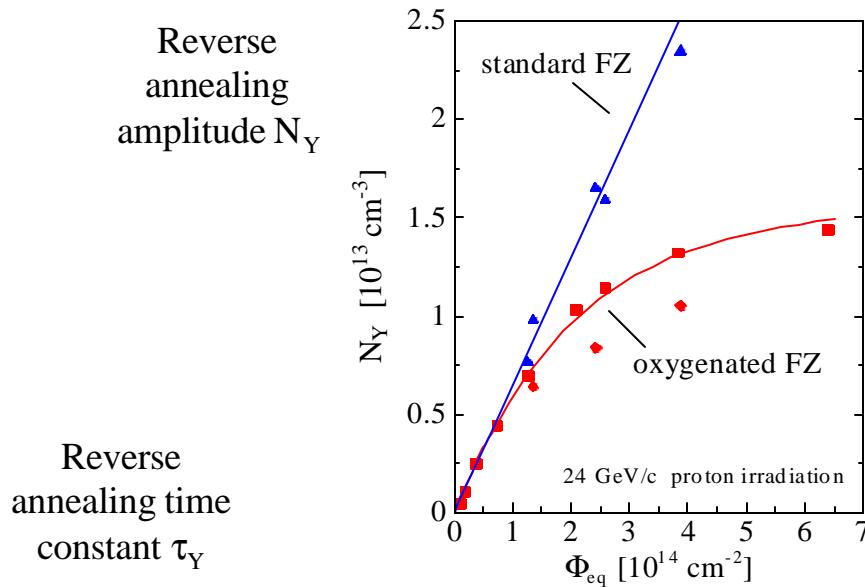
- Example: Annealing at 20°C and 60°C -

Damage component	time constant at 20°C	time constant at 60°C	Acceleration between 20°C and 60°C
N_{eff} : Beneficial annealing (N_a)	55 h	19 min	174
N_{eff} : Reverse annealing (N_v)	475 d	1260 min	544
Leakage current: exponential term	12 d	86 min	196
Leakage current: logarithmic term	-	-	482

- ◆ Annealing of different damage components arise from different microscopic processes.
⇒ It is not straightforward to compare data achieved after different annealing procedures !!
- ◆ Example: Statements like: “annealing into the minimum of the N_{eff} -annealing curve” have different meanings for different annealing temperatures
⇒ Standardized annealing procedures needed in order to compare data within HEP-community

Material dependence of annealing processes

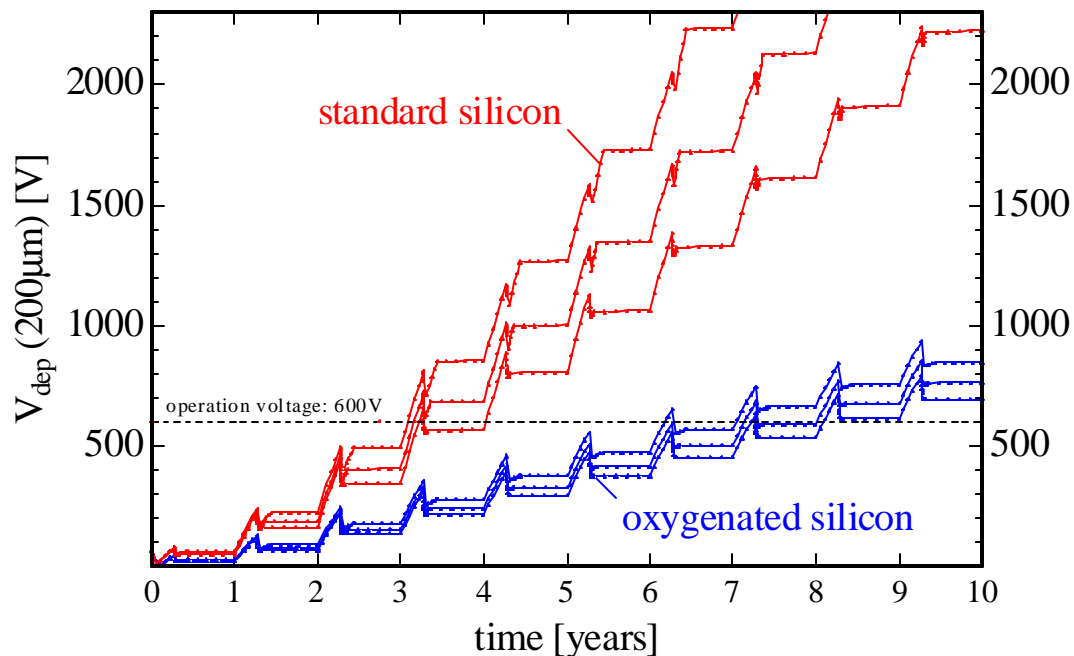
- **oxygenated** / **standard silicon** -



- ◆ **24 GeV/c proton irradiated**
- ◆ **O-enriched diodes**
- ◆ **g_c improved by a factor of 3**
- ◆ **saturation of reverse annealing**
- ◆ **delayed reverse annealing**

Damage Projection - ATLAS Pixel Detector - B-Layer (4cm)

- ◆ **Radiation level:**
 - $F_{\text{eq}}(\text{year}) = 3.5 \cdot 10^{14} \text{ cm}^{-2}$ (full luminosity)
> 85% charged hadrons
- ◆ **Three scenario:**
 - 1 year = 100 days beam (-7°C)
 - (1) 3 days 20°C and 14 days 17°C
 - (2) 30 days 20°C
 - (3) 60 days 20°C
 - Rest of the year: no beam (-7°C)



Conclusions

◆ Material dependence of damage effects

- **Leakage current** damage parameter and its annealing is material independent (no impurity, resistivity or conduction type dependence)
- Effective doping changes can be improved by oxygenation of the material (**factor 3 for stable damage parameter g_c**). Such improvement is only observed when the radiation environment contains a significant **charged particle component**.
- **Lower resistivity material is beneficial** for detectors that operate in a radiation environment dominated by reactor energy **neutrons**.
- **Reverse annealing saturates** at high fluences ($2 \times 10^{14} \text{p/cm}^2$) for oxygen enriched silicon. **Time constant larger by a factor of 2-4** allowing detectors to remain at room temperature for longer periods during maintenance periods: **additional safety margin**
- After proton irradiation a broad **variation** with respect to the radiation hardness of “**standard silicon**” has been observed while oxygenated silicon showed reproducible results.
⇒ **All materials have to be tested** ⇒ Best with sources that represent the LHC environment (at least p and n)

◆ Annealing of Radiation Damage

- Different damage parameters show different annealing behavior (activation energies, frequency factors)
⇒ It is not possible to scale all parameters between different annealing temperatures with only one factor !!
⇒ Systematic studies are needed (for each material) to scale the damage in temperature/time
⇒ In order to compare data within the HEP-community standardized annealing procedures are needed

◆ Damage Projections

- Uncertainties if the damage parameters were not achieved on exactly the same material the damage projection is made for
- NIEL-Hypothesis used in order to predict the damage (1 MeV neutron equivalent damage)
⇒ not tested for all particles / particle energies !

Workshop - For Discussion

Is there a way / interest to introduce standardized radiation tests

(particles, particle energies, annealing procedures, test structures, measurement procedures)

in order to compare results within the HEP-Community ?

Workshop - Discussion

- ◆ **How to implement Quality-Assurance/Control in HEP-experiments?**

- ◆ **Can we standardize (recommend)**
 - measurement procedures ?
 - test structures ?
 - irradiation procedures ?
 - annealing procedures ?

- ◆ **Future activities:**
 - Will we have a further workshop ?
 - Common activities ?

- ◆ **- and other topics.....**