Update on irradiation experiments with electrons of different kinetic energies (between 1.5 MeV and 27 MeV) performed on n-type silicon

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Outline

- Motivation&Goals&Strategy
- Experimental results:
  - electrical characterization
  - HRTEM and EPR investigations
- Summary and Future plans

Motivation: *Bridge the gap between the defect analyses and device performances as a crucial step for further device developments*

Goals: *Identify the chemical structure of the defects causing the change in the detector performance at the operating temperature and find possible ways of improving the radiation hardness of Si sensors*

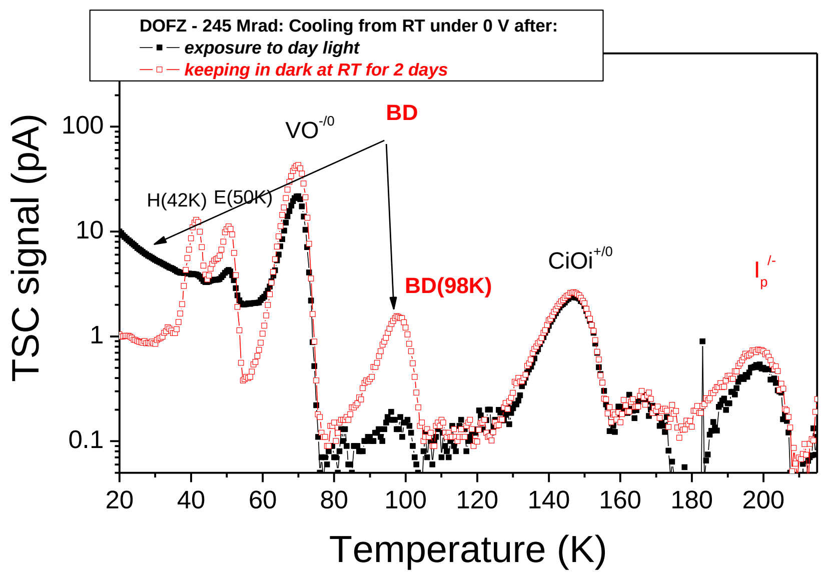
Strategy: *Irradiation experiments with electrons of different kinetic energies (between 1.5 MeV and 27 MeV), correlated studies of electrical characterization, EPR and HRTEM investigations* *(research started in 2012 with a romanian national funded project PNII-ID-PCE-2011-3 Nr. 72/5.10.2011)*

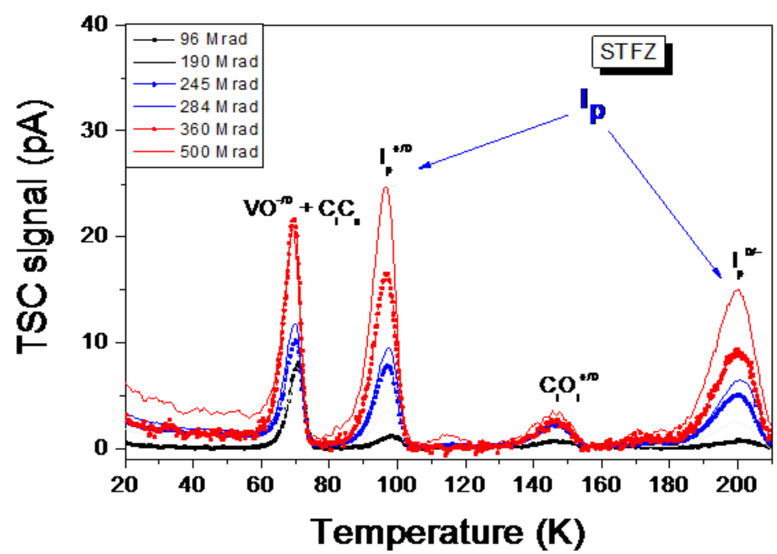
Defects induced by irradiations (forming and transforming at ambient temperatures)

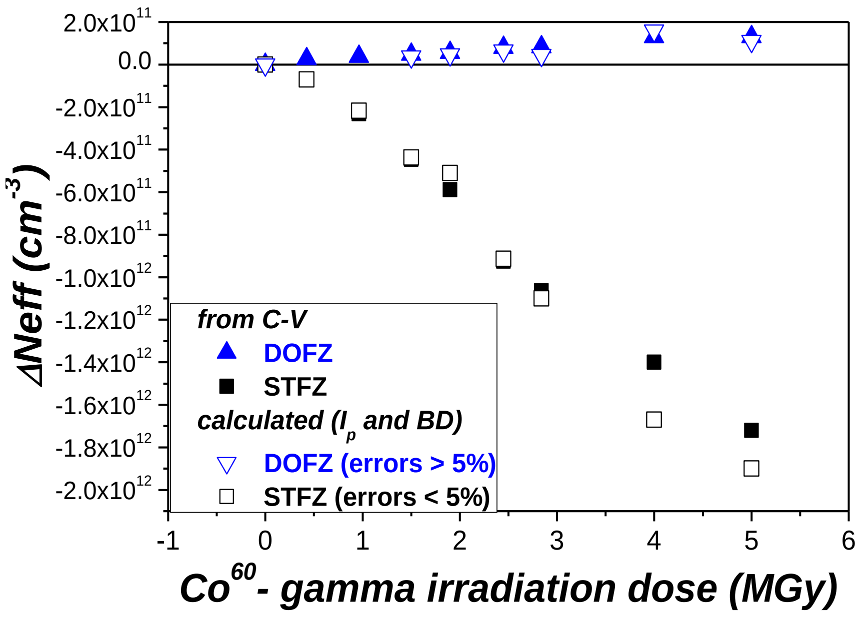
Defects	$\sigma_{n,p}$ [cm^2]	E_A [eV]	Assignment/References	Impact on electrical characteristics at RT
E(30K)	σ_n= 2.3 x 10^{-14}	E_C - 0.1	Electron trap with a donor level in the upper half of the Si bandgap /[Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52]	On the N_{eff} by introducing positive space charge *-It makes the difference between proton and neutron irradiations* *-More generated in O rich material*
$BD_A^{0/++}$ $BD_B^{+/++}$	σ_n= 2.3 x 10^{-14} σ_n= 2.7 x 10^{-12}	E_C - 0.225 E_C - 0.15	**Bistable Thermal double donor TDD2** (two configurations A and/or B) - Electron trap with a donor level in the upper half of the Si bandgap/ [Appl. Phys. Lett. 50 (21) (1987) 1500; Nucl. Instr. and Meth. in Phys. Res. A 514 (2003) 18; Nucl. Instr. and Meth. in Phys. Res. A 556 (2006) 197; Nucl. Instr. and Meth. in Phys. Res. A 583 (2007) 58]	On the N_{eff} by introducing positive space charge *-Strongly generated in O rich material*
$I_p^{+/0}$ $I_p^{0/-}$	σ_p= (0.5-9) x10^{-15} σ_n=1.7 x10^{-15} σ_p= 9 x 10^{-14}	E_V + 0.23 E_C - 0.55	Donor level of V_2O or of a still unkown C related defect / [Appl. Phys. Lett. 81 (2002) 165; Appl. Phys. Lett. 83, 3216 (2003); Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52] Acceptor level of V_2O or of a still unkown C related defect/[Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52, Appl. Phys. Lett. 81 (2002) 165]	On the N_{eff} by introducing negative space charge and on LC *-Strongly generated in O lean material*
E_4 E_5	σ_n=1 x 10^{-15} σ_n=7.8 x 10^{-15}	E_C -0.38 E_C -0.46	Acceptor in the upper part of the gap associated with the double charged and single charged states of V_3, respectively (**$V_3^{=/-}$ and $V_3^{-/0}$**) / [J. Appl. Phys. 111 (2012) 023715.]	On LC
H(116K)	σ_p=4 x 10^{-14}	E_V + 0.33	Hole trap with an acceptor level in the lower part of the Si bandgap - Extended defect (cluster of vacancies and/or interstitials) / [Appl. Phys. Lett. 92 (2008) 024101, Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52-68]	On the N_{eff} by introducing negative space charge
H(140K)	σ_p=2.5 x 10^{-15}	E_V + 0.36	Hole trap with an acceptor level in the lower part of the Si bandgap - Extended defects (clusters of vacancies and/or interstitials)/[Appl. Phys. Lett. 92 (2008) 024101, Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52-68]	On the N_{eff} by introducing negative space charge
H(152K)	σ_p=2.3 x 10^{-14}	E_V + 0.42	Hole trap with an acceptor level in the lower part of the Si bandgap - Extended defects (clusters of vacancies and/or interstitials)/[Appl. Phys. Lett. 92 (2008) 024101, Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52-68]	On the N_{eff} by introducing negative space charge
$VO_i^{-/0}$	σ_n= 1.44 x 10^{-14}	E_C -0.176	**$VO_i^{-/0}$** /[J. Appl.Phys.79(1996)3906 ; Mat. Sci. in Semic. Proc. 3 (2000) 227]	
$C_iC_s^{-/0}$	σ_n= 1.4 x 10^{-14}	E_C - 0.171	**$C_iC_s^{A\ -/0}$** /[Phys. Rev. Lett. 60 (1988) 460-463, Phys. Rev. B42 (1990) 5765]	
H(40K)	σ_p= 1.7 x 10^{-15}	E_V + 0.09	Hole trap/ [Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52-68]	
$C_i^{+/0}$	σ_p =4.3 x 10^{-15}	E_V + 0.284	**$C_i^{+/0}$**/ [M. Moll, PhD Thesis, University of Hamburg, DESY-THESIS-1999-040, 1999]	
$C_iO_i^{+/0}$	σ_p =4.3 x 10^{-15}		[J.Appl.Phys.79(1996)3906]	
$V_2^{-/0}$	σ_n=2.1 x 10^{-15}	E_C - 0.424	**$V_2^{-/0}$** / [J.Appl.Phys.79(1996)3906; M. Moll, PhD Thesis, DESY-THESIS-1999-040, 1999]	
H(87K)	σ_p=0.3 x 10^{-15}	E_V + 0.193	**$V_3^{0/+}$** /[Phys. Status Solidi A 208 (2011) 568.]	

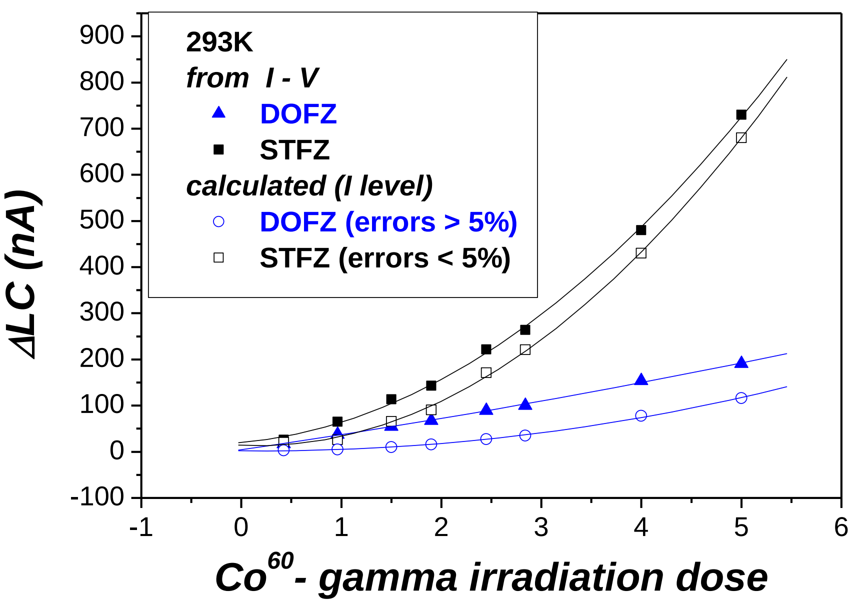
❑ Point defects – after irradiation with Co^{60} – gamma or low energy electrons

Defects	$\sigma_{n,p}$ [cm^2]	E_A [eV]	Assignment/References	Impact on electrical characteristics at RT
$BD_A^{0/++}$ $BD_B^{+/++}$	$\sigma_n = 2.3 \times 10^{-14}$ $\sigma_n = 2.7 \times 10^{-12}$	E_C - 0.225 E_C - 0.15	Bistable Thermal double donor TDD2 (two configurations A and/or B) - Electron trap with a donor level in the upper half of the Si bandgap/ [Appl. Phys. Lett. 50 (21) (1987) 1500; Nucl. Instr. and Meth. in Phys. Res. A 514 (2003) 18; Nucl. Instr. and Meth. in Phys. Res. A 556 (2006) 197; Nucl. Instr. and Meth. in Phys. Res. A 583 (2007) 58]	On the N_{eff} by introducing positive space charge *-Strongly generated in O rich material*
$I_p^{+/0}$ $I_p^{0/-}$	$\sigma_p = (0.5\text{-}9) \times 10^{-15}$ $\sigma_n = 1.7 \times 10^{-15}$ $\sigma_p = 9 \times 10^{-14}$	E_V + 0.23 E_C - 0.55	Donor level of V_2O or of a still unkown C related defect / [Appl. Phys. Lett. 81 (2002) 165; Appl. Phys. Lett. 83, 3216 (2003); Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52] Acceptor level of V_2O or of a still unkown C related defect/[Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52, Appl. Phys. Lett. 81 (2002) 165]	On the N_{eff} by introducing negative space charge and on LC *-Strongly generated in O lean material*





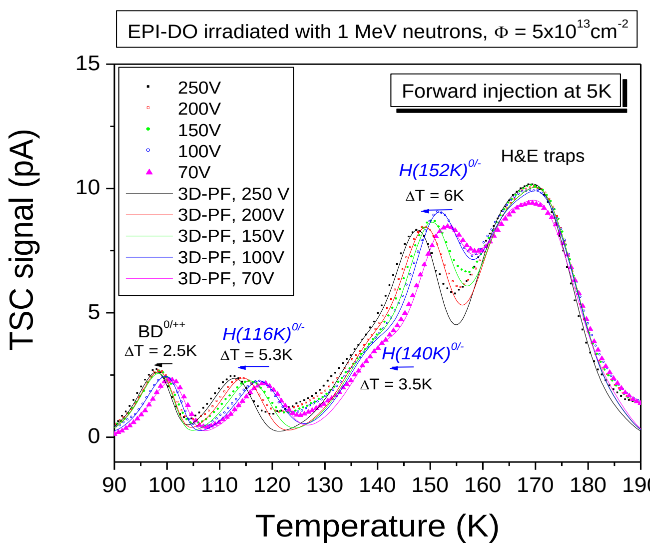


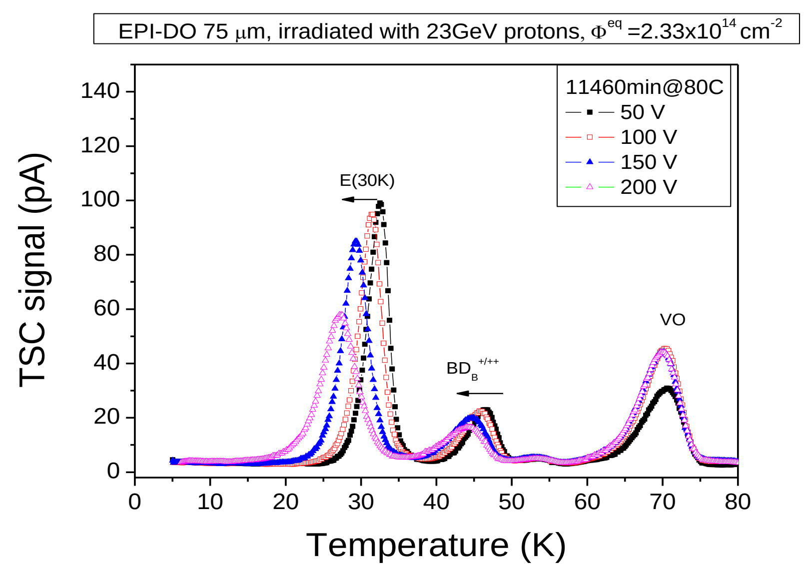


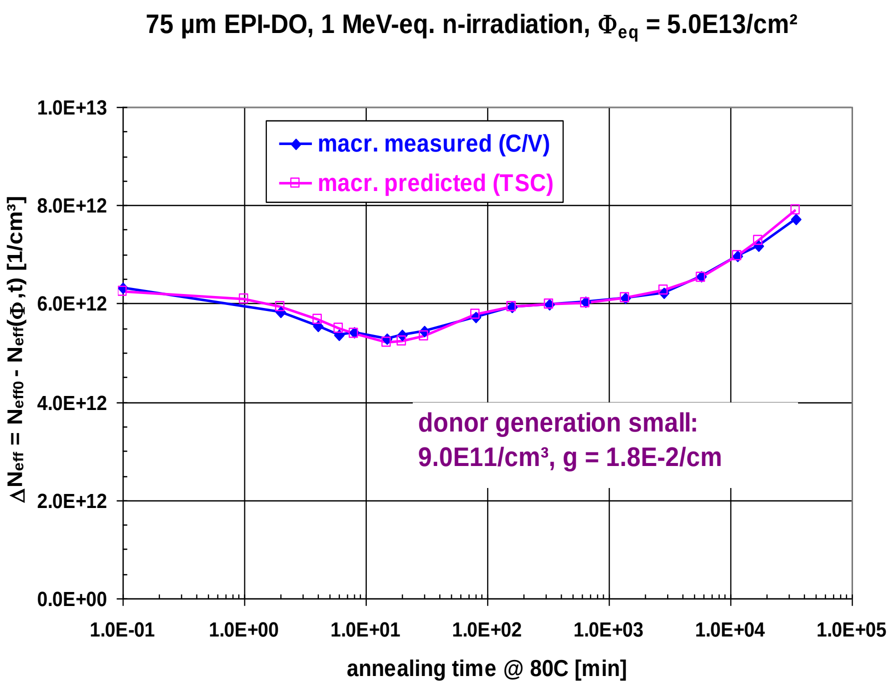
change of N_{eff} and leakage current well described by accounting only the BD (TDD2) and I_p (unknown chemical structure) defects

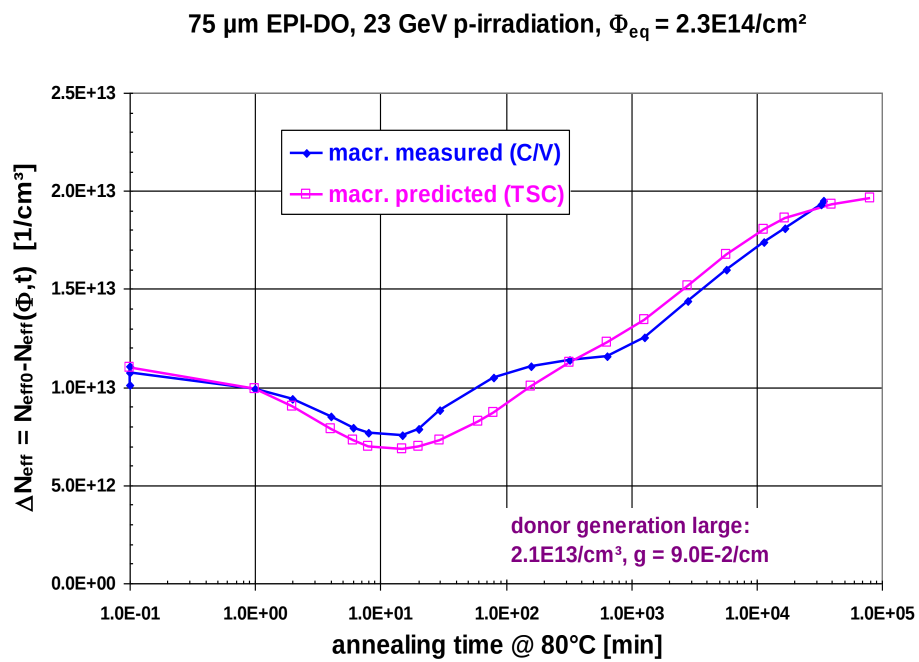
❑ Extended Defects – after hadron irradiation and high energy electrons

Defects	$\sigma_{n,p}$ [cm^2]	E_A [eV]	Assignment/References	Impact on electrical characteristics at RT
E(30K)	σ_n= 2.3 x 10^{-14}	E_C - 0.1	Electron trap with a donor level in the upper half of the Si bandgap /[Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52]	On the N_{eff} by introducing positive space charge *-It makes the difference between proton and neutron irradiations* *-More generated in O rich material*
E_4 E_5	σ_n=1 x 10^{-15} σ_n=7.8 x 10^{-15}	E_C -0.38 E_C -0.46	Acceptor double charged and single charged states of V_3, respectively ($V_3^{=/-}$ and $V_3^{-/0}$) / [J. Appl. Phys. 111 (2012) 023715]	On LC
H(116K)	σ_p=4 x 10^{-14}	E_V + 0.33	Hole traps with an acceptor level in the lower part of the Si bandgap - Extended defects (cluster of vacancies and/or interstitials) / [Appl. Phys. Lett. 92 (2008) 024101, Nucl. Instr. and Meth. in Phys. Res. A 611 (2009) 52]	On the N_{eff} by introducing negative space charge
H(140K)	σ_p=2.5 x 10^{-15}	E_V + 0.36		
H(152K)	σ_p=2.3 x 10^{-14}	E_V + 0.42		







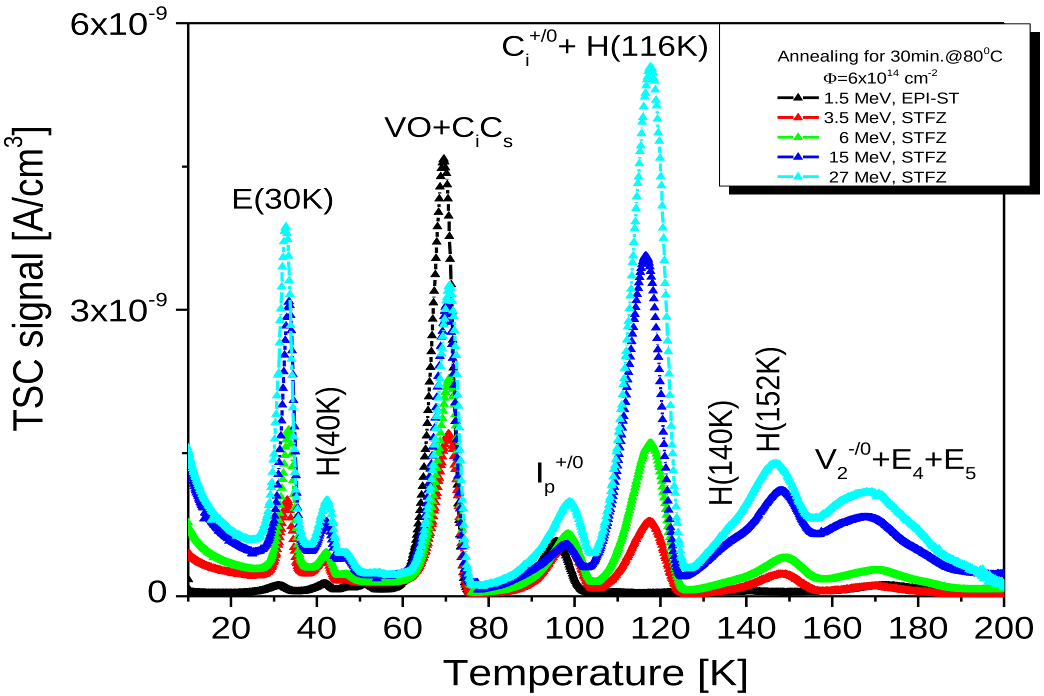


E_i^{30K} - enhanced generation after irradiation with charged hadrons

change of N_{eff} , with the irradiation fluence and annealing time, well described by accounting only the E(30K), H(116K), H(140K) and H (152K) (unknown chemical structure) defects

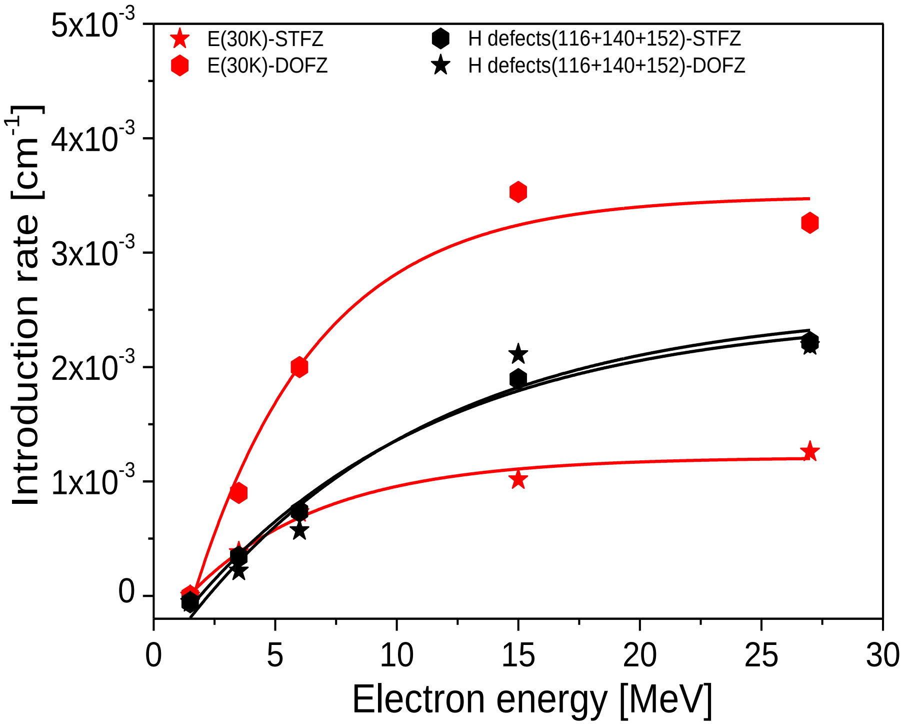
Experimental results:

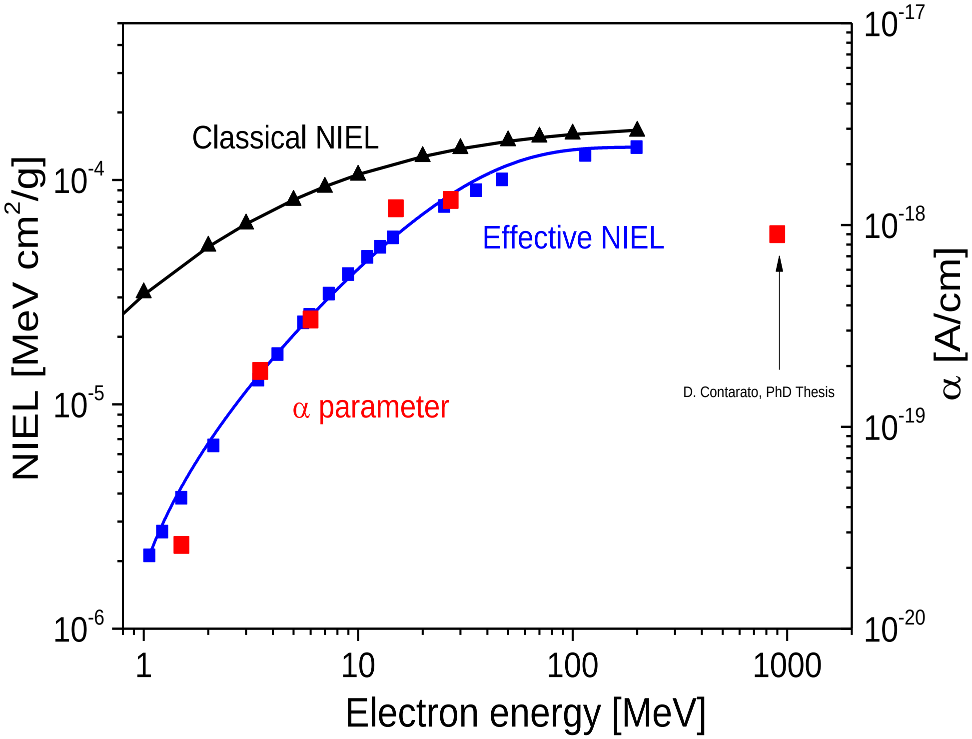
- electrical characterization



Defects	Assignment	Impact on the electrical characteristics of diodes
E(30K)	**Electron trap with a donor level in the upper half of the Si bandgap,**	**On the N_{eff} by introducing positive space charge**
I_p	**Defect of amphoteric nature (with both donor and acceptor levels), introduction rate almost independent of electrons energy**	**On the N_{eff} by introducing negative space charge and on LC**
H(116K)	**Hole trap with an acceptor level in the lower part of the Si bandgap - Extended defect (cluster of vacancies and/or interstitials),), introduction rate increases with electrons energy**	**On the N_{eff} by introducing negative space charge**
H(140K)		
H(152K)		

Introduction rates vs. electron energy



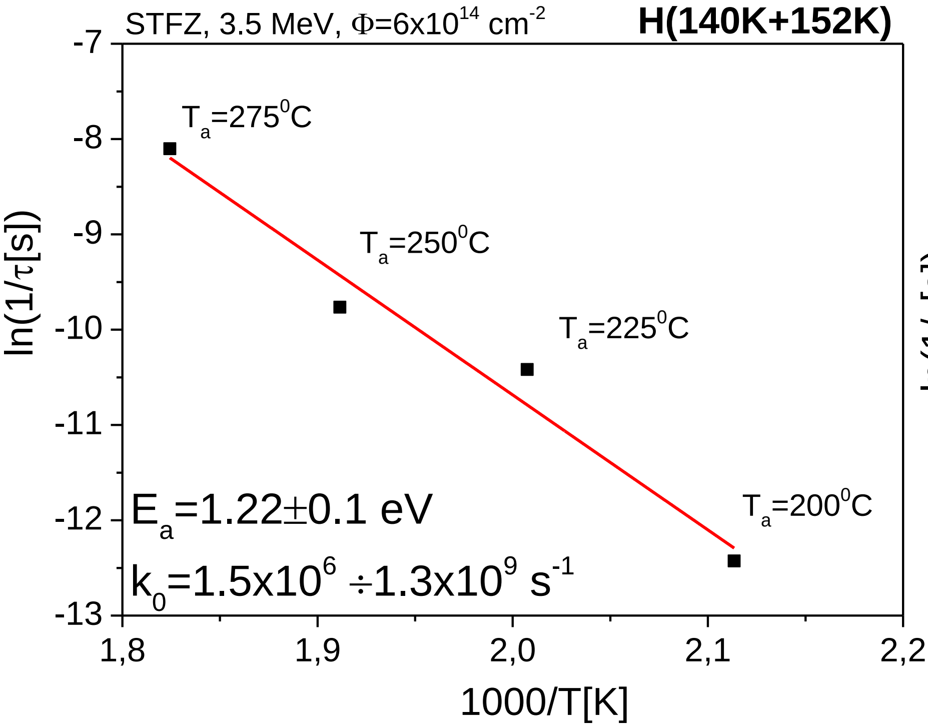


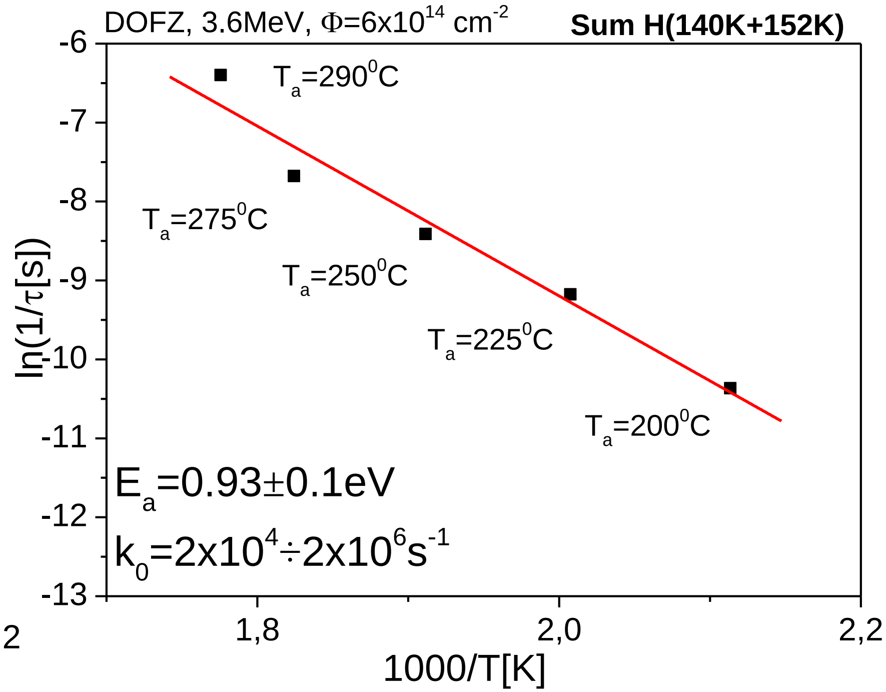
Introduction rates vs. the electron energy show a saturation tendency- expected from NIEL

Introduction rates for H defects for DOFZ & STFZ are similar → no [O] dependent, to be identified via HRTEM investigations

Introduction rate for E (30K) is 3 times larger in DOFZ material → [O] dependent, to be identified via EPR studies

Activation energy and frequency factor in STFZ and DOFZ

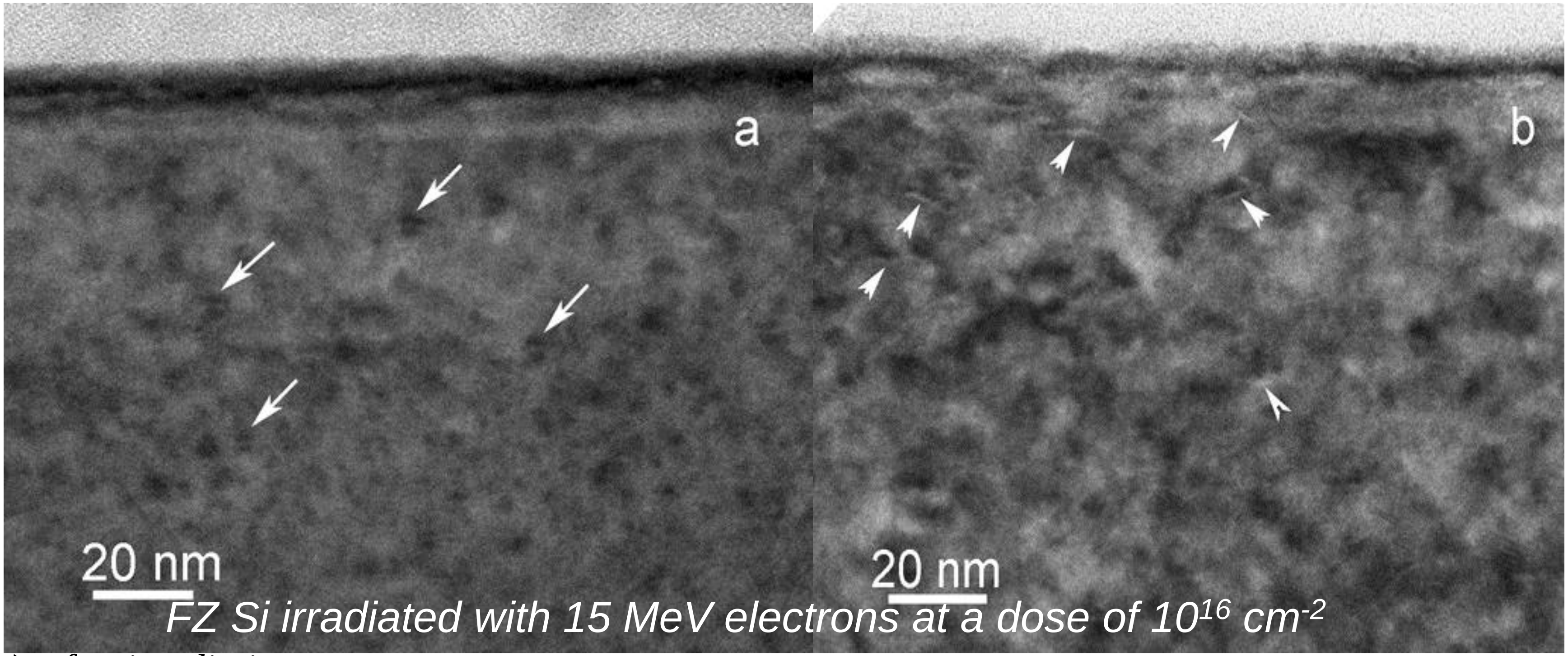




τ for H(140K+152K):	τ for STFZ [min]	τ for DOFZ [min]
@200°C	4160	529
@225°C	558	161
@250°C	290	75
@275°C	55	36
@290°C	-	10

Annealing out of H(140K+152K) defects strongly depends on O concentration !!!

Experimental results:- HRTEM investigations for identifying the structure of H type defects

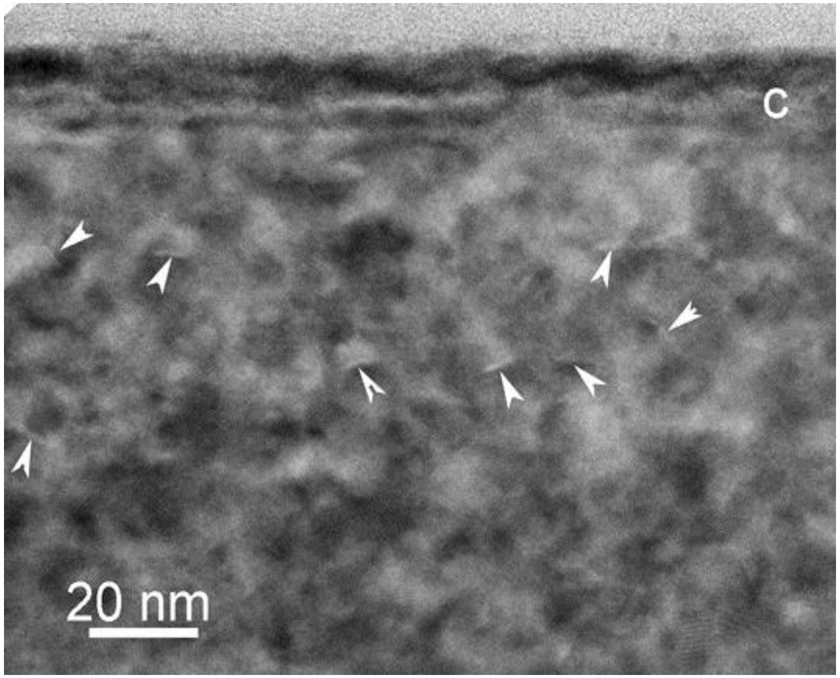


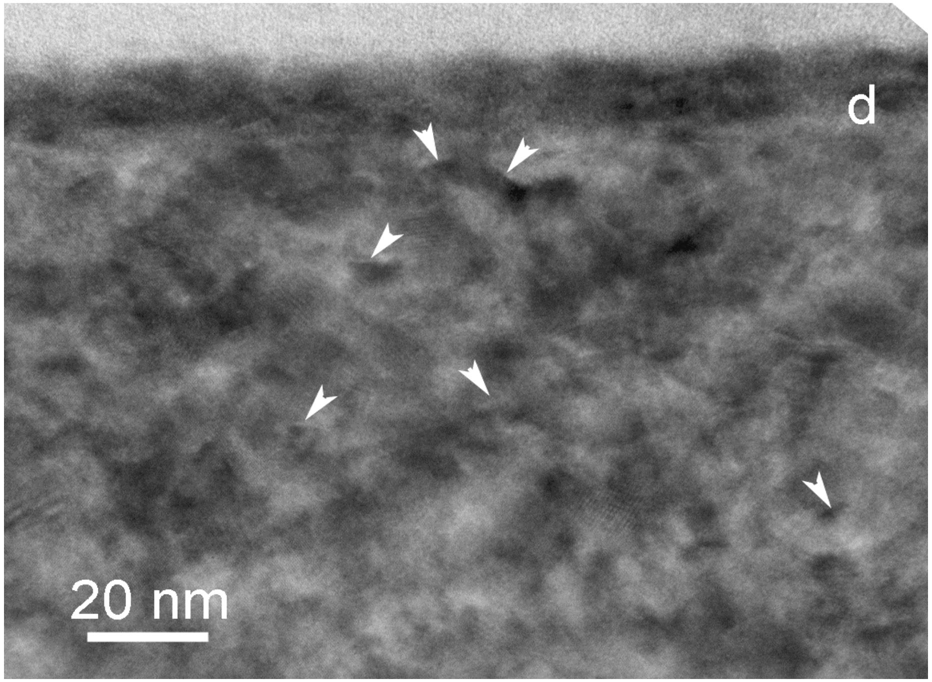
FZ Si irradiated with 15 MeV electrons at a dose of 10^{16} cm^{-2}

(a) *after irradiation*.

- Black contrast dots indicate the presence of clusters of point defects (generally vacancy clusters) which can form platelets revealed by the characteristic "coffee bean" contrast (arrows).
- Interstitial clusters are formed close to an impurity atom, for instance oxygen [R.C. Newman, Defects in silicon, Rep. Progr. Phys. 45, 1163-1210 (1982)]. The size of the extended defects is **smaller than 3 nm**.

(b) after irradiation and annealing at 80 °C for 73380 min.: By annealing, the clusters of point defects agglomerate forming larger extended defects (arrow heads). Their dimensions are generally **in the range 5 -7 nm**.





(c) after irradiation and annealing at 80 °C for 73380 min.+ 30min.@200 C:

• The annealing effect is most prominent revealing the highest concentration of extended defects.

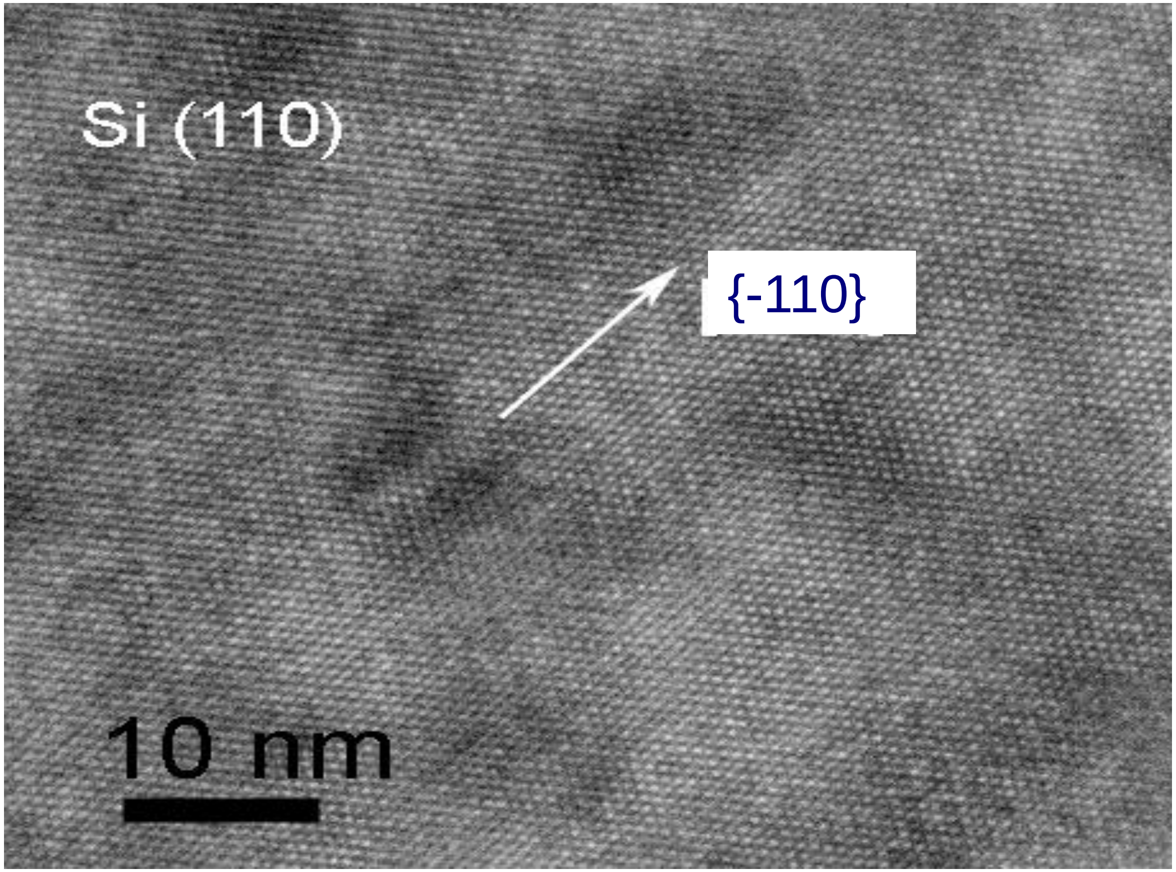
• The dimensions of extended defects remain in the range **5 -7 nm after the treatment at 200^0 C**

d) *after irradiation and annealing at 80 °C for 73380 min.+ 30min.@200 C + 60min.@275 C:*

•The further annealing at 275 °C for 60 min. produces an apparent decrease in concentration of the extended defects

• some very large defects (~30 nm long) seldom start to occur.

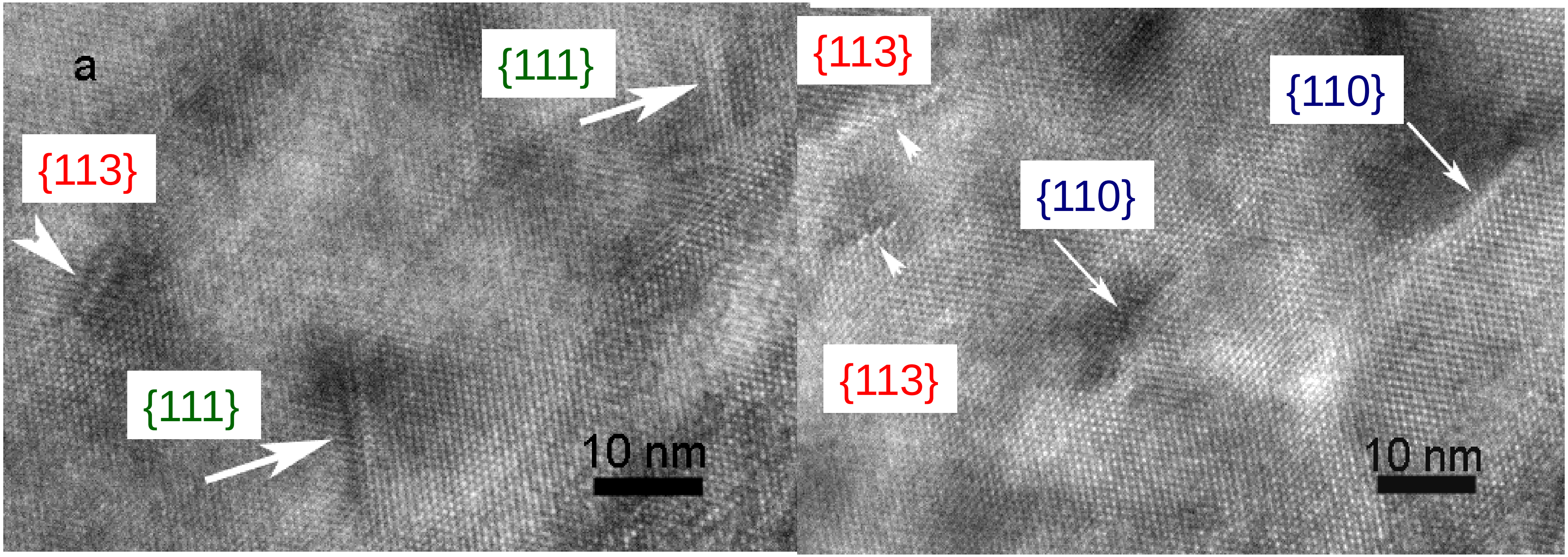
HRTEM images at higher magnifications (more information about the nature and structure of the extended defects) – *soon after irradiation with 15 MeV electrons*



***HRTEM images** along the [110] zone axis showing the most common types of extended defects*

- The plate-like defects ("coffee beans") are mainly oriented in the {-110} direction and they are the precursors for other extended defects formed during annealing.
- The defect clusters does not look amorphous. The Si lattice is disordered by the presence of defects but the crystalline lattice is not destroyed.

HRTEM images – after annealing at 80 C for 73380 min.

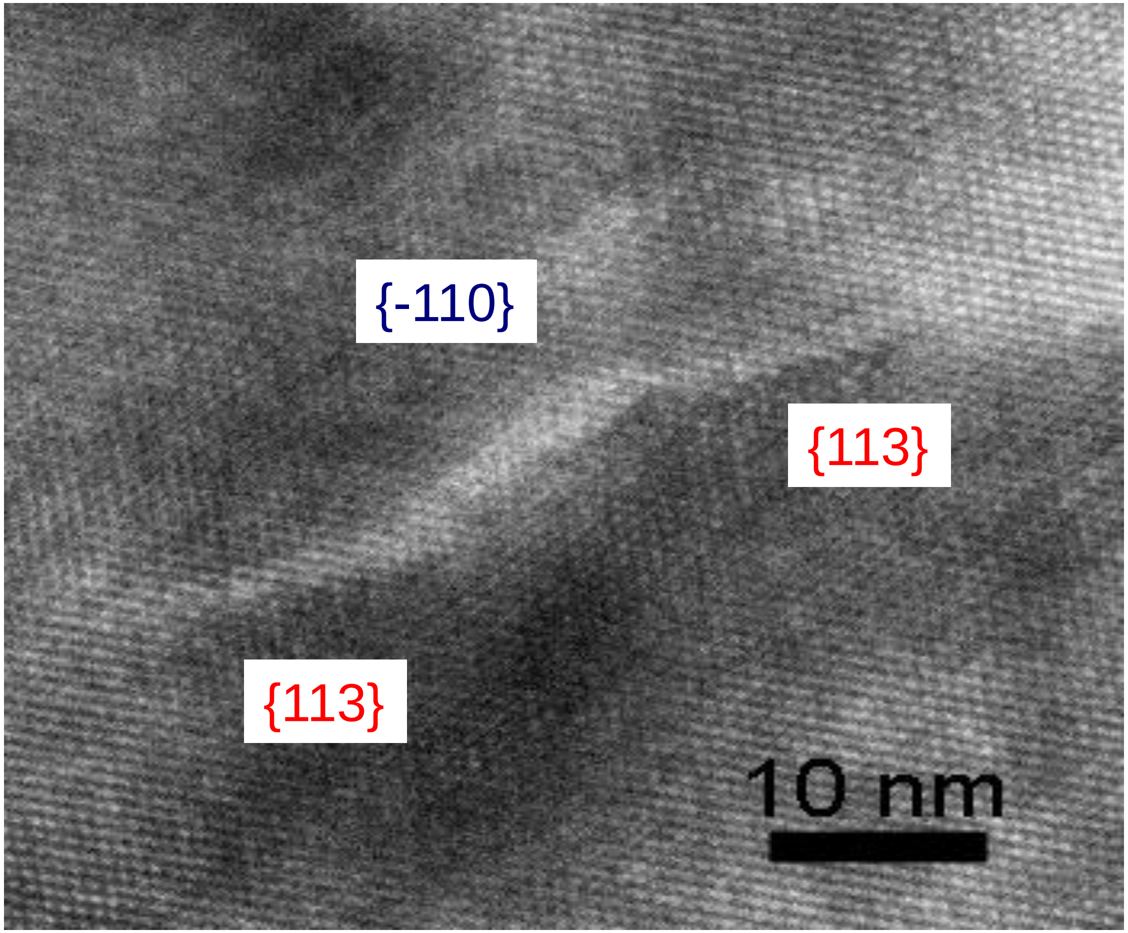


Two types of defects start to form:

- ***{111} type planar defects-*** are intrinsic partial Frank dislocation loop formed by the ***aggregation of vacancies*** [L Fedina et al. Phys. Stat. Sol. (a) 171, 147 (1999)]

- ***{113} type defect - planar defects*** - formed by ***agglomeration of interstials*** [S Takeda, T. Kamino Phys Rev. B 51 2148 (1995)] and has been the most often type of extended observed in the annealed sample. The defect size did not vary after a further annealing at 200^0 C. (the ***{110} defects are the precursors*** of the ***{113} defect***. [S. Takeda, Phys Rev. B 51 2148 (1995)])

HRTEM images – *after: 73380 min.@80 ^{0}C + 30 min.@200^{0}C + 60min.@275^{0}C*



HRTEM image along the [110] zone axis showing a large extended defect formed in the Si sample irradiated with 15 MeV electrons after last annealing step at 275^{0} C

The large extended defect revealed in the figure is of interstitial type. It is formed from three segments. The outer two segments are of {113} -type defects closed in the middle by an {110} type defect. The total extension of the defect is ~33 nm. Note that such large extended defects have been seldom observed.

EPR investigations on Si-FZ100 samples doped with ^{17}O or ^{13}C (June-Nov. 2013 data) for identifying the I_p and E(30K) defects

Investigated samples:

Set 1:

- Si-FZ100 with ^{16}O (10^{16}cm^{-3}) , irradiated with e^- (15MeV / 1x 10^{16}cm^{-2}).
- Si-FZ100 double implanted with ^{17}O[3MeV/(5x10^{13}cm^{-2}/each side)] + annealed 1300 ^{o}C/5 days
- Irradiated with e^-(3.5Mev / 1x10^{16}cm^{-2})
- Irradiated with e^-(3.5Mev / 2x10^{16}cm^{-2})

c.) Si-DOFZ100 with ^{16}O (10^{17}cm^{-3}) , irradiated with n (1 MeV / 2x10^{16}cm^{-2}).

d) Si-FZ100 implanted with ^{17}O(3MeV/4.5x10^{14}cm^{-2})+ annealed at 950^{o}C/5 days; unirradiated

These samples have been investigated from RT down to 10K

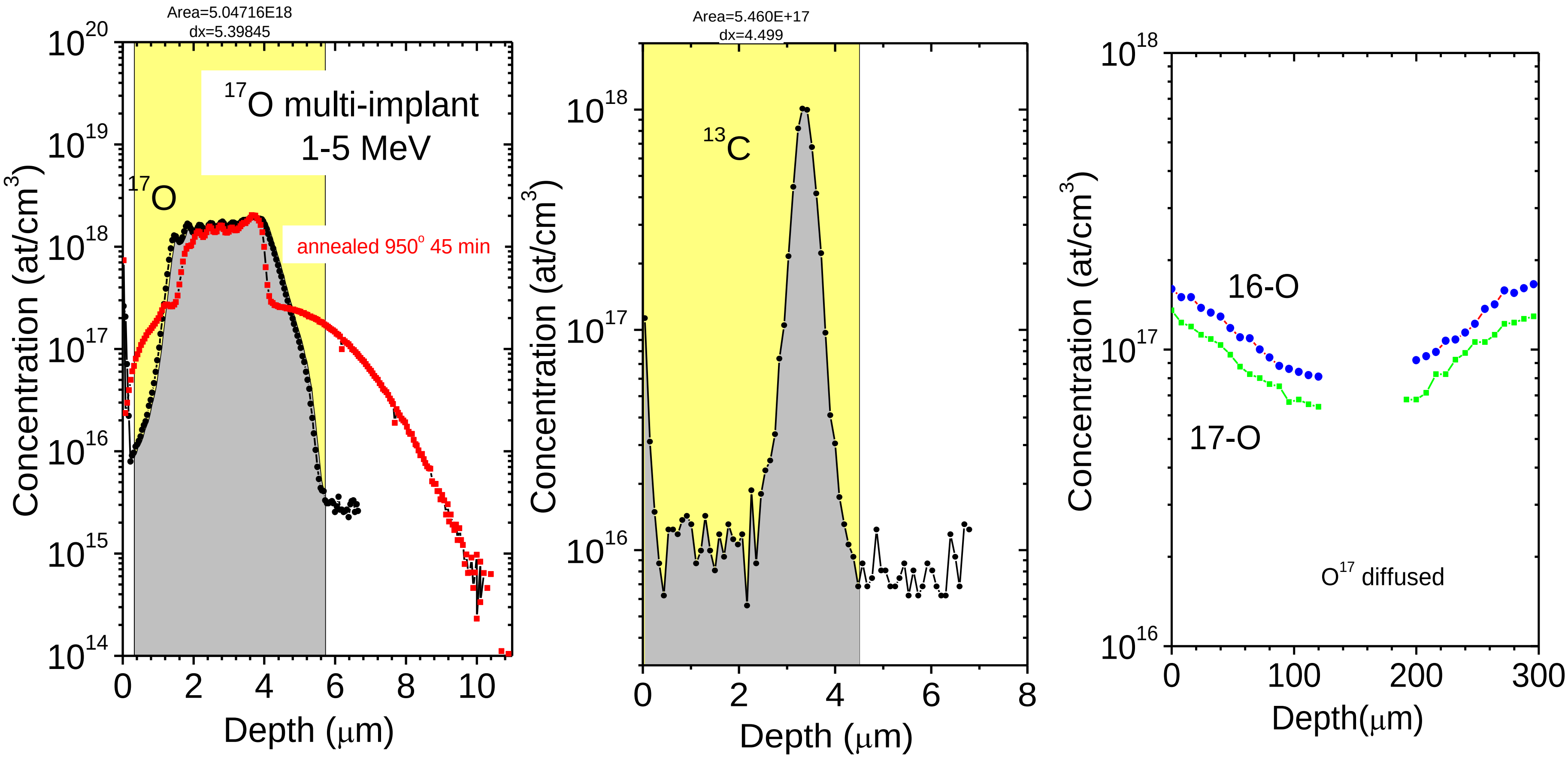
Set 2:

- FZ100-doped by diffussion with ^{17}O + e^- irradiated (27 MeV/ 1 and 2 x 10^{16} cm^{-2});

  Received 2 samples each of 5 x 5 x 0.3 mm^3
- FZ100- doped with 13C + e^- irradiated (27 MeV / 2 x 10^{16}cm^{-2}); Received 2 samples of 5 x 5 x 0.3 mm^3

These samples have been investigated up to now only from room temperature (RT) down to 100 K

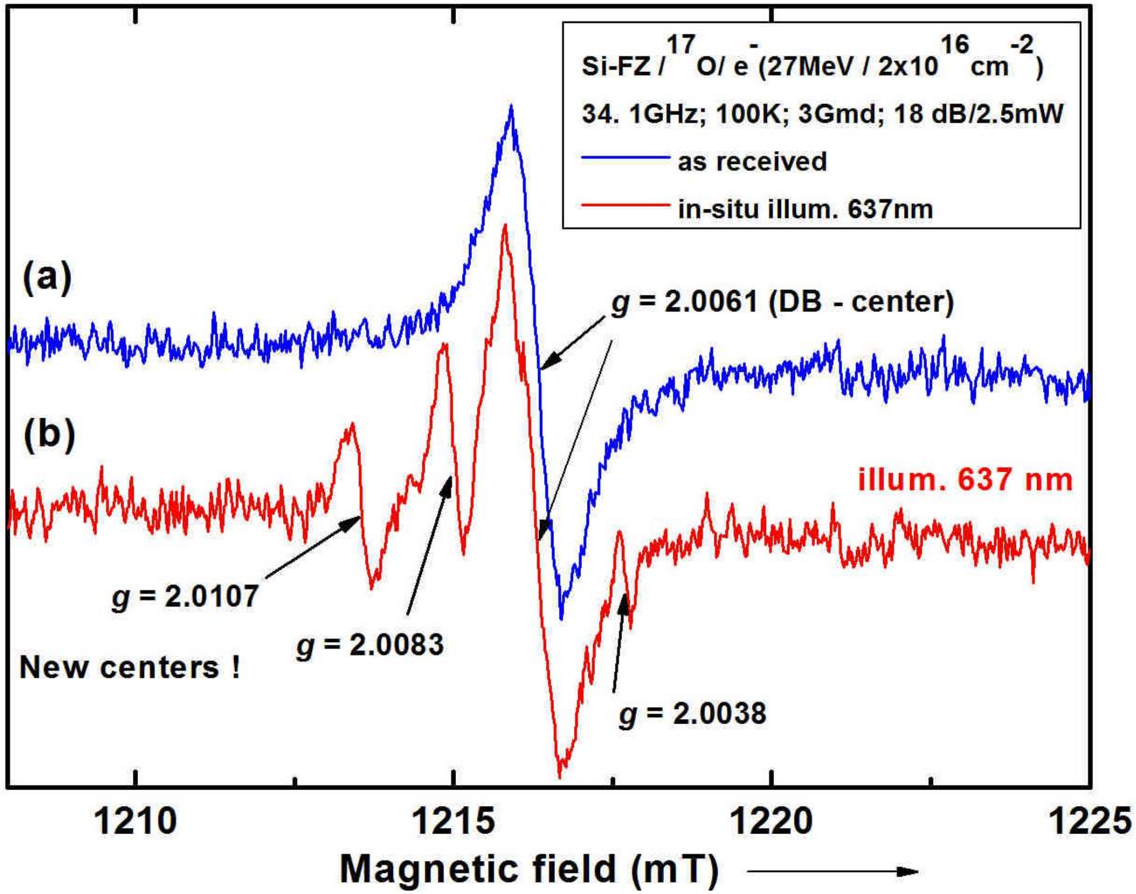
Profile of ^{17}O and ^{13}C in the investigated samples



• the ^{17}O concentration in the oxygen-diffused samples is ~ 7 larger than that reached in the implanted samples

• the concentration of ^{13}C achieved in the implanted samples is below the EPR detection limit

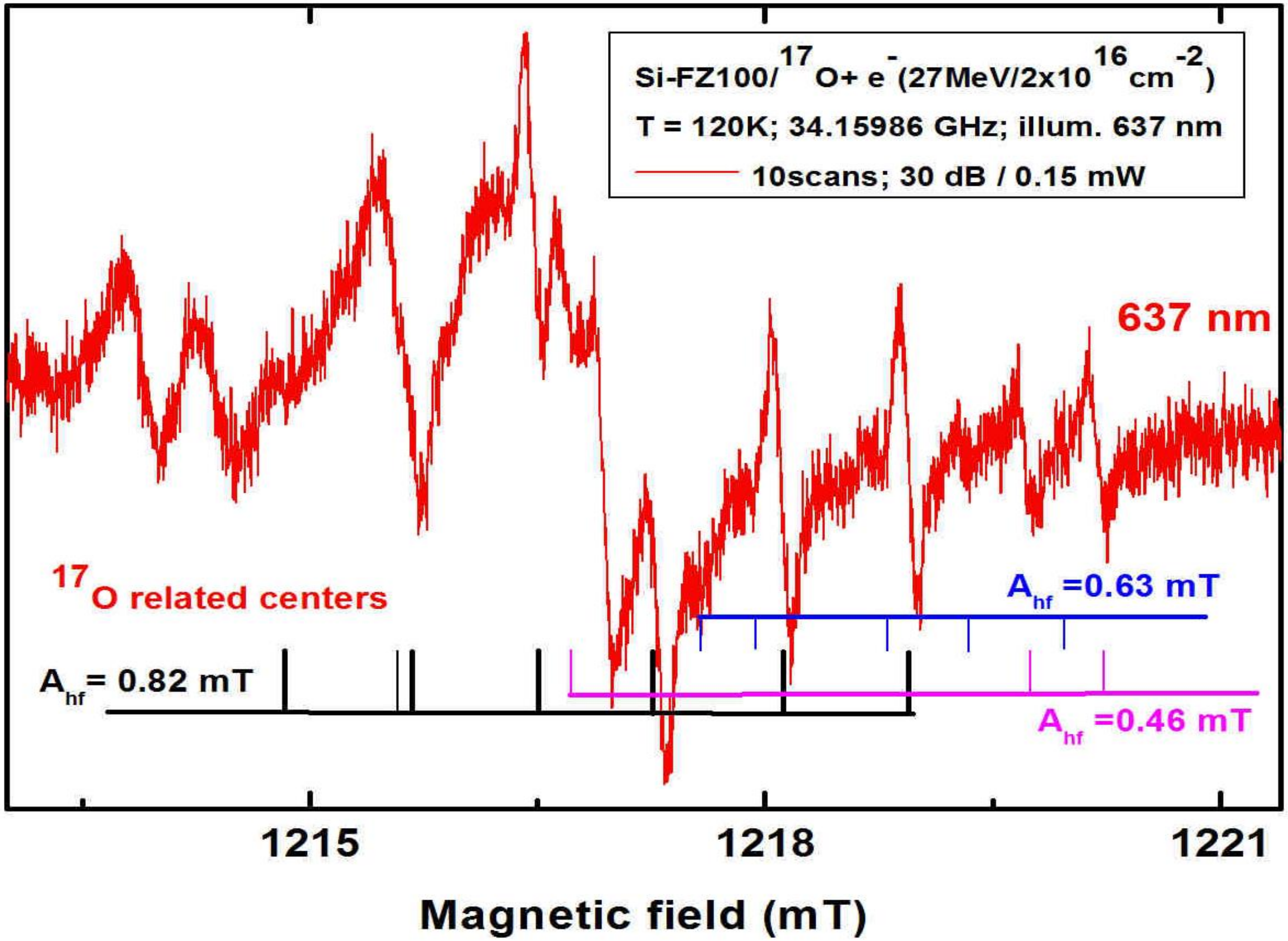
^{17}O-diffused sample



- the EPR spectrum of the as-received sample doped with ^{17}O by diffusion and irradiated with electrons of 27MeV exhibits only an isotropic line localised at g= 2.0060 +/-0.0002 , with peak-to-peak line width DHpp = 0.80 +/- 0.03 mT, visible already at RT, caused by unpaired electrons in dangling bonds. No changes were observed by in-situ 637nm illumination at RT.

- **Illumination at T < 120K resulted in the observation at <u>high microwave powers</u> of three new lines localised** at g-values: 2.0107; 2.0083 and 2.0038 with linewidths from 0.1 mT to 0. 4 mT

^{17}O-diffused sample



Measurements at <u>low microwave powers (0.15 mW) resulted</u> in changes in the EPR spectrum with the observation of several easily saturable narrow lines (~ 0. 1mT) of equal separations of 0.8 and 0. 6 mT, <u>due to the superfine interactions with ^{17}O nuclei (I =5/2).</u>

→ Further investigations (angular dependences) at T<100K to reveal their structure

Summary and future plans

- *Introduction rates of the defects determining the device performance vs. the electron energy show a saturation tendency - expected from NIEL*
- *Introduction rates for H defects for DOFZ & STFZ are similar → no [O] dependent but the annealing out of H(140K&152K) is different → High chances to be identified via HRTEM investigations*
- *Introduction rate for E (30K) is 3 times larger in DOFZ material → [O] dependent, to be identified via EPR studies*

- *HRTEM investigations revealed that during annealing at temperatures ≤ 200 C two types of extended planar defects are formed: {111} type (aggregation of vacancies) and {113} type (agglomeration of interstitials). Further studies on oxygenated samples are planned now, hoping that this way can differentiate between H(116K) and H(140K&152K) and identify both.*

- *EPR lines characteristic for hyperfine interactions from paramagnetic defects interacting with nuclei with I > 2, very likely ^{17}O, observed only in the samples doped by difussion. Further investigations (angular dependences) at T<100K to reveal their structure are planned.*

Thank You !