We have focussed on generation and annealing of the defects having a direct influence on the device characteristics. The radiation damage is primarily due to bulk damage, consisting in the generation of isolated point- and extended cluster defects. As point defects are produced by low energy silicon recoils, cluster effects are due to larger energy recoils which will then lead to a dense cascade of silicon atoms removed from their specific lattice sites. For investigating the generation process of point and extended defects we have used irradiation by mono-energetic electrons in a wide energy range from 1.5 to 27 MeV. The generation of point defects and defect kinetics involving impurities (as e.g. B, C and/or O) were investigated from electrical point of view after irradiation with low fluences of alpha particles or low energy electrons both n or p type silicon structures. The generation of small clusters of defects were investigated mainly after irradiation with high energy electrons and high irradiation fluences of n-type Si sensors. Also, after high irradiation fluences EPR/ENDOR and HRTEM studies were initiated. The main results are:

1) Low irradiation fluences

   a) Using forward current injection at room temperature we can eliminate the radiation-induced boron–oxygen complex, which is the main compensating center in irradiated p-type Si sensors. The process is called Forward Current Enhanced (FCE) annealing. To achieve a high enough elimination rate, relatively high current densities are required. Besides, new evidences have been obtained on the relation between interstitial carbon–oxygen complex growth at 350–400 °C and interstitial boron–oxygen complex annealing in Si crystals. When the FCE annealing temperatures are relatively low (lower than 290 K) the loss of the E1 peak amplitude is accompanied by the growth of the H2 peak only (compare curve 1 with curve 2 in Fig. 1). From its DLTS signature we identified the latter one as the interstitial carbon. This defect is known to be mobile at room temperature and can be trapped by interstitial oxygen. This reaction is also observed in our structures as the loss of the H2 peak (Ci) has a one-to-one relation to the growth of the H3 peak (CiOi) (not shown here). At higher FCE annealing temperatures we observe either the growth of both H2 (Ci) and H3 (CiOi) peaks (below 50 8C) or the growth of the H3 peak only (above 50 8C). At the end of both, the complete FCE annealing of BiOi and the trapping of all mobile carbon interstitials by oxygen, we detect only the divacancy (H2) and the CiOi (H3) peaks (curves 3 and 30 in Fig. 1). Following the increase of the H2+H3 peak amplitudes after different durations of the FCE annealing and comparing them with the decrease in amplitude of the E1 peak, a linear correlation is found for three different FCE temperatures. These results demonstrate that the annealing of the BiOi complex (E1 peak) is accompanied by the appearance of interstitial carbon (H2 peak). In our opinion, such a process can occur considering three consecutive defect reactions: (i) the recombination enhanced dissociation of BiOi and appearance of Bi, (ii) an inverse Watkins replacement reaction for interstitial boron.
The BiOi concentration decreases exponentially with annealing time (see Fig. 2). The rate of the FCE annealing depends on the forward current density (compare curves 1 and 2 in Fig. 2), annealing temperature and irradiation dose (compare curves 1 and 1’ in Fig. 2). Experiments at different temperatures showed that the FCE annealing rate changes approximately quadratically with the injection current density, similar to the FCE annealing of the interstitial boron. As seen from Fig. 2 the lower the irradiation dose is the higher is the annealing rate for the same forward current density and temperature. This fact can be explained as a result of the decrease of minority carrier lifetime with irradiation dose. We have performed isochronal annealing studies of BiOi and CiOi complexes, up to 400K for the FCE annealing and up to 700K for thermal annealing. The obtained defect concentrations (N) normalized to the defect initial density (N0, measured after the preliminary annealing at 120 °C) are given in Fig. 3. Some of the obtained results (curves 1 and 1’ in Fig. 3) are consistent with previous results on the thermal stability of BiOi and CiOi complexes. They also show that an essential growth of CiOi defect concentration takes place at 300–350 °C. Such an increase of CiOi concentration during annealing at Tann >300 °C was observed in many works. However, in our structures the CiOi increase is rather large compared to the one observed in Czochralski grown crystals. We consider that this difference is due to the relatively low carbon content in our structures that are based on epitaxial silicon.
According to the thermal annealing data presented in Fig. 3 (curves 1 and 1’), no direct correlation between the growth of CiOi and the annealing-out of BiOi exists. While the BiOi anneals-out completely at 200 °C, the increase in the concentration of CiOi is observed only above 300 °C. Under FCE annealing, however, the increase in CiOi concentration takes place already at room temperature and simultaneously with the decrease in the BiOi density (curves 2, 2’ and 3, 3’ in Fig. 3). Furthermore, the FCE-induced generation of CiOi leads to defect concentrations that are very close to those obtained after thermal annealing (in absence of FCE) at temperatures above 300 °C (compare curve 1’ with curves 2’ and 3’ in Fig. 3). Also, after the FCE annealing, a subsequent thermal annealing at T>300 °C produces only a relatively small increase of the H3 (CiOi) peak height (see curves 2’ and 3’ in Fig. 3). It means that the two reactions, dissociation of BiOi and formation of CiOi, taking place during the thermal annealing at different temperatures, are both enhanced by the forward current injection, resulting in much lower activation energies for the FCE annealing than those corresponding to the thermal annealing. All these energies can be evaluated by fitting the isochronal annealing data. The fitting results for the FCE annealing of BiOi complex are shown by lines 1–3 in Fig. 3. The resulting activation energies are in the range of Ea:0.4–0.6 eV for current densities between 15 and 30 A/cm² and are close to those reported for the slowly forming recombination center in illuminated p-type silicon. The activation energy increases when lowering the injection current densities. For zero current density, the activation energy for the annealing of BiOi was reported previously to be of about 1 eV larger, i.e., Ea =1.4 eV (curve 1 in Fig. 3). Thus, the presently reported FCE annealing procedure leads to a significant decrease in the activation energy for the annealing of the BiOi complex (ΔEa =1 eV), similar to the case of interstitial aluminum and boron in silicon.

b) Metastability of CiOi defect centers

It is shown experimentally that, in contrast to the stable configuration of (interstitial carbon)–(interstitial oxygen) complexes (CiOi), the corresponding metastable configuration (CiOi*) cannot be found in n-Si based structures by the method of capacitance spectroscopy. The rates of transformation CiOi are practically the same for both n- and p-Si with a concentration of charge carriers of no higher than 10¹³ cm⁻³. It is established that the probabilities of the simultaneous formation of stable and metastable configurations of the complex under study in the case of the addition of an atom of interstitial carbon to an atom of interstitial oxygen is close to 50%. This is caused by the orientation dependence of the interaction potential of an atom of interstitial oxygen with an interstitial carbon atom, which diffuses to this oxygen atom. The presence of the CiOi* complex manifests itself in a slight shift of the H₀35 peak to
lower temperatures and its certain or some broadening (see Fig. 4). A superposition of the two peaks related to $\text{CiOi}$ and will be here designated as $H035b$ (Fig. 5, curve 1). This is related to the fact that the rates of the emission of holes from the $\text{CiOi}$ and complexes are very close to each other. The property of the trap revealed by us makes it possible to explain the evolution of the DLTS spectra related to carbon centers in $n\_\text{Si}$ during the course of $\text{Ci}$ annealing. As is known, one can observe both acceptor (trap for majority charge carriers $E012$) and donor (trap for minority charge carriers $H029$) states of $\text{Ci}$ (Fig. 6) in diodes based on $n\_\text{Si}$. The donor state of $\text{CiOi}$ (peak $H035$, Fig. 6) manifests itself also as a trap for minority charge carriers. In addition, we can also trace the variation in the concentration of the complex consisting of interstitial carbon and a carbon atom in the lattice site ($\text{CiCs}$); this complex also contributes to the amplitude of the $E017$ peak. One can extract this contribution via measurements with different filling pulse durations. Thus, in $n\_\text{Si}$, we can trace variations in the $\text{Ci}$ concentration through measurements of the height of the $E012$ and $H029$ peaks. At the same time, it follows from the abovementioned data that we cannot observe the peak related to the complex. The presence of the metastable state of the $\text{CiOi}$ complex can be deduced only from an increase in the amplitude of the $H035$ complex after complete disappearance of the $E012$ and $H029$ peaks. This effect is well seen, for example, from comparison of the curves 1' and 2' in Fig. 7 and from the dependence of the height of the $H028$ and $H035$ peaks on the annealing duration for the $n+\text{p}$ and $p+\text{n}$ diodes (see Figs. 8 and 9).

**Fig. 4.** DLTS (lines) and MC_DLTS (dots) spectra of silicon $n+\text{p}$ diodes irradiated with electrons; the spectra were measured either before (curves 1 and 1') or after completion (curves 2 and 2') of the annealing of $\text{Ci}$.

**Fig. 5.** DLTS (lines) and MC_DLTS (dots) spectra of silicon $n+\text{p}$ diodes irradiated with electrons; the spectra were measured either after annealing at 290 K for 219 min (curves 1 and 1') or after completion of the annealing of $\text{Ci}$ (curves 2 and 2').
Fig. 6. DLTS (lines) and MC_DLTS (dots) spectra of silicon p+n diodes irradiated with electrons; the spectra were measured either before annealing (curves 1 and 1') or after completion (curves 2 and 2') of the annealing of C_i.

Fig. 7. DLTS (lines) and MC_DLTS (dots) spectra of silicon p+n diodes irradiated with electrons; the spectra were measured either after annealing at 300 K for 101 min (curves 1 and 1') or after the completion of annealing of C_i (curves 2 and 2').

Fig. 8. Variations in the concentration of carbon-containing defects during the course of the isothermal annealing of diodes formed on the basis of p_Si. The annealing temperatures were 290 K (t_{ann} = 0–219 min) and 310 K (t_{ann} = 219–419 min).
Thus, we used the DLTS method to study the kinetics of annealing of interstitial carbon $C_i$ in diodes formed on the basis of $n_-$ and $p$-$Si$ and irradiated with 6$_{\text{MeV}}$ electrons at $T = 273$ K. As a result of the annealing of $C_i$, complexes consisting of interstitial carbon and interstitial oxygen in the stable $C_iO_i$ and metastable $C_iO_i^*$ configurations are formed. It is established that, in spite of the close location of deep levels in the band gap of Si, the trap has a small (<1) ratio of the capture cross section of holes to the capture cross section of electrons (in contrast to $C_iO_i$). As a result of this, measurements of the DLTS spectra in $p$-$Si$ with the injection of minority charge carriers make it possible to exclude the component related to the metastable configuration and observe only the signal caused by the trap $C_iO_i$. We obtained additional confirmation of the simultaneous and equiprobable formation of and $C_iO_i$ complexes as a result of annealing of $C_i$. We determined the time constants for the transformation $C_iO_i$; these constants are found to be almost the same for diodes based on high-resistivity $n_-$ and $p$-$Si$.

c) Generation of $V_3$ defects, their bistability and trapping parameters

Low fluence irradiations with electrons of larger energies reveal the generation of small clusters of trivacancies ($V_3$) in PHR configuration. In this planar configuration the $V_3$ defect have two energy levels, corresponding to double and single acceptor states at 0.359 eV and 0.458 eV below the conduction band of silicon. Examples of DLTS spectra recorded after irradiation with electrons of 3.5 MeV and 27 MeV are shown in Fig. 10(a) and (b).

![Fig. 10. DLTS spectra measured on DOFZ (open symbols) and STFZ (filled symbols) diodes after irradiation with: (a) 3.5 MeV electrons; (b) 27 MeV . The positive spectra correspond to electron injection pulses and the negative ones to pulses of forward biasing. Measurements done with: $U_R = -10$ V, $U_{P,e} = -0.1$ V, $U_{P,Fw} = 3$ V, $t_p = 100$ ms, $T_W = 200$ ms.](image_url)
Thus, starting with electrons of 3.5 MeV the recoil energy is sufficiently high to knock out secondary atoms frequently. As shown in the literature, this defect is bi-stable, changing its configuration at ambient temperatures from a PHR configuration to a fourfold coordinated (FFC) one, while it is stable up to 220 °C. It has been shown previously that the recovery of both DLTS signals (from $V_3^{(-)}$ and $V_3^{(0)}$) is possible by injection of a high forward current (1A at 20 °C for 10 minutes). If the $V_3$ defect was found to be part of the origin of the leakage current, then both the leakage current and the defect concentration should have also a similar annealing behavior. Fig. 11 shows a comparison between the measured leakage current (open symbols) and the $V_3$ defect concentration (filled symbols). The leakage current at full depletion was extracted from the I-V measurements at 20 °C. As depicted in Fig. 11(b), the time constants for the annealing at 80 °C of the leakage current and of the concentration of $V_3$ defects in planar configuration (PHR) are very similar, $\tau = 44 \pm 3$ min. for LC and $\tau = 43 \pm 2$ min. for $V_3$. These experiments indicate that the variation seen in the leakage current is entirely related to the change in the concentration of $V_3$ in PHR configuration. In particular, a direct connection between the $V_3$ concentration and the leakage current can be done via the $V_3^{(-)}$ energy level that is located close to the middle of the band gap and may constitutes an effective generation center. Known for this energy level ($V_3^{(-)}$) are the activation energy and the capture cross section for electrons determined to be 0.458 eV below $E_C$ and $\sigma_n = 2.4 \times 10^{-15}$ cm$^2$, respectively. Using these trapping parameters as well as the experimental values obtained for the change in the leakage current and in the $V_3$ concentration, the capture cross section for holes of $V_3^{(-)}$ acceptor state can be determined as well, resulting in a value of $\sigma_p = 2.5 \times 10^{-13}$ cm$^2$.

II) High irradiation fluences
a) Electrical Characterization (TSC, CV, IV)

Other point and extended defects can be observed after irradiation with larger fluences, where the DLTS method is not applicable anymore. Examples of TSC spectra obtained after irradiation with electrons of different energies and for a fluence of $\Phi_e = 6 \times 10^{14}$ cm$^{-2}$ are given in Fig. 12(a). Known as point defects and detected after gamma-irradiation are: IO$_2$ (the interstitial oxygen dimer), the BD and the Ip defects. Several other defects, among which H(116K), H(140K) and H(152K) are acceptors in the lower part of the silicon bandgap and E(30K) is a shallow donor in the upper part of the gap, are observed after hadron irradiations and not after irradiations with $^{60}$Co-gamma-source. As can be observed in Fig. 12(a), these
electrically active defects can already be detected in very small concentrations after irradiation with high fluences of 1.5 MeV electrons and their generation increases with the electron energy suggesting that they are extended defects. It is worth mentioning that the E(30K) defect is not detected immediately after the irradiation. This defect anneals-in after irradiation is stopped and reach its maximum concentration after 200 min. annealing at 80 °C. The TSC signals from H(116K), H(140K) and H(152K) defects continue to grow much longer time after the irradiation is performed and tend to stabilize after 4000 min. -see Fig. 12(b).

The generation rates for different electron energies, calculated with the saturated values (after annealing of 4000 min. at 80° C) of the defect concentrations, are given in Fig. 13. A saturation of the rates starts to be observed around 15 MeV electron energy.

One can observe that only the generation of E(30K) defect is influenced by the oxygen content - its introduction rate is almost ~ 40% larger in DOFZ compared to STFZ material. In contrast, although a slight difference in the annealing-in time dependence exists between STFZ and DOFZ materials, the generation rates for H defects are very similar indicating that oxygen is not directly involved in the formation of these defects, leading to the supposition that they might be related to higher order vacancy complexes (\(V_{n>3}\)).

Relevant for applications is whether the radiation induced defects affect the sensors characteristics at their operational temperature and to what extent. The resulting Neff
calculated based on the defect concentrations determined from TSC experiments for a temperature of 20 °C is given in Fig. 14 for the STFZ and DOFZ diodes irradiated with 15 MeV electrons. In the same figure the $N_{\text{eff}}$ determined from the experimental C-V curves is given as well. One can observe that both type of diodes get “inverted” during the annealing time, resulting in negative values of $N_{\text{eff}}$ at the end of the annealing. As already known, the higher oxygen content in DOFZ compared with STFZ material favors the formation of shallow donors (as BD and E(30K)) while is suppressing the generation of close to midgap I_p acceptor like defect. This, together with the faster annealing in of the H defects in STFZ compared with DOFZ material (see Fig. 12b) leads to a delay in the apparition of “type inversion” effect in DOFZ diodes. The good agreement between the two methods of determining the $N_{\text{eff}}$ in irradiated diodes confirms that the defects determining the changes of the device performance at ambient temperatures after irradiation are not the point defects but the small clusters of defects as E(30K), H(116K), H(140K) and H(152K).

![Graphs showing effective doping concentration versus annealing time for STFZ and DOFZ diodes](image1)

**FIG. 14.** The effective doping concentration, as determined from C-V characteristics at 20°C (open symbols) or calculated with eq. 3 and accounting the defect concentration evaluated from TSC experiments, versus annealing time at 80 °C, after irradiation with 15 MeV electrons, $\Phi = 2 \times 10^{14}$ cm$^{-2}$ of: a) STFZ diode; b) DOFZ diode.

In addition, similar to $V_3$ small cluster, there are indications that these H defects change their configuration at ambient temperature. This phenomenon is observed in inverted diodes. An example is given in Fig. 15.

![Graphs showing TSC and C-V spectra](image2)

Fig. 15. Bistability of H(116K), H(140K) and H(152K) defects and corresponding change in the depletion voltage of a STFZ diode irradiated with 27 MeV electrons and annealed for 3960min. at 80 °C: a) TSC spectra; b) C-V curves. The measurements were performed consecutively, the first just after the annealing (curves 1), the
second 24 h after annealing while the sample was kept in dark at 290 K (curves 2) and the third ones after performing a 1A forward injection at 0°C (curves 3) for 30 min.

b) EPR/ENDOR investigations
The standard ESR measurements resulted in observation of a very limited number of irradiation induced paramagnetic defect (IPPD) species in as-irradiated samples. Many more IPPDs were observed by across the gap in-situ illumination at T < 150 K. The investigated samples were Float-zone Silicon (SiFZ) enriched with 17O or 1C isotopes and irradiated with electrons of 3.5 MeV and 27 MeV. After irradiation with 3.5 MeV electrons, the signal from IPPDs is at the detection limit. Further irradiation will be performed at the beginning of the next year and the EPR experiments will be redone. After irradiation with 27 MeV, the signals were somehow better and the spectra are now under evaluation. Two vacancy related centers where observed $V_2$ and $V_4$ in their single acceptor charge state. The spectra are shown in Fig. 17.

![Fig. 16. EPR spectra measured at 100K, during the in-situ illumination on the annealed samples at different temperatures: (a) SiFZ-17O, (b) SiFZ-13C.](image)

For these two centers the annealing behaviour is shown in Fig. 17.

![Fig. 17. Annealing behaviour of $V_2$ and $V_3$ during the isochronal annealing of 30 minutes at different temperatures up to 250°C.](image)

Many of other IPPDs are now under evaluations.
c) **HRTEM investigations**

STFZ and DOFZ silicon wafers irradiated with 15 MeV electrons at a fluence of $1 \times 10^{16}$ cm$^{-2}$ and 27 MeV electrons at a fluence of $2 \times 10^{16}$ cm$^{-2}$, respectively have been extensively studied by transmission electron microscopy. Fig. 18 shows a HRTEM image along the [110] zone axis of the 15 MeV electron irradiated STFZ Si sample revealing by the dark contrast the presence of clusters of defects. Small cluster of defects give the black dotted contrast better observed in the brighter zones of the image, while the dark patches reveal the presence of larger cluster defect agglomerates. A particular contrast at defects, the so called "coffee bean", is indicated by white arrows in Fig. 18. These are plate-like extended defects, appearing in the image as brighter lines surrounded by the dark contrast of the surrounding strain field. The plate-like defects are oriented along the $<110>$ directions and results from the agglomeration of a large number of point defects in the {100} planes parallel to the wafer surface. They are randomly distributed in the whole specimen. Their average size, considering also the surrounding strain field where the Si lattice is disturbed, is approximately 5 nm. As observed in the HRTEM images, in the zones with clusters of defects the Si lattice is not amorphous. Of course, the Si lattice is disordered by their presence, but the lattice remains crystalline. Fig. 19 shows HRTEM images at higher magnifications revealing more information about these defects.

![HRTEM image of STFZ Si sample](image)

**FIG. 18.** Low magnification HRTEM image along the [110] zone axis of the STFZ Si sample irradiated with 15 MeV electrons. White arrows indicate plate-like defects ("coffee beans")

Fig. 19(a) shows the clusters of point defects as revealed by their dotted black contrast. Few are single cluster, i.e. a single darker dot, but most of them are agglomerated, either along the principal crystallographic directions forming darker lines of clusters, or randomly giving rise to darker patches. The density of defect clusters is very high. They are rather uniformly distributed in the lattice, therefore there is almost no region in the Si lattice not affected by their presence. It is worth mentioning that these types of defect clusters produced by high energy (15 Mev) electrons in Si and observed by HRTEM are not mentioned in literature. Since interstitials are very mobile at room temperature, it is likely that the cluster of defects consists of cluster of vacancies, at least when they are not agglomerated. It is known that a study at atomic scale of small defect clusters is not straightforward. Correct information can be obtained for the one dimensional (1D) or the two dimensional (2D) clusters which have a periodic structure along the electron beam, meaning the $<110>$ direction, as in our case. The HRTEM images presented here were recorded close to Scherzer defocus where the column of atoms are seen in dark contrast while the bright dots correspond to channels in the Si structure. Therefore, in the HRTEM images the clustering of point defects is revealed by a dark contrast. A better understanding of the structure of these defect clusters need simulations of the HRTEM images on suitable models created with dedicated programs, which for the moment are not available in our laboratory. Fig. 20 shows a HRTEM image along the [110] zone axis of the 27 MeV electron irradiated DOFZ Si sample revealing the clusters of point defects and their agglomeration by the black dotted contrast and black patches, respectively. Unless the STFZ Si sample irradiated with 15 MeV electrons, in this sample no plate like defects ("coffee bean" type) were observed. At higher magnifications, Fig. 21, the clusters of defects in the DOFZ Si wafer irradiated with 27 MeV electrons show a similar contrast as in the STFZ Si sample irradiated with 15 MeV electrons (see Fig. 19(a)). As previously, they are distributed in the whole specimen at a comparable
high concentration, meaning that the density of the introduced defect clusters, as observed in the HRTEM images, does not apparently differ, although the two samples were irradiated at different electron energies and fluencies. It might exist an effect of saturation in the rate of introducing the clusters of defects at high energies, as indicated in figure 6 for the E(30K), H(116K), H(140K) and H(152K) clusters of defects. On the other hand, regardless the presence of diffused oxygen in the Si samples (STFZ vs. DOFZ) the clusters of point defects show similar contrast and distribution along the principal crystallographic directions. It looks like the clusters of defects observed by HRTEM are not related to the presence of O in the samples.

FIG. 19. HRTEM image along the [110] zone axis of the STFZ Si sample irradiated with 15 MeV electrons: (a) clusters of point defects revealed by the dotted dark contrast some agglomerated in dark patches or along the principal crystallographic directions are visible; inset detail of a star-like agglomerate of defect clusters; (b) a region of the specimen containing both clusters of defects and a "coffee-bean" plate like extended defect marked with white arrow; the black arrow indicates an agglomerate of defect clusters, i.e. a balck path.

FIG. 20. Low magnification HRTEM image along the [110] zone axis of the DOFZ Si sample irradiated with 27 MeV electrons.

Figure 21. HRTEM image along the [110] zone axis of the DOFZ Si sample irradiated with 27 MeV electrons. Clusters of point defects revealed by the dotted dark contrast some agglomerated in dark patches or along the principal crystallographic directions are visible; inset detail of the agglomerates of defect clusters.