AVOIDING BORON-OXYGEN RELATED DEGRADATION IN HIGHLY BORON DOPED CZ SILICON

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ABSTRACT: Lifetime degradation in Cz-grown silicon due to the formation of recombination active complexes consisting of boron and oxygen limits the efficiency potential of Cz-Si solar cells. In this contribution, a new approach to avoid this special type of degradation is presented. It is shown that there exists an additional state of the complex beside the 'annealed', weak recombination active state and the lifetime limiting, 'degraded' state, which is stable under the typical working conditions and, more important, only weakly recombination active. A simple model based on observations as well as some properties of this so called 'regenerated' state and its formation kinetics will be presented.

Keywords: Czochralski, Degradation, Regeneration

1 INTRODUCTION

Performance degradation of boron-doped Cz-Si solar cells plays an enormous role in modern photovoltaic, as they have a market share of about 38% [1]. The degradation of the electrical parameters of the solar cell originates from a degradation of the bulk lifetime due to the formation of complexes consisting of boron and oxygen, which act as recombination centres with an energy level deep within the band gap [2,3]. The saturation density of these complexes (after complete degradation) scales about linearly with the boron and quadratically with the oxygen concentration suggesting a stoichiometric composition of BO₂ [4,5,6]. Dependent on both concentrations, a fundamental lifetime limitation was proposed by Bothe et al. [7]. The degradation occurs in the course of time under illumination as well as under applied bias voltages [8] thus suggesting that free carriers enhance the formation process probably by increasing the diffusion velocity of an oxygen dimer involved in the complex [4]. Recently, a second type of boron-oxygen complex with a slightly different formation kinetic was reported [5] showing a very fast degradation. For longer times, this second type seems to convert into the well known type of complex responsible for the fundamental lifetime limit. At elevated temperatures and without external carrier injection, the reversal of the degradation can be observed, which is identified with the dissolution of the harmful complexes. The formation as well as the dissolution of the harmful complexes are thermally driven reactions described by an Arrhenius equation, allowing the extraction of an activation energy that is about 0.45 eV for the degradation [9,10,11] and about 1.32 eV for the anneal [2].

To soften the negative effect of degradation, it is necessary to reduce the density of the harmful boronoxygen related complexes. Therefore, the 'classical' ways of avoidance mainly target on the concentration of the involved components.

In general, commercially available Cz-Si has an interstitial oxygen content of about $5 \cdot 10^{17} - 1 \cdot 10^{18}$ cm⁻³, which arises from an unwanted but inevitable melting of the quartz crucible during the crystal growth with the Cz-technique. Lowering the oxygen concentration thus means to avoid the melting of the crucible, which is hardly to prevent at temperatures of about 1412°C (melting point of silicon).

In contrast, the reduction of the boron content is easily achievable and the loss caused by boron-oxygenrelated degradation declines with decreasing boron concentration (Fig. 4). Simultaneously, the efficiency potential of the solar cell in a standard screen printed process with the maximum at about 1 Ω cm [12] drops and hence a trade-off between both influences gives the maximum potential after degradation in Cz material with a resistivity of about 3-6 Ω cm, which is commonly used for industrial mass production of Cz-Si solar cells.

Other approaches like alternative p-type dopants (Gallium or Aluminium) or the usage of n-type base material result in other problems not discussed here.

In this contribution a recently reported [9] completely new approach is presented in more detail that can be applied to the finished solar cell independent from boron and oxygen concentration.

2 EXPERIMENTAL SETUP

2.1 Sample preparation

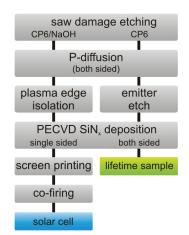


Figure 1: Schematic process flow diagram for the solar cells and lifetime samples used in the experiments.

For lifetime measurements boron-doped Cz-Si samples with a resistivity of 1.4 Ω cm were prepared by a CP6 saw damage and polish etch, a both sided 50 Ω /sq. phosphorous gettering treatment with subsequent

removal of the phosphorous doped layer followed by a both sided PECVD SiN_x :H-layer for surface passivation. The surface recombination velocity was calculated to be around 100 cm/s.

Furthermore, $125 \times 125 \text{ mm}^2$ semi-square borondoped Cz-Si solar cells were fabricated from standard commercially available material from three different suppliers with a base resistivity in the range of 1.4-14 Ω cm. A standard industrial process was applied including an alkaline or acidic saw damage etch, a both sided 50 Ω /sq. phosphorous emitter diffusion, plasma edge isolation, a PECVD SiN_x:H antireflection coating and a screen printed, co-fired aluminium back and silver front contact.

Former investigations [9] of the reaction kinetics have proven that the samples are mainly affected by the well-known boron-oxygen related degradation as described in the introduction.

2.2 Measurement techniques

The minority carrier lifetime of the homogeneous samples was measured by quasi-steady-state photoconductance decay (QSSPC) [13] at room temperature. Lifetime was extracted at a fixed injection level (low level injection) and not at a fixed illumination intensity to compensate the influence of the varying minority carrier concentration.

The measurements of the illuminated currentvoltage-characteristics took place under 1 sun (AM 1.5 current equivalent) at room temperature $(25.0 \pm 0.1^{\circ}\text{C})$. A gallium doped Cz-Si solar cell was used as reference, which was proven to be stable under illumination [14].

The measurements of the current-voltagecharacteristics in the dark were performed at a constant temperature of $24.0 \pm 0.1^{\circ}$ C. The collected data were afterwards evaluated using the 2 diode model to extract the saturation current density J₀₁ of the first diode.

The influence of the illumination (flash) during the measurements and handling regarding degradation is only of small importance.

3 EXPERIMENTS

3.1 First investigations on Cz-Si solar cells

For a first investigation a Cz-Si solar cell was annealed at 200°C to make sure that the main part of the harmful complexes was dissolved. Afterwards, it was placed under illumination at elevated temperatures (~70°C) and the development of the electrical parameters in the course of time was monitored by IV-measurements. The results for V_{oc} are shown in Fig. 2, the other parameters are shown in Fig. 3.

At first, V_{oc} drops due the formation of the harmful boron-oxygen related complexes. Later on, a contrary trend sets in and the electrical parameters begin to recover slowly. The increase of V_{oc} (and of the other parameters as well) saturates for longer times at a value close to the 'annealed' value that was measured directly after the annealing step at the beginning of the experiment. In contrast to the degradation, this recovery process of the electrical parameters (green line) was called 'regeneration' and as the light plays an important role, the effect should be called 'Light Induced Regeneration'.

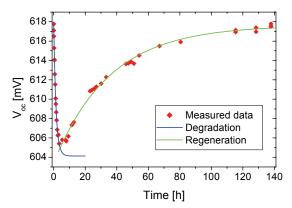


Figure 2: Temporal development of V_{oc} of a Cz-Si solar cell (2.8 Ω cm) under illumination at a temperature of ~70°C. The solid lines are simple exponential fitted curves. The dynamic can be identified as a relatively fast degradation (blue) and a subsequent slow regeneration (green) of V_{oc} .

The whole curve consisting of degradation and regeneration is called a regeneration cycle. As V_{oc} provides the most accurate data, further results are discussed regarding V_{oc} data.

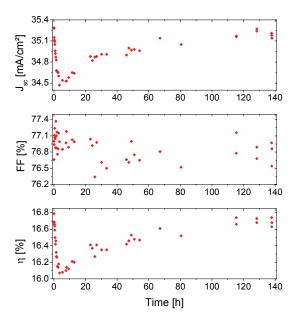


Figure 3: Temporal development of the other IV-parameters of the same solar cell shown in Fig 2. J_{sc} shows comparable changes as V_{oc} , the fill factor FF is not significantly affected. The efficiency η as being the product of all other parameters shows the regeneration cycle as well, but suffers from the error in the FF measurement.

3.2 Reproducibility and boron concentration

Further experiments were performed on Cz-Si solar cells processed on material from different suppliers and with different resistivities to check the reproducibility of the regeneration effect. Several samples were placed under illumination at elevated temperatures (70°C) and their electrical parameters were monitored by IV-measurements. The results for V_{oc} are shown in Fig. 4.

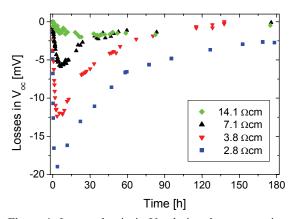


Figure 4: Loss and gain in V_{oc} during the regeneration cycle at 70°C for Cz-Si solar cells with different base resistivity. The maximal loss depends strongly on the doping concentration. The regeneration effect occurs in all samples.

The maximal drop of V_{oc} strongly depends on the base resistivity of the material. The higher the resistivity and thus the lower the boron doping concentration the smaller is the drop due to degradation as mentioned in the introduction even if the oxygen concentration is unknown. Regardless of the resistivity and the correlated amplitude of the degradation, the regeneration works on all samples and proves its reproducibility across Cz-Si material from several suppliers.

3.3 Lifetime investigations

As V_{oc} responds very sensitively to changes in the bulk lifetime, similar measurements were performed on lifetime samples to check the role of the bulk lifetime in the regeneration cycle.

At the beginning of the experiment, the sample was annealed at 200°C and then placed under illumination at elevated temperatures. Fig. 5 shows the results of the QSSPC measurements in the course of time.

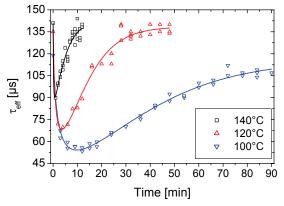


Figure 5: Multiple cycles of the effective lifetime of one sample $(1.4 \ \Omega cm)$ monitored by QSSPC during illumination at different temperatures. It is assumed that the surface passivation remains unaffected and that the observed changes originate from changes in the bulk lifetime. The solid lines represent an adapted lifetime fit function.

As the effect of regeneration shows up also in lifetime measurements, it seems not to be a property of the pn-junction or especially of the solar cell, but to be a fundamental intrinsic property of the material itself.

The bulk lifetime is determined by the recombination through all different possible channels represented by their specific lifetime τ_i and the total bulk lifetime τ_b may be written as

$$\frac{1}{\tau_b} = \sum_i \frac{1}{\tau_i} \tag{1}$$

As the bulk lifetime τ_b is inversely dependent on each recombination channel, it is dominated by the lowest lifetime τ_i and this channel is lifetime limiting. This means that the recombination channel of the boronoxygen related complexes, which is responsible for the drop in Fig. 5, must be neutralized in the course of time to allow the observed recovery of the bulk lifetime.

So far, there are three phenomenologically known 'states' of the boron-oxygen related complex. First, there is the only weak recombination active 'annealed' state, which is unstable under illumination or carrier injection. The second is the well known recombination active 'degraded' state responsible for the fundamental lifetime limit [7]. The third state is the final state of the so called fast degradation process [5], which probably could convert into the second state.

Based on the properties of these states, the data shown in Fig. 5 necessitate an additional 'regenerated' state with a recombination activity comparable to the annealed state, but which is, in contrast to the annealed state, not converted into the degraded state under illumination and carrier injection.

3.4 Investigations on stability at moderate temperatures

If the main part of the boron-oxygen related complexes resides in this new 'regenerated' state, the electrical parameters of the solar cell should be comparable to the annealed state but in contrast they should remain on this level under illumination as well as under external bias voltages that cause the degradation.

Therefore, a Cz-Si solar cell was almost completely regenerated under illumination at elevated temperature and then illuminated with 1 sun (AM 1.5 current equivalent) at 25°C for several hours. Alternatively, the same experiment was performed using external bias voltage in the dark instead of illumination. The results for V_{oc} are presented in Fig. 6, J_{sc} and in consequence η show an analogue behaviour.

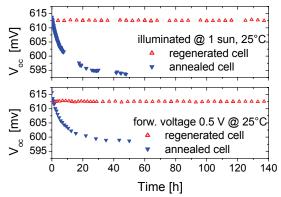


Figure 6: Stability test of the electrical parameters under illumination (top) and an applied bias voltage (bottom) at 25°C. Presented in blue is the development of a solar cell directly after an anneal, which results in the typical degradation. In contrast, the electrical parameters of the same solar cell after a regeneration cycle (red data) show no significant instability.

3.5 Current induced regeneration

The question arises, whether light is the real trigger for the conversion of complexes from the degraded state into the regenerated state, maybe by direct absorption at the complex. Alternatively, the generated excess carriers could play an important role.

Therefore, it was investigated whether the regeneration can also be triggered by external forward voltages in the dark. The solar cell was first annealed at 200°C and then connected via the busbars to a regulated DC power supply unit in constant current mode. The experiment took place in the dark to minimize the influence of ambient light. The results are shown in Fig. 7.

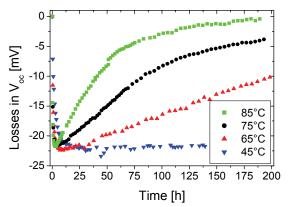


Figure 7: Degradation and regeneration of V_{oc} induced by applied forward voltages at different temperatures. With increasing temperature, the duration of the regeneration process decreases. At temperatures lower than 45°C the regeneration is barely detectable.

Obviously, the regeneration can also be triggered by external forward voltages thus meaning that the presence of electrons and holes at least accompanies the conversion process of the complexes. In analogy to the light induced case, this phenomenon should be called 'Current Induced Regeneration'.

3.6 Temperature dependence of regeneration

Both, Fig. 5 and Fig. 7 show a strong difference in the durations of the regeneration process ranging from several days to a few minutes at different temperatures from 45° C to 140° C. The changes in the initial degradation confirm the activation energy of about 0.45 eV already measured by other groups [9,10,11].

If the regeneration is a thermally activated process comparable to the degradation, an Arrhenius equation of the type

$$P_{conv} = v_{char} \cdot \exp\left(-\frac{E_a}{k_B T}\right)$$
(2)

can be deduced. This equation describes the probability P_{conv} of a conversion of one complex into another state. It

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includes a characteristic trial frequency ν_{char} and an energy barrier E_a in comparison with the mean thermal energy $k_{\rm B}T$. This relation can be adapted to describe the observable parameters of a macroscopic ensemble of complexes by its characteristic time constant t_0 even if a simple exponential decay (used later on for J_{01}) in the form

$$y(t) = y_0 \cdot \exp\left(-\frac{t}{t_0}\right) \tag{3}$$

is not the analytic correct function. A more complicated relation was developed for the evaluation of the lifetime measurements [15], which is not described here.

As it is shown in Fig. 2 the influence of the degradation (blue line) declines rapidly and, the pure regeneration (green line) can be investigated shortly after the minimum occurred. From the lifetime data in Fig. 5, an Arrhenius plot based on the extracted reaction constants can be derived as it is shown in Fig. 9.

An analogue procedure was accomplished on solar cell level using IV-measurements in the dark. The collected J_{01} data from different temperatures is shown in Fig 8.

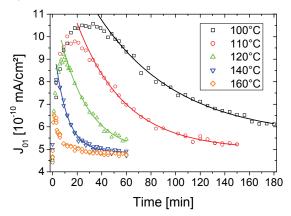


Figure 8: Degradation and regeneration of the saturation current density J_{01} derived from the 2-diode model under illumination at elevated temperatures. The data originate from the same solar cell with resistivity 1.4 Ω cm. The solid lines represent single exponential decay fits to the data.

As expected, the effect of degradation and regeneration is also clearly observable in J_{01} . In contrast to Fig. 4, the variation in the absolute amplitude of the effect originates from the different thermal development of the degradation ($E_a \sim 0.45 \text{ eV}$, [9,10,11]) and the regeneration ($E_a \sim 0.62 \text{ eV}$). With increasing temperature, the regeneration transforms the generated harmful complexes more quickly and the maximal number of harmful complexes decreases.

Based upon the time constants t_0 that can be extracted using Eqn. 3 to fit the data of each temperature, Fig. 9 shows in addition to the lifetime data an Arrhenius plot derived from Fig. 8.

The data in the Arrhenius plots derived from the lifetime data (Fig. 5) and the J_{01} data (Fig. 8) support a linear fit indicating that the regeneration is a thermally activated process. The activation energy E_a of the regeneration that can be extracted from both plots using Eqn. 2 agrees well with a value of about 0.62 eV.

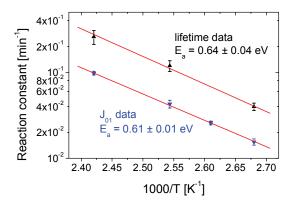


Figure 9: Arrhenius plot derived from the lifetime and J_{01} measurements at different temperatures. The solid lines represent linear fits allowing the determination of the activation energy E_a . The slope of both curves agrees well. The vertical shift results from different methods to determine the reaction constant.

3.7 Stability at higher temperatures

At temperatures of about 200°C, an instability of the regenerated state is observable if no further regeneration is enforced. It is found that samples treated at those temperatures show no instant changes in their electrical parameters, but become unstable regarding illumination or applied forward voltages at lower temperatures. The subsequent degradation after a longer treatment at ~200°C seems to be the same as observed before the regeneration. It is therefore assumed that the main part of the complexes is converted from the regenerated state to the annealed state. This conversion is expected to take time and thus a temporal variable part of the complexes resides in the regenerated and in the annealed state. But as those two states behave electrically indistinguishable (comparable low recombination), a measurement directly after the temperature treatment does not show any difference. Only a subsequent degradation reveals the occupation of states.

For the determination of a decay constant of this process, multiple very similar samples are treated in the dark at the same temperature for different durations. Afterwards, the samples are degraded under illumination until no more change in the IV-parameters could be detected. The resulting decay curves consisting of the data from the differently treated samples are shown in Fig. 10.

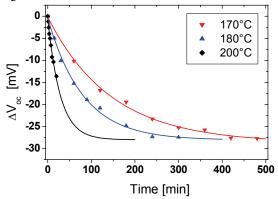


Figure 10: V_{oc} decay curves of the instability of the regenerated state towards the annealed state recorded after a subsequent degradation. The lines are exponential decay fits for determination of the decay constants.

The number of samples was limited to eight and therefore only eight different durations were investigated. Data in the strongly decaying initial phase grant a good fit of the exponential decay in this region but do not allow a determination of the value obtained after a long time. Therefore, the long time limiting value had to be given externally for the 200°C curve according to the other decay curves.

The extracted decay constants give rise to another Arrhenius plot with an extracted activation energy of about 1.0 eV, as shown in Fig. 11.

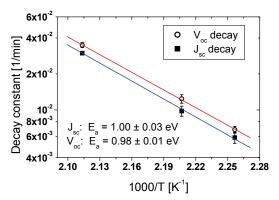


Figure 11: Arrhenius plot of the process responsible for the instability of the regenerated state. The data points were extracted from J_{sc} and V_{oc} decay curves.

The characteristic frequency of this process determined by the Arrhenius plot is about 10^7 Hz. This is significantly lower than phononic frequencies of about 10^{13} Hz, being expected for simple complex dissociation by lattice vibrations. It is conceivable that the conversion from the regenerated state to the annealed state passes over an intermediate stage.

The instability of the regenerated state allows the multiple usage of a sample that can be brought to the annealed state by heating at 200°C for longer times, as it was done in the beginning of the experiments.

4 A THEORETICAL APPROACH

4.1 A three state model

The stability of the electrical parameters as it is shown in Fig. 6 indicates the existence of an additional 'regenerated' state. Neglecting the degraded state of the harmful boron-oxygen related complexes, which is reached within the 'fast process' [5] and which is supposed to convert into the well known lifetime limiting degraded state reached in the 'slow process', a complex can reside in three fundamental different states.

The annealed state ('state A') of the complex shows only weak recombination activity and converts under illumination or more generally under carrier injection into a degraded state ('state B'). This degraded state shows a significantly higher recombination activity and limits the bulk lifetime. At elevated temperature, this state also becomes unstable and converts under illumination or applied forward voltages to the regenerated state ('state C'). This new state shows only a weak recombination activity comparable to the annealed state but is stable at least under the typical working conditions of the solar cell. If the regeneration process does not occur (e.g. in the dark), the regenerated state becomes unstable at elevated temperatures and the macroscopic system passes over to the annealed state. Whether this process uses the degraded state as an intermediate stage or whether it is a direct reaction from the regenerated to the annealed state, could not be clarified so far as both paths are hardly distinguishable at the investigated temperatures.

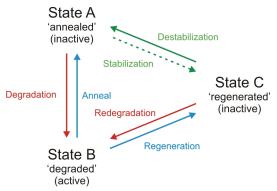


Figure 12: Reaction scheme for the degradation and regeneration based on three microscopic states of the boron-oxygen related complex. The conversion of large quantities of complexes into another state affects the observed lifetime.

Fig. 12 shows a reaction scheme of these three microscopic states with the 6 possible reaction paths named after the macroscopic (theoretical) observable reactions. The conversion of the regenerated state to the annealed state may be the direct destabilization path or a double reaction of redegradation and anneal. Alternative reactions exceeding this model might also be possible.

The direct reaction of 'stabilization' leading from the annealed to the regenerated state was not observed so far.

Based upon this model, theoretical calculations have been carried out, which describe the observed dynamics but are not presented here due to their complexity [15]. These calculations provide a correct fit function for the lifetime data shown in Fig. 5 to extract the reaction constants and predict thermal equilibrium states of the system.

4.2 Formation kinetics

Even if the exact microscopic states of the boronoxygen related complex are unknown, some basic conclusions are possible. A change in the recombination activity with a constant number of complexes means a change in the capture cross section for electrons and holes at the complex. This could be achieved by the change of the charge state, internal structural changes of the complex or a new composition of the complex.

The duration of the regeneration process is probably too slow for a change of the charge state of the complex as this process would be dominated by the fast reacting electronic system.

A simple structural change of the harmful complex like the splitting up of the involved oxygen dimer could be one explanation. On the other hand, this mechanism is assumed to occur on a shorter time scale.

Therefore, a more inert mechanism as the diffusion of impurities in silicon is more likely to cause the regeneration. In this case, the question arises whether the presence of electron-hole pairs triggers the process perhaps by a 'Bourgoin-Corbett-mechanism' [16], which describes an enhanced diffusion velocity of an impurity between different lattice sites by a continuously changing charge state. As a result, the moving impurity continuously causes a recombination of electrons and holes and the activation energy of about 0.62 eV would represent the activation energy drawn from the mean thermal energy of the system to support the diffusion.

A mixed approach would be imaginable wherein the injection of the excess carriers necessitates a change in the quasi-Fermi-level and thus the distribution of electrons across the density of states. This could result in an altered occupation of the electronic states of the harmful complex, which means a change of the charge state. This could strongly attract diffusing (inversely charged) impurities that bond to the complex. In this case, the activation energy of the regeneration would be identified by a shift of the quasi-Fermi-level. A change of only the charge state without other subsequent changes would probably not generate a complex, which remains stable and thus would be in contradiction to the investigations done so far.

Another possible explanation could be an energy barrier restricted structural change of the complex itself, which needs the amount of energy, which is released in a recombination event close to the complex in addition to a certain amount of energy drained from the mean thermal energy of the system. In addition, an impurity in the direct vicinity of this complex could be caught.

The possible candidates for the participating impurity have to fulfil a few requirements. The concentration of boron-oxygen related defects is of the magnitude $10^{12}-10^{13}$ cm⁻³. As the main part of the harmful complexes is neutralized within the regeneration, the concentration of the impurity has to be at least of a comparable magnitude, but in general a significantly higher concentration is needed. The second required property would be that the impurity itself does not reduce the lifetime significantly with a concentration of probably higher than 10^{14} cm⁻³. In addition, the effect could be proven on Cz-Si material from different suppliers suggesting that it is a generally present impurity in the silicon itself or a systematic contamination within the Cz-Si crystal growth process.

Possible candidates could be carbon ($\sim 10^{16}$ cm⁻³), nitrogen ($\sim 10^{15}$ cm⁻³) or oxygen ($\sim 10^{18}$ cm⁻³) as single atomic impurities or probably combinations like a dimer.

The last requirement is a relatively high diffusion velocity in silicon. For example, it was shown for an oxygen dimer that it can diffuse quickly by a carrier enhanced mechanism (Bourgoin-Corbett [16]). Other dimers could use an analogue mechanism.

4.3 Recombination activity of the regenerated state

The lifetime measurements in Fig. 5 show that the lifetime after a completed regeneration cycle reaches a comparable high level as in the beginning of the experiment. It is unlikely that the annealed and the regenerated states possess an equivalent recombination activity. Therefore, we assume that another unknown defect becomes lifetime limiting if the main part of the complexes residing in the degraded state has passed over to the regenerated state. This lifetime limiting defect might be the same as it was in the annealed state and thus represents the real limitation of the Cz-Si material.

The low recombination activity of the regenerated

state of the boron-oxygen related complex may be caused by either an energy level unsuitable for recombination due to its specific position within the band gap. A second possible reason might be a small and probably highly asymmetric capture cross-section for electrons or holes (or both). This would apply to a multiply charged defect that shields itself by the net charge against one type of carrier. The harmful boron-oxygen complex is suspected to be single or double positively charged giving rise to an asymmetric capture cross-section preferring the negatively charged electrons as it was observed [3].

The positive partial charge is assumed to be located at the oxygen atoms or dimer respectively. In contrast, boron acts as acceptor dopant in silicon and is therefore negatively charged at moderate temperatures. This negative partial charge of the boron atom within the boron-oxygen complex might attract other positively charged impurities that reside in the vicinity of the complex.

It is imaginable, that this boron-oxygen complex accumulates a highly positively charged cluster of impurities that surround homogeneously the boron atom (e.g. in a tetrahedral structure build up by two dimers). In consequence, the positive charge shields the complex from any hole needed for a successful recombination event and the complex loses its capability of recombination even if the energy level is deep within the band gap. A possible positively charged impurity could be an oxygen dimer.

5 CONCLUSIONS

In this contribution the reaction dynamic of the boron-oxygen related degradation and regeneration process was investigated. It was found that the harmful boron-oxygen complex responsible for the degradation can be converted into a regenerated state with significantly lower recombination activity by illumination or applied forward voltages indicating a process that is triggered or at least accompanied by free carriers.

This regenerated state was proven to be stable under the typical working conditions of the solar cell and to be instable at elevated temperature.

The investigation of the formation kinetics has shown that the regeneration process is a thermally activated process with an activation energy of about 0.62 eV. The process responsible for the instability of the regenerated state could not be clarified undoubtedly although an activation energy of about 1.0 eV was determined.

As the harmful boron-oxygen complexes are neutralized within the regeneration process, the fundamental lifetime limit [7] becomes invalid and the usage of highly boron-doped Cz-Si material without losses from this type of degradation is possible.

As the presented method can stabilize the efficiency at a high level and therefore opens a wide field of applications in using highly boron-doped Cz-Si for photovoltaics, the method of regeneration was applied for a patent.

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