

# From simulation to experiment: Understanding BO-regeneration kinetics

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## ARTICLE INFO

Keywords:  
Silicon  
Boron-oxygen  
Regeneration

## ABSTRACT

Regeneration of boron-oxygen related defects is investigated in differently compensated silicon wafers. It is shown for the first time that boron-oxygen defects can be transformed into a stable regenerated state also in compensated n-type silicon.

The coupling between regeneration rate and the completeness of the regeneration reaction is simulated based on the 3-state model of BO defects. Maximum regeneration temperatures that can be applied are determined for differently regenerating samples. The results are used to develop a high-speed process that can accelerate regeneration by two orders of magnitude without compromising neither the completeness of the regeneration process nor the stability of the resulting high-minority carrier lifetime values.

## 1. Introduction

Boron-doped crystalline silicon with high oxygen content, e.g., Czochralski (Cz) grown silicon, shows severe minority carrier lifetime degradation under carrier injection due to boron-oxygen related (BO) defects becoming recombination active [1–3]. This results in deterioration of lifetime-sensitive devices made from these materials, e.g., crystalline silicon solar cells [4–6]. The same effect occurs in boron-doped p-type [7–9] as well as n-type [10,11] silicon partially compensated or overcompensated with phosphorus or co-doped with gallium. BO defects can be transformed into a recombination-inactive state that is stable under carrier injection by the regeneration reaction that requires a combination of slightly elevated temperatures (typically 60–200 °C) and carrier injection [12,13], applied to hydrogenated silicon wafers [14–16]. This reaction may be identified (after the first occurring degradation reaction) by an asymptotically saturating recovery of the carrier lifetime or an asymptotical decrease of the defect density. For compensated p-type silicon it has been shown that the regeneration process also works in p-type samples co-doped with boron and phosphorus [17–20] with the limitation that the resulting BO state seemed to be only partially stable [19,20]. On

the other hand, n-type silicon doped with the same elements could not be successfully regenerated into a stable BO state [21].

Here, we present regeneration data measured in silicon wafers taken from Cz ingots that are (co-)doped with differing amounts of boron, gallium and phosphorus resulting in p-type as well as in n-type samples. An adapted high-speed regeneration process is developed based on the analysis of the interaction between the different transition reactions of the BO defect. The focus of this work is on the regeneration kinetics independent of the physical model that may explain the regeneration of recombination-active BO defects.

## 2. Three-state-model simulations

The BO defect undergoes different transitions described by the 3-state model (see Fig. 1) that depend on the external conditions [22]. BO degradation leading to the well-known reduction in minority carrier lifetime occurs in silicon under carrier injection if boron and oxygen are present in sufficiently high concentrations. Annealing as well as destabilization is purely thermally activated whereas regeneration additionally relies on carrier injection and the presence of hydrogen within the material, as introduced, e.g., via a belt furnace firing step from hydrogenated layers [23,24]. As long as low injection conditions are used, degradation is also only thermally activated when a certain minimum illumination (< 0.1 sun) is applied [25]. At typical regeneration conditions consisting of a combination of slightly elevated temperatures (e.g., 60–230 °C) and carrier injection, all four transition reactions are

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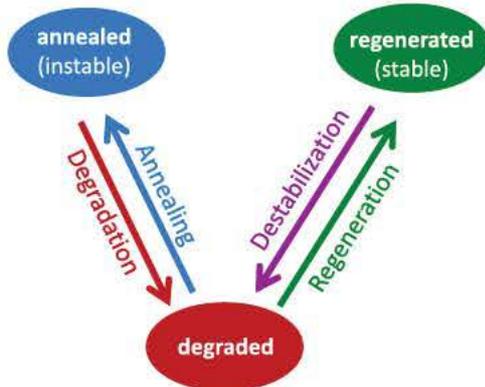


Fig. 1. Transition paths of the metastable BO defect. Annealing and destabilization occur in the dark, whereas degradation and regeneration only occur under carrier injection. All reactions are thermally activated.

**Table 1**  
Thermal activation energy  $E_A$  and transition rates at 200 °C used for simulation purposes. Time constant at temperatures deviating from 200 °C are altered according to Arrhenius' law.

	$E_A$ (eV)	Rate at 200 °C (min <sup>-1</sup> )
Annealing	1.3 [25,26]	4.0 [25]
Degradation	0.4 [27,28]	15.5 (extrapol. from 25 °C) [27]
Regeneration	1.0 [30]	0.03 [29] (slow), 24.0 [30] (fast)
Destabilization	1.25 [30]	0.015 [29]

active simultaneously. Hence, the rates of all of them determine how the occupancy of the three BO states evolves during such treatment. In the following section, this will be investigated by means of calculations using the 3 state model. The equations used for simulation purposes can be found in [22]. All transition reactions are thermally activated, but differ considerably in activation energy (see Table 1). Hence, the influence of the four transition reactions on the evolution of the occupancy of the different defect states will be evaluated at different temperatures. The time constants/transition rates at temperatures deviating from 200 °C are calculated according to Arrhenius' law. The activation energy of the regeneration reaction assumed for the simulation presented here was measured in uncompensated samples since there is no confirmed value for compensated wafers (see [19] for the only published value). Please note that regeneration rates can only be measured reliably if the regeneration process is complete and the resulting carrier lifetime is stable under further illumination at lower temperature. If this is not the case, the measured transition rate is very probably a superposition of the desired regeneration and an unwanted annealing reaction. Hence, neither the extracted rates nor activation energies calculated from such measurements can clearly be assigned to the regeneration of BO defects. The activation energy of the destabilization reaction has not been measured in compensated wafers, yet. Thus, the activation energy values for compensated samples might deviate slightly from the values assumed here without the basic message being changed.

### 2.1. Influence of regeneration rate on BO kinetics

In this section, the influence of different regeneration rates on BO kinetics is discussed. It is assumed that a silicon wafer containing boron as well as oxygen is exposed to carrier injection at 200 °C. The simulation shown in Fig. 2 compares BO kinetics for fast ( $R_{\text{Reg}} = 24 \text{ min}^{-1}$ ) and slowly ( $R_{\text{Reg}} = 0.03 \text{ min}^{-1}$ ) regenerating samples keeping all other transitions constant. Experimentally, this situation can be realized by comparing a well

hydrogenated sample (regenerating quickly) to a poorly hydrogenated one (regenerating slowly) or by changing the illumination intensity.

Apart from the latter samples reaching a stable distribution of BO states later, regeneration becomes incomplete in the slowly regenerating sample (Fig. 2, right): ~40% of the defects stay either in the degraded or in the annealed state implicating that lifetime is not permanently stabilized at the intended high level. If the time resolved progress is not monitored, this effect may be misinterpreted in the way that the treatment duration was simply chosen to short, e.g., stopping it at  $t=0.1 \text{ min}$  in Fig. 2 (left) and treating the sample for longer times ( $> 1 \text{ min}$ ) would still lead to the intended result. However, as can be seen in Fig. 2 (right) for the shown slow regeneration rate, once the system has reached its final equilibrium state, extending the treatment duration will not change the situation. Besides, illumination at lower temperature would result in renewed degradation of the unintentionally annealed defects. This renewed degradation can be mistaken for the regenerated state to be instable.

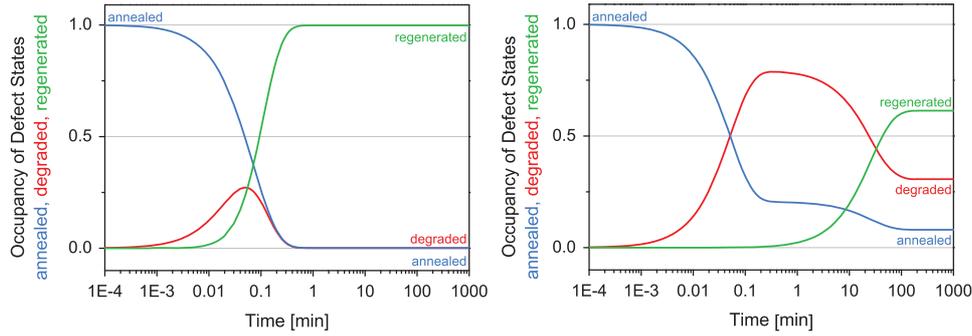
When having a look at the equations describing the evolution of the different defect states (see [22] for details), it becomes clear why the regeneration rate at a given temperature influences the completeness of the actual regeneration process: If the destabilization rate is in the same order of magnitude as the regeneration rate, e.g., for slowly regenerating samples, the regenerated BO state empties during the process to a considerable extent meaning that the long term equilibrium shifts away from the regenerated state. Thus, the occupancy of the regenerated state after saturation depends on the ratio of regeneration and destabilization rate meaning on the regeneration rate if temperature and therefore the destabilization rate is kept constant. Hence, the occupancy of the regenerated state in equilibrium changes with regeneration time constant and there exists a critical regeneration time constant for each temperature (e.g., ~1 min at 200 °C, see Fig. 3 left) beyond which the regeneration process cannot be complete ( $> 99\%$ ) anymore because regeneration is not fast enough compared to the destabilization reaction. To reach 99% complete regeneration, a ratio of at least  $R_{\text{Reg}}/R_{\text{Dest}} \approx 100$  is needed. This ratio automatically decreases for slowly regenerating samples so that, e.g., in samples with a regeneration time constant of 50 min at 200 °C only 50% of all BO defects can be transformed into the stable regenerated state, as depicted in Fig. 3.

### 2.2. Consequences and limitations for high speed regeneration processes

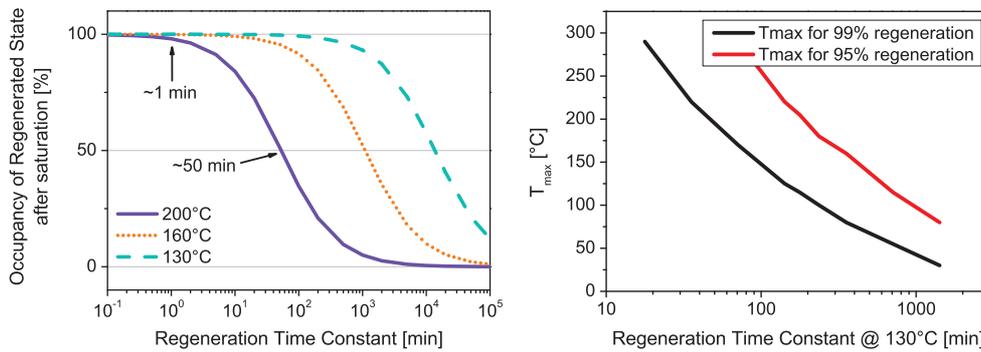
As explained in the last section, the completeness of the regeneration process depends on the ratio of regeneration ( $R_{\text{Reg}}$ ) and destabilization ( $R_{\text{Dest}}$ ) rates. To reach 99% complete regeneration, a ratio of at least  $R_{\text{Reg}}/R_{\text{Dest}} \approx 100$  is needed, 95% complete regeneration requires at least  $R_{\text{Reg}}/R_{\text{Dest}} \approx 25$ . Since destabilization has a higher ( $> 1.25 \text{ eV}$ ) activation energy than regeneration (1.0 eV) (see Table 1), the ratio of their rates decreases automatically with increasing temperature meaning that regeneration becomes more and more incomplete. Hence, temperature cannot be increased deliberately (without changing the sample processing or increasing illumination during the process). This is a very important result for industrial application of regeneration.

The maximum temperature that is possible without compromising the regeneration completeness can be determined by the following procedure:

1. Measure the regeneration time constant of a specific sample at a temperature that is low enough for the regeneration to be measurable and complete, e.g., 130 °C. This will determine whether the sample is fast (time constant in the order of



**Fig. 2.** Evolution of the occupancy of the different BO defect states during simulated regeneration treatment of Si wafers at 200 °C under carrier injection. (Left) Fast regenerating sample: saturates quickly at 100% regeneration. (Right) Slowly regenerating sample: saturation is reached later and only ~60% of all BO defects are in the regenerated state.



**Fig. 3.** (Left) At a given temperature, the regeneration time constant determines what ratio of BO defects is regenerated once equilibrium is reached. (Right)  $T_{\max}$  allowed for the regeneration process in dependence of regeneration time constant measured at 130 °C, 1.1 sun for 99% (95%) complete regeneration.

minutes at 130 °C) or slowly (time constant in the order of hours at 130 °C) regenerating.

2. Extrapolate the regeneration time constant to higher temperatures using the Arrhenius correlation. The destabilization time constant at the respective temperatures can be found in the literature [30] for several temperatures and can be extrapolated the like.
3. Calculate  $R_{\text{Reg}}/R_{\text{Dest}}(T)$ .  $T_{\max}$  is the temperature where  $R_{\text{Reg}}/R_{\text{Dest}}$  equals 100 (or any other desired value, e.g., 25 for 95% completeness).

Fig. 3 (right) shows how far temperature can be increased in the case of samples featuring certain regeneration time constants at standard regeneration temperature of 130 °C without reducing the completeness of the regeneration process.

These results are now used to understand the regeneration behavior of compensated p type as well as n type samples.

### 3. BO-regeneration measurements in compensated silicon

#### 3.1. Experimental details

Samples investigated in this experiment are made from Si wafers differently doped with B, P and Ga. The dopant concentrations are given in Table 2. They were calculated using Scheil's equation; the interstitial oxygen concentrations were measured with FTIR (Fourier transformed infrared spectroscopy). All wafers were laser cleaved into smaller pieces, saw damage etched in hot NaOH, followed by cleaning in HCl and dipping in diluted HF. Then, a  $\text{POCl}_3$  based gettering step was applied at high temperature (~1 h at ~840 °C). The diffused layer was removed thereafter in hot NaOH. After RCA cleaning, a hydrogen rich PECVD

**Table 2**

Concentration of dopants and interstitial oxygen in the investigated samples.

Sample	[B] in $10^{16} \text{ cm}^{-3}$	[P] in $10^{16} \text{ cm}^{-3}$	[Ga] in $10^{16} \text{ cm}^{-3}$	[O <sub>i</sub> ] in $10^{17} \text{ cm}^{-3}$
B	1.0	0.0	0.0	8
B+P	3.0	1.5	0.0	6
B+P+Ga	3.4	7.8	5.4	7
n-Type	5.7	12.2	5.0	8

$\text{SiN}_x\text{:H}$  was deposited on both sides (75 nm each) using the direct plasma, low frequency PECVD (Plasma Enhanced Chemical Vapor Deposition) technique in a Centrotherm system. Finally, all samples were fired in a belt furnace at around 800 °C peak temperature in order to activate the  $\text{SiN}_x\text{:H}$  surface passivation and to release hydrogen from the layer into the silicon bulk. From every material, the so processed lifetime samples underwent a procedure including the following steps:

1. BO annealing at 225 °C in the dark for 15 min.
2. BO degradation at 40 °C, 0.2 sun illumination for ~70 h.
3. BO regeneration at 130 °C, 1.1 sun illumination (standard regeneration) or at 180 °C, 2.7 sun (high speed regeneration) until saturation of minority carrier lifetime.
4. Stability check of minority carrier lifetime at 45 °C, 0.2 sun for 24 h.

Illumination was done by an incandescent halogen lamp. Effective minority carrier lifetime  $\tau_{\text{eff}}(t)$  was measured after each step and every few minutes during the regeneration process at an injection of 10% of the doping level using a WCT 120 lifetime tester from Sinton Instruments. Lifetime values were transformed into normalized BO defect concentrations  $N^*(t)$  using  $N^*(t) = 1/\tau(t) \cdot 1/\tau_{\text{ann}}$  with the lifetime in the annealed state  $\tau_{\text{ann}}$  not being affected by BO defects.  $N^*(t)$  is fitted using a single exponential function yielding the characteristic

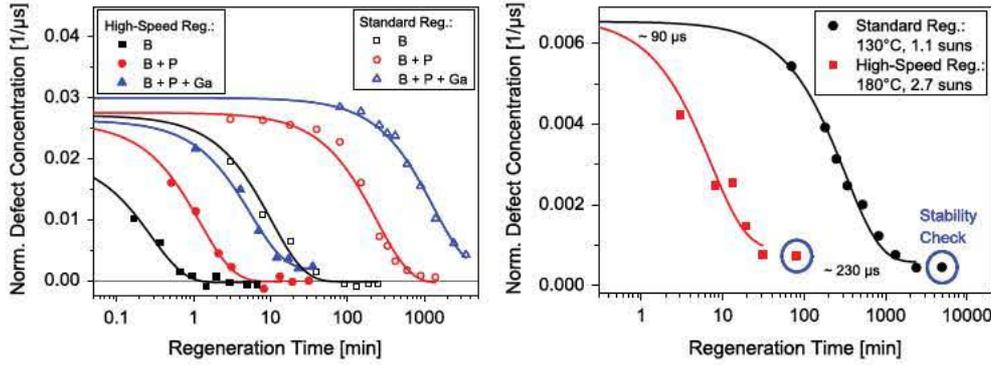


Fig. 4. Evolution of BO defect concentration during a standard (130 °C, 1.1 sun; open symbols) and an adapted high-speed (180 °C, 2.7 sun; filled symbols) regeneration process for p-type (left) and n-type (right) compensated silicon (different scaling!). The latter process leads to acceleration of the regeneration reaction by two orders of magnitude. The stability of the regenerated state has been proven for all samples.

regeneration time constant  $t_{\text{Reg}}$  and its inverse, the regeneration rate  $R_{\text{Reg}}$ , that are both used to compare the regeneration kinetics of different samples. To get a time resolved measurement of the high speed regeneration process, the respective samples were degraded at 45 °C, 0.2 sun until saturation after each regeneration step to ensure that no defects were unintentionally transformed into the instable annealed state that would at first glance result in the same increasing lifetime values. After that, minority carrier lifetime is measured and its evolution with time is analyzed as for the standard regeneration process.

### 3.2. Results

Fig. 4 shows the evolution of the BO defect concentration during the standard (130 °C, 1.1 sun) and an adapted high speed regeneration process that is characterized by the use of higher temperature and stronger illumination (180 °C, 2.7 sun). At first glance, a tendency can be seen that the non compensated samples regenerate the most quickly ( $R_{\text{Reg}} \approx 2.7 \text{ h}^{-1}$ ), followed by the samples partially compensated with phosphorus ( $R_{\text{Reg}} \approx 0.4 \text{ h}^{-1}$ ) and finally by the samples additionally containing gallium ( $R_{\text{Reg}} \approx 0.01 \text{ h}^{-1}$ ). Their regeneration rates differ from the first and the second group by one, respectively two orders of magnitude. Minority carrier lifetime of all regenerated samples is stable under further illumination (0.2 sun, 45 °C). The same is true for the n type compensated samples and in both cases the high speed process allows acceleration of the regeneration reaction by two orders of magnitude. Hence, our procedures are suited to regenerate successfully BO defects in both p and n type compensated silicon and the final state is characterized by high lifetime and stability under carrier injection.

The general trend found by Lim et al. [18] saying that regeneration is slowed down by increasing boron concentrations can be confirmed. This is what one would expect if the regeneration of BO defects can be explained by mobile hydrogen atoms passivating the recombination active defects because boron is known to trap hydrogen [31 and many others]. Hence, increasing boron concentration decreases the concentration of mobile hydrogen atoms within the wafer and can reduce its diffusivity. Therefore, higher boron concentrations would result in a slower regeneration process. But more data is needed to investigate a possible additional influence of phosphorus or gallium on the regeneration of BO defects. This question will be addressed in a separate publication [32].

The regenerated state being stable under illumination in n type silicon has direct consequences on the physical model of the BO regeneration. Sun et al. [33] explained that in n type Si the regenerated state could not be stable under carrier injection if regeneration is ionic interaction between hydrogen and BO defects. Since Niewelt et al. [34] recently presented convincing data on the long time stability of the minority carrier lifetime of

BO regenerated compensated n type wafers under illumination, we can conclude that the deactivation may rather be achieved by covalent binding between hydrogen and BO defect than by ionic interaction. Another explanation would be hydrogen having a catalytic effect on BO regeneration. See [34] for details.

### 3.3. Adapting the high speed regeneration process

The non compensated samples presented in this paper regenerate fast enough ( $t_{\text{Reg}} \approx 45 \text{ min}$  at 130 °C) for the regeneration temperature to be increased to  $T_{\text{max}} > 200 \text{ °C}$ . In contrast, for the samples compensated with boron and phosphorus,  $t_{\text{Reg}}$  is  $\sim 120 \text{ min}$  at 130 °C which corresponds to  $T_{\text{max}} \approx 130 \text{ °C}$ . Hence, temperature cannot be increased unless regeneration is additionally enhanced: Increasing the illumination intensity can compensate for otherwise decreasing completeness at higher regeneration temperature. E.g., using 2.7 sun illumination instead of 1.1 sun can halve the regeneration time constant. Then, temperature can be increased to  $\sim 180 \text{ °C}$  without problems. Therefore, the high speed regeneration process is adapted to these conditions and is carried out at 180 °C, 2.7 sun. As a matter of fact, samples that regenerate even more slowly, e.g., the p and n type samples co doped with B, Ga and P, cannot regenerate completely under those conditions. This can be seen in Fig. 4, where the samples co doped with boron, gallium and phosphorus saturate at a defect concentration level that is above zero.

### 3.4. Discussion on “incomplete” regeneration processes

The correlation between regeneration rate and its completeness can also explain why earlier studies [19,21] found that minority carrier lifetime was not stable after “regeneration” of compensated p and n type silicon. The “regeneration” process typically was carried out at  $\sim 200 \text{ °C}$ , 1 sun [21]. The determined regeneration rates in the order of  $1 \text{ h}^{-1}$  at 200 °C, e.g., due to poor hydrogenation of the silicon bulk, are apparently too slow to apply such high regeneration temperatures without compromising the completeness of the regeneration process, and a considerable number of BO defects will unintentionally transform into the instable annealed state. Hence, a following stability test under degradation conditions (e.g. 45 °C, 0.2 sun) would again lead to BO related degradation of the latter defect species; but this does not mean that the regenerated BO state is instable itself. This problem becomes the more important the lower the regeneration rates are within a sample, hence it might even seem as if regeneration was impossible in the extremely slowly regenerating compensated n type samples [21].

As opposed to this, in the investigations presented here, we applied two regeneration procedures that both meet the requirements for complete regeneration: a standard low temperature

regeneration process at 130 °C, 1.1 sun illumination and a high speed regeneration process at 180 °C, 2.7 sun illumination, both carried out on hydrogenated samples.

#### 4. Summary and conclusions

The regeneration of BO defects was investigated in hydrogenated lifetime samples made from differently compensated p and n type silicon wafers.

A procedure that is able to transform BO related defects into the regenerated state characterized by high lifetime and stability under subsequent illumination was presented for the first time for n type compensated silicon. Comparable to p type samples, the regeneration process could be accelerated by two orders of magnitude by applying an adapted high speed regeneration procedure. No differences in the stability of the regenerated state or in the completeness of the regeneration process were found on condition of choosing suitable regeneration parameters. Those parameters were determined by simulating the influence of different regeneration rates on the completeness of the regeneration process based on the 3 state model of BO defects. Maximum regeneration temperatures that can be applied were determined depending on the regeneration rate measured at standard regeneration conditions (130 °C). The results were used to adapt the high speed regeneration process without compromising neither the completeness of the regeneration process nor the stability of the resulting high minority carrier lifetime values.

#### Acknowledgments

The authors would like to thank ApollonSolar for providing the Si material, L. Mahlstaedt and T. Wiedenmann for technical support during sample preparation and S. Stoyanov for regeneration measurements. Part of this work was funded by the German BMU under Contracts 0325581 and 0325450A. The content is the responsibility of the authors.

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