



On improved passivation stability on highly-doped crystalline silicon and the long-term stability of regenerated Cz-Si

David Sperber*, Axel Herguth, Giso Hahn

Department of Physics, University of Konstanz, Universitätsstr. 10, 78464 Konstanz, Germany

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ABSTRACT

Different surface passivation approaches are applied on Cz-Si and FZ-Si samples and long-term stability is investigated during treatments at 60–80 °C and up to 1 sun equivalent illumination intensity. It is shown that $\text{SiN}_x\text{:H}$ and $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ surface passivation show a much more stable passivation quality when deposited on P-diffused and B-diffused surfaces, respectively. Long-term measurements lead to the conclusion that Cz-Si samples fired at measured peak temperatures up to 750 °C are very stable after regeneration of bulk defects. Samples fired at 850 °C show much stronger bulk-related degradation potentially linked to light and elevated temperature induced degradation (LeTID). Furthermore, Cz-Si samples fired at 850 °C express an instable behavior after a regeneration treatment.

1. Introduction

A high bulk minority carrier lifetime τ_b is required in solar cells to enable high conversion efficiencies. It is well known that light induced degradation (LID) during operation of solar cells may degrade τ_b significantly, e.g., due to the formation of boron-oxygen (BO) related defect centers [1–4] or copper related LID [4]. A few years ago, it was discovered that another type of LID, frequently called light and elevated temperature induced degradation (LeTID), may significantly decrease τ_b , too [5–7]. Both BO-LID and LeTID can be regenerated during or after sample preparation, allowing for high and supposedly stable τ_b [8–11]. However, the amount of studies on the long-term stability of regenerated Czochralski silicon (Cz-Si) samples is rather sparse. In [12–14], slight instabilities have been observed during stability tests of Cz-Si samples after a regeneration treatment and it has been suspected that the formation of defects not related to BO-LID are the main cause of these instabilities.

Usually, it is not τ_b but the effective minority carrier lifetime τ_{eff} which is measured in LID experiments and influenced by recombination in the bulk and at the surface of a sample. Accordingly, surface related degradation (SRD) as observed in [15–17] can pose a significant challenge for light induced degradation studies aiming at changes in the silicon bulk [16]. Especially in long-term stability studies, even slight changes in surface passivation quality can easily be misinterpreted as changes apparently occurring in the bulk. Fig. 1(a) shows a long-term stability measurement conducted after a regeneration treatment of a lifetime sample made from non-diffused B-doped Cz-Si passivated with

$\text{SiN}_x\text{:H}$. As can be seen, τ_{eff} decreases significantly after several thousand hours of treatment at 60 °C and 0.1 suns. This long-term decrease could easily be interpreted as an instability of the regenerated state of the BO related defect. However, the simultaneous rise of the surface related saturation current density J_0 indicates that this degradation is surface related.

Passivation with $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ stacks results in more stable passivation quality compared to single $\text{SiN}_x\text{:H}$ layers on non-diffused silicon [16]. After prolonged treatment at 80 °C and 1 sun, however, even an $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ passivated sample is slightly affected by SRD as can be seen by rising values of J_0 in Fig. 1(b) (note the different scale). Accordingly, both passivation approaches are not ideally suited for the investigation of long-term stability of τ_b .

Significant degradation of $\text{SiN}_x\text{:H}$ based passivation already at 60 °C and 0.1 suns additionally raises the question if solar cells could suffer from SRD, too. However, $\text{SiN}_x\text{:H}$ is usually not applied on B-doped silicon but on highly P-doped emitter layers. At treatment temperatures of 150 °C, we have recently observed a significant reduction of SRD on float-zone silicon (FZ-Si) samples with diffused surfaces ([17], to be published). Accordingly, heavily doped surfaces could be useful both for an enhanced stability of solar cells and for long-term LID studies on lifetime samples.

In this contribution, the stability of passivation quality of dielectrically passivated diffused surfaces will be examined at lower temperatures typical for solar cell operation or long-term stability studies. Advantages and disadvantages of using diffused layers in LID experiments will be discussed and the application of different passivation

* Corresponding author.

E-mail addresses: david.sperber@uni-konstanz.de (D. Sperber), axel.herguth@uni-konstanz.de (A. Herguth), giso.hahn@uni-konstanz.de (G. Hahn).

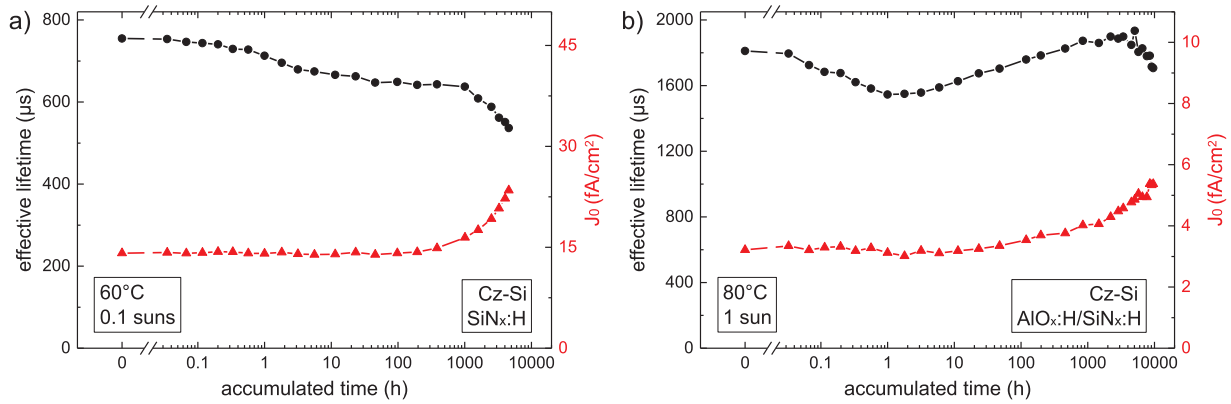


Fig. 1. Measurement of τ_{eff} and J_0 of B-doped Cz-Si samples (doping density $N_d \approx 7 \cdot 10^{15} \text{ cm}^{-3}$) passivated with (a) $\text{SiN}_x\text{:H}$ and (b) $\text{AlO}_x\text{:H/SiN}_x\text{:H}$. Both samples were fired at $\sim 750^\circ\text{C}$ and received a treatment at 150°C and 1 sun for 12 min to regenerate bulk defects. Samples were then tested for long-term stability at 60°C and 0.1 suns or 80°C and 1 sun as shown in the graphs.

schemes gives new insight into the long-term stability of regenerated Cz-Si samples fired at different temperatures.

2. Material and methods

If not stated otherwise, samples were made of B-doped FZ-Si or Cz-Si wafers with doping density $N_d \approx 7 \cdot 10^{15} \text{ cm}^{-3}$. The wafers were etched in KOH to a thickness $d \approx 180 \mu\text{m}$ and received a chemical polish (CP) in a solution of acetic acid, HNO_3 and HF. To clean wafer surfaces, the wafers were oxidized in a solution of H_2O_2 and H_2SO_4 at 80°C and the resulting surface oxide was removed in diluted HF (Piranha clean). Wafers were then processed as shown in Fig. 2. Diffusion in POCl_3 ambient was carried out at 840°C and resulted in heavily P-doped n^+ layers with sheet resistance $\sim 55 \Omega/\text{sq}$. The resulting phosphosilicate glass at the sample surface was removed in diluted HF and the emitter was etched back to $\sim 120 \Omega/\text{sq}$ in an aqueous solution of HF, HNO_3 and NaNO_2 to reduce the impact of the heavily P-doped surface layer (kink region) on recombination [18]. Other samples received a BBr_3 diffusion at a drive-in temperature of 910°C followed by etching in diluted HF to remove the boron silicate glass at the sample surface. This resulted in p^+ doped layers with sheet resistance $\sim 100 \Omega/\text{sq}$. Diffused samples received another Piranha clean. Other wafers received no diffusion to serve as reference samples.

P-diffused wafers and references were coated with $\sim 75 \text{ nm}$ $\text{SiN}_x\text{:H}$ using a direct plasma enhanced chemical vapor deposition (PECVD) at 450°C and 40 kHz. B-diffused wafers and references received a passivation stack consisting of 10 nm $\text{AlO}_x\text{:H}$ grown by atomic layer deposition (ALD) at 300°C capped with $\sim 75 \text{ nm}$ $\text{SiN}_x\text{:H}$ in a remote PECVD at 400°C and 2.45 GHz. $\text{SiN}_x\text{:H}$ layers had a refractive index $n \sim 2.0$ at 600 nm. All diffusions and coatings were applied to both wafer

sides, resulting in symmetrical samples. After laser-cutting to an edge length of 5 cm, samples were fired in a fast firing belt furnace at a belt speed of 6000 mm/min. Peak temperatures during firing were measured at the sample surface using a sheathed type K thermocouple of diameter 0.25 mm (Omega HKMQIN-IM025U-500). Due to varying hydrogen content in the different layers and differences in $\text{SiN}_x\text{:H}$ deposition temperatures, the degree of bulk and surface hydrogenation of $\text{SiN}_x\text{:H}$ and $\text{AlO}_x\text{:H/SiN}_x\text{:H}$ passivated samples may vary significantly after firing. After processing, samples were stored in darkness until measurement series were carried out.

In measurement series samples were treated on hotplates at constant temperature and halogen lamp illumination as indicated in each measurement. The illumination intensity is given in sun equivalents ("suns") where one sun equivalent illumination was achieved by matching the short circuit current of a solar cell to that under a solar spectrum simulator [19]. During treatment, τ_{eff} was repeatedly measured using the generalized mode [20] of a Sinton Instruments lifetime tester (WCT-120) [21] at 30°C . If not stated otherwise, τ_{eff} is shown at an excess minority carrier density $\Delta n = 0.1 \cdot N_d$.

The surface related saturation current density J_0 was extracted from lifetime data according to the method described in [22]. For samples with diffusion J_0 equals the emitter saturation current density J_{0e} , while for non-diffused samples J_0 equals the surface saturation current density J_{0s} [23].

Selected samples had their passivation layer removed after a measurement series by etching in 10% HF. In a next step, $\sim 4 \mu\text{m}$ were removed on each sample side using a CP etch before samples received two Piranha cleans. Afterwards, samples were wet-chemically passivated using a 0.08 M iodine ethanol solution [24,25] and τ_{eff} was measured immediately thereafter.

3. Results and discussion

3.1. Stability of $\text{SiN}_x\text{:H}$ passivated samples

Fig. 3 shows measurement series conducted on $\text{SiN}_x\text{:H}$ passivated samples with (full symbols) and without (empty symbols) P-diffused surface fired at $\sim 750^\circ\text{C}$ measured sample temperature. All samples were treated at 80°C and 1 sun illumination. While the Cz-Si samples (red) show a pronounced bulk related minimum I and maximum II in the first hours of treatment as expected due to BO degradation and regeneration, the FZ-Si samples (black) show only minor instabilities during the first hours. After prolonged treatment, samples without emitter (-p-) show significant SRD leading to a pronounced decrease of τ_{eff} correlated with a significant rise of J_0 in good agreement with results discussed in [15,16].

We have observed before that P-diffused (n^+pn^+) samples made of

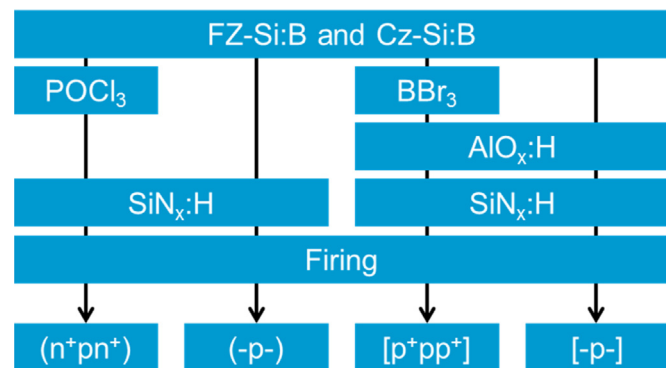


Fig. 2. Process flow and labeling of samples. Sample names indicate the sample structure and different brackets relate to different dielectric passivation layers.

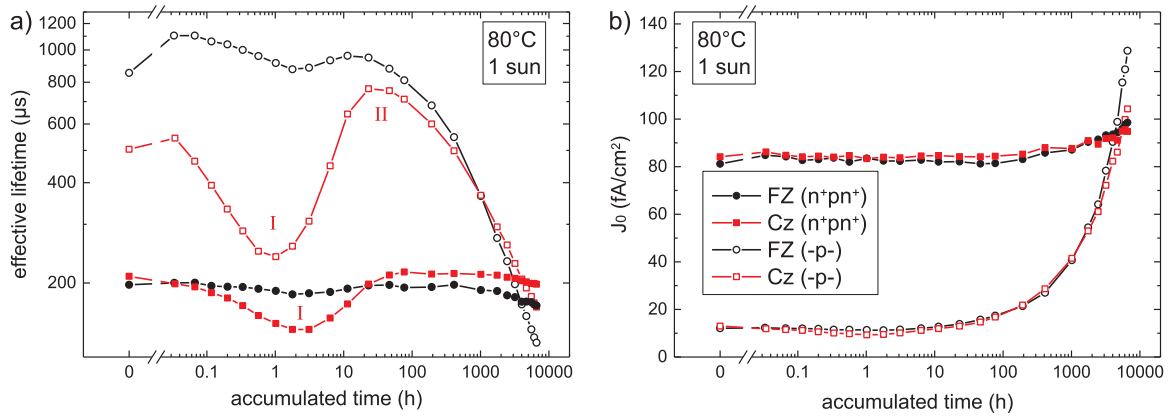


Fig. 3. (a) Measurement of τ_{eff} of samples with and without P-emitter during treatment at 80 °C and ~ 1 sun illumination. (b) Calculated values of J_0 of the same samples.

FZ-Si are significantly more stable in terms of SRD compared to (-p-) samples when treated at 150 °C and 1 sun ([17], to be published). As can be seen in Fig. 3, this remains true at 80 °C for both FZ-Si and Cz-Si samples: SRD of (n^+pn^+) samples is much weaker and after several thousand hours of treatment, both τ_{eff} and J_0 of (-p-) samples cross the values of (n^+pn^+) samples.

Although they show an improved stability, the (n^+pn^+) samples are very much surface limited right from the start which lowers the sensitivity to changes in τ_b significantly. Additionally, they still show a non-negligible degradation of up to 15 fA/cm² on long time scales, and hence this passivation approach is still not well suited for long-term stability studies aimed at changes in the silicon bulk on a high lifetime level. An (n^+pn^+) structure could, however, be useful for the investigation of LeTID: Formation of LeTID has been shown to depend strongly on choice of passivation layers and strongest degradation is observed for H-rich SiN_x:H passivation [26]. Hence, passivation with SiN_x:H is beneficial for LeTID studies and an (n^+pn^+) structure then improves passivation stability significantly. As lifetime drops well below 100 μ s in case of strong LeTID, the rather low sensitivity of an (n^+pn^+) structure is not a big problem anymore. Additionally, the enhanced stability might be helpful in solar cell concepts because potential SRD of SiN_x:H layers should be weaker on a phosphorous emitter.

3.2. Enhanced stability of AlO_x:H/SiN_x:H passivation on B-diffused silicon

As already discussed in [16], AlO_x:H/SiN_x:H stacks offer higher passivation stability compared to SiN_x:H on lowly doped silicon. AlO_x:H/SiN_x:H passivated FZ-Si samples with and without B-diffusion,

fired at 800 °C measured sample temperature, and treated at 80 °C or 150 °C and 1 sun are shown in Fig. 4. After an initial rise of τ_{eff} , which is probably caused by iron-boron pair dissociation [27], the FZ-Si samples show a bulk related degradation (I) and regeneration (II) as first described in [15], further discussed in [28,29] and probably related to LeTID [30,31]. Stronger degradation of the FZ-Si samples in Fig. 4 compared to Fig. 3 arises due to different sample fabrication: The choice of passivation layers [31], firing temperature [29], and thickness of samples [15] all affect the strength of supposed LeTID in FZ-Si as is the case for LeTID in multicrystalline silicon [26,32,33].

After regeneration of τ_b leading to maximum II, the [-p-] sample with AlO_x:H/SiN_x:H passivation treated at 80 °C and 1 sun shows only slight SRD after 2500 h of treatment, similar to samples investigated by Niewelt et al. [34]. A [p^+pp^+] sample with B-diffusion shows no degradation on this timescale but, on the contrary, a slight increase in τ_{eff} . At a higher treatment temperature of 150 °C, the [-p-] sample shows significant degradation starting at ~ 20 h of sample treatment while the [p^+pp^+] sample remains very stable up to several hundred hours of accumulated treatment time.

Values of J_0 are not shown because extraction of J_0 does not seem to work correctly after prolonged treatment of AlO_x:H/SiN_x:H passivated samples made of B-doped silicon. Capacitance-voltage (CV) measurements on similar samples suggest that a loss of fixed charge causes this behavior. Additionally, determination of very low J_0 values as achieved with AlO_x:H/SiN_x:H passivation has been found to be significantly influenced by strong bulk recombination even after correction measures as described in [22]. Although J_0 analysis is inconclusive in B-doped AlO_x:H/SiN_x:H passivated samples, there is strong evidence that the long-term decline is surface related: The observation that [p^+pp^+]

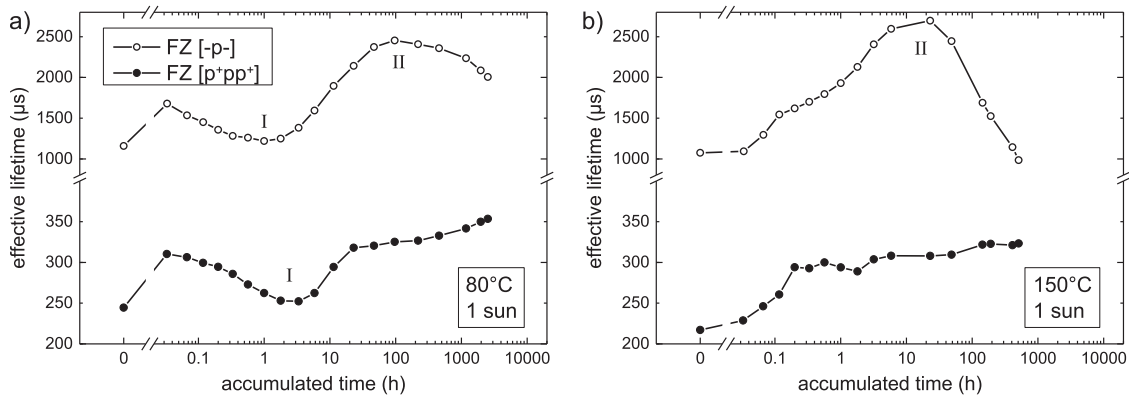


Fig. 4. (a) Measurement of τ_{eff} of B-doped FZ-Si samples with and without B-diffused layer, fired at 800 °C, and treated at 80 °C and 1 sun. (b) Identically processed samples treated at 150 °C and 1 sun. Data of (b) taken from [17]. All samples were made of B-doped FZ-Si ($N_d \approx 1.5 \cdot 10^{16} \text{ cm}^{-3}$, $d \approx 250 \mu\text{m}$) and only received a dip in diluted HF prior to sample processing. Further processing was carried out as described for [-p-] and [p^+pp^+] samples in Fig. 2.

samples show a completely different long-term behavior compared to [-p-] samples already indicates a surface related phenomenon as both sample types mainly differ in their surfaces. Furthermore, P-doped samples with the same passivation stack show a similar long-term decline of τ_{eff} correlated with a strong increase in J_0 . Subsequent wet-chemical re-passivation of the sample surface restored the former high level of τ_{eff} in these samples ([17], to be published).

Whether a B-diffused surface is advantageous may depend on the experiment: As long as the treatment temperature is rather low, $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ passivation is rather stable on both highly and lowly doped silicon and, if high sensitivity is most crucial, e.g., in long-term stability studies with only minor expected degradation effects, $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ on base doping should be an appropriate choice. If, however, stability of passivation quality is most important, $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ on a B-diffused surface is better suited, especially at higher treatment temperature. This is, e.g., the case for accelerated degradation and regeneration studies at elevated temperatures. One disadvantage of using a B-diffusion is the requirement of a drive-in step at high temperatures $\sim 900^\circ\text{C}$ which may significantly alter the degradation behavior of a sample compared to a non-diffused sample. It can also be advantageous to compare both diffused and non-diffused sample surfaces in the same experiment as will be shown in the next section.

3.3. Long-term stability of Cz-Si samples fired at different temperatures

Fig. 5 shows FZ-Si and Cz-Si samples passivated with $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ with and without B-diffused layer underneath. The samples were fired at different peak temperatures and first treated at 80°C and 1 sun to study degradation and regeneration in the silicon bulk. Afterwards, samples were treated at 60°C and 0.1 suns to investigate their long-term stability. As can be seen in Fig. 5(a), a measured peak firing temperature of 650°C leads to negligible bulk related degradation in FZ-Si samples. Cz-Si samples, on the other hand, show significant bulk related degradation and regeneration as expected due to BO-LID. The long-term stability is very good for all samples, and hence the regenerated state of BO defects can be considered stable in the Cz-Si samples fired at 650°C .

At 750°C measured peak firing temperature, all samples start at lower τ_{eff} compared to the samples fired at 650°C . The FZ-Si [-p-] sample now shows degradation and regeneration of τ_b to some extent, too. Degradation of the Cz-Si samples leads to degraded τ_{eff} similar to that of samples fired at 650°C , so the severity of BO-LID has not changed significantly in accordance with observations in [35]. Regeneration of the Cz-Si samples occurs faster due to higher firing temperature and/or cooling rate in agreement with [12,36,37]. The long-term stability of all samples is still very good and the regenerated τ_{eff} is on a similar level as in the samples fired at 650°C .

After firing at 850°C , the initial τ_{eff} is much lower for all samples compared to the samples fired at lower temperatures (note the different scale). Now, FZ-Si samples show significant bulk related degradation and regeneration. Cz-Si samples degrade heavily and take a longer time to regenerate. Levels of τ_{eff} after regeneration are still rather low for all samples and during the subsequent stability test, the Cz-Si samples show significant degradation during 2500 h.

As both [-p-] and [p^+pp^+] samples show similar τ_{eff} , it can be suspected that the samples are mainly limited by the silicon bulk. This assumption is further backed up by Fig. 6 showing a wide range of time and injection resolved data gathered during treatment of the Cz-Si samples with and without B-diffusion. As can be seen, both samples differ significantly at higher injection (higher Δn , reddish colors) as would be expected due to the different passivation approaches: The [p^+pp^+] sample is more limited towards higher injection due to additional recombination in the B-diffused layer. Both samples are, however, most limited and look very similar at lower injection (lower Δn , blueish colors).

Fig. 7 shows data of the same samples over an even wider injection

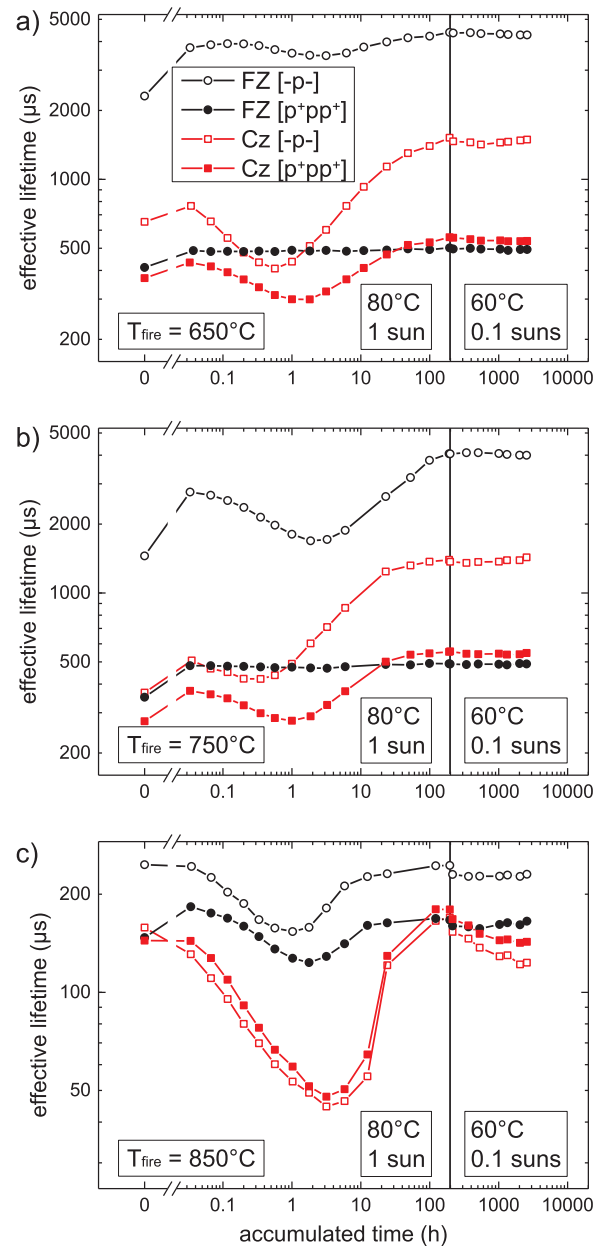


Fig. 5. (a) Measurement of τ_{eff} of B-doped samples with and without B-diffused layer fired at 650°C . Samples were treated at 80°C and 1 sun or 60°C and 0.1 suns. (b) Identically processed samples fired at 750°C . (c) Identically processed samples fired at 850°C .

range but only at characteristic points in time as defined in Fig. 6. The first point A is taken after 2 min of treatment to exclude effects of initial illumination such as FeB dissociation. B relates to the point of maximal degradation, C to data after regeneration and D shows values after the long-term stability test. As can be seen, the slope of the curves changes slightly when comparing A to C and D which indicates that the defect composition of the samples differs to some extent before and after treatment. However, it can be seen that the long-term degradation from C to D does not change the shape of the curve significantly. Again, it can be seen that both samples differ at higher Δn due to different surface passivation but resemble each other and are strongly limited at lower Δn . Such a limitation typically arises due to bulk recombination and indicates that the samples suffer from limited τ_b throughout the whole measurement series. This implies that already the low τ_{eff} at the start of sample treatment arises due to a bulk limitation and that degradation and regeneration, too, are caused in the bulk of the samples.

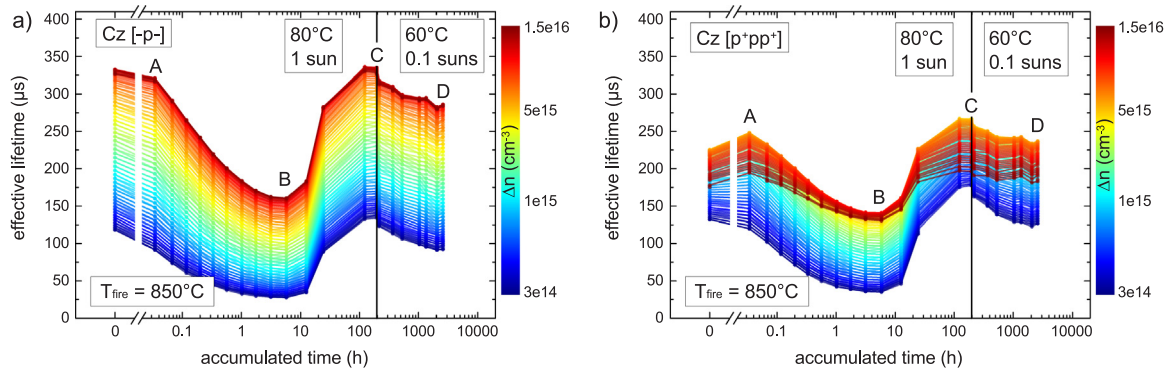


Fig. 6. (a) Injection resolved τ_{eff} of the Cz-Si [-p-] sample fired at 850 °C shown in Fig. 5. (b) Injection resolved τ_{eff} of the Cz-Si [p⁺pp⁺] sample fired at 850 °C shown in Fig. 5.

To verify this assumption, dielectric layers and $\sim 4 \mu\text{m}$ were etched off the sample surfaces after treatment and a wet-chemical iodine ethanol (IE) passivation was applied as described in Section 2. Table 1 shows that both FZ-Si and Cz-Si samples still show very low τ_{eff} after IE passivation. An unprocessed FZ-Si reference sample underwent the same etching and IE passivation procedure and achieved a τ_{eff} of 741 μs , proving that the re-passivation procedure resulted in good surface passivation. Hence, it is not the surface passivation which limits τ_{eff} in the other samples and differences in passivation quality of IE compared to the original sample passivation merely cause slight changes of τ_{eff} compared to values before re-passivation. It can thus be concluded that all samples fired at 850 °C are strongly limited by τ_b .

Fig. 8 shows values of the effective defect density N_{eff} calculated as the difference of inverse τ_{eff} at maximal degradation and after regeneration after ~ 200 h of sample treatment. As can be seen, both FZ-Si and Cz-Si samples show much stronger degradation after firing at 850 °C. It appears possible that both materials are affected by the onset of a similar degradation mechanism triggered by firing at high temperatures. A possible candidate for such a mechanism is LeTID as already proposed for Cz-Si samples in [38,39] and for FZ-Si samples in [30,31]. As a consequence, the long-term instability after regeneration of samples fired at 850 °C appears to not be related to BO-LID and could indicate potential issues with the long-term stability of LeTID affected samples after regeneration treatments. A possible reason for such a long-term instability could be a different reaction equilibrium of defect activation and deactivation at the different treatment conditions during regeneration treatment and testing of long-term stability. The stronger degradation of Cz-Si samples compared to FZ-Si samples could, on the other hand, be related to a different amount of a precursor species suspected to play an important role in LeTID defect formation (e.g., [32,40]).

Table 1

τ_{eff} (μs) of samples fired at 850 °C before and after re-passivation using IE.

	FZ [-p-]	FZ [p ⁺ pp ⁺]	Cz [-p-]	Cz [p ⁺ pp ⁺]	Reference ^a
Before IE	227	145	116	146	–
After IE	189	163	111	146	741

^a Unprocessed FZ-Si sample ($N_d \approx 7 \cdot 10^{15} \text{ cm}^{-3}$, $d = 250 \mu\text{m}$) passivated with IE.

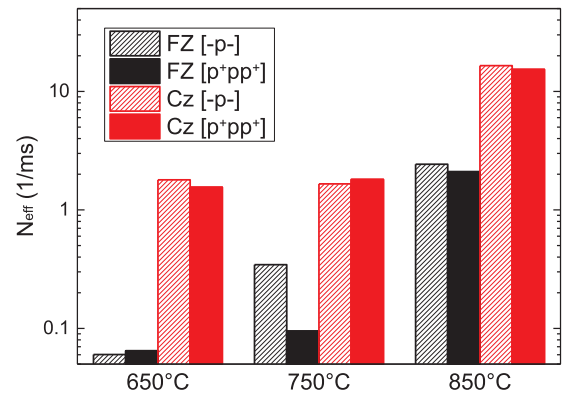


Fig. 8. Maximal N_{eff} reached during degradation of the samples shown in Fig. 5. N_{eff} was calculated using the degraded value of τ_{eff} and the regenerated value after ~ 200 h of sample treatment.

4. Conclusions

It has been shown that the stability of $\text{SiN}_x\text{:H}$ and $\text{AlO}_x\text{:H/SiN}_x\text{:H}$ passivation during illumination at elevated temperature is significantly

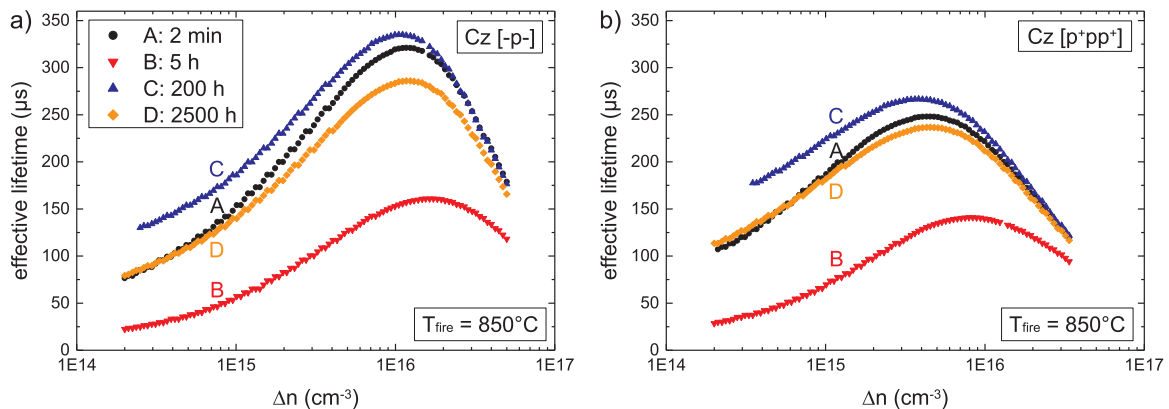


Fig. 7. τ_{eff} over Δn at selected points in time of the (a) Cz-Si [-p-] and (b) Cz-Si [p⁺pp⁺] samples fired at 850 °C and shown in Figs. 5 and 6.

enhanced on highly P- and B-diffused surfaces, respectively. Still, $\text{SiN}_x\text{:H}$ passivation on a P-emitter shows surface related degradation to some degree. Samples with $\text{AlO}_x\text{:H}/\text{SiN}_x\text{:H}$ on B-diffused surfaces are, on the other hand, very stable and well suited for long-term stability studies. Different passivation approaches have been used to investigate the long-term stability of Cz-Si samples after regeneration treatments. Up to measured peak firing temperatures of 750 °C, the only observed long-term degradation effects appeared to be linked to degradation of surface passivation quality while the bulk lifetime of the samples is stable. Firing at 850 °C leads, however, to much stronger LID in both FZ-Si and Cz-Si samples which is potentially linked to LeTID. Accordingly, at high firing temperatures, LeTID might affect even monocrystalline samples much stronger than BO-LID. Cz-Si samples fired at 850 °C furthermore showed a significant instability during long-term stability tests after a regeneration treatment and, thus, long-term stability after LeTID regeneration procedures should be investigated carefully.

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Declarations of interest

None.

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