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Surface Passivation Properties of HfO2 Thin Film on n-type Crystalline Si

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*Abstract*—Atomic layer deposited hafnium oxide is shown to provide good surface passivation of low resistivity, n-type crystalline Si wafers after a low temperature anneal. The surface passivation is related to a fixed negative charge, as well as an excellent interface with the crystalline Si wafer. In this work, the influence of four different deposition parameters on the HfO2 passivation properties, namely pre-cleaning, precursors, deposition temperature and post-annealing temperature, are discussed. Minority carrier lifetime of 1.9 ms (SRV 7.7 cm/s) on FZ n-type wafers and 1.7 ms (SRV 11 cm/s) on Cz n-type wafers under optimized deposition conditions and after a post-annealing process have been measured. These values are the lowest surface recombination velocities reported on this material so far. A significant improvement of the surface passivation is observed after 100 h light soaking, resulting in a carrier lifetime of 2.5 ms. Fitting of the results by a two-defect charge trapping/de-trapping model indicate that additional light-induced negative charges enhance the field effect passivation, which is also consistent with the experimental results. Due to its high refractive index and the obtained good surface passivation of Si wafers, HfO2 has a great potential as a surface passivation material, e.g. in the fabrication of high efficiency Si solar cells.

*Index Terms* — defect density, fixed carriers, atomic layer deposition, HfO2, photovoltaic cells, silicon surface passivation

# INTRODUCTION

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afnium oxide (HfO2, hafnia) deposited with atomic layer deposition (ALD) has been studied widely due to its high dielectric constant (k~25) and its good thermodynamic stability with silicon (Si), which makes it a candidate for replacing thermal SiO2 as the gate dielectric in MOSFETs [1]. In addition, compared to Al2O3, HfO2 has a higher refractive index of ~2 and, hence, it could be used simultaneously for antireflection and surface passivation in Si solar cells [2]. Nevertheless, results regarding HfO2 surface passivation are rarely reported. Lin et al. have demonstrated a good surface passivation with an effective surface recombination velocity (Seff) of 55 cm/s and 24 cm/s on 2.1 Ωcm p-type and 3.3 Ωcm n-type c-Si, respectively [3]. Dingemans et al. have presented surface recombination velocities Seff < 30 cm/s on 3.5 Ωcm n-type c-Si whereas a Seff of ~20 cm/s on 5 Ωcm n-Si was reported by Gope et al., both after annealing at 400 °C for 10 min [4], [5]. The surface passivation yielded by HfO2 seems to vary depending on several factors, most importantly the ALD process used. For instance, in some cases as-deposited HfO2 films have a positive fixed charge density, whereas in other cases it is negative. The postdeposition anneal has been shown to affect the final charge value [3], [7]. It has also been reported that the charge in as-deposited HfO2 is positive, and depending on the film thickness some samples may become negative after annealing [3]. Sreenivasan et al. have also demonstrated that the choice of Hf precursor affects the fixed charge formation in HfO2 using HfCl4 precursor leads to a Cl rich interface and a negative fixed charge that is not affected even by high temperature anneals [6]. However, positive fixed charges have also been reported when HfCl4 precursor was used [7]. This indicates that the charge formation in HfO2 does not depend only on the choice of Hf precursor but also on other parameters related to the ALD process.

Little has been published on the effect of oxidant precursors on HfO2 passivation properties, as well as the effect of deposition temperature, pre-cleaning procedures and post-annealing, which all play important roles on HfO2 films. In this work, influences of all these parameters on the surface passivation properties of HfO2 have been studied. In addition, many passivation materials for silicon wafers undergo light induced degradation effects e.g. SiOx and a-SiNx:H, which might degrade the solar cell performance during operation. It is therefore of interest to investigate how the light soaking influence the HfO2 films.

# Experiment Details

Double-side polished (100) oriented n-type 2.8 Ωcm resistivity 4 inch 290 um float zone (FZ) and 1.0 Ωcm resistivity 6 inch 380 um Czochralski (CZ) Si wafers were used in the experiments. Approximately 20 nm of HfO2 (250 cycles) was deposited by thermal ALD (Beneq TFS-500) on low resistivity n-type wafers using TDMAH (tetrakis(dimethylamino)hafnium) as the Hf precursor. In order to assess the passivation quality as a result of different process variables, the basic setting of four parameters are OH-terminated surface pre-cleaning, H2O as oxidant, 200 °C of deposition temperature and postdeposition anneal at 450 °C for 15 min, shown in Table 1 red. Each time only one variable was adjusted. The effect of different surface pre-cleaning procedures on surface passivation was studied on FZ wafers while CZ wafers were used for the other process parameters experiments, shown in Table 1. One 6 inch wafer was deposited with HfO2 on both sides at basis parameters and cut into 6 pieces. Two of the pieces were used as lifetime samples while the other four were used to measure CV at different times after illumination under a tungsten halogen lamp at 1 Sun (100 mW/cm2) [8]. A spectroscopic ellipsometer was used to measure the film thickness. Injection level dependent minority carrier lifetime was measured with quasi-steady-state photoconductance (QSSPC, Sinton Instruments WCT-120). Since the as-deposited state HfO2 provides very poor surface passivation and lifetime lower than 6 us, not shown here, all the lifetime measured are all taken after the postdeposition heat treatments. Assuming an infinite bulk lifetime, a maximum surface recombination velocity Seff,max was calculated from the lifetime measurements with Seff,max =W⁄(2τeff ), where W was the wafer thickness and τeff  the measured effective lifetime at injection level 1×1015 cm-3 [9]. The interface defect density (Dit) and total charge (Qtot) were measured with the contactless capacitance-voltage method (COCOS, Semilab PV-2000). [10] Lifetime and COCOS measurements were done both before and after the postdeposition heat treatments and also after different illumination times.

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| **Pre-cleaning** | RCA1+HF dip | | RCA1+HF dip+RCA2 | | | | RCA1+HFdip+N2 12h | |
| **Precursors** | O3 | | H2O | | | | O3+H2O | |
| **Deposition Temperature /°C** | 150 | 170 | | | 200 | 250 | |  |
| **Post-annealing Temperature /°C (15 min)** | 300 | 400 | | | 450 | 500 | | 600 |

Table. 1. Experiment parameters setup, each time focusing on one parameter, the other three using basic settings. Three different oxidant variants were used to find the best precursor for surface passivation: i) H2O, ii) O3 and iii) H2O+O3. Four different deposition temperatures of 150 °C, 170 °C, 200 °C and 250 °C were studied. Five postdeposition temperatures of 300 °C, 400 °C, 450 °C, 500 °C and 600 °C for 15 min were explored. (Basic settings in red)

# RESULTS AND DISCUSSION

## Pre-cleaning

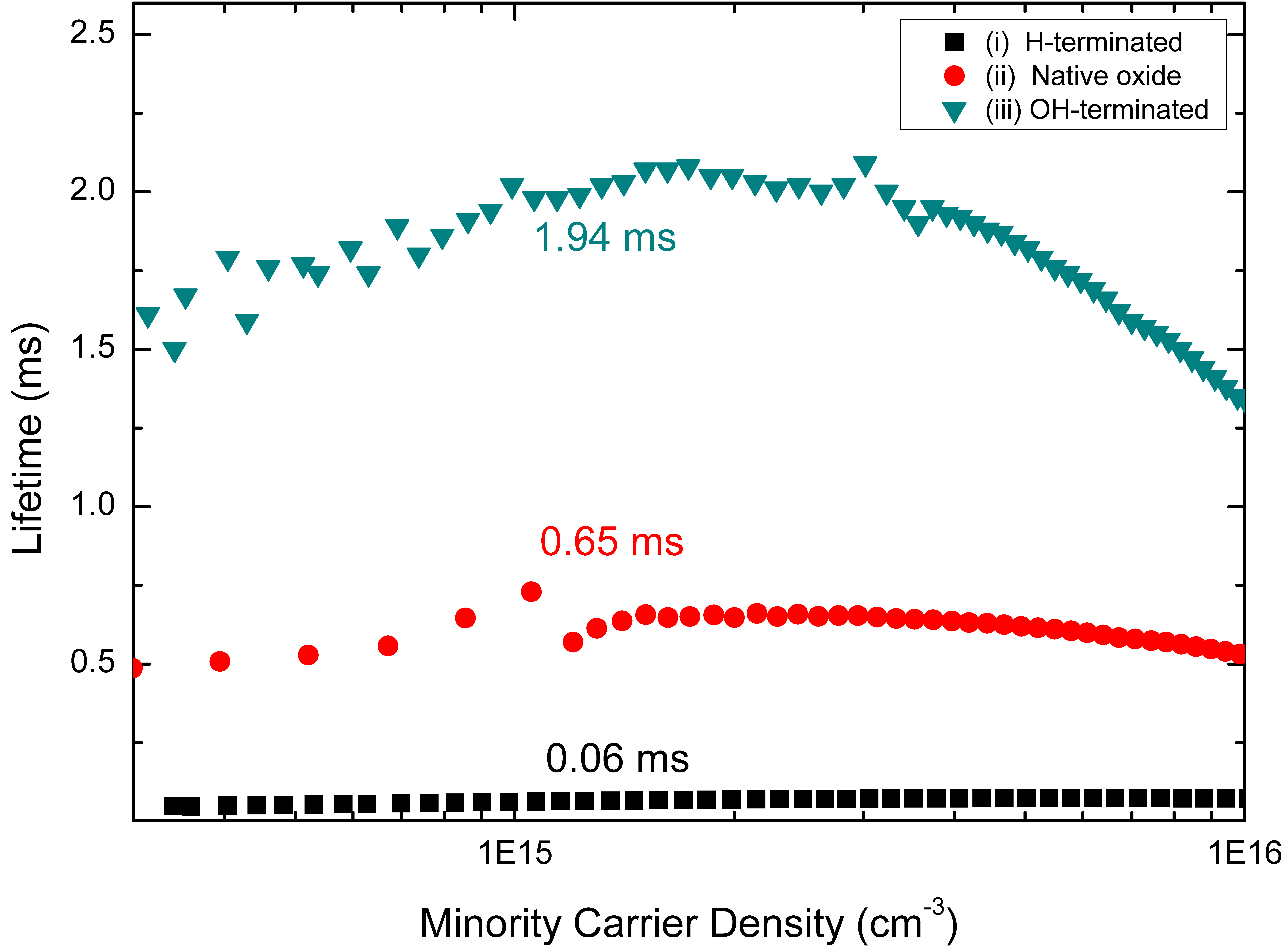
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Fig. 1. Injection level dependent minority carrier lifetime of HfO2 samples after four different pre-cleaning processes before the ALD deposition are shown: (i) H-terminated surface resulting from an RCA1 cleaning procedure followed by a HF dip; (ii) native oxide surface based on (i) and subsequently waiting for 12 h in N2 atmosphere; (iii) OH-terminated surface resulting from (i) plus a step of RCA 2. All samples were annealed at 450°C for 15 minutes. The stated lifetime values are at injection level of 1×1015 cm-3.

Figure 1 shows the injection level dependent lifetimes resulting from applying three different pre-cleaning procedures after postdeposition annealing. The surface passivation quality depends strongly on the applied cleaning procedure. For samples receiving the RCA1 + HF dip, virtually no passivation is obtained indicating that the H-terminated surface is not beneficial for HfO2 passivation of the Si surface. However, surfaces with native oxide (ii) substantially improve the passivation from 0.06 ms to 0.65 ms, indicating that an initially present thin oxide surface is better for HfO2 passivation. The highest lifetime in this work is 1.94 ms and is obtained on samples subjected to (iii) RCA1 + HF Dip + RCA2. The corresponding SRV is ~7.5 cm/s, which is the best reported value so far on HfO2 passivation Si. Although native oxide surface could improve the lifetime from 0.06 to 0.65ms, OH-terminated surface gives a better surface for the precursors to react with, thus forming HfO2 with better passivation quality. These results indicate that the OH- terminated surfaces resulting from the RCA2 treatment is beneficial for HfO2 passivation. This phenomenon can be related to the mechanism of deposition using TDMAHf and H2O on a Si surface during ALD deposition. During each cycle reaction there are two half-reactions. Firstly, the Hf precursor molecule reacts with chemisorbed OH- bonds at Si surface and produces two alkylamine molecules as by-products. In the second half reaction H2O removes the other two alkylamine ligands to form an OH- terminated surfaces for the next cycle of reaction. In a growth mechanism proposed by Liu et.al, a hydroxylated (OH-) surface is suggested to be necessary for initiating the HfO2 deposition. [11, 12] This was an important motivation for the selection of (iv) RCA1 + HF Dip + RCA2 as the standard pre-cleaning procedures.

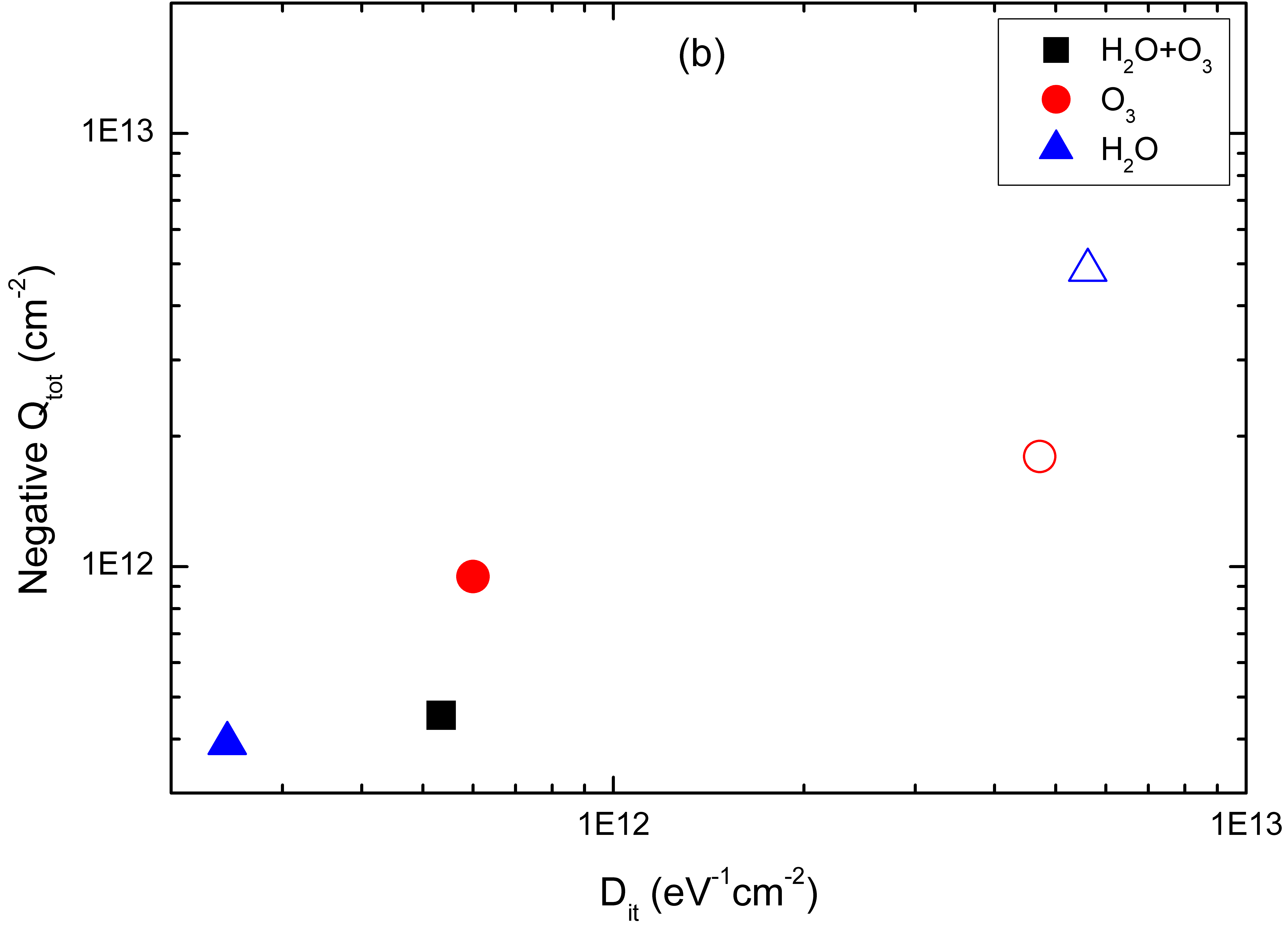
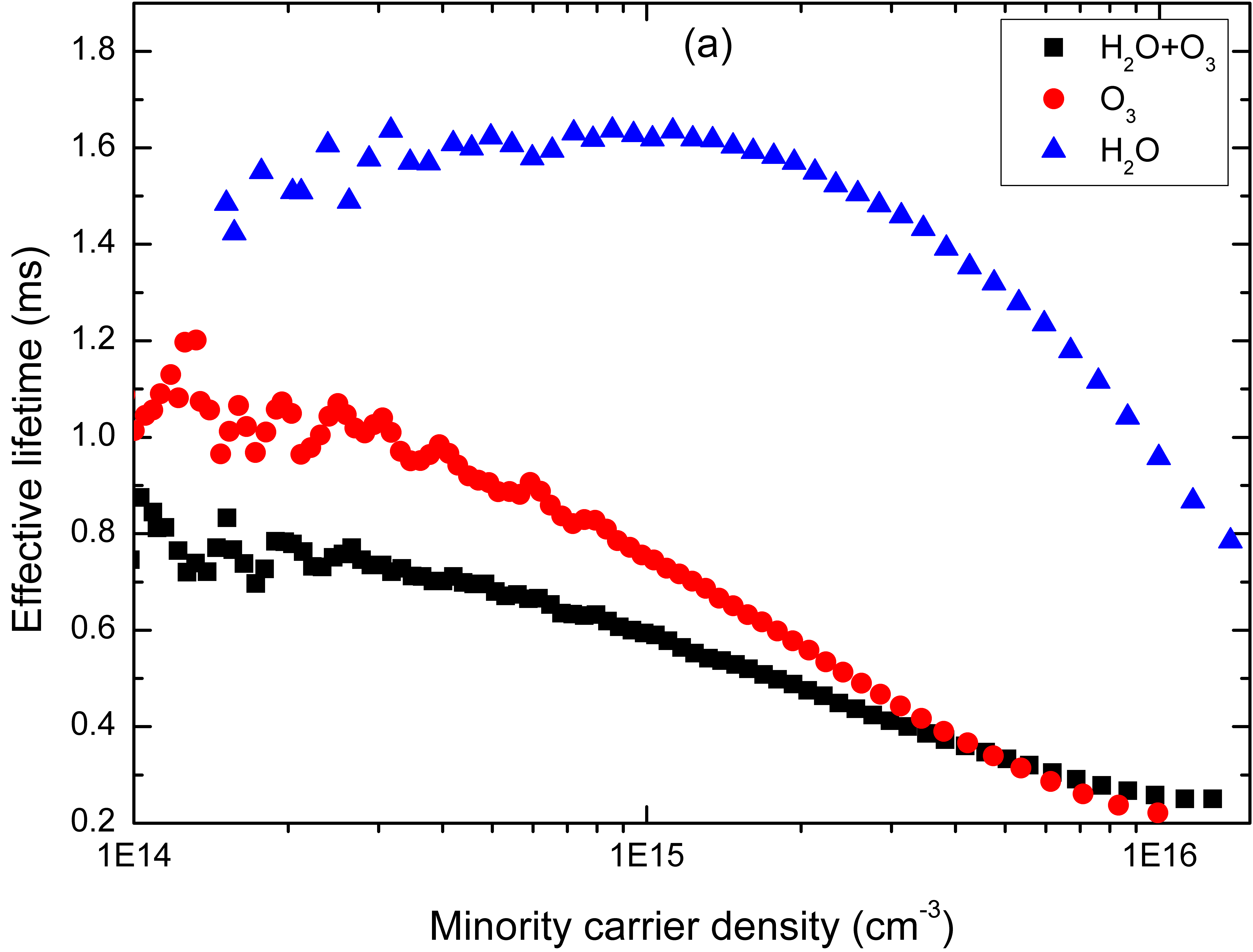


Fig. 2. a) Injection level dependent minority carrier lifetime of HfO2 samples after annealing at 450°C for 15 minutes. Three different oxidants were used in the ALD process: i) H2O, ii) O3 and iii) H2O+O3. The ALD temperature was ~170°C. b) Negative total charge and the interface defect density of HfO2 samples before (open symbols) and after (solid symbols) annealing

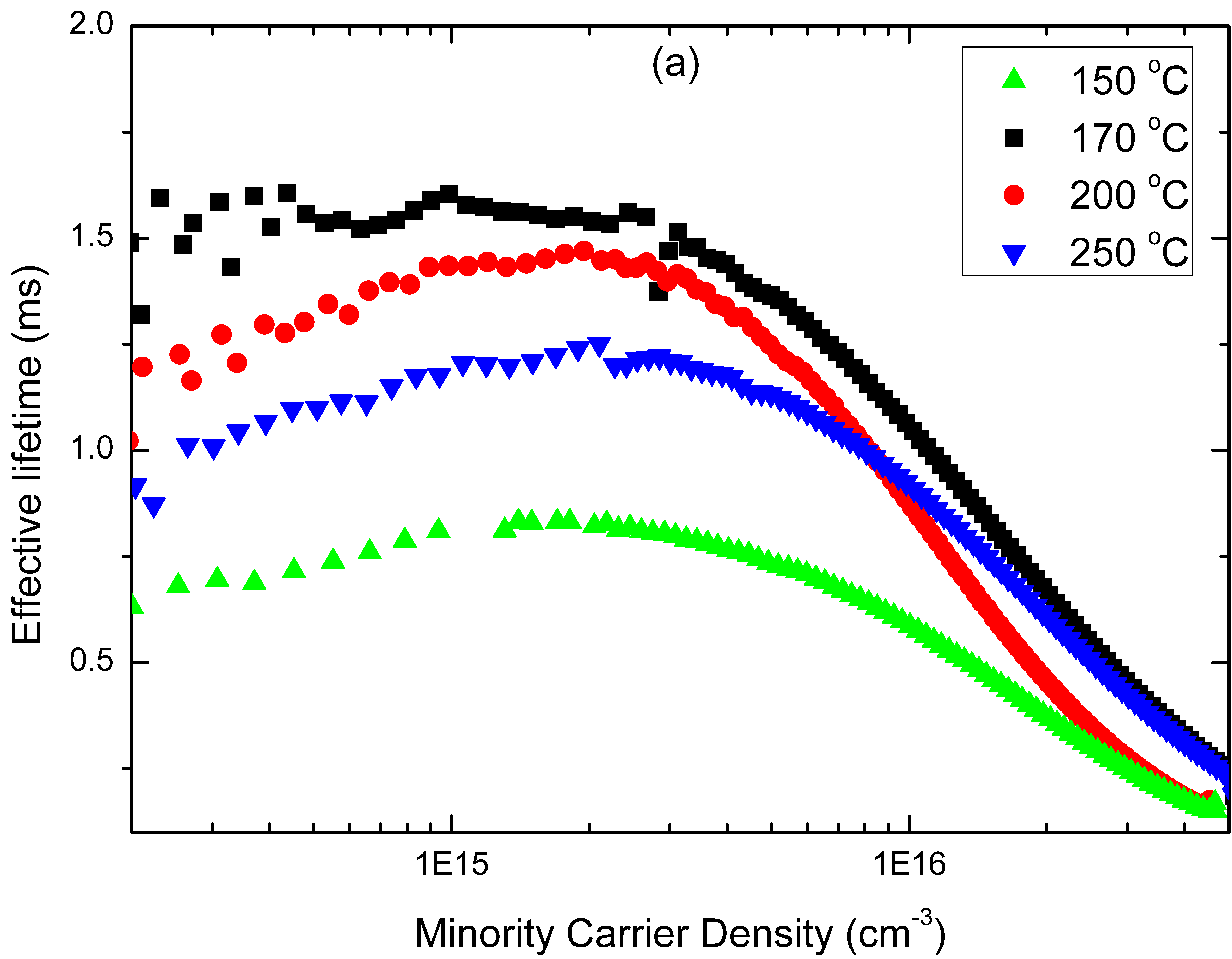
## Precursors

During HfO2 deposition, different precursors have a large influence on the chemical composition and electrical quality of the films. Several studies have been performed on different Hf precursors, however only few data are available on the oxidant precursors [6], [13], [14]. Here we focus on the influence of different oxidant precursors on HfO2 passivation property on silicon. Figure 2a shows the injection level dependent lifetimes of the three different oxidant precursor processes after post-deposition annealing on CZ n-type wafers. Both the O3 and H2O+O3 processes provide relatively good surface passivation with lifetime 0.74 ms and 0.59 ms (Seff,max values of ~30 cm/s and ~38 cm/s), respectively, at an injection level of 1×1015 cm-3. The best surface passivation is reached with the pure H2O process resulting in 1.62 ms (Seff,max = 14 cm-3).

The Dit and negative Qtot values are presented in Figure 2b. Before annealing, all three processes show high Dit values over 5×1012 eV-1cm-2. In the case of the H2O process, the Dit value before annealing is too high to be reliably characterized, and it is therefore not included in the figure. After annealing at 450 °C 15 min, the Dit decreases by one order of magnitude for all three processes. The improvement is strongest for the H2O process. The low Dit value of the H2O based sample is consistent with Al2O3 passivation studies: it is most likely caused by higher amount of hydrogen in the film deposited with H2O. During annealing this hydrogen will diffuse into the interface passivating the interface defects [15], [16]. This could also explain the difference between the pure O3 and H2O+O3 processes where slightly lower Dit value is observed with the combined process.

COCOS measurements show that regardless of the oxidant, HfO2 has a negative charge both in the as-deposited state and after annealing. After annealing, H2O based processes have similar negative Qtot values, whereas in the pure O3 process the charge is clearly higher (Figure 2b). Overall, the charge values in the annealed HfO2 samples are in the range of negative (5-10) ×1011 cm-2 and hence slightly lower than the fixed charge in Al2O3 [17]. Similar to Al2O3 deposition, the pure O3 process has the highest Qtot, however the lifetime of samples with HfO2 passivation is more sensitive to the interface, which deteriorate by the higher Dit. In the combined process the charge is in the same level as in the pure H2O process but the higher Dit value lowers the lifetime. The best surface passivation is thus obtained with the pure H2O process, which gives the lowest Dit value and relatively high fixed charge.

## Deposition temperature

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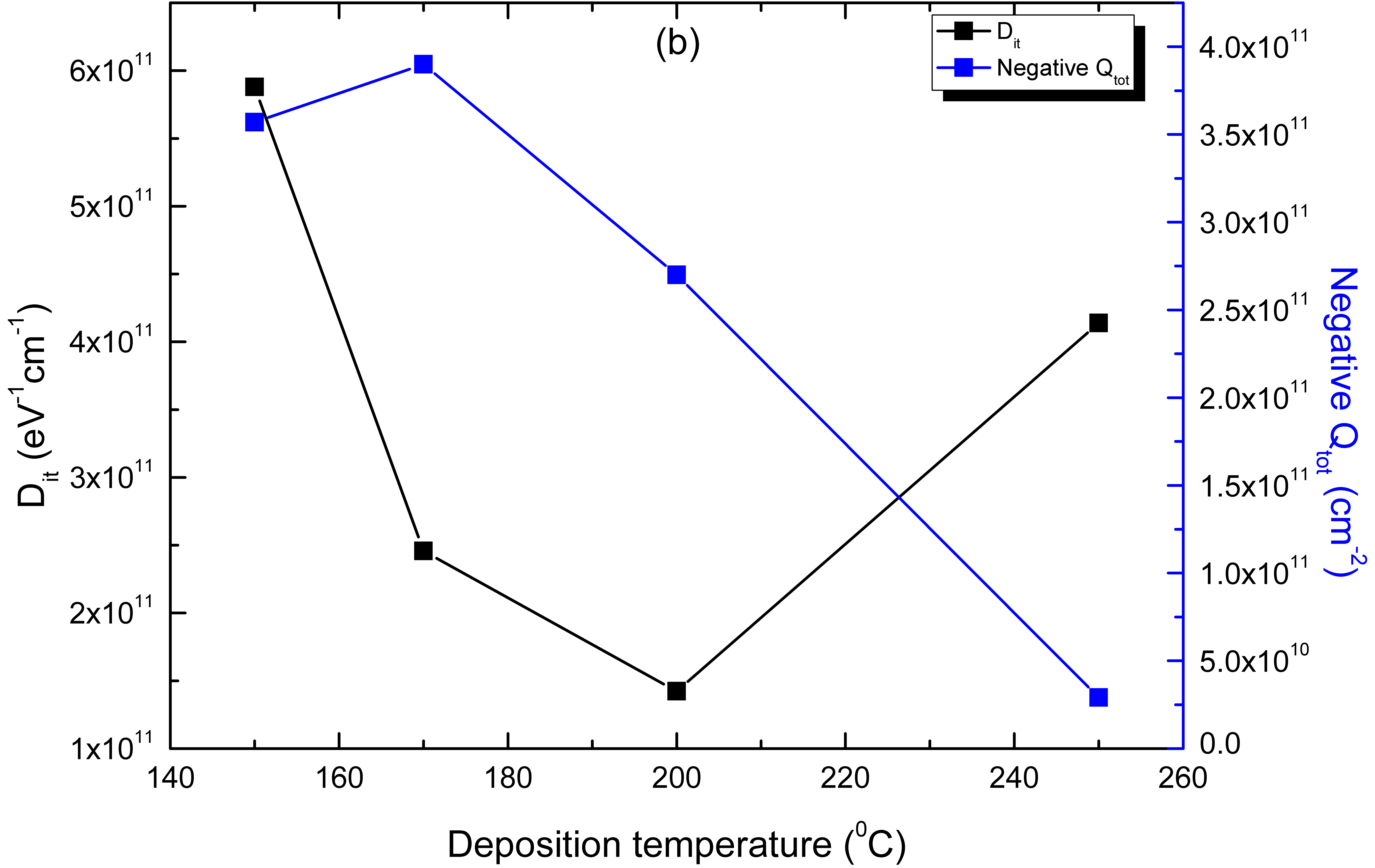
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Fig. 3. a) Injection level dependent minority carrier lifetime of HfO2 samples at different deposition temperatures: 150 °C, 170 °C, 200 °C and 250 °C. b) Negative total charge (right) and the interface defect density (left) of HfO2 samples as a function of deposition temperature.

For the ALD process, deposition temperature is one of the most important parameters determining the film properties. Here we explored four different deposition temperatures, 150 °C, 170 °C, 200 °C and 250 °C, and their effect on the passivation properties of HfO2. Figure 3 (a) gives the effective lifetime of these samples. We see that when increasing the deposition temperature from 150 °C to 170 °C, the effective lifetime increases from 0.81 ms to 1.58 ms and then slowly decreases to 1.1 ms at 250 °C. The Dit and negative Qtot results are shown in figure 3 (b). As the deposition temperature increases, Dit decreases to the lowest value of 1.42×1011 eV-1cm-2 at 200 °C. Thereafter, Dit increases with temperature. The magnitude of the negative Qtot increases to a maximum 3.9×1011 cm-2 at 170 °C, after which it decreases to 2.9 ×1010 cm-2 at 250 °C. This indicates that there is an optimum range of deposition temperature between 150 °C to 200 °C resulting in the beneficial combination of low Dit and high negative Qtot, providing both good chemical and field effect passivation of HfO2 for n-type Si wafers.

## Annealing temperature

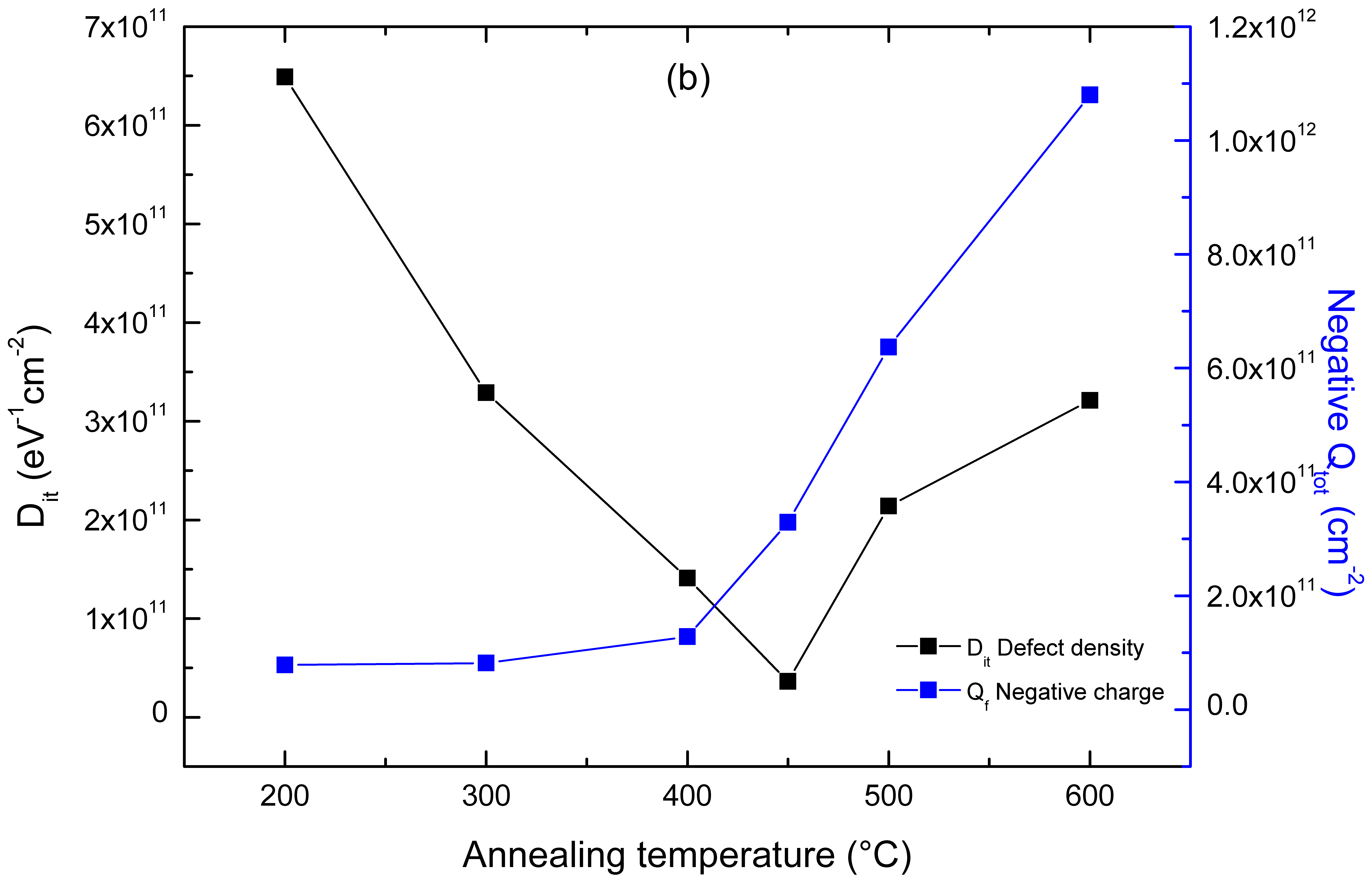
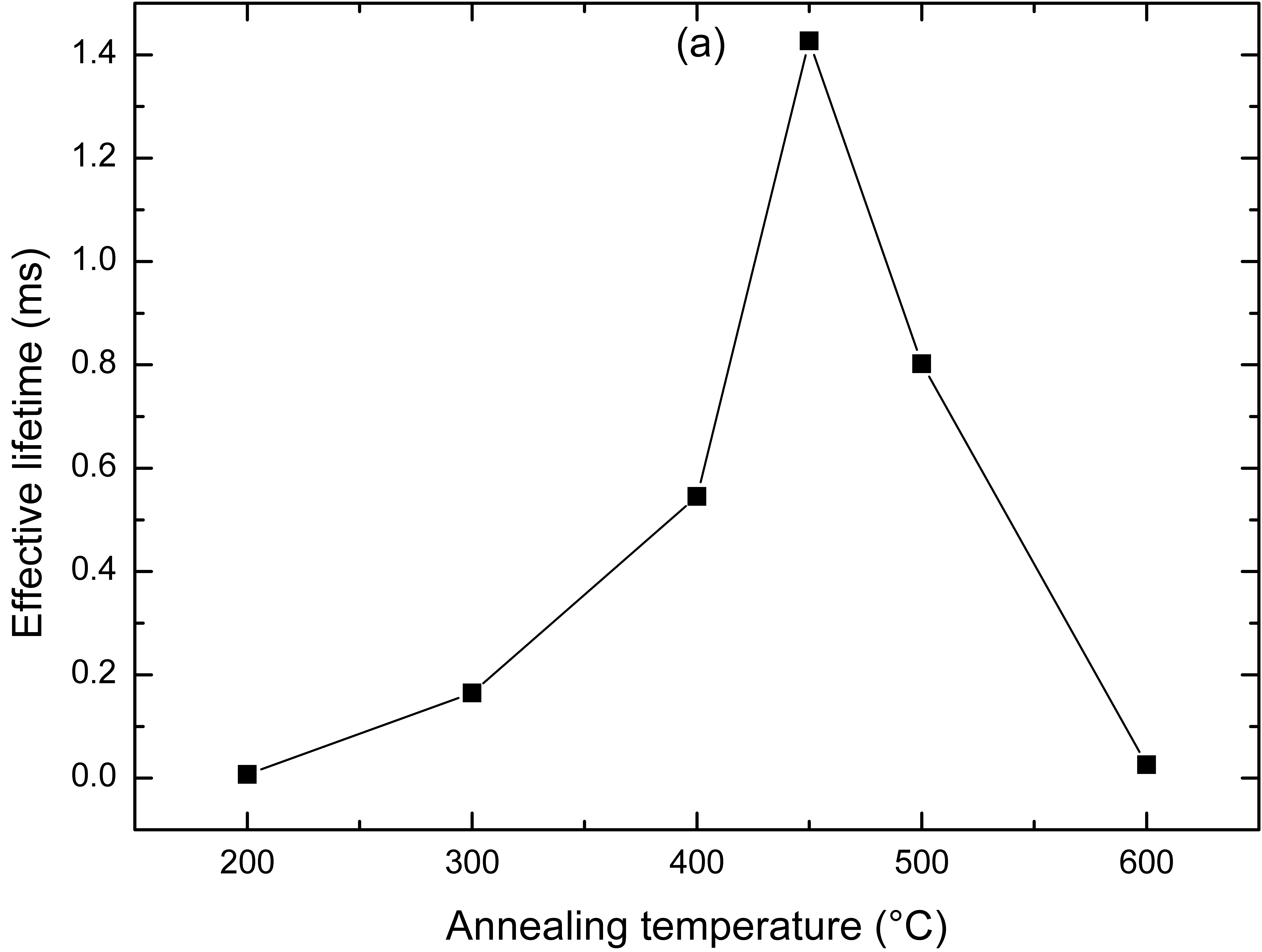


Fig. 4. a) Injection level dependent minority carrier lifetime of HfO2 samples subjected to five different postdeposition annealing temperatures: 300 °C, 400 °C, 450 °C, 500 °C and 600 °C for 15 min. b) Negative total charge (right) and the interface defect density (left) of each HfO2 sample after annealing.

Besides the deposition temperature, the postdeposition annealing temperature also affects the ALD deposited HfO2 films [18]. In order to obtain a good passivation process with HfO2, we therefore need to find the optimal post-annealing process. We fixed the annealing time to 15 min, which is experimentally tested to be enough for oxide layer (Al2O3, HfO2 etc.) annealing [4], [19]. Figure 4a shows the effective lifetime after each annealing process. The data at 200 °C is the as-deposited state value without annealing process, which results in a very low lifetime around 6 µs, verifying that the HfO2 film is similar to Al2O3 and yields no appreciable passivation without annealing process. As the annealing temperature increases, there’s a maximum value of effective lifetime of 1.4 ms at 450 °C. In addition to lifetime measurements, each sample has been characterized by COCOS to obtain Dit and negative Qtot. The results are shown in figure 4b. Consistently with the lifetime results, the Dit has a minimum value at 450 °C. At the same temperature, the negative charge Qtot increases as the annealing temperature increases, indicating that the annealing temperature affects both chemical and field effect passivation. The lowest Dit (3.63×1011 eV-1cm-2) is obtained at an annealing temperature corresponding to the highest effective lifetime. In addition, from figure 4a we find that when the sample undergoes annealing at a temperature higher than 600 °C, the effective lifetime degrades to a very low level similar to that of the as-deposited sample, almost no passivation is measured, although the negative charge is high 1.8×1012 eV-1cm-2, the corresponding Dit increases to 3.21×1011 cm-2, which could be related to H effusion during high temperature annealing [20] [21]. That is, as the post annealing temperature increase, negative charge increase while Dit have a lowest value around 450 °C corresponding to the highest lifetime, indicating that the chemical passivation play prominent role during annealing HfO2 passivation samples.

## Effect of illumination

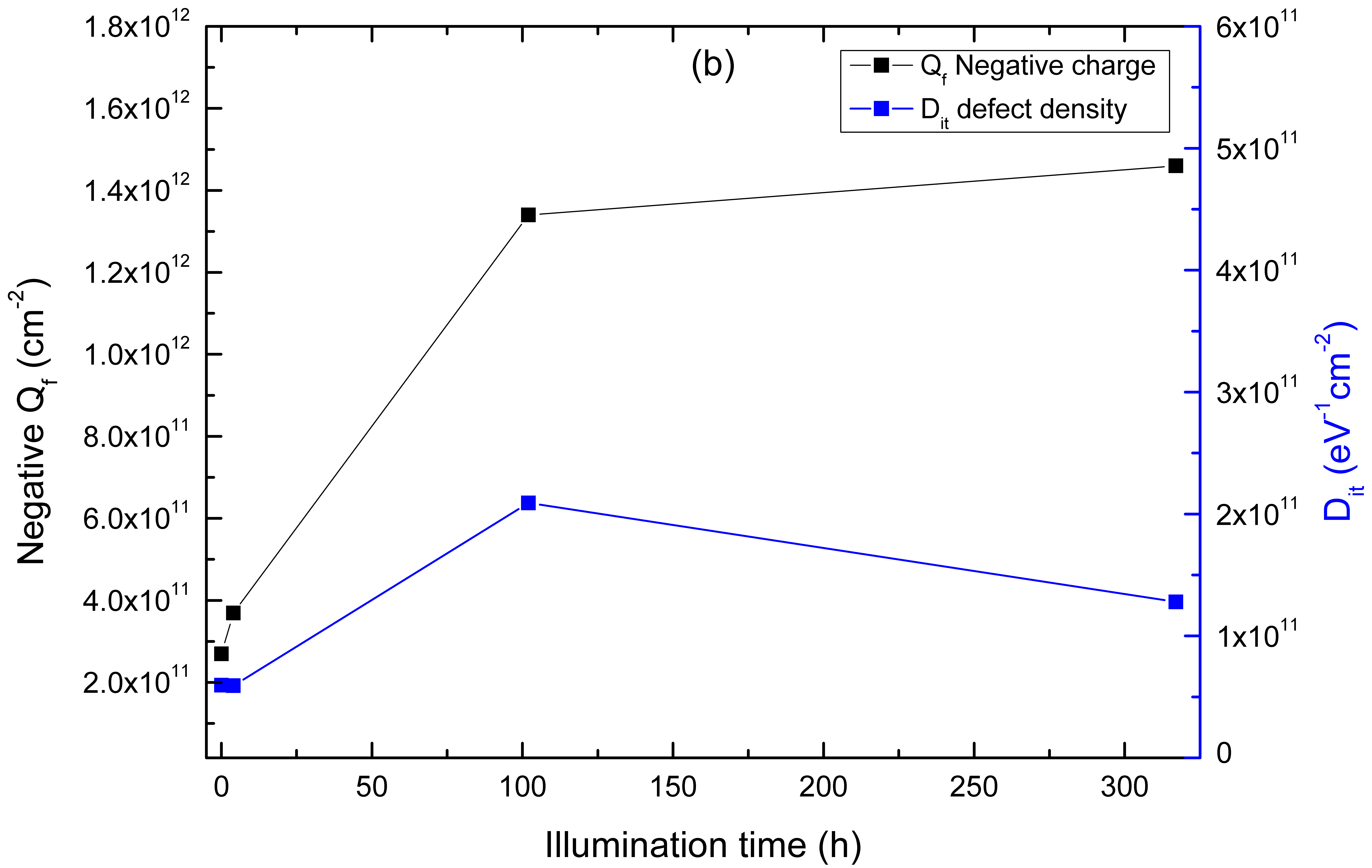
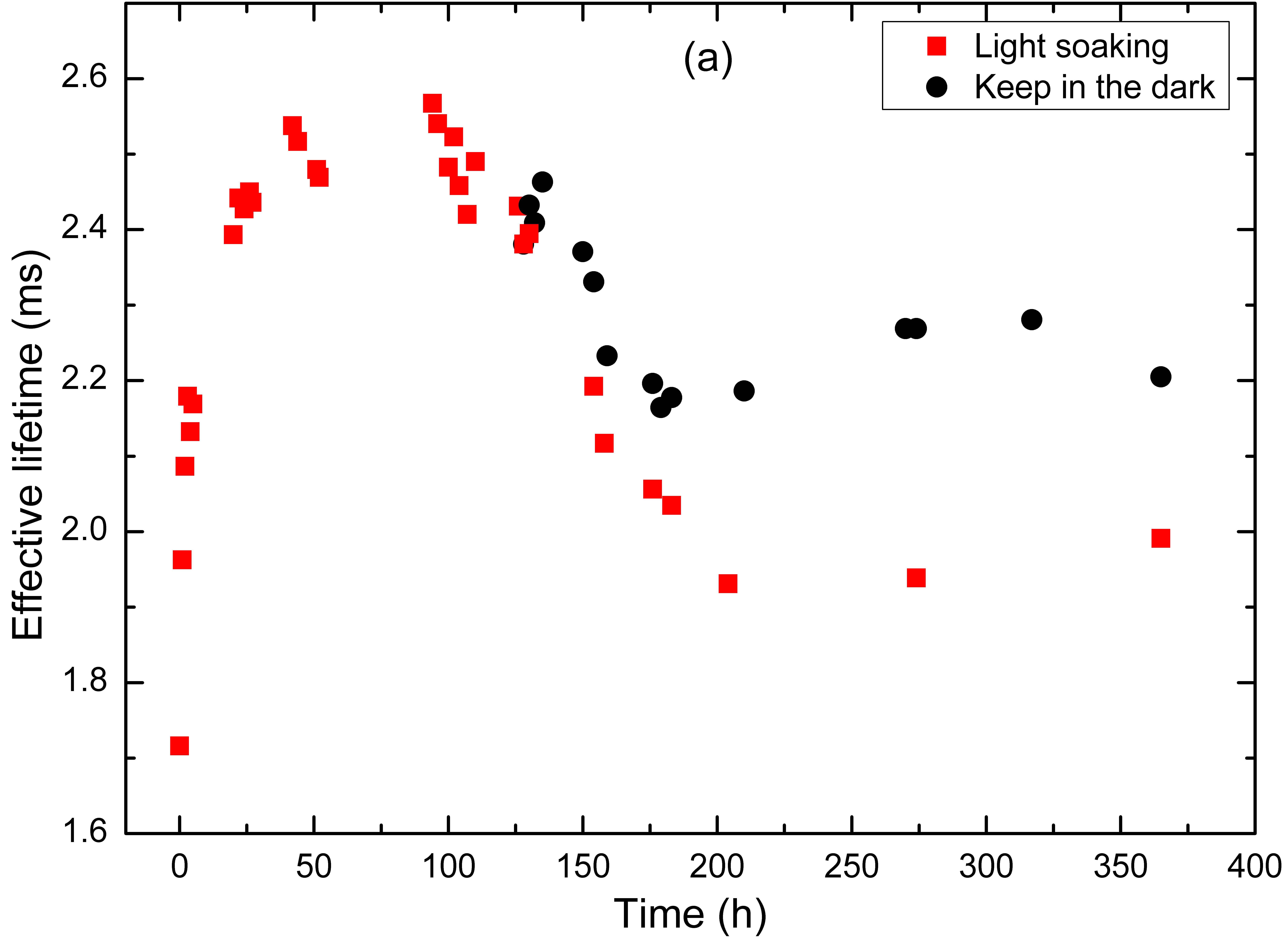
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Fig. 5. a) Time dependent effective lifetime at an injection level of 1×1015 cm-3 of two n-type CZ c-Si samples passivated on both sides by a 20 nm HfO2 film. All samples were exposed to a one-sun illumination light soaking system over 125 h, then one was chosen to be kept in the dark (black) while the other was kept under illumination (red). b) Negative total charge (left) and the interface defect density (right) of HfO2 samples after different illumination time.

Figure 5 shows the effect of light soaking on the surface passivation yielded by HfO2 deposited onto n-type CZ Si wafers. As shown in figure 5a, both lifetime samples were illuminated until 130 h, and then one of them was chosen to be kept in the dark while the other was kept under illumination (red), tracking the lifetime until 365 h. From figure 5a, we see that the effective lifetime of HfO2 passivated samples increases exponentially during light soaking and saturates after around 100 h from 1.7 ms to 2.5 ms (corresponding to a Seff decrease from 11.1 cm/s to 7.4 cm/s). After light soaking, the effective lifetime of illuminated and the samples kept in the dark decreases to around 2.2 ms and 2 ms, respectively, still higher than that before light soaking.

In order to explore the passivation at different illumination times, four small pieces of the passivated wafers were illuminated at the same time. Dit and Qtot were measured at the different stages of the illumination. From the results, shown in figure 5b, we find that as the illumination time increases, the magnitude of the negative Qtot increases from 3×1011 eV-1cm-2 before light soaking to 1.3×1012 eV-1cm-2 after 100 h light soaking. The lifetime remains high until 365 h, where the Dit has slightly increased as compared to the initial state, but still maintains an acceptably low level of around 1.3×1011 cm-2. This fact that the charge density increased under light soaking, is an advantageous effect of HfO2 for surface passivation of silicon wafers.

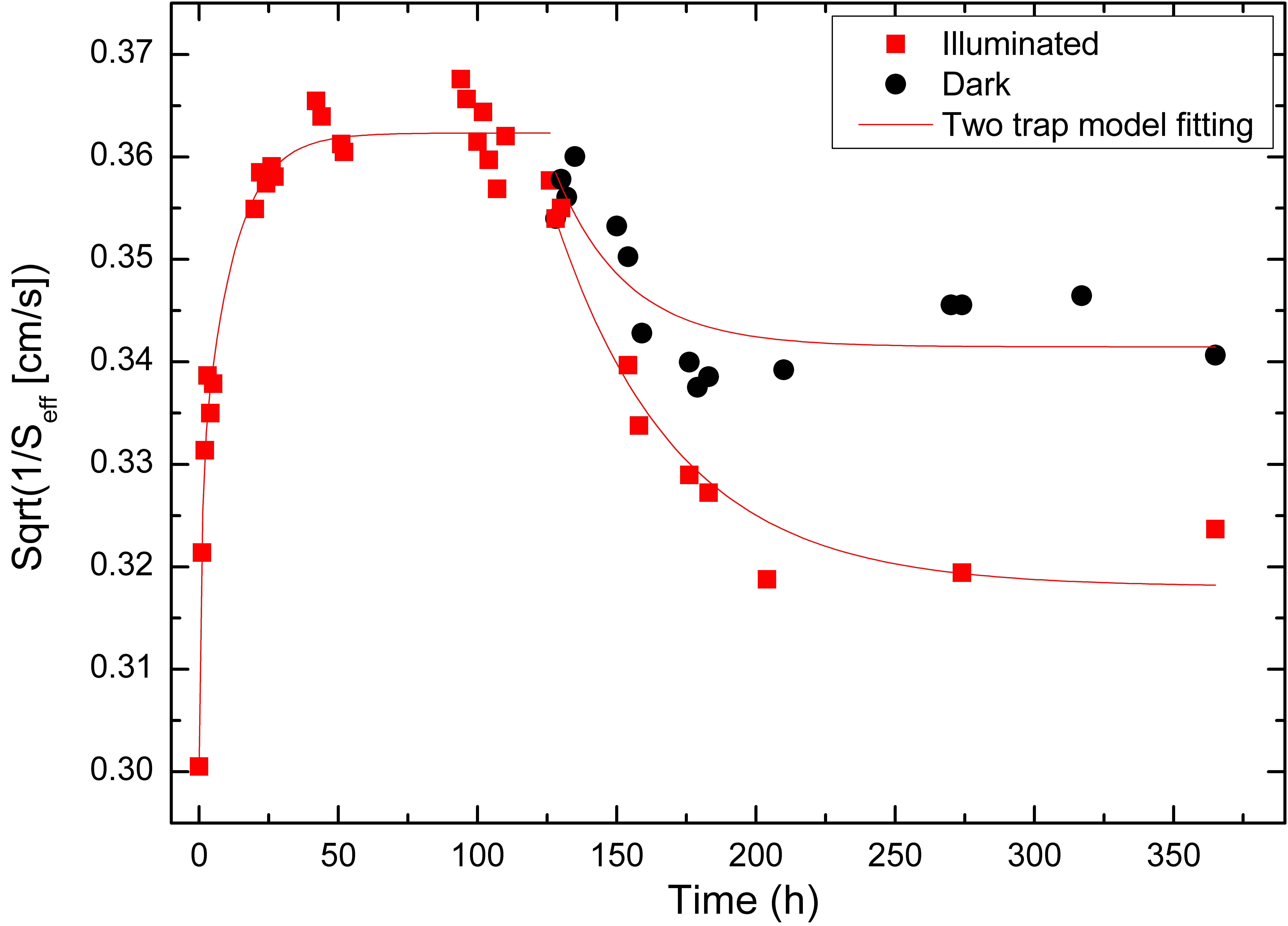


Fig. 6. Time dependent Seff of HfO2 passivated samples under illumination with two-trap model fitting curve.

Similar trends of increasing effective lifetime under light soaking are also observed on other metallic oxide materials, e.g. Al2O3 [22], TiO2 [23], [24]. The overall behavior under illumination strongly differs from that of other passivation materials such as SiO2 [25] and SiNx [26], where the effective lifetime decreases under light soaking.

To further understand the mechanism behind this phenomenon, a charge trapping/de-trapping kinetic model has been used on Al2O3 passivation of p-type Si, which was proposed by Bao et al. and for TiO2 passivation by Thomson et al. [22], [23] Since the HfO2 thin film exhibits negative charge and an ultrathin interfacial SiO2 layer (1~2 nm) between HfO2 and Si [7], similar to Al2O3 and TiO2, the charge trapping/de-trapping model could possibly explain the HfO2 light soaking behavior as well. Photoexcited excess electrons can be trapped/de-trapped by electron traps at the HfO2/ Si interface. The resulting effective surface recombination velocity Seff can be related to the rate of traps formed, the density of filled and unfilled traps at the interface in the following manner:

(1)

where is the time dependent effective surface recombination velocity, and are constants related to the initial and final , is time,  is the resulting net rate of trapping rate and de-trapping rate of the different defect types [23].

A two defect model is used to fit the data before/after light soaking saturation (125 h). As shown in figure 6, a good fit to the experimental data is obtained. Before saturation during light soaking occurs, the trapping rate dominates the net rate . Here we obtain a rate of 1.24 h-1 for the fast trap and 0.09 h-1 for the slow trap. During subsequent storage in the dark, the de-trapping rate dominates the net rate . Here we obtain 0.04 h-1 for both traps. The net rate under illumination after saturation is around 0.02 h-1 for both traps. We find that the before light saturation is much faster than during storage in the dark, which means it takes longer time to recover to the initial state before light soaking than it does to reach the saturated state. The net rate after saturation under illumination is smaller than the de-trapping rate kept in the dark, whereas the is a combination result between trapping and de-trapping, indicating that is close to after light saturation. The effective lifetime degenerate after light saturation but is still 20 % higher than the state before light soaking, indicating that the light soaking of HfO2 can improve the passivation properties on n type c-Si, which keeps consistence with the negative Qtot results. This light soaking enhancement is very important for Si solar cells passivation especially considering front surface passivation.

# Conclusion

The results of this work indicate that thermal ALD deposited HfO2 at low temperature can yield good surface passivation of n-type Si wafers after annealing at 450 °C. The highest effective lifetime is 1.94 ms. To improve our understanding of the passivation process the impact of different deposition parameters on HfO2 passivation were explored, including the pre-cleaning process, precursors, deposition temperature and annealing temperature on CZ n-type Si wafers. We find that an OH-terminated surface obtained by the proper pre-cleaning is beneficial. We show that H2O is a promising oxidant compared to O3 and O3 + H2O. An optimum deposition temperature between 150 °C to 200 °C was determined, as well as an optimum postdeposition annealing temperature of 450 °C to give good HfO2 passivation.

Moreover, an improvement of passivation under light soaking was observed. From Dit and Qtot results at different illumination time, we find that increasing the density of negative charge is a key factor for the observed passivation improvement, which is also consistent with the simulation results of the charge trapping/de-trapping model. More profound analysis of the film and interface properties is needed to fully understand the passivation mechanism of HfO2 on Si wafers. The results presented here verify that HfO2 is a promising material for Si surface passivation, especially for front surface providing better antireflection properties as compared to Al2O3. It also has potential for n+ surface passivation because of a lower density of negative fixed charge than Al2O3, but with excellent interface quality. It is also suggested to apply HfO2 on p type c-Si wafer for its negative fixed charge property.

In general, HfO2 deposited by thermal ALD provides good surface quality for n-type Si wafers, with low Dit, sufficiently high negative charge density as well as light illumination enhancement of the passivation quality.

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