

# Minority Carrier Lifetime Estimation by Photoconductance Decay for Silicon Wafers Immersed in HF

Ricardo Augusto Razera, Adriano Moehlecke, and Izete Zanesco

**Abstract**—The measurement of minority carrier bulk lifetimes of Si wafers immersed in HF is known to have an immersion time dependence, which is specially strong for high lifetime wafers and can vary significantly for different surface treatments performed previously. These complications can make the lifetime measurement become difficult to interpret and inaccurate. Therefore, a method for extracting the bulk lifetime from the measured curves of lifetime versus immersion time is suggested. A model of the reaction between Si and HF is considered, consisting in a first-order reaction between Si and HF for polished wafers and two first-order reactions for textured wafers. Experiments on polished and textured p-type Si wafers were conducted with the photoconductance decay technique to test the model proposed and to show that it is more adequate to estimate the bulk lifetime by using the reactions models than to report it as the lifetime measured at a particular immersion time, which is the usual procedure.

**Index Terms**—Immersion time in HF, minority carrier lifetime, photoconductance decay (PCD), Si wafer.

## I. INTRODUCTION

IT IS well established that the minority carrier lifetime ( $\tau$ ) and the surface recombination velocity ( $S$ ) are important parameters for characterizing photovoltaic cells [1]–[3]. Because of this importance, the dependence of  $\tau$  on the various microscopic parameters of Si wafers has been extensively studied in the past few decades, and effective and fast methods of measuring it have been proposed. The usual method is measuring the photoconductance decay (PCD) while the Si wafer surfaces are covered with a passivating thin film [3], [4], such as a-Si:H, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, or SiN<sub>x</sub>, or a passivating liquid [5], such as HF [6] or I<sub>2</sub>/ethanol [7]. Although thin film passivation may be a more practical route in most cases, this passivation scheme usually involves high-temperature steps, which may degrade the wafer bulk and change  $\tau$ . In these cases, liquid passivation may be more appropriate.

Manuscript received March 21, 2017; revised April 25, 2017; accepted May 14, 2017. Date of publication May 29, 2017; date of current version June 19, 2017. This work was supported in part by Eletrosul for the grant of the R&D project entitled “Development of Industrial Processes for the Manufacture of Solar Cells with Aluminum Paste and Passivation,” 1110140010, and in part by the National Council of Scientific and Technological Development (CNPq). (Corresponding author: Ricardo Augusto Razera.)

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Digital Object Identifier 10.1109/JPHOTOV.2017.2706420

Various authors have determined that HF provides an excellent surface passivation [6], [8]–[13], with surface recombination velocities as low as 0.25 cm/s [8]. The usual problem with HF passivation is, however, that the measurements of  $\tau$  with the wafer immersed in HF turn out having an immersion time dependence. In fact, Poortmans *et al.* [6] reported differences in surface recombination velocities of as much as 160 cm/s for immersion times of 2 and 10 min when the wafers are immersed in concentrated (49%) HF. For a high lifetime wafer ( $\approx 2$  ms), this difference in  $S$  can result in a change in  $\tau$  from  $\tau = 1760 \mu\text{s}$  for  $S = 1$  cm/s to  $\tau = 90 \mu\text{s}$  for  $S = 160$  cm/s, for a wafer thickness of 300  $\mu\text{m}$ . Thus, these differences in surface recombination velocity for different immersion times can render the measurement completely inaccurate. In fact, although values of  $S$  of order 0.25 cm/s are sufficiently low if the bulk lifetime to be measured is of order 100  $\mu\text{s}$  or less, in the cases where the lifetime is of order 1 ms or higher even these low values of  $S$  can cause significant changes in the bulk lifetime. Furthermore, the immersion time dependence observed in  $\tau$  can be complex, especially for textured surfaces, which are the common surface morphologies used in solar cells. These kinds of stability problems are reported by several authors [6], [10], [13]–[15], indicating that the interpretation of PCD measurements for wafers immersed in HF is still not clear.

The reaction between HF and Si has been studied in detail by various authors using infrared absorption spectroscopy (IRAS)[16]–[19]. In these studies, it was found that various kinds of compounds, of the form SiH<sub>x</sub> and SiF<sub>x</sub>, are generated during the reaction [16], leaving the final Si surface hydrogenated [8], [20]. These are the bonds that reduce the surface recombination velocity in comparison with a bare Si surface, where the high recombination rates are caused by the high concentration of dangling bonds. These studies also showed that the mechanism for HF and Si reactions depend strongly on the previous surface treatments performed on the Si wafer [6], [16]–[20]. Even differences between thermal and chemical SiO<sub>2</sub> have been noticed when the reaction is studied with IRAS [18]. These reactions between Si and HF tend to complicate the immersion time dependence of  $\tau$  even further, possibly making the PCD measurements unreliable.

The purpose of this work is to present a more systematic approach to evaluate the minority carrier lifetimes of silicon wafers immersed in HF. This way of extracting the bulk lifetime consists in a model for the Si and HF reactions, taking into

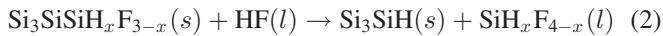
account the immersion time dependence of  $\tau$  and eliminating the problems associated with it. The model is then compared against *in situ* measurements of  $\tau$  for p-type Si wafers immersed in HF.

## II. HF AND SI REACTION KINETICS

When the wafer surfaces get first in contact with HF, a reaction occurs with the native oxide present, which immediately etches this oxide in the first few seconds of immersion. Trucks *et al.* [20] have deduced from quantum-chemical calculations that, immediately after the oxide is removed, the Si surface is terminated by fluorine atoms, through a reaction of the form:



Subsequently, the SiF bonds get further attacked by HF molecules through reactions of the form:



leaving the surface H-passivated. The presence of these reactions is confirmed experimentally by IRAS measurements of both SiH and SiF bonds during the immersion of the wafer in HF [16].

It is important to note that the reaction in (1) occurs virtually instantaneously, while the reaction in (2) is slower. This is a consequence of the higher activation energy associated to reaction (2) [20]. Thus, it is the second reaction that causes the immersion time dependence of  $\tau$ .

Even though the reactions in (2) have the same form, there is no reason to expect that the general reaction should be of first order, since there could be several different mechanisms for it to proceed. Nevertheless, in this paper, it is always assumed that the reaction that substitutes fluorine bonds by hydrogen ones is of first order, for a reasonable agreement with our experiments was obtained this way.

## III. DEPENDENCE OF THE SURFACE RECOMBINATION VELOCITY ON REACTION KINETICS

For a sufficiently low  $S$ , the effective lifetime ( $\tau_{\text{eff}}$ ) of the minority carriers measured by the PCD method is given by

$$\frac{1}{\tau_{\text{eff}}} = \frac{1}{\tau_b} + \frac{2S}{W} \quad (3)$$

with  $\tau_b$  the bulk lifetime and  $W$  the wafer thickness. Through this equation one can find the surface recombination velocity by measuring the effective lifetime of the Si wafer and knowing the bulk lifetime. On the other hand, one can obtain the bulk lifetime if one measures  $\tau_{\text{eff}}$  and has the surface recombination velocity. The latter case is the usual one, where  $\tau_{\text{eff}}$  is measured and  $S$  is assumed to be zero because of the passivating liquid.

Equation (3) is valid for  $SW \ll 2D$ , where  $D \approx 30 \text{ cm}^2/\text{s}$  is the minority carrier diffusivity. For  $300 \mu\text{m}$  wafers, this means that  $S$  has to be much lower than  $2.10^3 \text{ cm/s}$ . Fortunately, the SRV associated to HF passivated Si surfaces is at least one order of magnitude lower than this value [6], [8], thus validating the approximation.

The connection between the experimentally determined  $S$  and the reaction rate associated with (2) is made by means of the relation between  $S$  and the concentration of surface

traps,  $N$

$$S = \sigma v_{\text{th}} N \quad (4)$$

where  $\sigma$  is the capture cross section of minority carriers and  $v_{\text{th}}$  is their thermal velocity.  $\sigma$  is treated here as a phenomenological parameter which may depend on the concentration of minority carriers on the surface and the actual cross section associated with the surface bond that captures these minority carriers. Since there is more than one type of bond present in the surface, (4) should be substituted by

$$S = v_{\text{th}} \sum_i \sigma_i N_i \quad (5)$$

where the sum runs over all kinds of bonds present.

The reaction studied here is the substitution of SiF by SiH bonds. In the course of this reaction, both  $\sigma_i$  and  $N_i$  in (5) may contribute to the time dependence of  $S(t)$ . The dependence of  $N_i$  on immersion time is the direct consequence of the reaction. On the other hand,  $\sigma_i$  depends on minority carrier concentrations, which may change because of different surface charges associated to each type of bond. In other words, because  $\sigma_i$  depends on  $\{N_i\}$ , it may also end up depending on immersion time. Explicitly, therefore, (5) is written as

$$S(t) = v_{\text{th}} \sum_i \sigma_i(\{N_i\}) N_i(t). \quad (6)$$

However, as will be shown later, a reasonable (within error bars) agreement with experimental values was obtained assuming that all  $\sigma_i$  are constants and that  $N_i$  changes according to (2), which was assumed to be a first-order reaction. Thus, the equation for  $S$  is

$$S = v_{\text{th}} N_0 \sigma_F e^{-kt} + v_{\text{th}} N_0 \sigma_H (1 - e^{-kt}) \quad (7)$$

where  $\sigma_F$  and  $\sigma_H$  are the capture cross sections for SiF and SiH, respectively, and are both constants by assumption.  $N_0$  is the total concentration of bonds in the Si surface and  $k$  is the reaction rate constant.  $t$  is the immersion time.

A further simplifying assumption used in the analysis of this work is that  $\sigma_F = 0$ . This corresponds to the assumption of perfect passivation of the SiF bond. This is reasonable because, as will be shown in the results, the SiH bond presents a higher capture cross section than SiF. Since our experiments could not measure absolute values of  $S$ , we could say that  $S = 0$  at the highest  $\tau$  measured and study only changes in  $S$  as the reaction proceeded. However, using this assumption means that the lifetime obtained is actually a lower bound to the true  $\tau_b$ , which is a usual shortcoming of methods that cannot separate surface from bulk recombination. The difference is that using (7) we can obtain a higher lower bound to  $\tau_b$ , because we extract its value as an extrapolation for when  $t = 0^+$ , which is the instant where the surface recombination is the lowest.

With all these assumptions and approximations, the dependence of the effective lifetime on immersion time can be obtained through (3) and (7). This gives

$$\frac{1}{\tau_{\text{eff}}} = A - B e^{-kt} \quad (8)$$

TABLE I  
SURFACE MORPHOLOGY, RESISTIVITY, AND THICKNESS OF EACH SAMPLE

Sample	Surface Morphology	Resistivity	Thickness
FZ	Textured	2 $\Omega$ .cm	300 $\mu$ m
Cz	Mirror Polished	35 $\Omega$ .cm	525 $\mu$ m

with

$$A = \frac{1}{\tau_b} + \frac{2S_H}{W} \quad (9)$$

$$B = \frac{2S_H}{W} \quad (10)$$

and

$$S_H = v_{th} N_0 \sigma_H. \quad (11)$$

Equation (8) means that fitting the experimentally determined  $1/\tau_{eff}$  as a function of immersion time will give the constants  $A$ ,  $B$ , and  $k$ . With these constants,  $\tau_b = \frac{1}{A-B}$  can be obtained.

#### IV. EXPERIMENTAL METHODS

The measurements of minority carrier lifetime were carried out with a Sinton Instruments WCT equipment. It utilizes a Xe lamp with a 700 nm high-pass filter as a strobe lamp for the injection of minority carriers. The injection level was  $10^{15} \text{ cm}^{-3}$ . The conductivity of the sample is monitored by the usual technique of inductive coupling the wafer to a coil. The measurements were made with the transient PCD technique, since the Si wafers analyzed had a high lifetime ( $\approx 2.5 \text{ ms}$ ) compared with the lamp's decay time ( $\approx 30 \mu\text{s}$ ). A mono exponential decay of conductivity was found in all measurements. The lifetime was obtained through the inverse slope of the logarithmic plot of photoconductance versus time. The uncertainty was estimated previously to be approximately 5%.

The wafers were introduced in a small container that had 50 ml of HF 48%. New HF was used in each measurement to avoid contamination between each sample. Each measurement was performed in intervals of 1 min after the wafer first came into contact with the hydrofluoric acid, until 20 min of immersion time were reached.

Two different kinds of samples were used: 1) mechanically (mirrored) polished Czochralski (Cz) and 2) textured float zone (FZ) wafers. All wafers studied were p-type. These different sets of samples are summarized in Table I, along with informations on resistivity and thickness of each set. All the wafers had (100) orientation, except those which were textured, that had the micropylramids' (111) facet exposed.

The samples preparation consisted of an RCA cleaning procedure ( $\text{H}_2 \text{O} + \text{HCl} + \text{H}_2 \text{O}_2$ , 5:1:1, at approximately  $80^\circ \text{C}$  for 10 min), which produces a thin layer with approximately 2 nm of chemical  $\text{SiO}_2$ . The textured samples were produced introducing Si wafers in a mixture of KOH, isopropyl alcohol, and  $\text{H}_2 \text{O}$  prior to the RCA cleaning. The chemicals used were all of analytical grade.

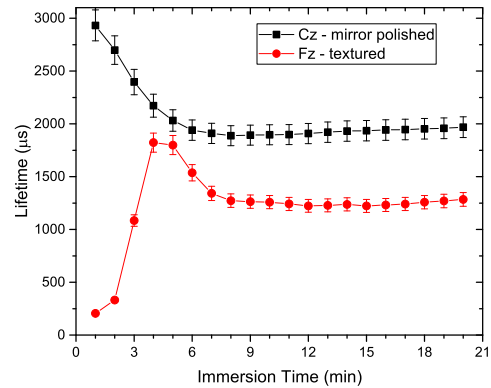


Fig. 1. Minority carriers lifetime versus immersion time curves found for each type of sample studied.

#### V. RESULTS AND DISCUSSIONS

Fig. 1 shows one curve for each type of sample in Table I. From inspection, one can already identify that each surface morphology presents a different behavior. The textured one first shows an initial fast increase of  $\tau$  with immersion time, a feature that is not present in the curve for the polished wafer [10]. Although Fig. 1 shows the measurement of only one sample of each type, several measurements were performed on different wafers with the same characteristics in Table I and the same behaviors were always found.

We suppose that the different behavior observed for the textured sample is caused by a delay in the reaction that substitutes the OH group at the surface (formed during chemical oxidation) for SiF bonds [reaction (1)]. The cause for the delay may be twofold: 1) a higher activation energy for the reaction of HF with the (111) surface compared with the (100) surface, or 2) an additional limiting factor related to the transport of HF to the surface of the micropylramids. This delay is not seen in the polished surface because in this case reaction (1) is fast enough to not be noticed. This explains the initial fast increase for textured samples and also explains why it is absent in the polished ones. In fact, the decrease in lifetime with immersion time for unprocessed polished Si wafers has been observed previously [6], [13], [15], although Zhao *et al.* [13] reported a decrease in lifetime also for textured surfaces. In that case, however, a dilute HF solution (1%) was used, which is known to cause different sets of reactions in comparison to concentrated HF, as reported by Niwano *et al.* [16]

Within this interpretation, the initial condition in the curve for the polished wafer, where the lifetime is highest, is the result of fluorine passivation of the surface. This can be seen only in the curve for the polished sample, because the delay effect on textured surfaces masks the passivation caused by fluorine. After fluorine gets substituted by hydrogen, at the end of the curve, the passivation is a result of SiH bonds. Thus, we conclude that fluorine passivation of Si surfaces is higher than hydrogen passivation.

It may seem odd that F passivates better than H. This may be the result of surface charges present during the reaction, which was observed and proposed by other authors in [11], [13], [15], and [21]. For p-type wafers, this would mean that either SiF bonds introduce negative charges, causing accumulation at the

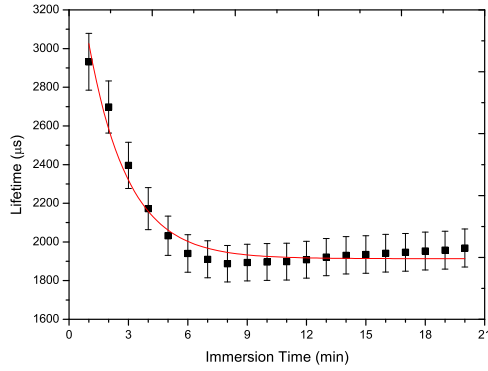


Fig. 2. Fitting of the experimental results obtained for the Cz-polished sample. The function fitted was the one in (8). The fitting resulted in the values  $A = (0.523 \pm 0.005) \text{ ms}^{-1}$ ,  $B = (0.287 \pm 0.022) \text{ ms}^{-1}$ , and  $k = (0.39 \pm 0.05) \text{ min}^{-1}$ .

surface, or that SiH bonds introduces positive charges at the surface, causing inversion. Both these possibilities would lead to an increase of  $S$  when SiF is substituted by SiH, and would lower  $\tau$ , as observed.

Fig. 2 shows the plot of the lifetime obtained from the measurement of the mechanically polished wafer, along with the fitted curve for (8). The fit so obtained has a coefficient of determination  $R^2 = 0.956$ . We thus conclude that (8) is a good model for the immersion time dependence of polished p-type Si wafers. The deviations from the points experimentally observed and the model proposed can, perhaps, be explained by the dependence of  $\sigma_H$  and  $\sigma_F$  on the concentrations of SiH and SiF bonds, which was ignored here.

The parameters  $A = (0.523 \pm 0.005) \text{ ms}^{-1}$  and  $B = (0.287 \pm 0.022) \text{ ms}^{-1}$  obtained with this fit gave  $\tau_b = (4.2 \pm 0.4) \text{ ms}$ . This is a higher lower bound than the value of  $\tau_b \approx 3 \text{ ms}$  that would be obtained if one just assumed  $S = 0$  for the highest value of  $\tau$  measured, which is the usual procedure. The reason for the higher value is that with this method we assume  $S = 0$  only when  $t = 0^+$ , since this is the instant where the surface recombination is lowest, as explained in Section III. This way, the immersion time dependence of  $S$  enabled a more precise determination of  $\tau_b$  for polished samples.

One can also model the initial increase observed for the textured wafer as a consequence of another first-order reaction that exchanges SiOH for SiF [reaction (1)], meaning that there are two reactions taking place in the first few minutes of immersion. In this scenario, SiOH is changed to SiF, which is then transformed into SiH through the same reaction as in the polished wafer case. The populations of each type of bond transform into one another according to the following set of coupled differential equations:

$$\frac{d}{dt}N_O = -k_1N_O \quad (12)$$

$$\frac{d}{dt}N_F = k_1N_O - k_2N_F \quad (13)$$

$$\frac{d}{dt}N_H = k_2N_F \quad (14)$$

and have the initial conditions  $N_O(0) = aN_0$ ,  $N_F(0) = (1 - a)N_0$  and  $N_H(0) = 0$ , where  $a$  is the initial fraction of the total number of bonds that has the OH group. Here,  $N_O$ ,  $N_F$ , and

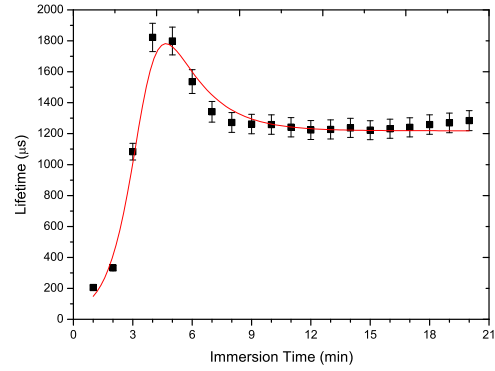


Fig. 3. Fitting of the experimental results obtained for the textured sample. The function fitted was the one in (16), giving the parameters  $C = (0.834 \pm 0.026) \text{ ms}^{-1}$ ,  $D = (16.7 \pm 1.9) \text{ ms}^{-1}$ ,  $E = (2.9 \pm 0.8) \text{ ms}^{-1}$ , and  $k_1 = (0.93 \pm 0.11) \text{ min}^{-1}$ .  $k_2$  was kept fix at  $0.39 \text{ min}^{-1}$ .

$N_H$  are respectively the concentration of SiOH, SiF, and SiH. Solving the differential equations and using (5), we obtain the total surface recombination velocity:

$$S = S_H + a \left( S_O + \frac{k_2}{k_1 - k_2} S_H \right) e^{-k_1 t} - S_H \left( 1 + a \frac{k_2}{k_1 - k_2} \right) e^{-k_2 t} \quad (15)$$

where we defined  $S_O = v_{th} N_0 \sigma_O$  in analogy to (11), with  $\sigma_O$  the cross section for the SiOH bond, and kept the assumption that  $S_F = v_{th} \sigma_F N_0 = 0$ .  $k_1$  and  $k_2$  are the rate constants associated to reactions (1) and (2), respectively. This equation for  $S$  gives  $\tau_{\text{eff}}$  as

$$\frac{1}{\tau_{\text{eff}}} = C + D e^{-k_1 t} - E e^{-k_2 t} \quad (16)$$

with

$$C = \frac{1}{\tau_b} + \frac{2S_H}{W} \quad (17)$$

$$D = \frac{2}{W} \left( S_O + \frac{k_2}{k_1 - k_2} S_H \right) a \quad (18)$$

$$E = \frac{2S_H}{W} \left( 1 + a \frac{k_2}{k_1 - k_2} \right). \quad (19)$$

From (15), it is clear that this model reduces to the previous one if  $a = 0$ , meaning that there are no OH groups present initially.

Fig. 3 depicts the fitting of the measured data for the textured sample against (16). This fit was obtained keeping  $k_2$  fixed at the same value of  $k$  obtained for the polished sample's fit, since these are the rate constants associated to the same reaction. We obtained  $R^2 = 0.880$ . We note that we tried fitting  $k_2$  again for this model in order to check that both reactions have the same rate constants in each case, but no significant changes were obtained and the fitting was not improved substantially. Presumably, the cause for deviation that is not contained within error bars is, again, an ignored time dependence of both  $\sigma_H$  and  $\sigma_O$ .

From (17) through (19), it can be seen that in this case  $\tau_b$  cannot be found from a simple relation between the fitting co-

efficients, since

$$\tau_b = \left( C - E + \frac{2S_H}{W} \frac{k_2}{k_1 - k_2} a \right)^{-1}. \quad (20)$$

However, if we assume that the concentration of SiH bonds is the same for both cases after the reactions are completed, and since the parameter  $B$  in (10) depends only on the thickness of the sample, we can still use it to obtain an estimate for the lower bound of  $\tau_b$  of the textured sample by  $\tau_b = \frac{1}{C-B \cdot W_p/W_t}$ , where  $W_p = 525 \mu\text{m}$  and  $W_t = 300 \mu\text{m}$  are the thicknesses of the polished and textured sample, respectively. Using the value  $C = (0.834 \pm 0.026) \text{ms}^{-1}$  and the same value of  $B$  as before, we obtain  $\tau_b = (3.0 \pm 0.4) \text{ms}$  for the textured sample.

We note that we also tried to fit the polished wafer case with a two reactions model, since we expect that, although too quickly, reaction (1) also occurs for this case. However, the fit was not significantly improved. Therefore, we kept the one reaction model for this case for the sake of simplicity.

Since the lifetime varies by such a significant amount with immersion time, as a result of the changes in surface recombination velocity as the reaction between Si and HF proceeds, it is necessary to establish a definite procedure for the evaluation of  $\tau_b$  when the Si wafer is immersed in HF. In principle, one could think of the following three ways to make this estimation.

- 1) Measure the lifetime until it stabilizes and report  $\tau_b$  as this value at stability.
- 2) Report  $\tau_b$  as the highest lifetime measured.
- 3) Use the kinetic model to estimate  $\tau_b$  as the value that would be obtained if the lifetime was measured when the concentration of fluorine bonds is highest.

For polished samples, it means that  $\tau_b$  should be reported as the value that would be measured when  $t = 0^+$ . For textured samples, it means that  $\tau_b$  should be reported as the value that would be found when  $t = 0^+$  if reaction (1) was infinitely fast. Now, since fluorine passivation was shown to provide a lower  $S$  than hydrogen passivation, we conclude that using the kinetic model is the most accurate method for the determination of  $\tau_b$ . Therefore, we find  $\tau_b = 4.2 \text{ms}$  for the polished sample and  $\tau_b = 3.0 \text{ms}$  for the textured sample, even though the highest lifetimes measured for these two wafers were 2.9 and 1.8 ms, respectively.

## VI. CONCLUSION

This work presented kinetic models for the reaction of Si wafers with HF that causes the immersion time dependence of the minority carrier lifetime. For the polished sample's model, a single first-order reaction that substitutes SiF bonds to SiH was sufficient to explain the time dependence observed. For the textured samples, a second reaction was needed to fit the experimental data, which was assumed to be the one that substitutes hydroxyl groups by fluorine bonds on the silicon surface.

Since we observed that the passivation was highest when the concentration of fluorine bonds was highest, we conclude that the best estimation of the bulk lifetime is given by the use of the kinetic model, since this way the bulk lifetime is given at the point where the surface recombination velocity is lowest. In fact, for both polished and textured samples, this procedure gave considerably higher  $\tau_b$  values than the highest lifetimes measured. We emphasize, however, that the  $\tau_b$  values

reported with this method are actually lower limits for  $\tau_b$ , since the passivation given by SiF bonds may be, although very small, different than zero.

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Authors' photographs and biographies not available at the time of publication.