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Research

Fundamental Boron–Oxygen-related Carrier Lifetime Limit in Mono- and Multicrystalline Silicon

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Boron-doped crystalline silicon is the most relevant material in today's solar cell production. Following the trend towards higher efficiencies, silicon substrate materials with high carrier lifetimes are becoming more and more important. In silicon with sufficiently low metal impurity concentrations, the carrier lifetime is ultimately limited by a metastable boron–oxygen-related defect, which forms under minority-carrier injection. We have analysed 49 different Czochralski-grown silicon materials of numerous suppliers with various boron and oxygen concentrations. On the basis of our measured lifetime data, we have derived a universal empirical parameterisation predicting the stable carrier lifetime from the boron and oxygen content in the crystalline silicon material. For multicrystalline silicon it is shown that the predicted carrier lifetime can be regarded as a fundamental upper limit. Copyright © 2005 John Wiley & Sons, Ltd.

KEY WORDS: silicon; recombination; lifetime; boron-oxygen; defects; Czochralski; multicrystalline

1. INTRODUCTION

The global market share of silicon solar cells made on boron-doped crystalline silicon exceeded 90% in 2002. Hence, crystalline silicon is by far the most relevant material in today's solar cell production. Following the trend towards higher efficiencies, large diffusion lengths are becoming more and more relevant. Thus, the carrier lifetime of the silicon wafers is of crucial importance.

When the concentration of lifetime-killing metal-related impurities, such as iron, iron–boron or chromium–boron pairs, can be neglected, the lifetime is ultimately controlled by a metastable boron- and oxygen-related defect complex.[1,2] This material-related defect is always formed due to illumination[3] or, more generally, due to minority-carrier injection if boron and oxygen are simultaneously present in crystalline silicon.[4,5]

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Recent studies have shown that all available experimental results can be consistently explained by a defect reaction model based on the diffusion of a fast-diffusing oxygen dimer (O_{2i}), which is captured by a substitutional boron atom, forming a B_sO_{2i} complex.[6] The relevance of this defect was realised only as conversion efficiencies of solar cells made on Czochralski-grown silicon (Cz-Si) were pushed towards 20% in the mid-1990s and bulk recombination began to limit the solar cell performance.[7] Details of the research activities within the last decade including studies of the electronic properties and the molecular structure of the defect were recently reviewed by Schmidt.[8]

Most experimental studies performed up to now focused on investigations of the relative metastable defect concentration. Since the absolute carrier lifetimes might be influenced by other impurities as well, the normalised defect concentration $N_t^* = 1/\tau_d - 1/\tau_0$ (τ_0, τ_d: lifetime before and after degradation) was used as a measure of the concentration of the metastable defect. Undoubtedly, the latter quantity is extremely useful for fundamental investigations. However, for solar cells only absolute values of the stable carrier lifetimes after degradation are relevant. Unfortunately, few data are available on the correlation of the absolute values of the stable carrier lifetime τ_d with the substitutional boron concentration [B_s] (doping concentration)[9,10] and to our knowledge so far no data have been reported on the correlation of τ_d with the interstitial oxygen concentration [O_i]. Therefore, to fill this lack of information and to find a general empirical parameterisation of the stabilised carrier lifetime τ_d as a function of the boron and oxygen content in crystalline silicon, we have performed carrier lifetime measurements on 49 different Czochralski materials with interstitial oxygen concentrations varying between 10^{17} and $10^{18}\,cm^{-3}$ and substitutional boron concentrations varying between 7×10^{14} and $2 \times 10^{17}\,cm^{-3}$. Optimised high-temperature treatments have been shown to increase the stable carrier lifetime permanently.[9,11] Since in most of the present solar cell manufacturing sequences a phosphorus emitter diffusion step is included, we have also investigated the influence of a conventional phosphorus diffusion step on the stable carrier lifetime.

On the basis of these measurements, we have performed a two-dimensional fitting of the stable carrier lifetime to find a parameterisation predicting τ_d for crystalline silicon on the basis of a given set of substitutional boron and interstitial oxygen concentrations. Compared with Cz-Si, mc-Si suffers from its non-ideal crystalline structure and often from the incorporation of metal impurities resulting in additional recombination paths. Thus, for mc-Si the lifetime values predicted by this empirical equation can be regarded as an upper limit. Our parameterisation allows manufacturers of silicon ingots or blocks a more accurate assessment of the quality of their material.

2. EXPERIMENTAL DETAILS

An important characteristic of the metastable boron–oxygen-related defect is the existence of two lifetime states. During annealing at 200°C for 10 min the metastable defect dissociates and the carrier lifetime τ_0 is a measure of the residual recombination in the silicon sample. After illumination the metastable defect is fully activated and the stable carrier lifetime τ_d, corresponding to the degraded state, can be measured. Depending on the boron concentration, the time necessary to achieve full degradation varies between a few minutes for $[B_s] \simeq 10^{17}\,cm^{-3}$ and a few days for $[B_s] \simeq 10^{15}\,cm^{-3}$.

In this contribution, all lifetime measurements have been performed at the same injection level $\eta = \Delta n/N_{dop} = 0{\cdot}1$. This ensures that the influence of Auger recombination and trapping is kept at a minimum. All lifetime measurements have been performed by the quasi-steady-state photoconductance (QSSPC) method.[12]

Before performing an emitter diffusion step at 860°C in a quartz-tube furnace, the samples have been cleaned in a standard RCA sequence. After diffusion the phosphorus-glass as well as the diffused emitter region were removed by wet chemical etching.

In order to minimise surface recombination, the wafer surfaces of each sample have been RCA-cleaned and electronically passivated by means of remote plasma silicon nitride films deposited at $\sim$400°C.[13] The doping concentrations have been determined from four-point probe measurements. FTIR measurements have been performed according to DIN 50438-1 using a Bruker Equinox 55 FTIR spectrometer to determine the interstitial oxygen concentration.

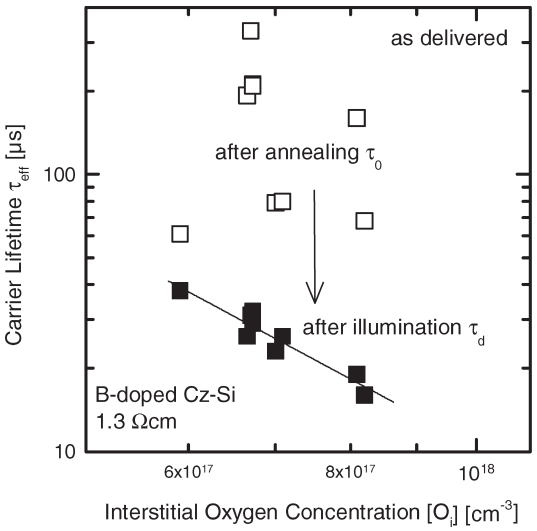

Figure 1. Measured carrier lifetime of various Cz-Si materials as a function of the interstitial oxygen concentration [O_i] with a doping concentration of $1{\cdot}1 \times 10^{16}\,\mathrm{cm}^{-3}$. After annealing (200°C, 10 min), the metastable defect is deactivated and the scatter in the data indicates different lifetime-limiting residual defect concentrations

3. RESULTS AND DISCUSSION

Figure 1 shows the measured carrier lifetimes of Cz-Si materials after annealing (τ_0) and illumination (τ_d) as a function of the interstitial oxygen concentration [O_i]. The scatter in the τ_0 lifetime data is extremely large, indicating different 'background' defect concentrations in the different materials. After sufficiently long illumination, the metastable defect is fully activated. In this case, the scatter in the data is considerably reduced and the metastable boron–oxygen complex limits the carrier lifetime. Thus, in order to find an empirical parameterisation of the carrier lifetime, the stable τ_d values measured after illumination have to be analysed. For the determination of the lifetime-limiting character due to the simultaneous presence of boron and oxygen in crystalline silicon, we focus on the analysis of Cz-Si samples, since magnetically confined Cz-Si (MCz-Si) is currently not commercially available. Multicrystalline silicon (mc-Si) materials are not included as their lifetime is usually strongly influenced by other defects. However, it will be shown in Section 3·4 that our analysis will also be partly applicable to mc-Si.

First, we will study the impact of boron and oxygen on the carrier lifetime separately. Here it is important to keep the concentration of one species constant while the other is varied.

3.1. Oxygen dependence

The role of oxygen has been analysed for three sets of samples with boron concentrations of around 0·3, 1 and $2 \times 10^{16}\,\mathrm{cm}^{-3}$ and oxygen concentrations varying between 3 and $9 \times 10^{17}\,\mathrm{cm}^{-3}$. While all samples with resistivities of 1·3 and 5 Ω cm come from different suppliers, the 0·7 Ω cm samples have been cut out of one ingot with an interstitial oxygen concentration intentionally varied between 3 and $6 \times 10^{17}\,\mathrm{cm}^{-3}$. For these samples, the same post-growth anneal to destroy thermal donors (TD) has been performed. The TD annihilation step is important to minimise the scatter in the lifetime data, because a residual TD concentration hampers a correct determination of the boron doping concentration by the four-point probe method.

The measured oxygen dependence of the stable carrier lifetime is shown in Figure 2. With increasing oxygen concentration, τ_d shows a nearly inversely quadratic decrease of the stable carrier lifetime.

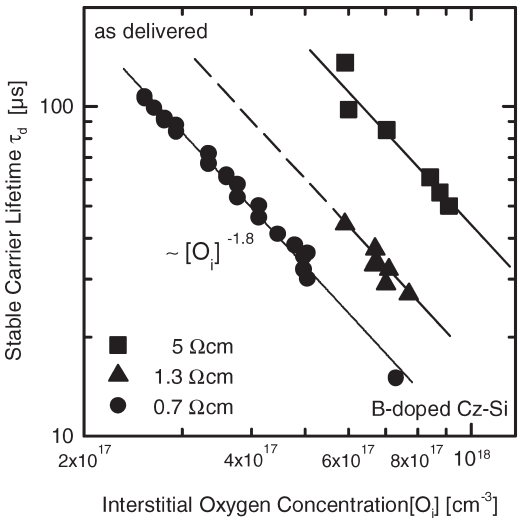

Figure 2. Measured oxygen dependence of the stable carrier lifetime τ_d for 0·7, 1·3 and 5 Ω cm Cz-silicon samples. For all doping concentrations, the carrier lifetime shows a nearly inverse quadratic decrease with increasing oxygen content

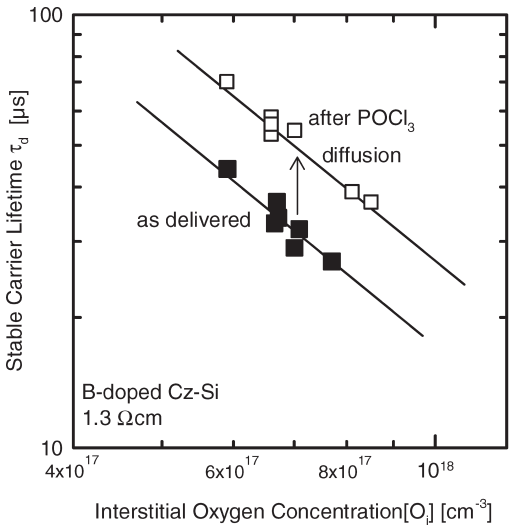

Figure 3. Influence of a high-temperature (860°C) phosphorus emitter diffusion step on the stable carrier lifetime. The lines are fits showing that the nearly inverse quadratic decrease of τ_d with $[O_i]$ is not altered, however, the absolute lifetime values are increased by a factor of 2

The impact of the high-temperature phosphorus emitter diffusion step on the stable lifetime is shown in Figure 3. While the oxygen dependence is not altered, the absolute value of τ_d is increased by a factor of $\sim$2. Note that the diffusion step has not been optimised for a maximum reduction in the concentration of the fundamental metastable boron–oxygen defect. As we have shown earlier,[11] using an *optimised* phosphorous diffusion step, the concentration of the boron–oxygen complex can be decreased by up to a factor of 3·5.

3.2. Boron dependence

In order to clarify the effect of boron, a set of samples with similar oxygen concentrations (around $7 \times 10^{17}\,\mathrm{cm}^{-3}$), but varying boron concentrations (ranging from 10^{15} to $10^{17}\,\mathrm{cm}^{-3}$) has been investigated.

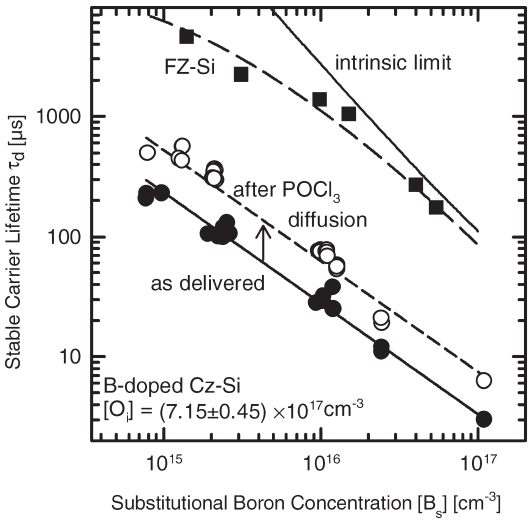

Figure 4. Boron dependence of the stable carrier lifetime τ_d before (as delivered) and after phosphorus diffusion at 860°C for samples with comparable oxygen concentrations. The stable carrier lifetime decreases approximately inversely with increasing boron concentration $[B_s]$. The phosphorus diffusion increases τ_d by a factor of ~ 2. Also shown are experimentally determined carrier lifetime values of float-zone silicon reported in the literature.[14] These values indicate the carrier lifetime limit as a function of the boron content in case of negligible oxygen concentrations. The intrinsic limit determines the theoretical upper limit

As shown in Figure 4, the stable carrier lifetime decreases approximately inversely with increasing boron concentration. As expected from the results of the oxygen dependence, the non-optimised phosphorus diffusion results in an increase in the carrier lifetime by a factor of ~ 2. Also shown in Figure 4 are experimentally determined carrier lifetime values of oxygen-lean float-zone silicon ($[O_i] < 10^{16}\,cm^{-3}$) reported elsewhere.[14] These values indicate the carrier lifetime limit as a function of the boron content in case of negligible oxygen concentrations. The intrinsic limit of the carrier lifetime due to Auger[15,16] and radiative[17] recombination is also included for comparison.

3.3. Lifetime parameterisation

After analysing the boron and oxygen dependence of the stable carrier lifetime separately, we performed a two-dimensional fitting to derive a complete empirical parameterisation predicting the carrier lifetime for silicon materials of a given oxygen and boron content. This statistical analysis was performed on a database containing stable lifetime values of 49 different Cz-Si samples of numerous suppliers with various boron and oxygen concentrations. The result of the fit for the as-delivered samples can be seen in Figure 5 as a grey plane. The measured data points are shown as circles that are connected to the plane by perpendicular lines to indicate their distance from the plane. The black crosses in Figure 5(a) indicate data points positioned behind the plane. In Figure 5(b), the same data points are shown from a different perspective to highlight the surprisingly small deviations between the fit and the data points. The small deviations indicate that the boron and oxygen contents predict the carrier lifetime very accurately.

The stable carrier lifetime is found to depend almost inversely on the boron concentration and approximately inversely quadratically on the interstitial oxygen concentration. In order to simplify the two-dimensional analysis, the logarithmic data were fitted by using the equation $f = c_1 + c_2 \cdot x + c_3 \cdot y$ with $x = \ln([O_i])$, $y = \ln([B_s])$ and $f = \ln(\tau_d)$. $[O_i]$ and $[B_s]$ are given in units of cm^{-3} and τ_d in µs. Back-transformation gives the following empirical equation

$$\tau_d = 7{\cdot}675 \times 10^{45} [B_s]^{-0{\cdot}824} [O_i]^{-1{\cdot}748} \quad (1)$$

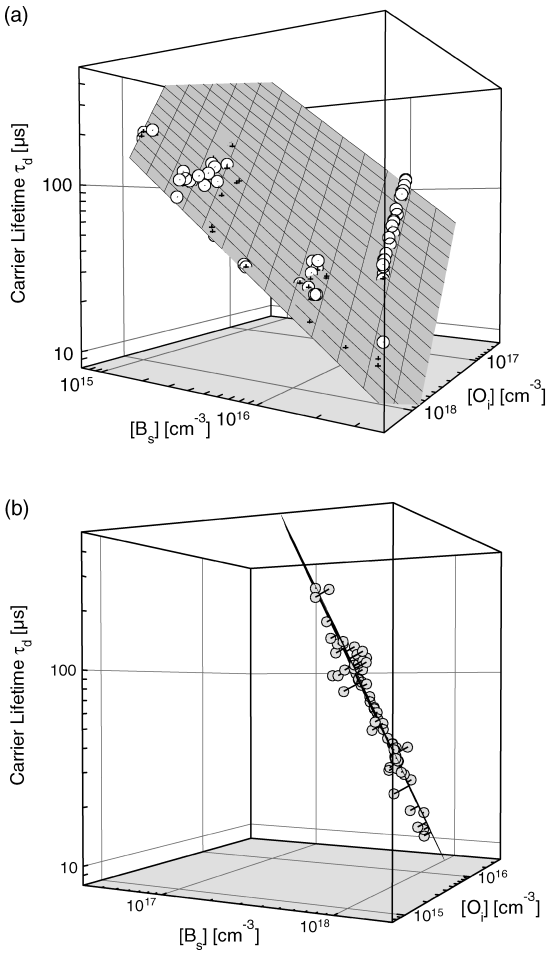

Figure 5(a,b). Simultaneous fit of the stable carrier lifetime taking numerous samples with different boron and oxygen concentrations into account. The result of the fit is shown as a grey plane while the data points are shown as circles. In order to indicate their distance from the plane, each data point is connected to the plane by a perpendicular line

for the stable carrier lifetime of samples in the *as-delivered* state. These predicted lifetime values correspond to experimentally determined carrier lifetime values at an injection level of $0{\cdot}1 \times N_{\mathrm{dop}}$ by the QSSPC method. The empirical equation was determined by leaving c_1,c_2 and c_3 unrestricted, which yield a correlation coefficient $R^2 = 0{\cdot}93$, indicating again a very reliable prediction of the stable carrier lifetime. Restricting c_2 and c_3 to the results determined by the separate analysis of the boron and oxygen dependence (see Sections 3·1 and 3·2) yields a less precise correlation with $R^2 = 0{\cdot}87$. Thus, for the full parameter space, Equation (1) gives the most precise values. However, we cannot exclude that in some special cases a slightly modified equation might give a better result. Note that the reliability of the parameterisation is restricted to the range of analysis with $[B_s]$ between 7×10^{14} and $2 \times 10^{17}\,\mathrm{cm}^{-3}$ and $[O_i]$ ranging from 10^{17} to $10^{18}\,\mathrm{cm}^{-3}$.

Figure 6 shows a contour plot of the carrier lifetime predicted by the parameterisation for different interstitial oxygen concentrations and resistivities. Taking a common solar cell resistivity of $1\,\Omega\,\mathrm{cm}$ gives a typical value of $\sim 30\,\mu\mathrm{s}$ for Cz-Si and an upper limit of $\sim 100\,\mu\mathrm{s}$ for mc-Si. Thus, mc-Si would be the better choice in case crystallographic defects and metallic impurities could be largely eliminated. Alternatively, Cz-Si with a resistivity around $8\,\Omega\,\mathrm{cm}$ would also give stable carrier lifetimes around $100\,\mu\mathrm{s}$.

For oxygen concentrations below $10^{17}\,\mathrm{cm}^{-3}$, which can be realised with magnetically confined Czochralski silicon (MCz), we have found τ_{d} to lie below the predicted values. Thus, we assume that a different residual defect limits the carrier lifetime in this low-oxygen regime.

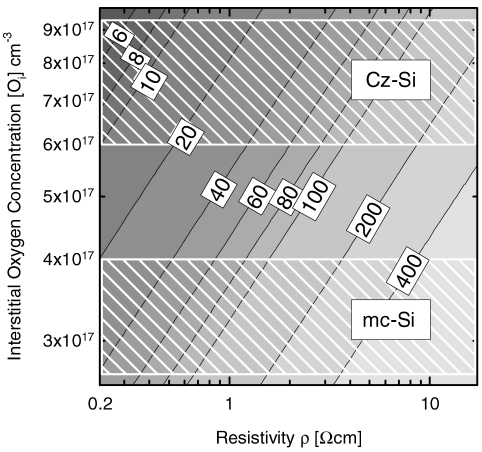

Figure 6. Contour plot of the carrier lifetime predicted by the empirical equation for different oxygen concentrations and resistivities. Highlighted are typical oxygen ranges for Cz-Si and mc-Si

The τ_d values after a non-optimised $POCl_3$ diffusion can be assumed to increase by factor of ~ 2. The origin of this increase is still under investigation. We assume a reduction of the oxygen dimer concentration, which is the oxygen-related part of the metastable boron–oxygen defect. The oxygen dimer concentration increases with the interstitial oxygen concentration, but the absolute value critically depends on the thermal history of the silicon ingot or block.[18] This suggestion is supported by the results of an earlier study.[11] We reported that the reduced concentration of the metastable defect after $POCl_3$ diffusion (i.e., the increase in the carrier lifetime) is a result of the thermal treatment alone and that faster cooling rates result in lower defect concentrations.

3.4. Carrier lifetime limit for mc-Si

In multicrystalline silicon the interstitial oxygen concentration is typically lower by a factor of ~ 5 compared with Cz-Si. Thus, the fundamental boron-oxygen complex limits the carrier lifetime on a much higher level. This can be seen in the contour plot shown in Figure 6, where typical ranges of oxygen concentrations for mc-Si and Cz-Si are shaded. Figure 7 shows a collection of experimental data of carrier lifetimes available in the literature[19–23] for multicrystalline silicon with different oxygen and comparable boron concentrations. Note that all these values have been measured on as-delivered wafers which have neither been gettered nor hydrogen passivated. Usually, the interstitial oxygen concentration of mc-Si blocks decreases from the bottom to the top. High $[O_i]$ concentrations exceeding $5 \times 10^{17} \times \mathrm{cm}^{-3}$ can be found in the bottom-near region of the mc-Si block. If not indicated explicitly, all lifetime values are area-averaged values determined by the QSSPC measurement technique and have been measured at an injection level of approximately $0{\cdot}1 \times N_{dop}$. This allows a direct comparison with the values predicted by the empirical lifetime Equation (1). All lifetime data, even the values which have been measured on mc-Si made out of a special virgin high-quality silicon feedstock,[22] lie well below the limit. This indicates that the boron–oxygen-related carrier lifetime parameterisation can be regarded as fundamental upper limit to the carrier lifetime in multicrystalline silicon.

3.5. The influence of thermal donors

During the growth process and the subsequent cooling of the Czochralski silicon ingot electrically active centres[24] are formed at temperatures around 450°C. These oxygen-related centres act as donors and because of their generation during thermal treatment they are named 'thermal donors' (TD). For the use in the microelectronics industry, TDs are annihilated at temperatures above 650°C by the silicon manufacturers. This thermal treatment can be omitted for solar-grade Cz-Si, because the TDs are destroyed during the first high-temperature

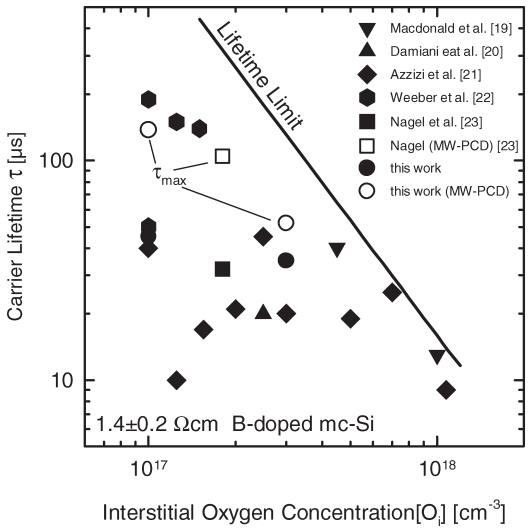

Figure 7. Carrier lifetime values of different mc-Si materials with different oxygen and comparable boron concentrations.[19–23] If not indicated explicitly, all lifetime values are area-averaged values determined by the QSSPC method and correspond to an injection level of approximately $0{\cdot}1 \times N_{dop}$. All values are below the boron–oxygen-related carrier lifetime limit, which therefore can be regarded as a fundamental upper limit for multicrystalline silicon

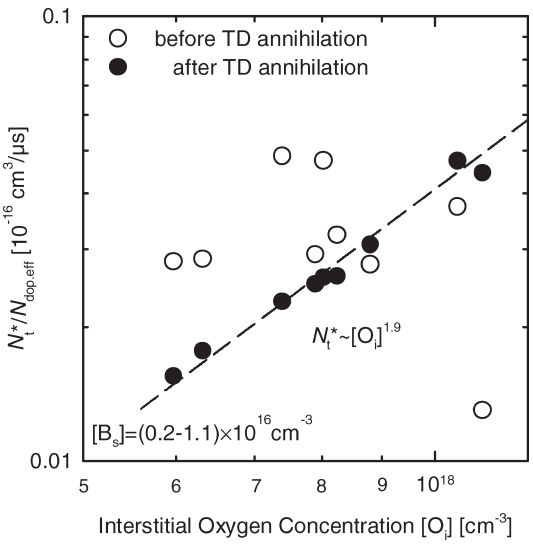

Figure 8. Normalised defect concentration of various Cz-Si samples with different oxygen concentrations before and after thermal donor (TD) annihilation. The specific resistance due to the doping with boron is considerably reduces by the TD concentration. As a consequence, the determination of the doping concentrations from four-point probe measurements underestimates the true boron concentration. Depending on the TD concentration, this effect leads to a large error in the determination of the normalised defect concentration

step in the solar cell manufacturing process. Because of this omission, care has to be taken when analysing unprocessed solar-grade Cz-Si wafers by means of lifetime measurements. In regions of high interstitial oxygen concentrations, the thermal donor concentration can be as high as $10^{16}\,cm^{-3}$, leading to an overcompensation of 2–3 Ω cm boron-doped silicon materials, which even becomes *n*-type. In any case the resistivity of the material

can be considerably altered by the TDs. As an important consequence, the determination of the doping concentration from four-point probe measurements underestimates the true boron concentration because this procedure only measures the *effective* doping concentration $N_{\mathrm{dop.eff}}$. The determination of the carrier lifetime by means of the QSSPC method requires the correct values for the mobilities of electrons and holes. As the mobility is a function of the concentration of charged impurities, both the absolute boron and the absolute TD concentration need to be known to determine the correct mobility values. A more pronounced error in the determination of the carrier lifetime occurs when not measuring the resistivity at all, but using the resistivity values specified by the silicon manufacturer and thus ignoring any change in conductivity due to thermal donors.

If one analyses the normalised defect concentration $N_t^*/N_{\mathrm{dop.eff}}$ of the metastable boron–oxygen-related defect, all these errors sum up. This is shown in detail in Figure 8 for Cz-Si samples from two ingots without post-growth TD annihilation treatment. The variation in the interstitial oxygen concentration is due to a gradient in the oxygen concentration along the growth direction. Before TD annihilation the scatter in the data is extremely large and no correlation between the normalised defect concentration and the interstitial oxygen concentration is found. After the TD annihilation this scatter in the data is considerably reduced and a clear dependence between $N_t^*/N_{\mathrm{dop.eff}}$ and $[O_i]$ can be observed. This dependence has already been analysed in detail elsewhere.[25]

4. CONCLUSIONS

We have presented a comprehensive analysis of the oxygen and boron dependence of the stable carrier lifetime in crystalline silicon. By simultaneously fitting the oxygen and boron dependence of the stable carrier lifetime of 49 different Czochralski silicon materials from numerous suppliers with various boron and oxygen concentrations, we have derived a universal empirical parameterisation predicting the carrier lifetime τ_d. For the lifetime measurements the QSSPC method has been used to determine τ_d at a fixed injection level of $\eta = \Delta n/N_{\mathrm{dop}} = 0{\cdot}1$ in order to minimise the influence of Auger and trapping effects. By combining the results of this study with an advanced QSSPC system adapted to measure the carrier lifetime in silicon ingots and blocks, manufacturers get access to a very useful tool for quality control. In case the level of contamination with lifetime-killing metal impurities is sufficiently low, the boron–oxygen-related defect limits the carrier lifetime in silicon. In order to minimise errors in the determination of the carrier lifetime, the concentration of thermal donors should be minimised. For multicrystalline silicon with additional recombination paths due to crystal defects and metal-related recombination centres, the presented empirical lifetime parameterisation was shown to be a fundamental upper lifetime limit.

Acknowledgements

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