

Lifetime studies of multicrystalline silicon

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Abstract: The application of photoconductance measurements to investigate the electronic properties of multicrystalline silicon is illustrated with selected experiments, ranging from process control to fundamental research. Carrier trapping effects are discussed, and a technique based on controlled cross-contamination of hyper-pure silicon wafers with multicrystalline wafers is used to separately study the effect of metallic impurities. The variability of the effective lifetime with injection level is explained in terms of the Shockley-Read-Hall recombination model. Finally, the implications of effective lifetime on device voltage are pointed out.

1. INTRODUCTION

In spite of its extensive, and increasing, use in the photovoltaics industry, multicrystalline silicon (mc-Si) is still a relatively poorly understood material, even after twenty years of research [1]. Inherently, it is a complex semiconductor system where the crystalline lattice is interrupted at the grain boundaries and the volume is frequently populated by foreign atoms, micro-defects and dislocations. It is fundamentally non-homogeneous, with changing properties across the surface of a wafer and within the volume of an ingot. The growth conditions can have a strong effect on its quality and there can be significant differences between ingots grown with the same technique. Furthermore, several different methods are currently used to grow the ingots; we should, properly speaking, say that there are several multicrystalline silicon materials.

The characterisation of the electronic properties of mc-Si by means, for example, of lifetime testing, is complicated, owing to its non-homogeneity and to the co-existence of several different recombination mechanisms. Yet, lifetime measurements are the most useful tool available to study the physical limitations of mc-Si and overcome them. It is particularly important to study the possible changes induced by processing [2], a task that is facilitated by the use of simple, contactless measurement techniques. It has recently been realised that the lifetime of mc-Si can be nearly as high as that of single crystal silicon [3,4], that high temperature processing is not necessarily harmful to mc-Si [5,6], that the surfaces can be well passivated [7] and that high efficiency solar cell designs can be implemented in mc-Si wafers [5]. These advancements have led to conversion efficiencies over 18% [5,6,8] and open-circuit voltages over 650 mV [9]; the 20% efficiency mark has practically been reached [10]. These impressive achievements in the laboratory do not mean, however, that we have come to the end of the journey. Besides prime quality material and ultimate performance, research should be directed towards the mid and low quality mc-Si commonly used for mass production of mc-Si solar cells.

The measurement principles and the data analysis the quasi-steady-state photoconductance method (QSSPC) used preferentially in this study for lifetime testing are quite straightforward [11, 12]. Its

application to different single crystal and multicrystalline silicon (mc-Si) wafers with different dopant densities, contamination levels and crystallographic quality is described here. The advantages of the QSSPC technique over the classical transient PCD method to characterise non-homogeneous materials are first illustrated with a simple experiment. The QSSPC method is then used to monitor the effect of a phosphorus gettering process. The variability of the *effective* minority-carrier lifetime with excess carrier density concentration is explored in some detail. Controlled cross-contamination of FZ wafers is used to separately study the effect of metal impurities on carrier recombination mechanisms. This technique combined with gettering, which leaves crystallographic defects as the primary cause of recombination, and the systematic application of lifetime testing lead to a better understanding of multicrystalline silicon.

2. AVERAGE LIFETIME OF NON HOMOGENEOUS MATERIALS

Agreement between the transient and steady state methods is not to be expected in all cases. On the contrary, we have found that the transient PCD method frequently overestimates the lifetime in non homogeneous materials, in particular, mc-Si. To demonstrate this, we prepared a 1 Ω cm FZ silicon wafer with passivated phosphorus diffusions on both sides and dipped half of it in HF to strip the passivating oxide. We measured the effective lifetime on both halves of the wafer and obtained 280 μ s for the passivated side and 6.5 μ s for the de-passivated side. The sample was then centred on the inductive coil of the photoconductance instrument, which is about 2 cm in diameter and the lifetime was measured sequentially using the transient PCD and the QSSPC techniques. The PCD lifetime was almost identical to that of the best half of the sample, about 280 μ s. The QSSPC lifetime was significantly lower, 165 μ s, very close to the average of the lifetimes of the good and bad regions of the sample. (The small discrepancy with this average, $(280+6.5)/2=143$ μ s, can be explained by the 14% error of our QSSPC set up when trying to measure lifetimes as high as 280 μ s).

Clearly, the transient technique can have a tendency to emphasise the highest lifetime present in the sample since the contribution from low-lifetime areas to the photoconductance vanishes quickly and is no longer present in the region of the decay curve typically used to determine the lifetime. The experiment described above confirms the theoretical expectation [13] that a steady state measurement should give an area-weighted average of the different lifetimes present in a non-homogeneous material. This is particularly relevant for multicrystalline silicon, where the QSSPC method gives a more realistic representation of its quality. Table I compares the QSSPC and transient PCD measurements of one single crystal FZ wafer and two multicrystalline silicon wafers (gettered). The QSSPC method can be expected to over-estimate the lifetime by about 10% for lifetimes in the range of 200 μ s, which is actually observed in the FZ sample. Despite this tendency to over-predict the lifetime, the QSSPC method gives lower, more realistic, lifetimes for the mc-Si wafers.

<i>Substrate</i>	<i>QSSPC measurement,</i> $\tau_{eff}(\mu s)$	<i>PCD measurement,</i> $\tau_{eff}(\mu s)$
FZ (1 Ω cm)	180	170
mc-Si (1.5 Ω cm)	190	260
mc-Si (1.5 Ω cm)	120	220

Table I. Comparison between transient PCD and quasi-steady-state photoconductance (QSSPC) measurements of single crystalline and multicrystalline silicon wafers. From [13].

3. DEPENDENCE OF THE EFFECTIVE LIFETIME ON EXCESS CARRIER DENSITY

In the quasi-steady-state technique it is easy to sweep through a range of light intensities by means, for example, of a xenon-bulb flash and take hundreds of data points within a flash duration of about 7 ms. If the decay rate of the light is 2.3 ms, the discrepancy between this quasi-steady-state measurement and a true steady-state one is less than 10% for effective lifetimes lower than 200 μs . This facilitates the study of the injection dependence of the effective lifetime. The data contain much more information than just a single lifetime value; studying the dependence of τ_{eff} with carrier density can give insight into the nature of different recombination mechanisms.

3.1 A phosphorus gettering experiment

Phosphorus gettering is a well-known process to improve the electronic quality of mc-Si wafers. Measurements of the lifetime before and after the gettering treatment are essential to assess its efficacy and to optimise the process. Several mc-Si and CZ wafers were cleaned and subjected to a light POCl_3 diffusion at 840 $^\circ\text{C}$, “in-situ” oxide passivation at 900 $^\circ\text{C}$ and forming gas anneal 400 $^\circ\text{C}$; the sheet resistance of the n-type diffusion was 460 Ω . The effective lifetime was measured using the quasi-steady-state (QSSPC) method. The wafers were then etched to eliminate the previous lightly diffused regions and subjected to a phosphorus gettering treatment at 900 $^\circ\text{C}$ for three hours. Subsequently, the heavy phosphorus diffusion was removed (about 10-15 μm of silicon were etched) and a new light diffusion was performed in the same conditions as the initial one.

Fig. 1 shows the inverse of the measured effective minority carrier lifetime as a function of the excess carrier density for one CZ and two mc-Si wafers. These graphs visualise the overall recombination rate at a given carrier density. The beneficial effect of the gettering treatment on the mc-Si wafers is remarkable, although it is not identical for all wafers. A 0.2 Ωcm wafer (not shown) remained essentially unchanged. Samples M1 and M2 have almost the same resistivity, 1.5 Ωcm , although they come from different ingots; notably, the resistivity of M1 was obtained with a compensation factor of 1.5. The lifetime of M2 increased with gettering a factor of 10, to about 200 μs , while that of M1 doubled, to about 50 μs .

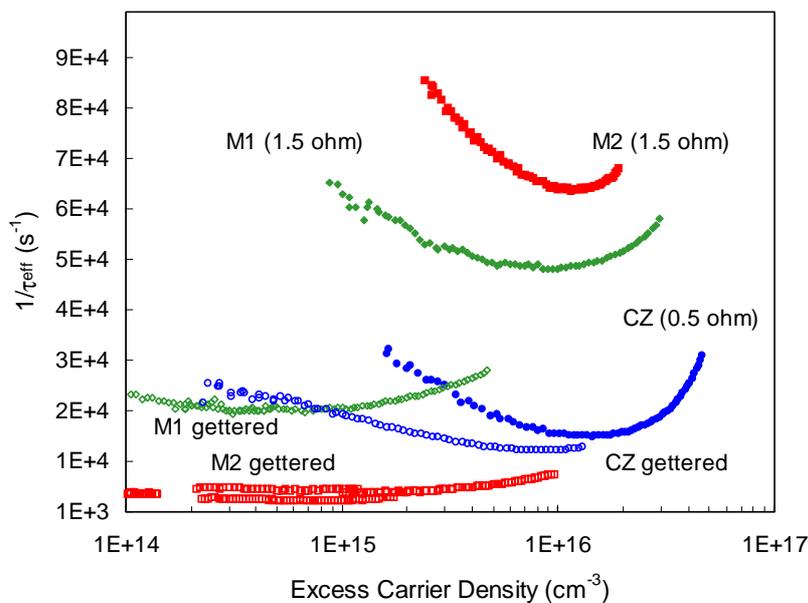


Figure 1. Pre- and post-gettering measurements of one CZ and two multicrystalline silicon wafers.

It is also interesting to note that the shape of the curves changes with gettering. In particular, the variability of τ_{eff} with injection level becomes much weaker. This indicates that the initial strong variability of τ_{eff} at low injection levels is very likely a fingerprint of the metallic impurities present in the wafers. The efficacy of the gettering treatment seems to be highest for wafers that had a stronger variation of the lifetime at low injection levels before gettering. In the following sections we investigate the variability of the lifetime in more detail.

3.2 The Shockley-Read-Hall recombination mechanism

The variability of τ_{eff} at low injection shown in Fig. 1 for the pre-gettered wafers can be explained by the classical treatment of bulk recombination developed by Shockley, Read and Hall [14]. The expression for the bulk minority carrier lifetime, simplified to the case of a p-type region and recombination centres located in the middle of the energy gap, is

$$\tau_{(SRH)} = \tau_{no} + \tau_{po} \frac{\Delta n}{\Delta n + N_A} \quad (1)$$

According to Eq.1, $\tau_{(SRH)}$ would increase with injection level from τ_{no} to $\tau_{no} + \tau_{po}$. If the electron and hole capture cross sections were identical, this could represent a doubling of $\tau_{(SRH)}$. Larger changes are actually possible if, for example $\tau_{po} \gg \tau_{no}$. At very high light intensities, the effective lifetime is affected by recombination at the phosphorus diffusions used to passivate the surfaces of these wafers. This produces, together with the Auger recombination mechanism, the upward trend in the $1/\tau$ vs. Δn curves shown in Fig. 1. The overall variability of the effective lifetime can be described with the following expression:

$$\frac{1}{\tau_{\text{eff}}} = \frac{1}{\tau_{(SRH)}} + \frac{J_o}{qn_i^2 W} (N_A + \Delta n) \quad (2)$$

Fitting the experimental data with Eq. 1 and 2 it is possible to determine the fundamental electron and hole lifetimes. The results corresponding to the curves in Fig. 1 are given in Table I.

	<i>Max. τ_{eff} (μs)</i>	<i>τ_{no} (μs)</i>	<i>τ_{po} (μs)</i>
M1 (mc-Si)	20	16	30
M2 (mc-Si)	15	7.5	25
CZ (pre-getter)	62	15	250
CZ (post-getter)	75	45	400

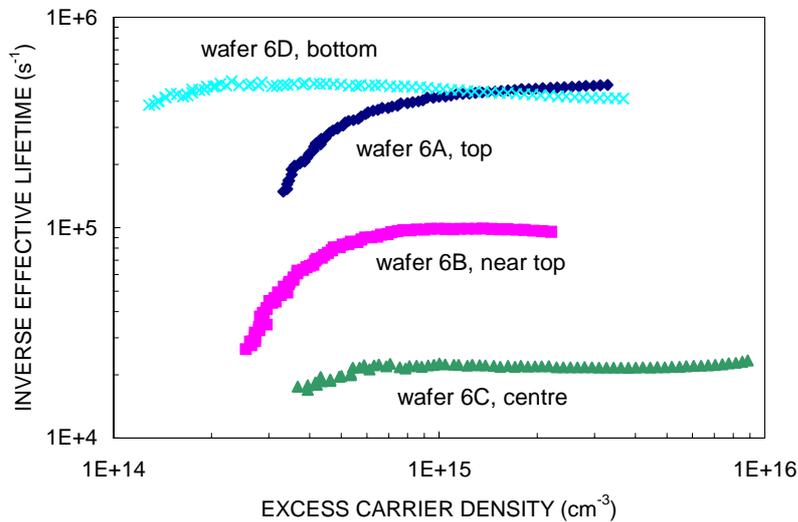
Table I. Fundamental electron and hole lifetimes for the multicrystalline and CZ wafers of Fig. 1.

4. LIFETIME MEASUREMENT OF MULTICRYSTALLINE SILICON

4.1 Lifetime map of a mc-Si ingot

Measuring the lifetime of wafers from different regions of a multicrystalline silicon ingot is an interesting exercise. A diversity of situations with different degrees of contamination and crystallographic quality naturally happen in a typical ingot growth process. In this experiment [15] we used wafers provided by Eurosolare Spa. The wafers came from two different ingots grown by

directional solidification. A light POCl_3 diffusion at 840°C for 25 minutes followed by thin oxide layer growth at 900°C for 30 minutes and FGA at 400°C was used to minimise recombination at the surfaces. The effective lifetimes of several $0.9\ \Omega\text{cm}$ wafers from ingot #6, measured with the QSSPC technique, are shown in Fig.2. Wafers from the central part of the ingot showed a significantly better electronic quality than wafers from the top and bottom regions. Without the additional information provided by the cross-contamination experiment described below, it is



impossible to determine the physical origin of the low lifetimes measured for some of the wafers; at this stage both crystallographic defects and metallic impurities are possible (and likely) reasons.

Figure 2. Experimental measurement of the effective lifetime of wafers from different regions of a cast multicrystalline silicon ingot (ingot #6).

4.2 Carrier trapping effects

Although the most immediate way of summarising the measurements shown in Fig. 2 is to report a single lifetime value corresponding to the range where τ_{eff} is reasonably constant, the data show an apparent increase of τ_{eff} as the light intensity and the carrier injection level decrease. Because of this, a direct measurement of the lifetime at the normal operating conditions of a solar cell (below 1 sun) is practically impossible. An advantage of the QSSPC set up is that it facilitates the measurement at high light intensities, where the effect saturates, or is swamped, and a realistic lifetime can be observed. Obviously, extrapolating the lifetime measured at high light intensities to predict the performance at one sun can be inaccurate, but it is better than having no information at all or believing in an anomalously high lifetime.

The behaviour of the effective lifetime of many mc-Si wafers at low intensities can be explained by a carrier trapping effect consisting of the temporary retention of electrons in relatively shallow energy levels in the band gap. We have adapted a previous theoretical model to the practical situation of a QSSPC measurement [16]. The model is based on the simultaneous presence of deep traps, primarily responsible for carrier recombination, and shallow traps that produce a distortion in the measured photoconductance while contributing negligibly to carrier recombination. In essence, the shallow traps reduce the number of free electrons available for recombination and conduction processes. The external illumination generates electrons and holes in equal numbers; many electrons are trapped and do not contribute to the photoconductance, although the holes do. The number of free electrons is thus reduced and the overall recombination rate is lower. The effect is more noticeable at relatively low illumination levels, when the fraction of trapped electrons is significant.

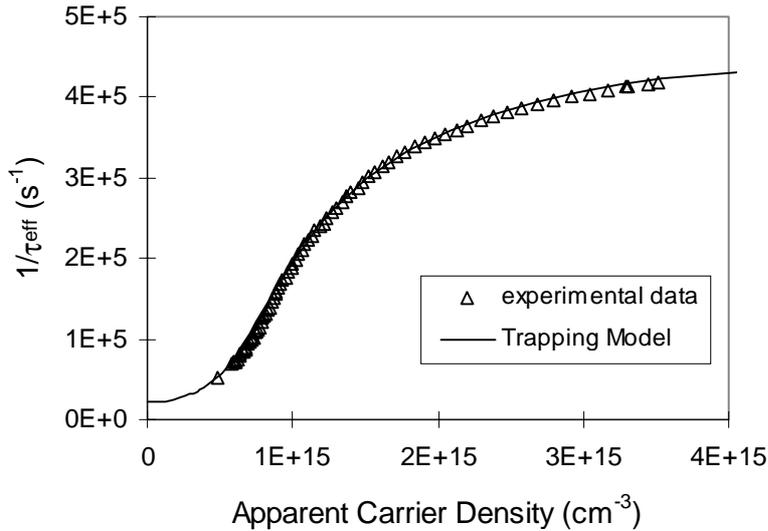


Figure 3. Photoconductance measurement of multicrystalline Si wafer 8A, from the bottom of ingot #8 showing trapping. The continuous line is a fit using the trapping theoretical model.

The trapping model explains the experimental dependence of the effective lifetime on injection level very well and allows the trap density to be determined. As an example, Fig.3 shows the model fitted to a mc-Si wafer from ingot #8. Preliminary evidence suggests that this “trapping” effect might

be related to crystallographic defects in the material. Metallic impurities produce the opposite type of behaviour, as we shall see later. Ingot #8, which was suspected to be defective due to problems during the growth process, proved to be of inferior quality and showed much more pronounced trapping. Wafers from the top of ingot #6 also showed more pronounced trapping than wafers from the central region (see Fig. 2), and this is probably correlated to the higher dislocation density measured in them.

5. RECOMBINATION DUE TO METALLIC IMPURITIES

Cross-contamination between mc-Si and ultra-pure FZ wafers can be used to detect the presence of metallic impurities in the mc-Si wafers [17]. The cross-contamination occurs when high purity float zone wafers are placed very close to the mc-Si samples during a high temperature step: a proportion of the mobile impurities present in the multicrystalline wafers effuse out of them and is absorbed by the adjacent float zone wafers. The process is very much like phosphorus (or boron) doping using solid sources; in this case the mc-Si wafers are the dopant sources and the dopant species are transition metals.

Several controlled cross-contamination experiments consisting of a light POCl₃ diffusion at 840°C for 25 minutes followed by thin oxide layer growth at 900°C for 30 minutes, were performed on wafers from various regions of two commercial cast mc-Si ingots. The lifetime of the control wafers was directly correlated to the lifetime of the adjacent mc-Si wafers and the corresponding ingot region, as can be seen in Table II. Considering that the typical lifetime of a clean 1 Ωcm FZ wafer processed in an identical way is of the order of 400 μs, the results indicate that the concentration of mobile impurities is low in the central region (control wafer lifetime 330 μs), while it is high at the bottom of the ingot (most likely due to contamination from the crucible), and also at the top (caused by transition metal segregation during ingot growth) [15]. Subsequent SIMS measurements identified the presence of Fe and Cr both in the mc-Si wafers and in the cross-contaminated FZ wafers.

In an elegant way, the cross-contamination technique transfers the metallic impurities to a crystallographically perfect medium where their effect on carrier recombination can be studied without the combined influence of grain boundaries, dislocations and other defects present in the

mc-Si wafers. In fact, trapping effects make it impossible to explore the low carrier density range in many mc-Si wafers, particularly those from the defective ingot #8. Figs. 4 and 5 show the results of intentional cross-contamination experiments that used 1 Ωcm FZ wafers as controls, a resistivity that is quite common for commercial solar cells. It is reasonable to think that, for a given level of contamination, the lifetimes of the 1 Ωcm FZ wafers are an upper bound of those achievable with mc-Si wafers of the same resistivity. Nevertheless, the concentration of metals in the FZ wafers is lower than that of the source mc-Si wafers and, very likely, it is higher near the surfaces than in the volume of the wafers.

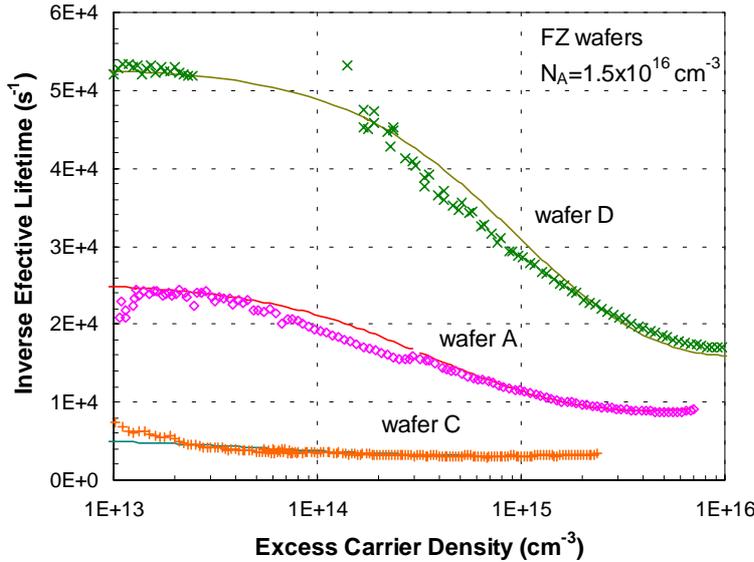


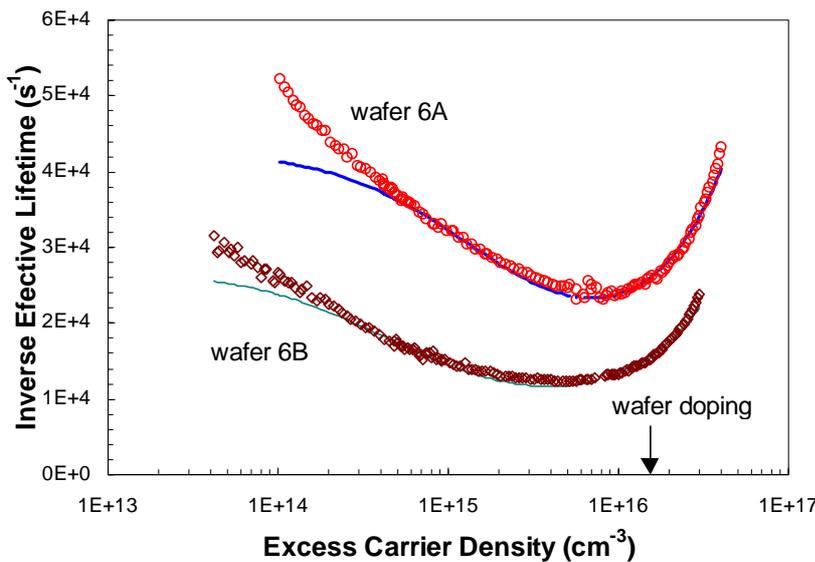
Figure 4. Inverse effective lifetime as a function of carrier density for three float-zone 1 Ωcm silicon wafers having different degrees of contamination from top (wafer FZ8D), centre (wafer FZ8C) and bottom (wafer FZ8A) regions of multicrystalline silicon ingot #8. The continuous lines are theoretical fittings using the S-R-H recombination model.

The experimental lifetimes shown in Figs.4 and 5 for FZ control wafers corresponding to the central and end regions of ingot #8 (crystallographically defective) and ingot #6 (standard quality), respectively, show a dependence with carrier density that can be modelled with a single S-R-H recombination level. The data can be fitted with Eqs. 1 and 2 using the fundamental τ_{no} and τ_{po} given in Table II. The ratio between τ_{no} and τ_{po} is not the same for all the wafers, indicating that the recombination centres are probably different.

	<i>Max. τ_{eff} of control FZ (μs)</i>	<i>of τ_{no} (μs)</i>	<i>τ_{po} (μs)</i>	<i>Contamination source</i>	<i>Max. τ_{eff} of mc-Si source wafer (μs)</i>
FZ 8A	110	50	2000	bottom, ingot #8	1.9
FZ 8D	60	21	300	top, ingot #8	1.2
FZ 8C	330	400	10^5	Centre, ingot #8	4
FZ 6A	45	30	250	Top, ingot #6	2.3
FZ 6B	100	50	1500	Near top, ingot #6	10

Table II. Recombination parameters of 1 Ωcm FZ wafers cross-contaminated by mc-Si wafers from different regions of two multicrystalline silicon ingots.

To complete the experiment, the mc-Si wafers (and also some cross-contaminated FZ wafers) were subjected to a POCl_3 gettering treatment followed by silicon etch and an additional light diffusion to passivate the surfaces. The FZ controls recovered the high lifetimes typical of the uncontaminated state. The gettered mc-Si wafers did not produce cross-contamination, indicating that the 3h, 900 °C gettering was sufficient to extract the majority of the mobile metal atoms. Most of the mc-Si wafers that had been identified as containing a high density of mobile impurities by the previous cross-contamination experiment improved markedly with phosphorus gettering. Nevertheless, wafers from the top region of both ingots had a very high density of dislocations ($>10^6 \text{ cm}^{-2}$) and did not improve with gettering [5]. The final lifetimes of the gettered mc-Si wafers can be considered to be a measure of their crystallographic quality. This quality proved poorest at the top of the ingots. To



predict the response to gettering the cross contamination experiment needs to be complemented with a measurement of the dislocation density.

Figure 5. Experimental (open markers) variation of the effective lifetime with excess carrier density for two 1 Ωcm FZ wafers contaminated with mc-Si wafers from top (wafer FZ6A) and near top (wafer FZ6B) regions of ingot #6. The continuous lines are the corresponding S-R-H fits.

6. DEVICE IMPLICATIONS OF METALLIC CONTAMINATION

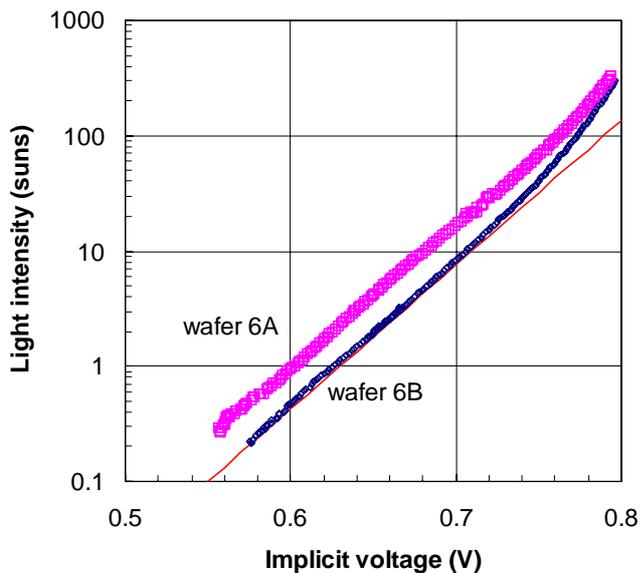


Figure 6. Implicit voltage vs. light intensity of two FZ wafers (FZ 6A and FZ 6B) with phosphorus diffusions on both sides and different degrees of metallic contamination.

As shown in the previous sections, the detailed analysis of the photoconductance data can be quite complex. A much more straightforward way of presenting them is to use the well-known language of device voltage [18]. This is shown in Fig. 6 for two 1 Ωcm FZ silicon wafers having phosphorus diffusions on both sides. These particular wafers were intentionally

cross-contaminated with the mc-Si wafers from ingot #6 (their corresponding effective lifetimes are shown in Fig.5) and exhibit a relatively high recombination rate, higher than standard 1 Ω cm FZ material. The implicit open-circuit voltages at one-sun illumination are 600 mV and 625 mV for wafers F6A and F6B, respectively. The straight line in Fig. 6 is a single-exponential fit to the implicit I_{sc} - V_{oc} characteristics with a $J_0 \approx 1.3 \times 10^{-8}$ $A\text{cm}^{-2}$ and an ideality factor of 1.35. The ideality factor decreases to nearly 1 only at very high light intensities. This relatively high ideality factor can be expected to have a detrimental effect on the fill factor of a solar cell fabricated with this wafer. Interestingly, the origin of high ideality factor and low fill factor can be traced to the presence of metallic impurities in the material.

7. CONCLUSIONS

The experimental fact is that carrier recombination in a semiconductor is complex and can not, in general, be accurately described by a constant lifetime value except within a restricted range of carrier densities. The *effective* minority-carrier lifetime can be affected by several physical mechanisms simultaneously that can have different dependencies on carrier injection level. The analysis of the injection dependence of the effective lifetime provides insight into these mechanisms.

The specific dependence of the effective lifetime on injection level can be quite different for multicrystalline silicon wafers. Frequently, it *increases* with injection level at very low carrier density levels, goes through a maximum and then decreases at high injection levels. The most likely physical reason for the measured dependence at low injection levels is that the bulk minority carrier lifetime *increases* due to the nature of the SRH recombination mechanism in the volume of a semiconductor. The strong change of τ_{bulk} observed in many samples can be explained with a high asymmetry in the electron and hole fundamental lifetimes. We have found evidence that, at least in some cases, the magnitude of SRH recombination is related to the concentration of metallic impurities in the material. Translated to the language of device voltage, the injection-dependence of the effective lifetime can produce increased ideality factors in the current-voltage characteristics and reduced fill factors and output voltages.

There are, nevertheless, cases where the apparent effective lifetime increases monotonically as the injection level is decreased. This behaviour, frequent in multicrystalline silicon, can be explained in terms of carrier trapping effects. The origin of these traps is still uncertain, with preliminary evidence indicating that they might be related to the crystallographic quality of the material, including the grain size and the dislocation density. If such correlation can be confirmed, measurements of the trapping effect might be very useful to diagnose the crystallographic quality of multicrystalline silicon.

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