



Acceptable contamination levels in solar grade silicon: From feedstock to solar cell

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ABSTRACT

Ultimately, alternative ways of silicon purification for photovoltaic applications are developed and applied. There is an ongoing debate about what are the acceptable contamination levels within the purified silicon feedstock to specify the material as solar grade silicon. Applying a simple model and making some additional assumptions, we calculate the acceptable contamination levels of different characteristic impurities for each fabrication step of a typical industrial mc-Si solar cell. The acceptable impurity concentrations within the finished solar cell are calculated for SRH recombination exclusively and under low injection conditions. It is assumed that during solar cell fabrication impurity concentrations are only altered by a gettering step. During the crystallization process, impurity segregation at the solid-liquid interface and at extended defects are taken into account. Finally, the initial contamination levels allowed within the feedstock are deduced. The acceptable concentration of iron in the finished solar cell is determined to be 9.7×10^{-3} ppma whereas the concentration in the silicon feedstock can be as high as 12.5 ppma. In comparison, the titanium concentration admitted in the solar cell is calculated to be 2.7×10^{-4} ppma and the allowed concentration of 2.2×10^{-2} ppma in the feedstock is only two orders of magnitude higher. Finally, it is shown theoretically and experimentally that slow cooling rates can lead to a decrease of the interstitial Fe concentration and thus relax the purity requirements in the feedstock.

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1. Introduction

The requirements on the silicon quality for photovoltaics (PV) are not as high as for the electronic industry. Therefore, alternative and cheaper methods to purify silicon are being developed for the PV market. The key question is what contamination levels in silicon can be considered as acceptable, i.e. what contamination levels are allowed to maintain the performance of the solar cell? Criteria for the specification of solar grade silicon (SoG-Si) that have been published by different authors differ in orders of magnitude and the origin of these values is not always clearly justified [1–8]. Some of the data together with the contamination levels found in electronic grade silicon (eg-Si) are given in Table 1. In the early eighties, the well-known *Westinghouse* study was carried out: the performance of solar cells that were made of intentionally contaminated Czochralski-grown single crystalline silicon (sc-Si) wafers were investigated [1]. Recently, Geerligs et al. [7] have started to repeat these extensive experiments for mc-Si.

The objective of this work is to calculate the acceptable contamination levels in silicon at different stages of the solar cell fabrication cycle (see Fig. 1) employing a simple theoretical model and some additional assumptions. An actual industrial process for mc-Si has been considered. Cañizo et al. [6] have done similar calculations for single sc-Si solar cells. In contrast to the sc-Si process, the effects of impurity precipitates in mc-Si have to be taken into account as they can act as impurity sources or as internal gettering sites. The allowed impurity concentration in the finished solar cell C_c , in the silicon wafer C_w and in the silicon feedstock C_u have been calculated for some typical impurities. Finally, several process modifications are discussed that have the potential to relax the previously established requirements on the silicon purity.

2. Theoretical model

Calculations have been done for p-type silicon devices. The starting point for our model calculations is an electron lifetime in the finished device of $\tau_e = 20 \mu\text{s}$. *PC1D* simulations, which corresponds to a typical industrial cell structure, have shown that this lifetime value results in an efficiency of about 15%.

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Table 1

Literature threshold values of different impurity types for the specification of SoG-Si feedstock material (C_u) and wafers (C_w); multi- (mc) and single-crystalline (sc) wafers materials are considered; the corresponding contamination levels found in electronic grade silicon (eg-Si) are also shown

Element	C_u [36] eg-Si [ppma]	C_u [8]- [ppma]	C_u [7] mc [ppma]	C_u [6] sc [ppma]	C_w [6] sc [ppma]	C_w [1] sc [ppma]
Ti	<0.003	0.4	0.17	2×10^{-3}	4×10^{-7}	5×10^{-6}
Cr	<0.003	3	–	0.1	1×10^{-5}	8×10^{-4}
Fe	<0.01	20	>16	9	5×10^{-4}	1×10^{-3}
Cu	<0.003	6	–	8	0.02	1

2.1. Contamination level C_c in the silicon solar cell

In a first step, the acceptable concentration C_c^j of the impurity type j in the finished solar cell has been calculated. Only Shockley Read Hall (SRH) recombination at atomic point defects as well as recombination at their precipitates have been considered. To account for the presence of other impurity types, it has been assumed that the sum of all other impurities has the same recombinative impact as the impurity type j :

$$\frac{1}{\tau_e} = \frac{1}{\tau_j} + \frac{1}{\tau_{\text{other}}} = \frac{2}{\tau_j} \quad (1)$$

In Section 4 the influence of this assumption will be discussed. The lifetime τ_j itself is composed of the following two terms:

$$\frac{1}{\tau_j} = C_{c,i}^j \sigma_j v_{\text{th}} + 4\pi r_0 D_e N_p^j. \quad (2)$$

The first term describes the SRH recombination at atomic point defects of concentration $C_{c,i}^j$, which can have interstitial or substitutional form. σ_j is the electron capture cross-section of the impurity type j and v_{th} is the thermal electron velocity. Following a model applied by Cañizo and Luque [9], the second term accounts for recombination caused by precipitates of the considered impurity type. The recombination at precipitates has been assumed to depend only on the electron diffusion coefficient D_e , the density of precipitates N_p^j and their radius r_0 (precipitates are assumed to be spherical). For our calculations, a precipitation radius of 100 nm has been chosen, in accordance with results of Buonassisi et al. [10,11]. Hence, there are two unknown variables, $C_{c,i}^j$ and N_p^j , that determine the electron lifetime in the solar cell. Further assumptions that depend on the particular contaminant type have had to be made during calculation. They are discussed in detail in Section 3.

The concentration $C_{c,p}^j$ of precipitated impurity atoms is proportional to the density N_p^j and radius r_0 . In our model, Cu, Fe and Cr are supposed to precipitate in form of silicides [11,10,12] while Ti is supposed to form an oxide [13].

All in all, the acceptable concentration C_c^j of the impurity type j in the finished solar cell is the sum of dissolved and precipitated impurities:

$$C_c^j = C_{c,i}^j + C_{c,p}^j. \quad (3)$$

This acceptable contamination level is mainly determined by the contaminant electron capture cross-section and the precipitate density. Table 2 shows the capture cross-sections of some metal contaminants in silicon at room temperature.

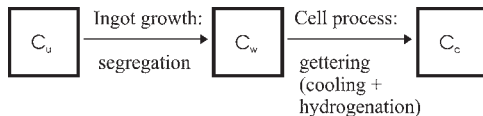


Fig. 1. Scheme of the solar cell fabrication cycle considered in the calculations; impurity concentrations C_u , C_w and C_c correspond to concentration in the silicon feedstock, in the silicon wafer and in the finished solar cell.

2.2. Contamination level C_w in the silicon wafer

In a second step, based on the obtained values of C_c^j , the acceptable contamination levels C_w^j in the silicon wafer have been calculated. It has been assumed that, during solar cell fabrication, the impurity concentration is only altered by a gettering step at 850 °C during 20 min. The gettering takes place during the phosphorous emitter diffusion and allows impurity atoms (in interstitial or substitutional form) to be partly extracted from the silicon wafer [14,9].

The mathematical model applied for the gettering process is described in detail in reference [9]. The effectiveness of the gettering step mainly depends on the diffusivity D_j and the solid solubility S_j of the particular impurity. Fast diffusing impurity atoms (e.g. Co, Ni, Cu) are more easily extracted than “slow” impurity atoms of low diffusivity (e.g. Ti, Zn). Furthermore, a high solid solubility facilitates the extraction as only mobile impurities in form of atomic point defects can be gettered. Table 2 displays the diffusivities and solid solubilities of several metal contaminants in silicon at 850 °C.

Again, the acceptable contamination level C_w^j in the silicon wafer before solar cell fabrication is the sum of the concentrations of dissolved $C_{i,w}^j$ and precipitated $C_{p,w}^j$ impurities.

2.3. Contamination level C_u in the silicon feedstock

In the third step, the acceptable contamination level C_u^j within the feedstock material before crystallization has been calculated. During directional solidification (DS), a cast process for the production of mc-Si, the ingot solidifies from the bottom to the top of the crucible. Due to a much higher solubility in the liquid phase, the vast majority of impurities stays in the molten fraction and thus is driven to the top of the ingot. The segregation profile for a fixed volume of solidifying liquid follows the Scheil equation [15,16]:

$$C_w^j = C_u^j k_{\text{eff}}^j (1 - f_s)^{k_{\text{eff}}^j - 1} \quad (4)$$

Here, k_{eff}^j is the effective segregation coefficient of the element j . It depends on the equilibrium segregation coefficient k_0^j and on the

Table 2

Electron capture cross-sections of several metal impurities in silicon at room temperature and their corresponding diffusion coefficients and solid solubilities at 850 °C [37,38]

Element	σ_e (RT) [cm ²]	D (850 °C) [cm ² /s]	S (850 °C) [cm ⁻³]
Ti	5.1×10^{-14}	1.35×10^{-10}	6.97×10^{10}
V	1.2×10^{-16}	1.0×10^{-9}	2.21×10^9
Cr	1.4×10^{-14}	3.48×10^{-8}	1.65×10^{12}
Mn	2.4×10^{-15}	1.04×10^{-6}	1.66×10^{13}
Fe	3.75×10^{-16}	1.16×10^{-6}	1.16×10^{13}
Co	1.7×10^{-15}	2.12×10^{-5}	1.99×10^{13}
Ni	6.0×10^{-17}	1.56×10^{-5}	3.54×10^{16}
Cu	8.5×10^{-18}	5.53×10^{-5}	1.13×10^{17}
Zn	3.0×10^{-15}	5.4×10^{-11}	1.36×10^{15}
Pd	3.2×10^{-15}	8.13×10^{-6}	1.43×10^{15}
Pt	7.0×10^{-15}	9.12×10^{-18}	6.15×10^{14}
Au _i	1.3×10^{-16}	5.86×10^{-12}	2.66×10^{13}

growth parameters of the crystallization process. f_s is the solidified fraction of the ingot which. Macdonald et al. [17] found that the concentration profiles of some elements (Cu, Fe, Co) deviate significantly from the profile predicted by the Scheil equation, mainly at the top and bottom of the ingot. In our model, the top 20% of the ingot has been discarded ($f_s = 0.8$) and the deviation at the bottom due to contamination from the crucible has not been taken into account.

In comparison to a sc-Si growth process, the k_{eff} of metal impurities in mc-Si is higher due to an enhanced segregation at grain boundaries [17,18]. Pizzini and Acciarri [18] have published effective segregation coefficients of different metal impurities in DS grown mc-Si ingots from purified metallurgical grade feedstock. These values have been employed in the Scheil equation to calculate the acceptable contamination levels C_u^j within the silicon feedstock.

3. Model results

The acceptable contamination levels from feedstock to solar cell have been calculated for four different metal contaminants of different recombination activity and diffusivity (see Table 2): titanium (harmful and slow), chromium (harmful and intermediate velocity), iron (intermediate harmfulness and fast) and copper (benign and very fast). All these metal contaminants form interstitial point defects in silicon. In order to solve Eq. (2), the following assumptions have been made for the particular contaminant types.

Due to the high solid solubility and the high diffusivity of Cu in silicon, our model suggests that all Cu precipitates are dissolved during the gettering step and that the interstitial concentration increases. Thus, we have assumed that only Cu interstitials determine the electron lifetime of $\tau_e = 20 \mu\text{s}$ in the solar cell.

Ti and Cr atoms are relatively slow diffusers in silicon. Calculations have shown that their respective precipitate concentrations do not vary noticeably during the gettering step and thus have been assumed to be constant. The density of precipitates has been chosen such that the lifetime is only reduced by less than 1%.

Iron is a relatively fast diffuser in silicon. We assume that in the presence of Fe precipitates the interstitial impurity concentration at 850°C immediately reaches its solid solubility value, $S^{\text{Fe}} = 1.16 \times 10^{13} \text{ cm}^{-3}$. This value has been taken as starting point for our calculations. The results are shown in Fig. 2 where the concentration of interstitial and precipitated impurity atoms have been drawn separately. During crystallization, the total amount of every contaminant is reduced by 2–3 orders of magnitude. The total impurity concentration after crystallization is dominated by precipitated impurity atoms. Their concentration is 1–3 orders of magnitude higher than the concentration of interstitial point

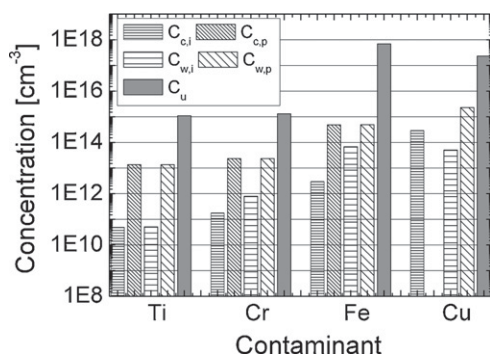


Fig. 2. Acceptable concentration of interstitial and precipitated Ti, Cr, Fe and Cu atoms, respectively, in a p-type solar cell ($C_{c,i/p}$) with $20 \mu\text{s}$ electron lifetime, in the silicon wafer ($C_{w,i/p}$) and in the silicon feedstock ($C_{u,i/p}$).

Table 3

Acceptable contamination levels of metal impurities with distinct capture cross-sections and diffusion coefficients (see Table 2)

Element	C_c [ppma]	C_w [ppma]	C_u [ppma]
Ti	2.7×10^{-4}	2.7×10^{-4}	0.022
Cr	4.7×10^{-4}	4.8×10^{-4}	0.026
Fe	9.7×10^{-3}	0.010	12.5
Cu	5.9×10^{-3}	0.046	4.6

defects for all four impurity types considered. During the solar cell fabrication process, only the concentration of dissolved Ti, Cr and Fe impurities is noticeably reduced as mentioned before. Due to the gettering step, the concentration of dissolved Fe is reduced by 74%, that of dissolved Cr by 77% and that of dissolved Ti by only 4%. The concentration of dissolved Cu is increased almost by a factor 6 because all precipitates are dissolved.

The total acceptable contamination levels in ppma are shown in Table 3. They correspond to the sum of interstitial and precipitated impurity concentrations that are given in cm^{-3} in Fig. 2. The total amount of any of the impurity types is not noticeably decreased during the gettering step except for Cu. The total amount of Fe is reduced by 2%, that of Cr by 3% and that of Ti by only 0.01%. In contrast, the total amount of Cu is reduced by 87% during the gettering step.

4. Model discussion

Comparing our model results with literature, a similar trend is observed: the metallic impurity that has to be less abundant in silicon is Ti, closely followed by Cr, while quite big amounts of Fe and somewhat lower amounts of Cu are acceptable. The acceptable Fe concentration in the feedstock C_u is of the same magnitude as the experimental result for mc-Si solar cells by Geerligs et al. [7]. Their threshold value for Ti is by a factor 8 higher than our calculated one.

The main difference with literature values for sc-Si is observed for C_u values of Ti and Cr in the silicon feedstock. Our values are at least one order of magnitude smaller than the literature ones. This is due to the enhanced segregation coefficient that has been applied in our model for mc-Si.

In contrast, our C_w values are higher than the values published by Davis et al. [1] and Cañizo et al. [6] for sc-Si. This is explained by the fact that in our model impurity precipitates are considered, as in mc-Si extended defects (e.g. grain boundaries) act as heterogeneous nucleation sites for supersaturated impurities [18]. Precipitates contain a great amount of impurity atoms (10^8 – 10^9 atoms/precipitate in silicides with $r_0 = 100 \text{ nm}$) but due to their lower density, these precipitates have a lower recombinative impact than dissolved metal impurities. Therefore, higher total amounts of impurities can be allowed in the mc-Si wafer. It has to be mentioned that the decoration of extended defects with impurities may increase their recombination activity. But in the case of large grain mc-Si, the electron diffusion length is not limited by recombination at grain boundaries and internal gettering of impurities to grain boundaries is advantageous [19].

The impurity reduction that results from our gettering model is consistent with experimental results by Macdonald et al. [20] except for Cu. As Cu is a very fast diffuser and its solid solubility decreases drastically with decreasing temperature it must be assumed that Cu forms precipitates immediately during cooling even for high cooling rates. This has not been considered in our model calculations and will relax somewhat the purity requirements for Cu.

It has been shown by Heuer et al. [21] that co-precipitations of different metal types take place. Intermetallic phases containing Cu,

Ni, Fe and Si have been observed, partly showing the characteristics of the corresponding metal silicides. However, in our model, only the precipitate radius and density are assumed to have an influence on the interaction between precipitates and dissolved impurities. Chemical interactions between different impurity types have not been taken into account as this is a complex subject on which more investigation has to be carried out.

In the end, the influence of the previous assumptions on the final results have been checked. Calculations have been repeated under the new assumption, that all other impurities present in the solar cell have three times the recombinative effect as the considered impurity type j (see Eq. (1)). The resulting acceptable contamination level C_c^j in the silicon feedstock is approximately half of C_c^j calculated before. This shows that more restrictive lifetime requirements decrease the acceptable contamination levels but not their order of magnitude.

5. Relaxation of purity requirements

There are mainly three tools to relax the purity requirements on the silicon wafer that can be applied during the solar cell process: passivation of recombination active point defects, extraction of impurities by means of external gettering and reduction of the interstitial impurity concentration by means of internal gettering.

In a typical industrial solar cell process, a co-firing step is included at the end of the process to form the front and back metallic contacts. During this fast annealing step at about 800 °C, hydrogen contained in the silicon nitride anti-reflection coating diffuses into the bulk of the silicon substrate. After hydrogenation, an

increase of the minority carrier lifetime between 50% and 400% has been reported for mc-, dendritic web, EFG and string ribbon silicon [22–26]. A hydrogenation step is not included in our calculations as it seems to be mainly crystalline defects (e.g. dislocations) and not metallic impurities that are passivated by the hydrogen [27,26].

It has been observed, that impurity extraction by means of a standard gettering step is not as efficient for mc-Si as for sc-Si [28,29,27]. Several research groups have proposed an extended gettering for mc-Si [30,31,10,32,26]. Buonassisi et al. [10] proposed a two-step gettering process for mc-Si and its effectiveness has been confirmed by Manshanden and Geerligs [32] and Tan et al. [26]. An increase in lifetime up to a factor 10 for low quality mc-Si was reported. The inclusion of an extended gettering step into our model is under investigation.

After a high temperature step, slow cooling is another tool to relax the purity requirements by improving the intragrain diffusion length in mc-Si. While fast cooling leads to supersaturation, spontaneous nucleation and thus to a high density of precipitates, a low cooling rate is supposed to increase the size of existing precipitates while maintaining the total precipitate density low. In other words, existing precipitates act as internal gettering sites. This hypothesis has been recently confirmed by Buonassisi et al. [33] in cooling experiments with intentionally doped mc-Si. We have included a slow cooling after the gettering step into our model. The following time dependence of the concentration of dissolved impurities is assumed [9]:

$$\frac{\delta C_i}{\delta t} = 4\pi r_0 N_p D^j(T(t)) [S^j(T(t)) - C_i(t)]. \quad (5)$$

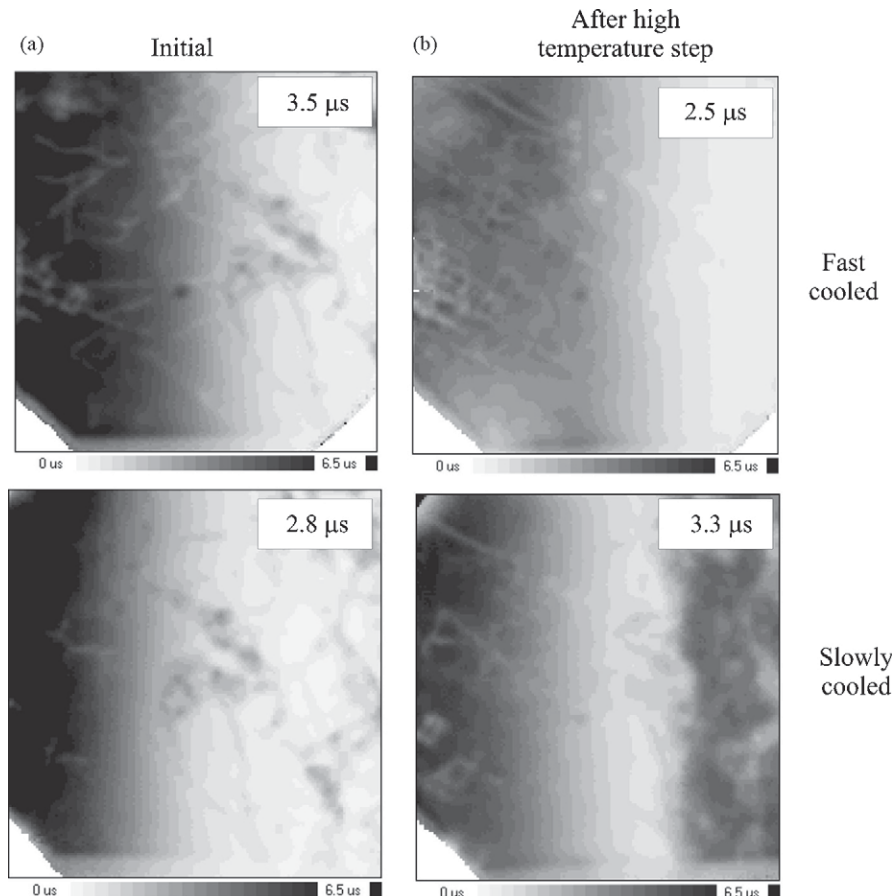


Fig. 3. Effect of fast and slow cooling on lifetime; μ -PCD mappings and average lifetime values of (a) initial and (b) final lifetime of two adjacent mc-Si wafers subjected to a high temperature step at 850 °C.

Here, S^j and D^j are the solid solubility and the diffusivity of the impurity type j . The density N_p and radius r_0 of precipitates are assumed to be constant. An exponential decay function has been used as temperature profile.

Fe is the only of the four contaminant types considered for which the final concentration of interstitials can be decreased noticeably. The diffusivities of Ti and Cr within the silicon are too low to make these impurities form clusters during cooling. On the other hand, Cu is such a fast diffuser that the clustering will be the same for slow and fast cooling.

Eq. (5) has been solved numerically via finite elements. It results that, maintaining the electron lifetime at $\tau_e = 20 \mu\text{s}$, the acceptable Fe concentration in the wafer is raised from 0.010 to 0.011 ppm and that in the feedstock from 12.5 to 13.9 ppm.

5.1. Experimental, results and discussion

A pair of bare, cleaned, adjacent mc-Si wafers have been subjected to an oxidation step at 850°C during 45 min. Subsequently, one wafer has been taken out of the furnace ("fast cooled"), the remaining wafer has been taken out after a slow cool down to 400°C during 3 h. Then, the oxide has been removed. Before and after the oxidation step, wafers have been cleaned, surface passivated with silicon nitride and the bulk lifetime as well as the concentration of interstitial Fe via Fe–B pair dissociation [34] have been measured by means of QSSPC. Furthermore, differential lifetime mappings have been measured by means of μ -PCD and are shown in Fig. 3.

The "fast cooled" wafer shows a lifetime degradation of 41% from its initial lifetime of $2.4 \mu\text{s}$ down to $1.7 \mu\text{s}$. In contrast, the slowly cooled wafer shows a lifetime enhancement of 36% from $1.4 \mu\text{s}$ up to $1.9 \mu\text{s}$. Average differential lifetime values are consistent with the actual lifetime values measured by means of QSSPC although the latter are somewhat lower. An explanation for the different results of the two methods is given in reference [35]. For the fast cooled wafer an increase of the interstitial Fe concentration $[\text{Fe}_i]$ from $(3 \pm 2) \times 10^{13}$ to $(5 \pm 2) \times 10^{13} \text{ cm}^{-3}$ is found. For the slowly cooled wafer a decrease from $[\text{Fe}_i] = (8 \pm 2) \times 10^{13}$ to $(5 \pm 2) \times 10^{13} \text{ cm}^{-3}$ is observed.

Results indicate that a slow cool down of a mc-Si wafer after a high temperature step can lead to an enhancement of the minority carrier lifetime. This lifetime enhancement seems to be correlated with an apparent decrease of the interstitial Fe concentration $[\text{Fe}_i]$. In contrast, a fast cool down of the wafer leads to a lifetime degradation and an apparent increase of $[\text{Fe}_i]$.

The resulting $[\text{Fe}_i]$ in both, the fast and the slowly cooled wafer, are higher than the literature value of the solid solubility of Fe at 850°C (see Table 2). This is contradictory to our hypothesis that $[\text{Fe}_i]$ immediately reaches its solid solubility value at 850°C . However, the uncertainty resulting from the applied method is of the same magnitude as the resulting $[\text{Fe}_i]$ values themselves. This is due to the low initial lifetime of the small grain mc-Si wafers used for the experiment. In such small grain mc-Si wafers, the electron diffusion length is probably limited by recombination at grain boundaries. Nevertheless, the trends observed for $[\text{Fe}_i]$ are the same as for the lifetime values measured by means of the two distinct methods applied.

Introducing the measured initial value $[\text{Fe}_i] = 8 \times 10^{13} \text{ cm}^{-3}$ into the slow cooling model (Eq. (5)), the final measured value $[\text{Fe}_i] = 5 \times 10^{13} \text{ cm}^{-3}$ is reached during a 3 h cool down to 400°C in the presence of $N_p = 1.5 \times 10^6 \text{ cm}^{-3}$ precipitates of radius 100 nm. This corresponds to a total Fe concentration of $C_{\text{Fe}}^{\text{tot}} = 2.5 \times 10^{14} \text{ cm}^{-3}$, a value in accordance with findings of several researchers [28,17]. Assuming that the electron lifetime is exclusively limited by Fe, the measured decrease of $[\text{Fe}_i]$ leads to an increase in lifetime from $3.3 \mu\text{s}$ to $5.2 \mu\text{s}$ according to Eq. (2). This

calculated enhancement of 63% is of the same magnitude as the actual enhancement we have found experimentally.

6. Conclusions

We have calculated the acceptable contamination levels of Ti, Cr, Fe and Cu in a p-type mc-Si solar cell, in the initial mc-Si wafer and in the silicon feedstock. These four impurity types have been chosen due to their different capture cross-sections and diffusivities within silicon. It results that Fe and Cu may be present in the silicon feedstock in amounts of several ppm whereas Cr and Ti may only have concentrations of some hundredths ppm. Because of the presence of precipitates in mc-Si, a higher total impurity concentration is allowed in mc-Si wafers in comparison to sc-Si wafers. This is because precipitates contain a great amount of impurity atoms but are less recombination active than interstitial impurities due to a lower density. On the other hand, the total impurity concentration in the feedstock has to be lower for the growth of mc-Si as the segregation coefficient in mc-Si growth is enhanced due to grain boundary segregation.

An effective tool to relax the purity requirements in mc-Si is slow cooling after a high temperature step. Due to a slow cool down of low quality mc-Si wafers, an increase in lifetime of 36% has been observed. This increase is consistent with results from our model.

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