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Significant minority carrier lifetime improvement in red edge zone in n-type multicrystalline silicon

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Abstract

We have carried out experiments on both boron diffusion gettering (BDG) and phosphorus diffusion gettering (PDG) in n-type multicrystalline silicon. We have focused our research on the highly contaminated edge areas of the silicon ingot often referred to as the red zone. Due to poor carrier lifetime attributed to these areas, they induce a significant material loss in solar cell manufacturing. In our experiments, the red zone was found to disappear after a specific BDG treatment and a lifetime improvement from 5 μs up to 670 μs was achieved. Outside the red zone, lifetimes even up to 850 μs were measured after gettering. Against the common hypothesis, we found higher dopant in-diffusion temperature beneficial both for the red zone and the good grains making BDG more efficient than PDG. To explain the results we suggest that high temperature leads to more complete dissolution of metal precipitates, which enhances the diffusion gettering to the emitter.

Keywords: n-type mc-Si, lifetime, gettering, red zone

1. Introduction

Since the discovery of a bunch of superior characteristics (see e.g. [1, 2, 3]), the possibility of using n-type silicon as a base material for solar cells has attracted plenty of interest. Nevertheless, nearly all the recent studies related to n-type material have concentrated on monocrystalline silicon as it is often speculated that n-type multicrystalline silicon (mc-Si) is not a viable material for solar cell manufacturing. However, there is only very limited data in the literature regarding n-type mc-Si, and therefore, it may be too early to make such conclusions.

One of the major benefits of n-type silicon is the insensitivity to some common metal impurities, such as iron. However, there are metal impurities which have been found to have a very strong recombination activity in n-type silicon. [4, 5] In addition, n-type mc-Si ingots are well-known to suffer from the red zone area that results from crucible contamination and raises the need for metal gettering in n-type as well. The most common gettering method in the current solar cell industry is phosphorus diffusion gettering (PDG), which works also in mc-Si, e.g. [6, 7, 8]. In addition, PDG is well-known to improve further when using a so-called low-temperature tail after the phosphorus in-diffusion. [9, 10, 11] However, PDG in n-type mc-Si is covered only in a small number of studies [12, 13, 14, 15, 16], very few of which deal with the problematic red zone area.

In an n-type silicon solar cell the formation of the emitter is often realized by boron diffusion. This conveniently opens the door for boron diffusion gettering (BDG).

However, typical temperature needed in boron diffusion is higher than for phosphorus and the gettering capability of the boron doped area is not considered to be as high as in PDG [17]. Thus there is an external contamination risk during the boron emitter formation. On the other hand, BDG is reported to be efficient when the boron concentration is high enough to form boron precipitates or boron rich layer. [18, 19, 20, 21] Indeed, in n-type mc-Si, Libal et al. [18] observed more than a 30% increase in lifetime after boron diffusion, although they did not report any results on the red zone. As in the case of PDG, a low temperature tail is observed to improve the BDG efficiency further in monocrystalline silicon. [20] For n-type mc-Si there is no data on this. However, since chromium, cobalt and nickel are fast diffusers [22], similarly to iron, there is no specific reason why adding a low temperature tail after the diffusion should not work in BDG as well.

In this paper we address the above issues by carrying out BDG and PDG experiments in n-type mc-Si with a specific emphasis on the reduction of the red zone area. In addition to comparing the minority carrier lifetime directly after the in-diffusion, we also study the effect of a low temperature anneal. The obtained results are discussed from the point-of-view of total metal concentration and the chemical state of the metals during and after the gettering treatments. We also examine the possibility of adding an optimized BDG or PDG step in a high efficiency n-type solar cell process. Finally the superiority of n-type silicon over p-type regarding the sensitivity to metallic impurities is discussed.

2. Experimental

Wafers used in this study were 200 μm thick $12.5 \times 12.5 \text{ cm}^2$ n-type multicrystalline silicon wafers cut in half. The wafer resistivity was 2 Ωcm . The wafers were taken from an edge position brick of the ingot at 30% brick height. Thereby, the wafers contained the red zone, i.e. the highly contaminated area at the wafer edge. All the wafers were so-called sister-wafers.

At first, the wafers were cleaned in an SC1 solution and dipped in a diluted HF solution. Before the actual process, the wafers were divided into three groups: (i) wafers experiencing BDG, (ii) wafers experiencing PDG and (iii) reference wafers experiencing no diffusion gettering treatment. For boron and phosphorus diffusion the boron and phosphorus sources were Filmtronics B154 spin-on dopant and Filmtronics P509 spin-on dopant, respectively.

The actual process began by a diffusion gettering treatment. Four different treatments were used, each consisting of a 60-min diffusion step followed by unloading to room temperature or an optional lower temperature tail. Boron and phosphorus diffusion times and temperatures were chosen in such a way that the resulting sheet resistances were comparable: approximately 40 Ω/sq in the case of boron and 25 Ω/sq in the case of phosphorus. As a summary, Table I presents the four different diffusion gettering treatments used in the experiments.

Table I. The diffusion gettering treatments used in the experiments.

Treatment	Temperature profile (and annealing ambient)
BDG_S	60 min at 930°C (95 % N ₂ , 5 % O ₂) + ramp down 4°C/min (100 % N ₂) + pullout at 800°C
BDG_LT	60 min at 930°C (95 % N ₂ , 5 % O ₂) + ramp down 4°C/min (100 % N ₂) + 3 h at 700°C (100 % N ₂)
PDG_S	60 min at 870°C (100 % N ₂) + ramp down 4°C/min (100 % N ₂) + pullout at 800°C
PDG_LT	60 min at 870°C (100 % N ₂) + ramp down 4°C/min (100 % N ₂) + 3 h at 700°C (100 % N ₂)

After diffusion the boron and phosphorus glasses were removed in an ammonium fluoride etching mixture and the wafers were again cleaned in an SC1 solution. The resulting boron and phosphorus doped silicon layers were then removed in order to get rid of the recombination in the heavily doped area that would disturb the bulk lifetime measurements. This silicon etching was done in a CH₃COOH:HF:HNO₃ solution, the thickness of the etched layer was approximately 12 μm. Next the wafers were cleaned again in an SC1 solution and dipped in a diluted HF solution. Finally, the wafers were oxidized for 40 min in oxygen atmosphere and subsequently annealed for 20 min at 900 °C in nitrogen atmosphere to passivate the wafer surfaces. The reference samples were prepared by etching 12 μm from the sample surface and subsequently oxidizing the samples similarly as the boron and phosphorus diffused samples. The minority carrier lifetimes in all samples were measured with microwave detected photoconductance decay (μ-PCD) by Semilab.

3. Results and discussion

Figure 1 (a) presents the minority carrier lifetime map of the reference sample, i.e. just after the crystal growth followed by surface passivation. The lifetime map is rather typical showing separate regions of different qualities resulting from the crystal

growth and crucible contamination. Metal behavior during gettering is likely to differ from region to region and therefore, it makes sense to study the regions separately. We have marked the regions with dashed lines and numbers 1-3 in Fig. 1. Figure 2 presents the response of various wafer regions to the different diffusion gettering treatments. The presented lifetime values are arithmetically averaged over the selected regions. Similar trends appear also in harmonically averaged lifetime values.

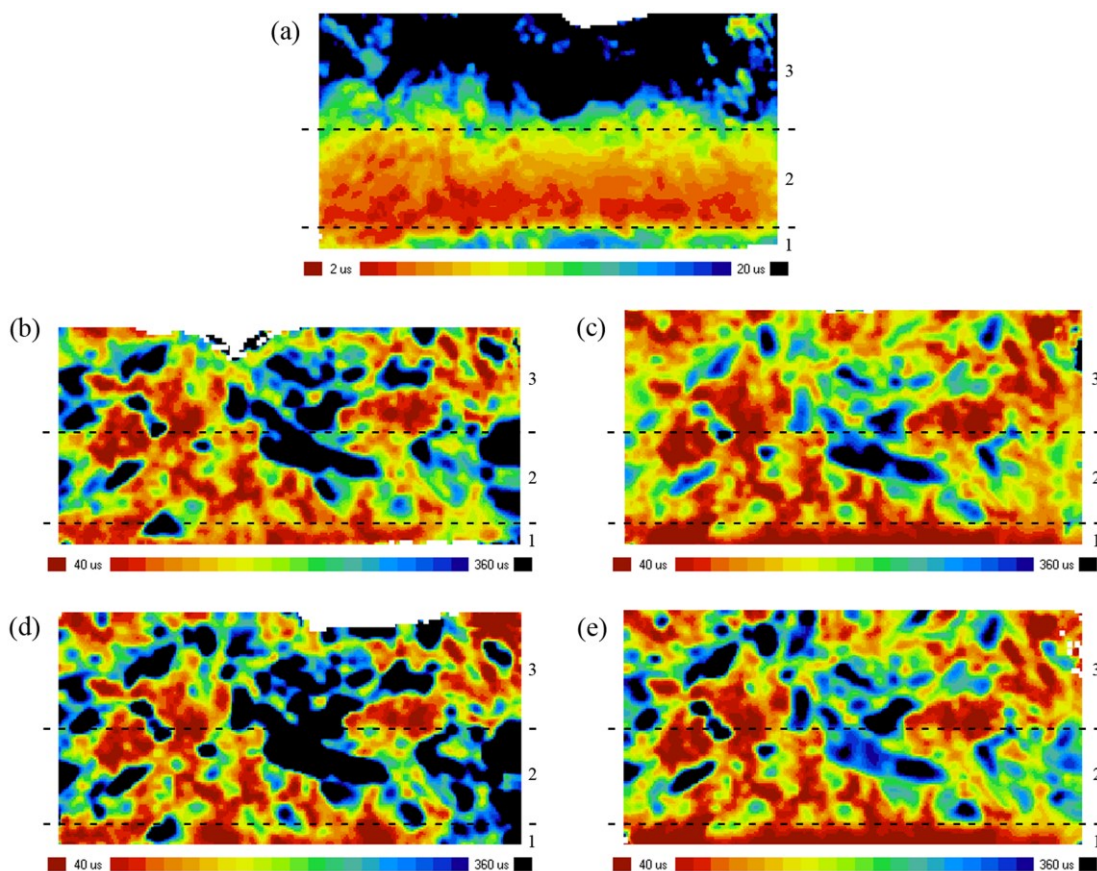


Figure 1. The lifetime maps of the samples after various treatments: (a) reference sample, (b) BDG_S, (c) PDG_S, (d) BDG_LT, and (e) PDG_LT. The sample size is $6.25 \times 12.5 \text{ cm}^2$. Notice the different scale in the lifetime maps.

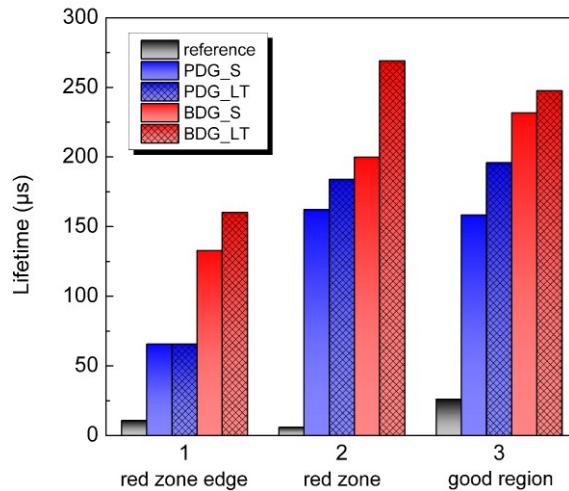


Figure 2. The response of various wafer regions to the different diffusion gettering treatments. The presented lifetime values are arithmetically averaged over the selected regions.

The lifetime in the reference sample (no diffusion gettering, Fig. 1 (a)) is quite low throughout the wafer and, as mentioned, a strong red zone (lifetime $\sim 5 \mu\text{s}$) is clearly visible. However, the lifetime can be seen to increase slightly at the outermost edge of the red zone (region 1). We attribute this increase in lifetime to the reduced concentration of dissolved metals through precipitation [23], i.e. to internal gettering. The total metal concentration increases towards the edge due to crucible contamination and thereby metals tend to precipitate in that area during the ingot cooling. Similar phenomenon has been observed earlier in p-type as-grown silicon, where iron has been shown to cause the red zone. [24, 25, 26] The possibility of trapping artifacts was ruled out by surface photovoltage measurements.

Notice that our initial lifetime map (Fig. 1 (a)) is not actually the as-grown lifetime map since we have passivated the surfaces by thermal oxidation at 900°C . During this high temperature step, as-grown metal precipitates are dissolved in the areas that contain metals less than their solubility at 900°C . Presumably, as-grown metal

precipitates remain insoluble only at the edge of the wafer (region 1), where the metal concentration is high enough. During cooling down from 900°C, internal gettering to the remaining precipitates takes place. Thereby, in our samples, the dissolved metal concentration is greatly reduced near the edge and the corresponding increase in lifetime is further enhanced.

The open question is what metal is limiting the lifetime here. Recent analysis by Schmidt et al. suggests that cobalt, chromium and nickel are the most deleterious metals in n-type material. [5] Shabani et al. have shown that all of these metals, with concentrations increasing towards the edge of the wafer, can be found from commercial multicrystalline silicon. [27] Based on the diffusivities and the solubilities of these metals [22], cobalt and chromium are quite probable candidates for the contaminants here. The issue was studied further by deep level transient spectroscopy (DLTS) measurements. DLTS measurements revealed two peaks with a concentration of around $2 \times 10^{12} \text{ cm}^{-3}$ in the red zone before gettering. After both BDG and PDG no peaks were seen indicating that the concentrations were decreased below the detection limit. The peaks could explain the changes in lifetime, but a precise identification of the defects was impossible.

Lifetime maps after boron and phosphorus diffusion gettering treatments (Fig. 1 (b) - (e)) show that both BDG and PDG are able to significantly increase the lifetime as expected. In the initially good region 3 BDG is slightly more effective than PDG: with the LT treatments the average lifetime is increased from 25 μs up to 195 μs and 250 μs for phosphorus and boron respectively. In good grains lifetime values even up to 650 μs and 850 μs are achieved correspondingly.

In the red zone, BDG is more clearly better than PDG, see Fig. 2 (regions 1 and 2). After BDG_LT the average lifetime in the red zone is increased from 5 μs up to 270 μs and locally values even up to 670 μs are achieved (corresponding values after PDG_LT 180 μs and 400 μs). This is the result of two things. Firstly, here boron is most likely supersaturated leading to a gettering capability comparable to PDG. Secondly, boron was diffused in at higher temperature (930°C) than phosphorus (870°C) (Table I). The higher temperature leads to faster dissolution of precipitated metals. [28] The dissolved metals move easily and thereby can be gettered by the boron layer. Consequently, the total metal concentration in the bulk is greatly reduced, which finally results in higher lifetime.

The above phenomenon also supports our low temperature tail results. In general, the low temperature tail enhances the diffusion gettering of dissolved metals but has only a minor effect on the precipitated metals. [28] Thereby, at low temperature the metals approach the dynamic equilibrium between dissolution and segregation to the emitter. The amount of getterable metals through dissolution differs from region to region but also depends on the in-diffusion temperature. Therefore, in PDG samples LT tail has almost no impact on the red zone as the precipitates remain insoluble. In the good region 3 there are soluble metals to be gettered, so the benefit of using LT tail is clear. In the case of boron, the impact of the low temperature tail extends to the red zone. At region 1, not all precipitates are dissolved (but still significant amount) so the improvement is not as pronounced as in region 2, where the initial concentration of precipitated metals is lower and the improvement is higher due to the more complete

dissolution during in-diffusion. In region 3, the total amount of metals after BDG_S is already so low that the impact of LT is again reduced.

It seems that our lifetime results from the red zone samples can be completely understood by utilizing the dissolution-gettering idea proposed by Plekhanov et al. [28]. However, all such experiments have been done on p-type only and, in addition the results are controversial regarding the general effectiveness and the benefits of such a high temperature process. For instance, Macdonald et al. [29] observed lifetime degradation due to generation of dislocations at high temperatures, when they applied the high-low temperature PDG process. Hartman et al. [30] obtained just the opposite result: in their experiments an extremely high temperature-anneal increased the quality of multicrystalline silicon by the reduction of dislocation density. However, they did not report any lifetime improvement. More promising results were obtained by Joshi et al., who observed an improvement in diffusion length, especially in areas with initially short diffusion length, after aluminum gettering at 1100°C. [31] Recently, also Schön et al. [32] and Michl et al. [33] observed a better lifetime in a process containing a temperature peak at 900°C before a standard PDG with multicrystalline silicon containing a total iron concentration of $3.5 \times 10^{13} \text{ cm}^{-3}$ and $2 \times 10^{14} \text{ cm}^{-3}$. Schön et al. attributed the increase in lifetime to a reduced density of iron precipitates [32] while Michl et al. showed that a reduction of interstitial iron concentration takes place due to the dissolution peak [33]. These results are actually related to each other since the reduced density of iron precipitates leads to the lower final interstitial iron concentration. This effect was most pronounced after a firing step [33], i.e., after a similar albeit much shorter high temperature step as the surface passivation used here. What we observed here was that typically the lifetime after

BDG was higher than after PDG, also in the initially good region 3. Although the reduction of dislocations does not occur at temperatures used in this study, it might be that the recombination activity of dislocations or other as-grown defects (e.g. oxide precipitates [34]) can be reduced at our temperature range. This phenomenon might also be an alternative explanation for the free surface or oxide layer gettering that has been proposed as an explanation for the lifetime improvement observed just after annealing or oxidation. [35]

In an n-type silicon solar cell, the emitter is usually formed by boron diffusion and the back surface field by phosphorus diffusion. Thus both BDG and PDG naturally occur in the fabrication process and, in theory the obtained gettering efficiency from both could be very high. However, the most optimal BDG or PDG treatments do not result in the most optimal emitter saturation current or back surface field operation. Thus trade-offs are necessary for directly diffused emitter or BSF profiles. One interesting option, especially in the case of high impurity concentrations, would be simultaneous boron and phosphorus diffusion gettering with a low temperature anneal. The time needed for an effective co-gettering could be noticeably reduced compared to plain BDG. [36] Obviously, if the etch back method [37] is used to form the emitter, it is possible to use high dopant concentrations and, in principal, separately optimize the gettering efficiency and the doping profile.

Our results show that in n-type multicrystalline silicon similar advanced gettering is needed as in p-type to minimize the effect of the red zone. In this respect, n-type seems not to have a significant benefit over p-type. On the other hand, in wafers with initially high lifetime (e.g. no red zone), the final solar cell performance is limited by

the contamination resulting from the actual cell process. In that case the main question is what the most common metal contaminants are in the solar cell process. If the main contaminant during processing is iron, then n-type has a clear benefit compared to p-type, but, e.g. in the case of chromium, the situation is different.

4. Conclusions

Our results clearly show that in n-type multicrystalline silicon both boron and phosphorus diffused layers can be used as effective sinks for impurities during gettering leading to a drastic increase in lifetime and a substantial reduction of the red zone area. We found BDG to be clearly more efficient due to the higher in-diffusion temperature. With the BDG_LT treatment the average lifetime in the red zone was increased from 5 μs up to 270 μs and locally values even up to 670 μs were achieved. Thus the formerly unusable ingot edge area was significantly reduced. Our results also indicate that n-type mc-Si could be a promising material for solar cell manufacturing even though the benefit of high tolerance to metal impurities (over p-type) is case sensitive.

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