Tradeoffs Between Impurity Gettering, Bulk Degradation, and Surface Passivation of Boron-Rich Layers on Silicon Solar Cells

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Abstract—The suitability of using a boron-rich layer (BRL) formed during boron diffusion as a gettering layer for n-type silicon solar cells is investigated. We have studied the gettering effectiveness, generation of dislocations and associated bulk lifetime degradation, and the impact of the BRL on the saturation current density, for different thickness of BRL and postoxidation conditions. Our results show that a BRL deposited using BBr_3-based furnaces is very effective at gettering interstitial Fe, removing more than 99.9% of Fe, but that the gettered Fe is released back into the wafer when the BRL is oxidized thermally. While we have detected no significant bulk degradation due to dislocations for the diffusion conditions used, there remains a tradeoff between the gettering effect and the recombination in the boron-doped region. Although the BRL can be oxidized chemically at low temperature using boiling nitric acid without losing the gettering effect, the lowest saturation current density is obtained by means of thermal oxidation, thanks partly to a lower boron surface concentration in thermally oxidized samples.

Index Terms—Boron diffusion, emitter saturation current, impurity gettering, silicon photovoltaic cells.

I. INTRODUCTION

PHOSPHORUS-DOPED n-type silicon for solar cells has received great interest in recent years, mainly due to its lower sensitivity to contamination from some common interstitial metal impurities and also immunity to the light-induced boron–oxygen defect [1], [2]. The greater tolerance of metal contamination makes n-type multicrystalline silicon solar cells an attractive idea [3], as the detrimental effects of metal impurities in the ingots are reduced [4], [5], [6]. Nevertheless, impurity gettering on n-type multicrystalline silicon is still important to achieve very high lifetimes, as demonstrated in [4] and [7].

The most commonly applied gettering method for multicrystalline silicon solar cells is phosphorus diffusion. Phosphorus diffusion gettering is well studied [8]–[11] and has the advantage that, for standard p-type cells, the gettering process occurs during the formation of the n+ region. As such, phosphorus gettering does not require any extra processing steps and is able to remove moderate levels of contamination. On the other hand, the gettering properties of boron diffusions are not so well established in practice and are more sensitive to the process conditions [12]. When applied to n-type multicrystalline silicon, the higher thermal budget for boron diffusions has been reported to cause precipitate dissolution and degradation of wafer lifetime [3]. However, it has also been reported separately that boron diffusions can improve bulk lifetime in n-type multicrystalline silicon through the growth of a boron-rich layer (BRL) [13]. Here, we investigate the conditions that are required for effective boron gettering of Fe in intentionally contaminated single-crystal float zone (FZ) silicon wafers. Although the motivation for BRL gettering is mainly for n-type multicrystalline silicon, monocrystalline silicon samples are used in this experiment so that the results will be dominated by the introduced metal species, and that the gettering will not be limited by precipitate dissolution. In other words, using monocrystalline silicon allows the results to be focused on the segregation coefficient and the capture potential of the BRL.

The BRL is a silicon boride layer grown between the boron silicate glass (BSG) and the heavily boron-doped region [14], when there is a "piling up" [15], [16] of boron dopants at the surface. The BRL is a hydrophilic layer that is also insoluble in hydrofluoric acid. Although the presence of the BRL has been linked to better sheet resistance uniformity [14], the BRL can also be undesirable under certain conditions, because the BRL can generate dislocations and cause bulk degradation. Kessler *et al.* reported that a BRL thicker than 10 nm can lead to thermal stress during cooling down, leading to degradation of bulk lifetime, due to the mismatch of the thermal expansion coefficient [15]. In addition, the presence of a BRL is expected to impede high-quality surface passivation of boron-diffused surfaces, which is essential for high-efficiency n-type solar cells. In order to remove the BRL, *in situ* thermal oxidation during the temperature ramp down [13] or low-temperature oxidation after

deglazing the BSG [17] are commonly used to convert the BRL into BSG. However, our previous results [18] have shown that BRL gettering is very sensitive to the postoxidation treatment, and the gettering effect can be completely negated after oxidation. As such, there are potential tradeoffs between the desired goals of efficient impurity gettering, minimized bulk degradation, and high-quality passivation of boron-diffused surfaces. In this paper, we explore these tradeoffs and aim to develop processing methods that allow all three goals to be achieved simultaneously.

II. EXPERIMENT METHODS

All samples were of size 45 mm × 45 mm and etched to about 250 μm thick using alkaline etching. The metal gettering effectiveness of the BRL was investigated using Fe as a model impurity, because interstitial Fe is a well-studied metal impurity whose concentration can be easily measured. Furthermore, although the exact values of the segregation coefficient will differ between elements, the gettering of different transition metals is expected to behave in a similar way qualitatively [19]. Fe gettering effectiveness was measured on 0.8-Ω·cm p-type FZ silicon wafers that were ion implanted with ^{56}Fe to a target volume dose of 1×10^{13} cm^{-3} Fe, while samples for bulk lifetime measurements were 1.0-Ω·cm n-type Czochralski silicon wafers that were not ion implanted. Sample preparation methods and conditions for ion implantation are the same as previous works [18], [20]. Bulk interstitial Fe concentrations ([Fe$_i$]) before and after gettering were measured using the FeB pair breaking method [21] and captured cross-sectional values from [22] and [23]. Quasi-steady-state-photoconductance-based lifetime measurements [24] taken at an excess carrier density of 1×10^{15} cm^{-3} were used for the results presented.

All of the boron diffusions were for 55min at 940 °C, followed by *in situ* thermal oxidation with variable O$_2$ concentrations during the 24-min ramp down to 700 °C. The two main parameters for the boron diffusion conditions investigated are the BBr$_3$ concentration during deposition, with N$_2$ carrier gas for BBr$_3$ (N$_2$-BBr$_3$) flow rate ranging from 15 sccm (0.043% BBr$_3$) to 60sccm (0.17% BBr$_3$), and the oxygen concentration during the *in situ* postoxidation ranging from 0% to 100%. Each of the parameters were varied one at a time, and increasing the BBr$_3$ concentration is expected to increase the BRL thickness [14], while increasing the oxygen concentration will increase the rate of oxidizing the BRL postdiffusion. All the other parameters, including the temperature time profile, the total gas flow rate during deposition and *in situ* postoxidation, and the oxygen concentration during the deposition, were kept constant.

After boron diffusion, all samples were deglazed in 10% HF for at least 5 min. The BRL is considered present if the wafer remains hydrophilic after rinsing. The resulting sheet resistances measured using the four-point probe were between 48 and 55 Ω/□ across the range of the diffusion conditions used. Unless stated otherwise, the BRL is then removed from the hydrophilic samples using boiling nitric acid [14]. The 10-min boiling nitric acid oxidation is expected to oxidize less than 10 nm of silicon [25], and the diffusion length of boron at the
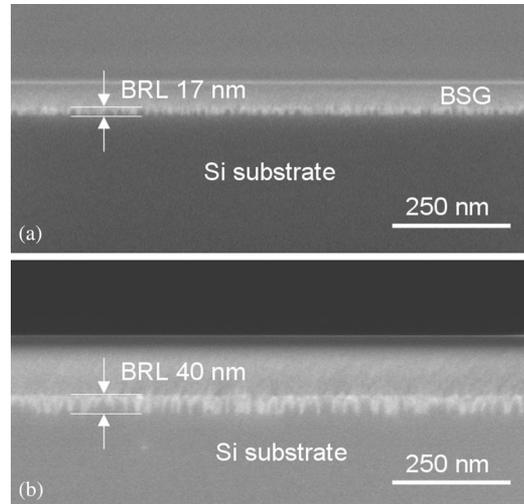

Fig. 1. Cross section SEM images [26] for determining the thicknesses of the boron rich layers (BRL) between the silicon substrate and the boron silicate glass (BSG) for a N$_2$ carrier gas flow of (a) 15 sccm and (b) 60 sccm.

boiling point of nitric acid of 121 °C is minimal; therefore, it will not cause significant changes to the boron diffusion profile or to the Fe distribution. This is further supported by the minimal increase in sheet resistance of less than 2 Ω/□ after the removal of the BRL, which is mainly due to the removal of the slightly conductive BRL [16]. The BRL thickness was measured by cross-sectional imaging with a field emission scanning electron microscope (SEM) [15].[1] The thicknesses were 17 and 40 nm for N$_2$ carrier gas flows of 15 and 60sccm, respectively, as shown in Fig. 1.

For Fe gettering and bulk recombination samples, the boron-doped regions were etched away using HNO$_3$:HF solution so that the measured lifetime would not be affected by emitter recombination. The surfaces were then passivated using plasma-enhanced chemical vapor deposition (PECVD) SiN$_x$ layers. The effective lifetime measured on PECVD SiN$_x$ passivated as-cut p-type control samples averaged 1.0 ms. Therefore, surface recombination is not a limiting factor for the p-type samples used to measure [Fe$_i$]. Emitter saturation current density J_{0e} [27] was measured using the Kane and Swanson method [28] on nonimplanted 100-Ω·cm n-type FZ silicon wafers. The boron-diffused surfaces were passivated by 30-nm-thick plasma-assisted atomic layer deposition (PA-ALD) Al$_2$O$_3$, after removing the BRL by boiling nitric acid. The dopant diffusion profiles of some samples were determined using electrochemical capacitance–voltage measurements (ECV) after measurement of the J_{0e}.

III. RESULTS AND DISCUSSION

A. Fe Gettering

The results in Fig. 2 show that the presence of the BRL is very effective at gettering Fe$_i$, removing more than 99.9% of the [Fe$_i$] for diffusion conditions with N$_2$ ramp down, comparable with

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[1]Images were taken at the Institute of Solar Energy Research Hamelin (ISFH) using a Hitachi S-4800 FESEM with accelerating voltages of 2 kV, working distances in the range of 1.5–3 mm, and emission currents of 10 μA.
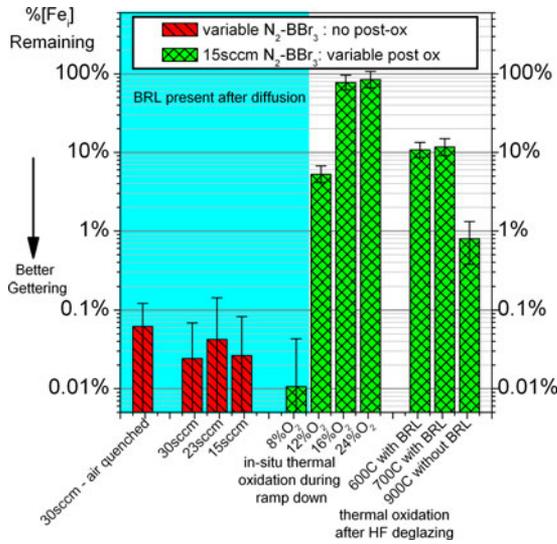
Fig. 2. Fraction of [Fe$_i$] remaining after gettering as a percentage of the initial bulk Fe$_i$ concentration. The blue shaded section represents samples that retained a BRL after high temperature processing, while the remaining samples had no BRL after processing.

phosphorus diffusion gettering with extended low-temperature annealing [18], [29]. The data on the left of Fig. 2 show that decreasing the BBr$_3$ concentration during the diffusion had no significant effect on the gettering; hence, the flow rate for nitrogen carrier gas for BBr$_3$ (N$_2$-BBr$_3$) was set to the lowest practical value of 15 sccm for subsequent diffusions with varying postoxidation conditions.

The results show that increasing the O$_2$ concentration during the postoxidation reduces the gettering effect significantly, with the gettering effect almost completely negated for O$_2$ concentrations above 16%. There is also a clear correlation between the presence of a BRL at the end of the diffusion and the gettering effect. When the O$_2$ concentration is increased to 12% from 8%, the BRL is fully oxidized by the end of the diffusion and the gettering effect diminishes rapidly.

In addition to BRL gettering, three other mechanisms for boron diffusion gettering have been reported. First, the higher solubility in the heavily boron-doped region due to Fermi level effect and ion pairing of Fe and B [30] can segregate Fe from the bulk to the p+ region. The second gettering mechanism is surface precipitation during cooling down, which is enhanced by the increased impurity concentration segregated to the p+ region due to the first mechanism [31]. The third mechanism is the segregation of impurities into boron precipitates within the heavily boron-doped region [19], [32]. As the boron-doped region is much thinner compared with the wafer, the first mechanism is expected to remove less than 1% and 20% of the [Fe$_i$] at 940 °C and 700 °C, respectively [30]. The second and third mechanisms require the supersaturation and precipitation of Fe and B, respectively; hence, it would require a sufficiently long duration of low-temperature anneal to be effective. For example, annealing times of more than 1 h were used in [31] and [32]. In this experiment, the diffusions are cooled down from 940 °C at a rate of 10 °C/min and unloaded once it cools down to 700 °C;

therefore, the gettering effects of other mechanisms are expected to be largely untapped in our experiment. Therefore, BRL gettering can be considered as the dominant gettering mechanism, with the exception of the samples with low-temperature oxidation (LTO) at 600 °C and 700 °C.

The results suggest that the Fe impurities are strongly segregated into the BRL during the deposition process. Our previous results with air quenching of the samples from diffusion temperature (included in Fig. 2 for reference) showed comparable gettering effectiveness [18], which indicates that the BRL gettering is effective at 940 °C, and most of Fe is gettered before the postoxidation process during cooling down. The lack of dependence on the cooling rate and the available time for precipitation of either B or Fe shows that the second and third boron gettering mechanisms are not effective in the diffusion conditions used. Furthermore, a more direct indication of the segregation effect of the BRL can be seen when the sample is oxidized thermally at 900 °C for 30 min after removing the BRL chemically, as indicated by the result "900 C without BRL" in Fig. 2. The boron-doped region is not removed during the thermal oxidation; hence, any Fe gettered in the boron-doped region would be driven back into the bulk. The results show that more than 99% of the Fe was gettered into the BRL.

Although the BRL is an excellent gettering layer, once it is oxidized into BSG, the segregation effect is lost and the Fe gettered in the BRL is released from the surface of the wafer. A small proportion of the Fe is trapped in the boron-doped region due to the higher solubility, but most of the Fe will be driven back into the bulk of the wafer. Considering that the diffusion length of Fe during a ramp down from 940 °C to 700 °C at a cooling rate of 10 °C/min is about 350 μm [22], most of the released Fe will be able to diffuse back into the bulk. The release of gettered Fe can also be clearly observed in the samples with LTO. The LTO samples were diffused and then ramped down in N$_2$ only to keep the BRL at the end of the diffusion, similar to the samples with no postoxidation and good gettering. However, instead of removing the BRL using boiling nitric acid at 121 °C, the samples were annealed in O$_2$ at 600 °C or 700 °C for 30 min. Although most of the [Fe$_i$] is gettered after the diffusion, as shown by the samples with boiling nitric acid oxidation, the bulk [Fe$_i$] is significantly higher after the low-temperature thermal oxidation, because the gettered Fe is released from the BRL back into the bulk. Nevertheless, around 90% of the Fe$_i$ is still gettered from the bulk and remains trapped in the heavily boron-doped region near the surface, due to the improved segregation to the p+ region at lower temperature.

B. Bulk Degradation

Heavy boron diffusions have previously been associated with significant degradation of the bulk lifetime, thought to be due to the generation of dislocations caused by lattice mismatch in heavily doped regions [33]–[35]. This is a potential disadvantage of using boron diffusions for solar cell fabrication, particularly in localized emitters. Furthermore, thick BRLs can also lead to bulk degradation during cooling down, possibly due to mismatch in thermal expansion coefficients [15]. This effect of the BRL is
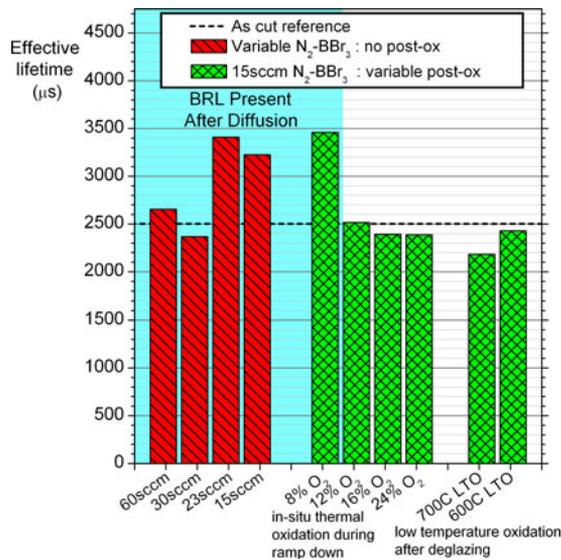
Fig. 3.   Effective lifetime of non-implanted n-type 1 Ω·cm CZ wafers after various diffusion conditions. The BRL and boron diffused regions were etched off before surface passivation.

separate to the lattice mismatch in heavily doped regions. This is demonstrated in the results of Kessler on boron-diffused samples with uniform sheet resistance, but nonuniform BRL thickness [15]. Despite having similar sheet resistance, significant bulk degradation is avoided in regions without BRL, but bulk lifetime degradation is found to be significant and proportional to the thickness of BRL when the BRL is present.

In this paper, the potential bulk lifetime degradation by BRL was investigated on n-type 1-Ω·cm nonimplanted wafers. Samples with postoxidation and no effective boron diffusion gettering received phosphorus diffusion gettering at 860 °C after etching off the boron-doped region to minimize the risk and effect of furnace contamination on the measured bulk lifetime. While results in Fig. 3 show that the bulk lifetime measured was lower in samples with higher BBr_3 concentration and thicker BRL, the effective lifetimes remained above 2 ms even for boron diffusions with 60 sccm of N_2-BBr_3 and BRL thickness of 40 nm. The extent of bulk degradation is less significant compared with previously reported results [15], possibly due to differences in the composition of the BRL or deposition conditions. In any case, for the diffusion conditions used in this study, there was no evidence of severe bulk degradation, even for samples with relatively thick BRLs.

C. Recombination in the Boron-Doped Region

An important aspect of implementing boron diffusions in silicon solar cells is the compatibility with high-quality surface passivation. Recombination in the boron-doped region is evaluated via the emitter saturation current density after surface passivation using PA-ALD Al_2O_3; the results of which are shown in Fig. 4. The first point to be noted is that the BRL prevents the passivation of the surface, limiting the J_{0e} to above 700 fA/cm^2, comparable with an unpassivated diffused surface. Therefore, the BRL should be removed before surface passiva-
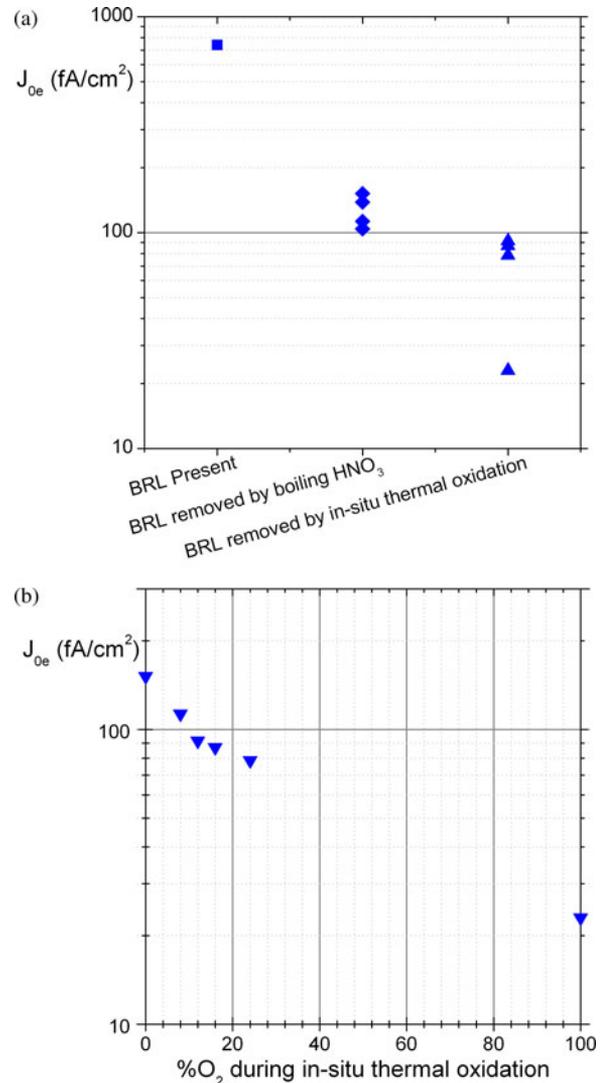

Fig. 4.   Emitter saturation current J_{0e} of (a) different BRL conditions and (b) as a function of O_2 concentration during in-situ thermal oxidation, measured on 100 Ω·cm n-type silicon wafers, passivated using PA-ALD Al_2O_3. Sheet resistance of the diffusions shown ranged from 48 Ω/□ to 55 Ω/□. Note that the BRL was present after diffusion and was removed chemically using HNO_3 for processes with O_2 concentration of 8% or less.

tion is applied. However, as noted above, if the BRL is oxidized thermally, this causes reinjection of gettered iron. Therefore, we have investigated the suitability of chemical oxidation of the BRL at low temperature to preserve the gettering effect while allowing high-quality surface passivation. The J_{0e} improves to between 100 and 150 fA/cm^2 after the removal of the BRL using boiling nitric acid, and application of surface passivation. As discussed previously, the boiling nitric acid does not alter the diffusion profile significantly; therefore, the improvement in J_{0e} after the removal of the BRL is not due to a lighter boron diffusion.

On the other hand, for samples where the BRL was removed thermally, the J_{0e} decreases as the O_2 concentration and rate of oxidation is increased during the ramp down. The J_{0e} was less than 30 fA/cm^2 for postoxidation with 100% O_2. The lower
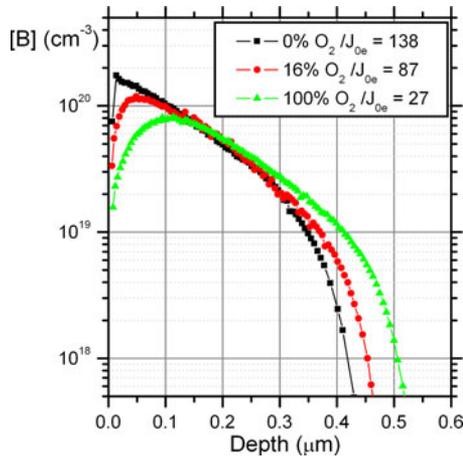
Fig. 5. Boron diffusion profiles after in-situ thermal oxidation at different concentrations of O_2, measured using ECV. The measured J_{0e} in fA/cm^2 is included for reference. Increasing the concentration of O_2 increases the rate of oxidation and lowers the boron surface concentration.

J_{0e} in samples with higher O_2 concentration is partly due to lower surface concentration [36] caused by surface depletion of boron during oxidation, as shown in Fig. 5. Although thermal oxidation can improve the J_{0e} values, the gettering effect is also negated by it. Therefore, there exists a compromise between J_{0e} and gettering, and ultimately, the boron diffusion should be optimized based on the quality of the silicon substrate and the requirement for gettering.

IV. CONCLUSION

The results presented here have shown that the BRL is very effective at gettering Fe impurities, even at $940\,^{\circ}$C. However, oxidizing the BRL thermally will release the gettered Fe back into the bulk, hence negating the gettering effect. Therefore, low-temperature chemical oxidation such as boiling nitric acid is required to maintain the gettering effect, while allowing surface passivation to be achieved. Our results showed no significant bulk degradation across all diffusion conditions, demonstrating that it is possible to achieve good gettering without significant bulk degradation. However, the J_{0e} results show that there remains a tradeoff between effective gettering and high-quality passivation of diffused surfaces, with the J_{0e} being higher when the BRL is chemically oxidized, partly due to the higher surface concentration. These results are useful in developing suitable boron diffusion recipes for high-efficiency n-type multicrystalline silicon solar cells.
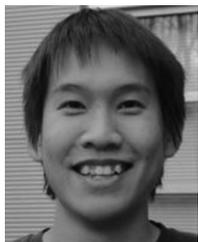
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