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Investigation of hydrogenation in n-type wafers with ring- and disc-shaped defect zones

Petra Manshanden[a], Paula C.P. Bronsveld[a]

[a]*ECN Solar Energy, Westerduinweg 3, NL-1755ZG Petten, the Netherlands*

Abstract

This paper investigates hydrogenation processes for improvement of the bulk recombination lifetime in n-type Czochralski (n-Cz) silicon wafers. Since in the near future an increase in the market share of n-type monocrystalline silicon solar cells is expected, research into the specific issues of using n-Cz Si wafers is rising. One of the widely recognized issues is the occurrence of ring- and disc-shaped defect regions, visible in recombination lifetime maps of the wafers or cells, that are responsible for significant efficiency reduction in solar cells. In this paper, it is demonstrated that these defect regions can be effectively passivated by an NH_3 plasma treatment. Also after deposition of silicon nitride, used for passivation and antireflection coating on both sides of n-PERT solar cells, the recombination activity in the defect regions is reduced.
The paper also reports on the evolution of the defect regions, after the passivation, in subsequent thermal processes. If wafers with passivated defect regions are subsequently exposed to 500-600 °C, the patterns reappear in lifetime maps. After exposure to temperatures above 650 °C for only seconds their recombination also increases. However, after these losses of passivation, the passivation can to some extent be restored by re-exposure to an NH_3 plasma or by annealing at lower temperatures up to 450 °C in the presence of a hydrogen source.
We correlate these phenomena to hydrogen sources and diffusion. Thus, the restoration of passivation depends on the presence of a hydrogen source in the wafer coating layers. For very strong ring shaped defect patterns, complete restoration of the passivation cannot be achieved in a stable fashion.

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Keywords: n-type Cz Si, hydrogenation, striation rings

1. Introduction

The PV market share of n-type monocrystalline silicon solar cells is increasing, with a possible share of 35% in 2025 [1]. This shift can be attributed to higher conversion efficiencies of n-type monocrystalline-Si based cells as

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compared to p-type mono- and multi-crystalline solar cells due to, among others, absence of boron-oxygen complex (B-O complex) related light-induced degradation and a lower sensitivity to common recombination active impurities, such as Fe. N-type silicon solar cells exist in various types, from cells with diffused junctions and front and rear metallisation grids (n-PERT cells) [2], to IBC [3], and HIT [4], and are in industrial production based on Czochraski (Cz) grown n-type silicon wafers.

For p- and n-type silicon wafers for PV, the research focuses on different aspects. For example, the lifetime of p-type silicon wafers is dominated by the B-O complex and impurities like Fe, whereas for n-type silicon the interaction of oxygen and intrinsic defects (such as vacancies and self-interstitials) is a key problem. In n-type silicon these lead to the formation of oxygen clusters that are associated with recombination active defects [5]. Within a wafer, regions with similar size and density of these clusters are practically always ring- or disc-shaped, due to the curved growth interface during ingot growth. Therefore, also regions in which the oxygen clusters cause high recombination activity, appear as ‘dark’ rings or disc shapes in lifetime maps. The presence of ring shaped defects could be a remnant of the P-band transition at the top of the ingot [6] or caused by fluctuations in the growth parameters (striations [7,8]).

Irrespective of their origin, extended exposure of wafers containing oxygen clusters to high temperatures (above ~600 °C) will dissolve oxygen clusters with a size smaller than a certain critical size, and increase the size of larger oxygen clusters. [9] This shift in cluster size distribution causes a net increase in recombination activity of the oxygen clusters, resulting in the appearance of ring-shape defect patterns in lifetime maps for wafers after thermal processing, that are either not present or less strong in the same wafers not exposed to thermal processing. Hence, issues with ring-shaped defects are mostly restricted to n-type Si solar cells with diffused junctions, which are exposed to high process temperatures.

A significant fraction (e.g. 10-20% [10]) of the n-type Cz Si wafers display stronger or weaker ring patterns in lifetime maps after processing into solar cells. In previous work, it was shown that for n-type Si solar cells an anneal at 200 °C could reduce or remove low minority carrier lifetime rings, both in cells and half-fabricates [11]. In the same paper it was shown that these changes were due to bulk defects. Similar effects have been demonstrated after illuminated annealing [12]. For both processes, hydrogen passivation of the defects composing the rings was postulated as an explanation for the mitigating effect. In this paper, the characteristics of the hydrogen passivation process on n-Cz wafers and its boundary conditions are further explored. The hypothesis of hydrogen passivation of the defects is used in this work as well.

2. Experimental details

2.1. Preparation of samples

Neighboring sample wafer sets were selected from sections of 3 different n-type Cz ingots for which rings were observed after cell processing. Based on the ring patterns observed in the lifetime maps of wafers from these sections, two typical types of wafer sets were selected. For the first type, separate rings can be distinguished in lifetime maps after high temperature processing (see e.g. figure 3). This is the type of ring pattern that is most commonly observed for diffused n-type solar cells. For the second type, that is less frequently observed, a large area in the center of the wafer is affected, although depending on the thermal exposure additionally some slightly darker or brighter rings can be recognized. This type may be present in a weak form before any processing. In the following, wafers with the first type of ring pattern will be referred to as ‘ring wafers’ and wafers with the second type as ‘disc wafers’. The resistivity of the ring wafers and disc wafers was 5 and 7 Ohm cm, respectively. Reference wafers with high bulk lifetime, and not susceptible to formation of ring or disc patterns after high temperature processing, were used to check the quality of the surface passivating coatings employed for measuring recombination lifetimes. These also had a resistivity of 5 Ohm cm. The ring wafers originated from a region in the ingot body close to seed end. The disc wafers originated from a region at about half the ingot length. All wafers in this test were 6” semi-square with a thickness of about 180 μm as cut.

Alkaline texture		
P+B co-diffusion		
HF glass removal		
Chemical polish		
NH_3 plasma	NH_3 plasma	NH_3 plasma
Surface cleans		
a-Si:H	ALD AlO_x	PECVD SiN_x:H
	Post Deposition Anneal at 500 °C	Short high T step
	NH_3 plasma	NH_3 plasma
		Anneals

Fig. 1.: schematic representation of the processing steps that both sample sets (ring and disc wafers) received in this work. Processing was sequential from top to bottom.

In figure 1 a schematic overview is given of the processing steps for the samples in this paper. Both sample sets received a simplified n-Pasha process [13], consisting of alkaline texture, co-diffusion of phosphorus and boron, and glass removal. Subsequently, the diffused regions were stripped by wet chemical polishing of about 10 micron per side, to exclude their contribution to the total recombination in the lifetime analysis.

After polishing, half of the wafers were subjected for 20 minutes to an ammonia (NH_3) plasma at 375 °C in a MW-PECVD Roth & Rau tool designed for deposition of silicon nitride coatings for solar cells. Subsequently, the wafer surfaces were cleaned and all wafers received surface passivating coatings on both sides. Three different surface passivating coatings were used, to test their interaction with the hydrogenation processes and stability of the bulk defect passivation. The tested coatings (always the same on both sides) were: 10 nm intrinsic hydrogenated amorphous Si (a-Si:H) deposited by PECVD, 72 nm hydrogen rich silicon nitride (SiN_x:H), deposited by MW-PECVD (microwave PECVD), or 6 nm aluminum oxide (AlO_x) deposited by ALD [14]. The AlO_x samples were subjected to a post deposition anneal at 500 °C, which is necessary to activate the surface passivation of AlO_x.

After lifetime measurements, the SiN_x coated wafers were subjected to a short high temperature step in a belt furnace, that is normally used to make contact between the screen printed metal grids and the diffused regions to test whether the passivation of ring shaped defects can withstand this step. Some AlO_x and SiN_x coated wafers, for which the rings were visible after surface passivation or after firing, respectively, were subjected again to an NH_3 plasma with the passivating layers on, to test whether the ring defects can also be passivated at the end of the solar cell process. This treatment was not applied to the a-Si:H coated samples, since it was expected that the surface passivation of these samples would not withstand exposure to 375 °C for 20 minutes of the NH_3 plasma. Also, for the SiN_x coated samples in which rings re-appeared after a short high temperature step, it was tested whether passivation of the rings could be restored by annealing at different temperatures, anticipating that the SiN_x:H coating could provide (additional) hydrogen for the bulk defect passivation.

2.2. Measurements

Photoluminescence images were made of all wafers using a home built photoluminescence (PL) set-up. In this set-up, the wafer is illuminated from the rear by LED lamps at a wavelength of 630 nm and an IR image is recorded from the front side with a Si CCD camera. Minority carrier lifetime values were obtained by performing transient and quasi-steady state photoconductance measurements using a Sinton WCT-120 system.

3. Results

3.1. Surface passivation quality

The quality of the surface passivating coatings was assessed from the effective lifetimes of reference wafers. For our highly charged AlO_x and SiN_x layers, could be expressed as J_0 using the Richter correction factors [reference]. Assessment of aSi:H quality in this manner is impossible and therefore the quality was assessed from the effective minority carrier lifetime.

The J_0 of the AlO_x coating was shown to be 5-10 fA/cm^2 after post deposition anneal (PDA). For the SiN_x samples the J_0 was found to be 15-20 fA/cm^2, which shows that the AlO_x coating is better than the SiN_x coating, despite leading to lower effective minority carrier lifetimes in the experimental wafers with ring and disc defects below. Hydrogenated amorphous silicon (aSi:H) contains too little fixed charges to induce a sufficient accumulation or depletion region for J_o analysis, however, the reference wafers give over 2 ms effective lifetime with aSi:H passivation which is the highest of all three coatings and therefore it can be inferred that the surface recombination of the aSi:H coating is the lowest.

3.2. Mitigation of ring shaped defects by NH_3 plasma treatment

Wafers known to be prone to ring- and disc-shaped regions of lower minority carrier lifetime received diffusion steps to activate the defects. Then they were polished and subjected for 20 minutes to a NH3 plasma. The wafers were coated with surface passivating coatings and analysed by PL and lifetime measurement.

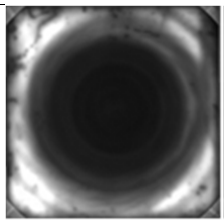
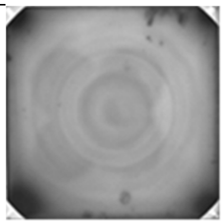
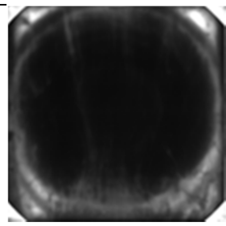
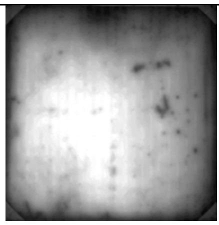
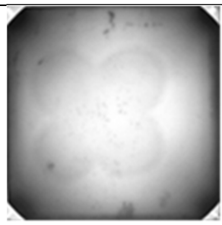
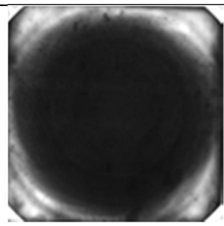
	aSi:H	SiN_x:H	AlO_x
No exposure to NH_3 plasma			
Lifetime in centre (τ_{eff})	184 μs	345 μs	21 μs
Exposure to NH_3 plasma			
Lifetime in centre (τ_{eff})	674 μs	513 μs	11 μs

Fig. 2.: Photoluminescence images depicting the effect of an NH_3 plasma treatment on **disc wafers** after the high temperature cell processing steps, preceding the passivation coating. Images were taken after the subsequent surface passivation coatings, which are indicated in the column headers (aSi:H, SiN_x, and AlO_x). The image greyscales are scaled for maximum contrast. The grayscales are very different between the different images.

In Fig. 2 photoluminescence images are shown for a neighboring set of disc wafers that have received the three different types of passivating coatings, comparing with and without the NH_3 plasma treatment before the surface passivating coating.

For the a-Si:H surface-passivated samples a strong disc pattern is observed if no NH_3 plasma treatment was applied after the high temperature steps. If the wafer was exposed to an NH_3 plasma after the high temperature steps, no disc pattern is visible, and a much higher lifetime is achieved. It is inferred that the NH3 plasma treatment is indeed effective to hydrogenate the bulk and thereby passivate the defect regions. Apparently the a-Si:H coating process, which occurs at low temperature, is not effective to passivate the defect regions.

For the SiN_x surface-passivated samples, the difference between the samples with and without NH_3 plasma treatment after the high temperature steps is much less pronounced, although still a more complete reduction of the ring pattern can be observed when the NH_3 plasma treatment was applied. The small difference implies that the effects of SiN_x deposition and the NH_3 plasma treatment (in terms of hydrogenation of the bulk of the wafer) are comparable. It is not surprising that the effects of SiN_x deposition and the NH_3 plasma treatment are comparable, since both processes happen at similar temperature in a hydrogen-rich plasma.

Wafers surface-passivated with AlO_x show the disc pattern very strongly with only a slight improvement when subjected to an NH_3 plasma after the high temperature steps. A possible explanation lies in the fact that the AlO_x coated samples were subjected to a post-deposition anneal (PDA) of 500 °C for 20 minutes to activate the passivating effect of the AlOx coating. This temperature step appears to have lowered the bulk minority carrier lifetime of these wafers.

In Figure 3 the same overview is given for the typical ring wafers. For this type of wafers, the minority carrier lifetime values are higher for all treatments and coatings. Qualitatively the same behavior is observed as for the disc wafers, except for the fact that for typical ring wafers the deposition of the surface passivating SiN_x:H coating is in itself already sufficient to eliminate the ring pattern, even without a preceding NH_3 plasma treatment.

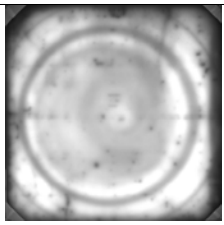
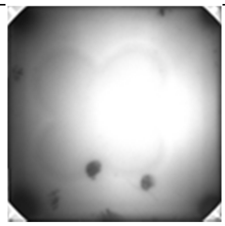
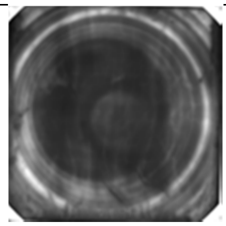
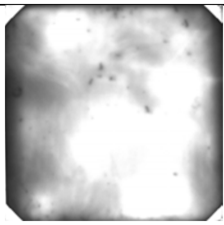
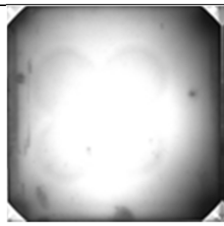
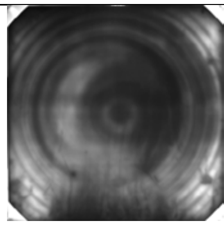
	aSi:H	SiN_x	AlO_x
No exposure to NH_3 plasma			
Lifetime in centre (τ_{eff})	1323 μs	791 μs	51 μs
Exposure to NH_3 plasma			
Lifetime in centre (τ_{eff})	2461 μs	803 μs	52 μs

Fig. 3.: Photoluminescence images depicting the effect of an NH_3 plasma treatment on **ring wafers** after the high temperature cell processing steps, preceding the passivation coating. The pictures are scaled on maximum contrast to make sure that rings are visible if present. The grayscale is, however, very different for the different samples.

3.3. Short high temperature exposure of the SiN_x coated wafers

Solar cells are commonly subjected to a short high temperature step after screen printing to sinter the metal contacts, called firing. This firing step can be expected to have an influence on the solar cells. The temperature investigated for this short high temperature step was ~650 °C for a few seconds. This step was applied only to SiN_x coated wafers as this coating is a standard part of the solar cell.

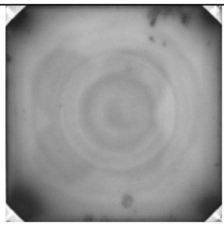
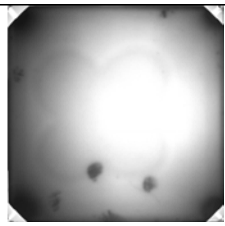
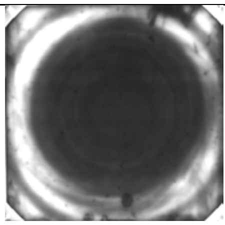
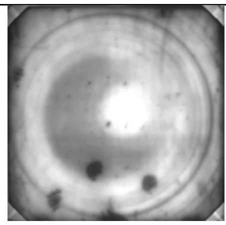
	Disc wafer	Typical ring wafer
SiN_x coated; before firing		
Lifetime in centre (τ_{eff})	345 μs	1092 μs
SiN_x coated; after firing		
Lifetime in centre (τ_{eff})	99 μs	899 μs

Fig. 4.: Photoluminescence images and minority carrier lifetime values before and after a short high temperature (firing) step of a few seconds at 650C, for the SiN_x coated wafers. Grey scale is individually optimized for maximum contrast.

As was already shown in figures 2 and 3, both types of ring defect patterns are practically absent after SiN_x coating. However, after application of the firing, ring patterns are clearly visible and similar to those for the samples with a-Si:H coating without NH_3 plasma treatment, assumed to be representative for the activity of the defect regions as caused by the high temperature diffusion. Apparently, the mitigating effect of the hydrogen containing plasmas is eliminated by the high T exposure in the firing step.

3.4. Effect of NH_3 plasma treatment after surface passivation coating

Applying a treatment after coating has as potential practical advantage that it could be applied to a solar cell after all thermal steps are completed. The NH_3 plasma of this study cannot be used for the wafers with the aSi:H coating, however, as this is less resistant against elevated temperature than SiN_x and AlO_x. Lowering the temperature of the plasma will ensure the aSi:H coating retains its surface passivating properties, but it will also decrease the reaction rate of the passivation.

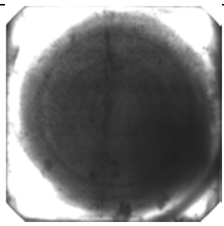
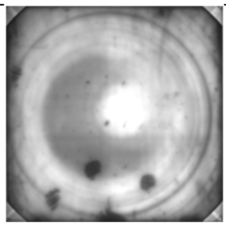
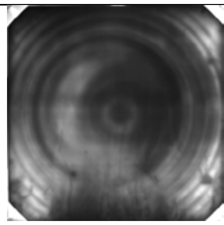
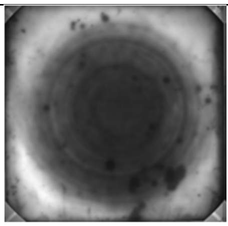
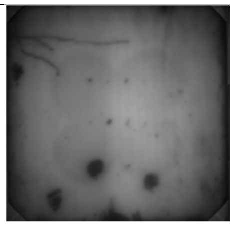
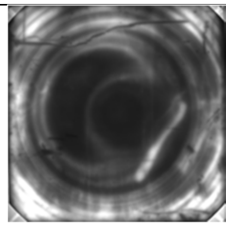
	Fired SiN_x (disc wafer)	Fired SiN_x (ring wafer)	AlO_x (ring wafer)
Initial condition			
Lifetime in centre (τ_{eff})	71 µs	899 µs	52 µs
After additional exposure to NH_3 plasma			
Lifetime in centre (τ_{eff})	162 µs	2051 µs	105 µs

Fig. 5.: Photoluminescence images and minority carrier lifetime values showing the effect of additional NH_3 plasma treatment, applied at back-end of processing, to the fired wafers with SiN_x , and to the AlO_x coated wafers. Grey scale is individually optimized for maximum contrast.

Since the ring and disc shapes appeared in the SiN_x:H-coated samples after the firing, and from the a-Si:H-coated samples it was observed that the NH_3 plasma treatment is effective to hydrogenate (passivate) the ring and disc shapes, an attempt was made to reduce the intensity of the defects again by performing an additional NH_3 plasma treatment on the fired samples with the SiN_x coating on. Application of a NH_3 plasma treatment after SiN_x coating leads to complete removal of the pattern for typical ring wafers, but does not eliminate the pattern completely for disc wafers. It was tested whether a similar reducing effect could be observed for the AlO_x coated wafers. However, here the additional NH_3 plasma treatment seemed to have only minimal effect and the ring pattern was still clearly visible. To separate the thermal effect (driving in hydrogen from the SiNx coating) from the plasma effect (supplying hydrogen from the NH_3 plasma), we investigate anneal in the next section.

3.5. Anneals of wafers with SiN_x coating at different temperatures

Since the ring/disc patterns in the SiN_x coated wafers are known to become less, or disappear, when exposed to elevated temperatures [11,12], this thermal dependence of the ring pattern was investigated in detail. SiN_x coated typical ring wafers which had been subjected to the firing step, in which the ring pattern was therefore clearly visible, were annealed at different temperatures in the range of 250-600°C in a box furnace for 10 minutes. To avoid the influence of previous annealing steps, separate (neighbouring) wafers from the ingot were used for each temperature.

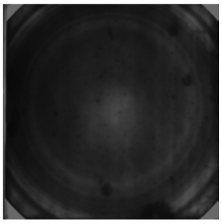
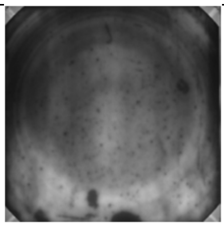
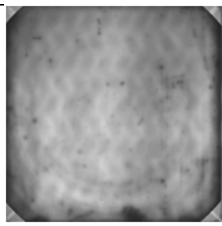
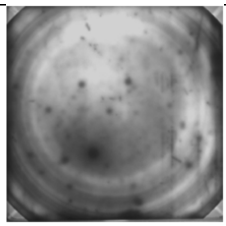
Temperature applied			
250 °C	350 °C	450 °C	600 °C
			

Fig. 6.: Photoluminescence images of SiN_x coated wafers that were fired, and subsequently annealed at different temperatures. The scaling is identical in all pictures.

In Fig. 6 the effect of the annealing subsequent to firing is shown in photoluminescence images. By raising the temperature of the anneal, the rings pattern first becomes less visible. (The pattern visible in the sample annealed at 450 °C is caused by the belt of the furnace used for the firing). If the anneal temperature is raised to 600 °C, the ring pattern becomes visible again. Previously [11], a different of SiN_x was used, hence the low temperature effect is not the same.

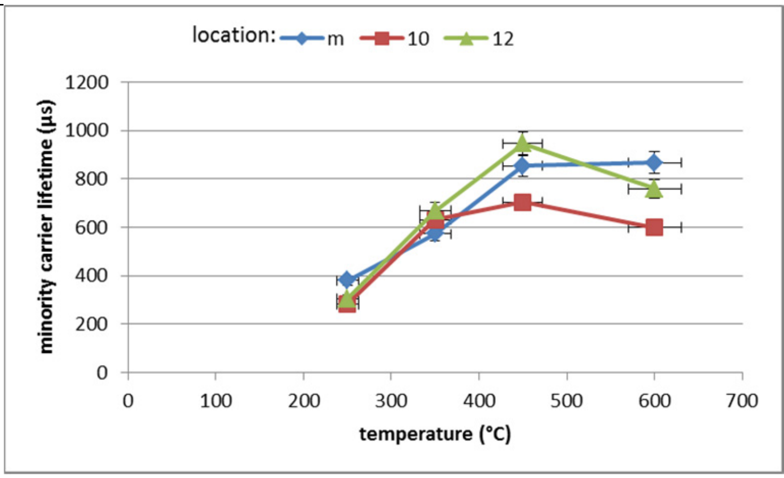
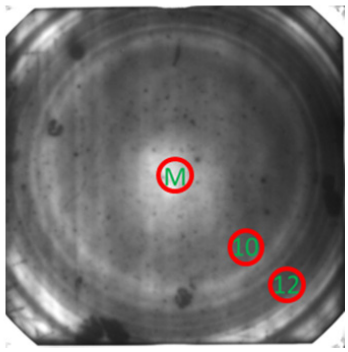
Minority carrier lifetimes on various locations on the wafer	Location on the wafer
	

Fig. 7.: Effect of various anneal temperatures on the minority carrier lifetime at several locations on the wafer (M, 10, and 12; respectively the middle, 10 cm from the edge and 12 cm from the edge)

To get a more detailed picture of the effect of the subsequent annealing on the minority carrier lifetime of these wafers, the lifetime has been measured on different locations along the diagonal where some broad rings were visible.

As the anneal temperature rises the minority carrier lifetime at all measured locations on the wafer increases. The rings that are re-appearing after anneal at high temperature do not suppress the lifetime to the initial status after firing. Probably higher anneal temperatures or longer times would result in lower minority carrier lifetime.

4. Discussion

The observed effects point at a process of reversible hydrogenation of bulk defects which is an interplay of hydrogen supply (from ambient or deposited coating), diffusion, release of hydrogen from defects, bulk, or coating, and transport barriers such as possibly AlO_x, with their respective temperature dependencies. In addition a change of bulk defects during these processes cannot be ruled out, but the experiments didn't clearly indicate that this happened.

Both the plasma used to deposit SiN_x and the plasma used to deposit a-Si:H contain hydrogen, which could have some influence on the bulk lifetime of the samples. As the deposition temperatures are different, the influence is not expected to be equal. Indeed, for the SiN_x it is clearly shown that the application of the coating can fully passivate the defects responsible for the ring shapes, while for the aSi:H the defects are only fully passivated in combination with hydrogenation from the preceding NH_3 plasma.

Comparing the aSi:H samples to the AlO_x samples, keeping in mind that the surface passivation in both cases should be sufficient, we see lower lifetimes and more clearly defined rings for the AlO_x samples even when the samples haven't been subjected to any other process than the diffusion, chemistry, and coating– and the PDA in the case of AlO_x.

Application of a NH_3 plasma before coating leads to elimination of typical ring patterns in combination with aSi:H coating or a SiN_x coating as deposited. The NH_3 plasma treatment was demonstrated to be also reasonably effective for $SiN_{x:}$H-coated wafers subjected to a short high temperature step, but it cannot be applied on a-Si:H coated wafers and is only minimally effective on AlO_x coated wafers. In the literature AlO_x is proposed as a suitable hydrogen permeation barrier [15]. Hence our inability to diffuse hydrogen through it is not unexpected.

From separate studies it is known that the hydrogen effusion for SiN_x layers peaks at 600 °C[16]. This means that at the temperature at which the hydrogen-passivation of the bulk defects starts to be lost, the maximum amount of hydrogen is available from the SiN_x coating. From the results of defect intensification after firing or box furnace anneal, it seems the net effect is negative for the defect passivation. The process of effusion of hydrogen from SiN_x is dominated by the breakage of the Si-H bond. The passivation mechanism of the bulk defects appears to have bindings with similar energy.

The surface passivation mechanism of the AlO_x needs a post deposition anneal (PDA) to drive out excess H_2O [17]. This PDA occurs at elevated temperatures, in our case 500 °C. Although it could be expected that hydrogen would be able to diffuse into the wafer during this PDA, no benefit for bulk lifetime is observed. Apparently the AlO_x layer doesn't constitute a source with sufficient concentration of hydrogen, and an AlO_x layer before post deposition anneal is not a sufficient barrier. This leads to ring wafers with poor bulk lifetime. The contrast to the anneal results with SiN_x coating should be noted here, where the efficiency of passivation decreased only at ~600 °C but no apparent loss of defect-hydrogenation took place at lower temperatures. (However, 500 °C is a temperature missing from the data here). SiN_x is known to be a large reservoir of hydrogen, which can clearly replace the hydrogen lost during anneal with some effectiveness, especially at lower temperatures.

5. Conclusion

A hydrogen containing plasma can decrease the recombination in ring shaped regions in n-Cz wafers, probably by hydrogen passivation of bulk defects. The specific type of plasma seems to be secondary to the effect, as this decrease has been clearly seen for NH_3 plasma, PECVD SiN_x deposition, and even is apparently present for low temperature PECVD aSi:H deposition process.

High temperature negates passivation of the rings. This reversible effect is starting to show from 600 °C for SiN_x coated wafers. For AlO_x coated wafers the effect is shown already at 500°C. Since a high temperature step is necessary for the firing of screen printed contacts, this can reduce solar cell quality. Avoidance of this step by alternative contacting processes is feasible, and also we showed that a post-firing anneal of SiN_x-coated samples can restore the passivation.

An anneal can decrease the effect of rings again. The temperature can be limited to temperatures not detrimental to cell efficiencies. It has been shown this anneal will not be sufficient for wafers with discs and very low minority carrier lifetimes. However, wafers of such low quality are a minority of the ring wafers.

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