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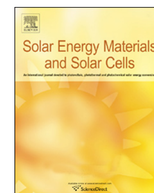

Taking monocrystalline silicon to the ultimate lifetime limit

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ABSTRACT

A central quantity to assess the high quality of monocrystalline silicon (on scales beyond mere purity) is the minority charge carrier lifetime. We demonstrate that the lifetime in high purity float zone material can be improved beyond existing observations, thanks to a deeper understanding of grown-in defects and how they can be permanently annihilated. In a first step we investigate the influence of several process sequences on the lifetime by applying a low temperature superacid passivation treatment. We find that a pre-treatment consisting of an oxidation at 1050 °C followed by a $POCl_3$ diffusion at 900 °C can improve the lifetime by deactivating or eliminating grown-in defects. Then, pre-treated wafers of different float zone materials are passivated with three state-of-the-art layer stacks. Very high effective lifetime values are measured, thereby demonstrating the high quality of the surface passivation schemes and the pre-treated silicon wafers. The measured effective lifetimes exceed previous records, and we report an effective lifetime of 225 ms measured on a 200 µm thick 100 Ω cm n-type silicon wafer symmetrically passivated with a layer stack of a thin thermally grown oxide and a poly-crystalline layer (the TOPCon layer stack).

1. Introduction

In the recent years the photovoltaic community has seen significant improvements in the efficiency of both industrially produced solar cells and record efficiencies of elaborate research scale cells, e.g. [1–3]. A major share of this development is due to ongoing improvements in surface passivation strategies and process advancements to utilise the high electrical quality of today's silicon wafers. This high quality has in turn been achieved by improvement of crystallization technologies (e.g. [4]) and optimised process routes that deactivate recombination-active bulk defects in commercially feasible material (e.g. [5–7]).

Float zone (FZ) silicon wafers are commonly used as a reference to study dielectric surface passivation, intrinsic recombination in silicon, and the performance limit of device structures. FZ silicon is typically assumed to be virtually free of recombination-active bulk defects, and thus it is the gold standard material to measure the maximum attainable effective charge carrier lifetimes τ_{eff} and assess the influence of surface recombination. However, this assumption is weakened by the finding that FZ wafers are often affected by bulk defects introduced during crystal growth or typical sample fabrication, e.g. [8,9]. The yet unpredictable occurrence of the defects introduces experimental scatter and can reduce reproducibility. The underlying defects are known to be affected by thermal treatments and thus distort the comparability of different process schemes due to different thermal budget [8,10]. Such unexpected changes of the bulk lifetime during processing, including dielectric deposition and subsequent annealing, can hinder the optimization of surface passivation layers, the quantification of intrinsic recombination in silicon and loss analyses in high performance solar cells.

In order fairly to compare advancements in surface passivation, the underlying bulk material must be well understood and it's bulk lifetime τ_{bulk} reliably obtained. FZ silicon provides a means to achieve this. However in moving forward, the PV industry must take into consideration, that depending on very specific growth conditions, different FZ silicon wafers of the same resistivity (i.e. different manufacturers) can feature significantly different τ_{bulk}. Fortunately, the grown-in defects responsible for this effect can be annihilated by high temperature processing [8], which in principle, will provide a means to quantify surface passivation quality accurately.

In this contribution we demonstrate that common thermal

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treatments can be used to improve the bulk lifetime of various typical FZ silicon materials. A recently developed superacid passivation treatment [11] is applied to wafers after different processes to demonstrate their impact on the wafer bulk. We suggest a process sequence that deactivates the bulk defects and transfers FZ silicon wafers to a stable state. FZ wafers should undergo such pre-treatment before the application as reference substrates. To support this suggestion the treatment is applied to a range of different FZ materials that are subsequently passivated with state-of-the-art surface passivation layers. The sample processing is performed at two institutes on wafers of the same material to demonstrate that the process sequence allows a direct comparison of passivation layers. We present record lifetimes on all investigated materials. This indicates that the pre-treatment improved the quality of the bulk material and demonstrates the quality of the surface passivation processes.

2. Sample processing

The investigated FZ silicon wafers from commercial suppliers represent a typical quality level used in photovoltaic research. The materials were chosen to cover a large doping concentration range and feature both p- and n-type doping. The specifications of the investigated materials are listed in Table 1. Exact resistivities were measured using the Van der Pauw technique [12] in rectangular $5 \times 5\,mm^2$ samples cut off one wafer after the experiments had been completed. The wafers of each material originate from the same box or batch to make sure that they originate from the same FZ crystal for best comparability. The three performed experiments are discussed in the following subsections and summarised in Fig. 1. The experiments were performed on full 4″ wafers, unless stated otherwise.

2.1. Experiment 1: influence of processes on the bulk lifetime

We investigated the influence of the following processes on the bulk lifetime τ_{bulk} of FZ Si wafers:

Pre-treatments:

– $POCl_3$ diffusion gettering in a quartz tube furnace

(60 min at 900 °C, resulting P-doped regions ($R_{sheet} \approx 18\,\Omega/\square$) etched off)

– Thermal oxidation in a quartz tube furnace

(60 min at 1050 °C, resulting oxide layer ($t_{ox} \approx 105$ nm) etched off)

Passivation processes:

– Al_2O_3 passivation
(plasma-assisted ALD of 20 nm Al_2O_3 on both sides at 180 °C in an Oxford Instruments OpAL reactor followed by a 25 min 440 °C forming gas anneal, see [13] for more details)
– ONO-passivation
(thermal oxidation at 1000 °C in a quartz tube furnace followed by PECVD deposition of SiN_x and SiO_x at/below 400 °C, see [14] for more details).

Several combinations of sequential application of these processes were investigated on materials A-F. Sequences featuring dielectric passivation were assessed via τ_{eff} measurements and photoluminescence imaging (PLI). To assess the impact on τ_{bulk} more directly and investigate process sequences without dielectric passivation, further investigations were performed on material E. Wafers of material E in the as-grown state and after different process sequences were cleaved to quarters and subsequently etched back to bare silicon. Then, the samples were all passivated with the same passivation. Using this approach the relative comparison of τ_{eff} is sufficient to assess τ_{bulk} changes, because all sister samples feature similar surface recombination. The passivation after etching was realised via immersion in a superacid (non-aqueous solution of bis(trifluoromethane)sulfonimide (TFSI) in dichloroethane (DCE)). This treatment allows for a most direct comparison of the material quality as it provides an effective surface passivation with negligible thermal budget and no relevant introduction of hydrogen to the bulk. The process and its features are discussed in more detail in [11].

2.2. Experiment 2: direct comparison of different dielectric passivation schemes

Suitably chosen processing sequences provide FZ wafers with high and stable bulk lifetimes. While an improvement in the bulk lifetime by introducing mobile species (i.e.hydrogen) and associated thermal budgets are important features of modern processing conditions (cf. the introduction of hydrogen from [15] or impurity gettering to [16] a-SiN_x layers) they can superpose the actual surface passivation quality. If achieved τ_{eff} or extracted parameters (such as saturation currents J_0 or surface recombination velocities S_0) are directly compared every unnoticed change of τ_{bulk} distorts the result. Therefore, a direct comparison of different surface schemes can only be fair, if they are applied to stable material. Based on the results of Experiment 1 the wafers underwent a pre-treatment consisting of an oxidation step at 1050 °C and a subsequent $POCl_3$ diffusion at 900 °C prior to passivation.

We processed samples to compare the performance of

A) 20 nm of ALD-Al_2O_3 (processed at Fraunhofer ISE as specified above and in [13])
B) the passivation ONO stack (processed at ANU as specified above and in [14])

on materials A-F listed in Table 1. The samples were subjected to photoluminescence imaging to check for lateral inhomogeneities that could severely affect the measured τ_{eff} (cf. [17,18]). Lifetime measurements using Sinton Instruments WCT-120 lifetime testers in transient mode were repeated on the same samples at multiple institutes for an inter laboratory comparison to assess reproducibility.

Kho et al. report that the passivation performance of ONO layer stacks can be improved further via corona charging [14]. Therefore, the samples passivated with ONO layer stacks were subjected to an enhancement procedure using corona discharge at University of Oxford. A point-to-plain set up with a steel pin at 30 kV was used to deliver

Table 1
Investigated FZ materials.

Material	*A*	*B*	*C*	*D*	*E*	*F**	*P1*	*N1*
dopant species	B	B	P	P	P	P	B	P
nominal resistivity (Ω cm)	0.5	100	1.5	5	10	100	1	1
measured resistivity (Ω cm)	0.48 ± 0.02	107.9 ± 4	1.77 ± 0.07	4.97 ± 0.2	8.93 ± 0.35	86.7 ± 3.4	–	–
nominal thickness (μm)	400	250	400	400	200	300*	250	200

* The material F samples passivated with TOPCon originate from the same crystal but are 200 μm thick.

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