

Shielded Hydrogen Passivation – a potential in-line passivation process

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Shielded hydrogen passivation (SHP) is a recently developed technique for introducing atomic hydrogen into materials and offers significant advantages over other hydrogenation techniques. Hydrogen de-activation of boron followed by electrochemical CV profiling was used to demonstrate that substantial quantities of atomic hydrogen can permeate through palladium/silver alloy foils which are 10 microns thick. It is thought that such thickness will be sufficient to withstand pressures up to 1 atmosphere allowing passivation in an in-line process. Further it is shown that poisoning of the foil using sulphur increases the flux of atomic hydrogen released. SHP delivers extremely good passivation of SiO₂-Si interfaces as demonstrated using thermally oxidised 1 ohm cm, n-type silicon where the lifetime, at 10¹⁵/cm³ injection level, was found to increase from 12 micro seconds to 1.05 ms after SHP processing. Upon application of corona charge, the lifetime further increased to 6.3ms, equivalent to SRV ≤ 0.17 cm/s.

1 Introduction

Introducing hydrogen into silicon is a well-recognized method to achieve excellent chemical passivation of surface and bulk defects [1], [2] and has been widely employed in the study of defects in silicon. It is particularly relevant to silicon PV where, historically, the manufacture of cell architectures such as screen printed cells involved a high temperature firing step in order to release hydrogen present in dielectrics such as SiN_x and AlO_x [3]. However, several high efficiency cell structures, such as those based on silicon heterojunctions and passivated contacts [4], require hydrogen introduction at low temperatures. Two well-known, low temperature hydrogenation sources are remote hydrogen plasma and forming gas anneal (FGA). However, these are

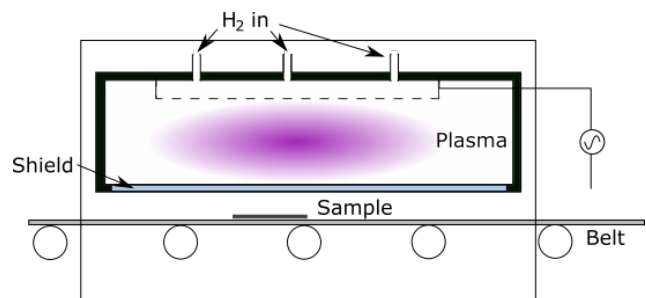


Figure 1 Schematic representation of SHP apparatus adapted for in line processing.

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not without their challenges. Remote plasma hydrogen exposure requires specialist equipment which is not widely available. Additionally, it requires the use of a vacuum chamber and therefore batch processes which is not optimal for use in industry. The alternative, FGA, is conventionally done above 400 °C and has demonstrated only moderate quantities of atomic hydrogen, which has been shown to impact the effectiveness of the hydrogenation process [5], [6].

In a recent letter Shielded Hydrogen Passivation (SHP) was introduced as an alternative method for introducing significant amounts of atomic hydrogen at low temperatures [7]. The approach uses a plasma, in this case produced within a PECVD machine, to generate atomic hydrogen with a metal shield placed between the sample and the

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plasma. The shield is held in place by an aluminium holder, which in turn is placed on the lower electrode, effectively grounding the whole structure. The shield stops the damage usually associated with direct plasma exposure by preventing hard UV photons and energetic ions from reaching the sample whilst letting atomic hydrogen through. In the approach described, a 100 nm layer of 99% pure palladium, in the form of a palladium leaf, was used as the metal shield and was shown to be capable of introducing significant atomic hydrogen [8].

Although SHP already has great potential in a research laboratory environment, its greatest advantage in an industrial setting would be its ability to completely separate the plasma chamber from the environment of the sample, as shown in figure 1. This would allow the process to be inline, and thus applicable to cheap industrial production. For this separation between sample and plasma to be plausible, the shield must be capable of withstanding pressure differences of up to 1 atm. This will require a thicker film than the 100 nm leaf previously used. However, an increase in film thickness may affect hydrogen permeation through the film and thus the process time necessary to maintain outstanding results. It is therefore important that this is better understood so that an appropriate film thickness may be selected.

2 Background

2.1 Atomic Hydrogen Release from Metallic Films Many studies have reported the permeation of hydrogen through metallic films, mostly using Pd. However, to the authors' knowledge the emission of atomic hydrogen from the film in such experiments has not been observed, except in the special circumstances detailed below. Pd is completely impermeable to molecular hydrogen. Thus the accepted understanding is that the transmission of hydrogen through a film consists of several distinct steps [9]. The first step is dependent on the catalytic property of the metal; if the metal has the ability to split molecular hydrogen into its atomic form it will result in dissociative adsorption onto the metallic surface - producing atomic hydrogen. Next, the diffusion of atomic hydrogen into the bulk of the metal will occur until it reaches the opposite surface. It is widely reported, and generally accepted, that upon reaching the opposite surface, atomic hydrogen undergoes recombinative desorption whereby it is released into the atmosphere as molecular hydrogen [10].

The observation of atomic hydrogen being released from Pd has only very seldom been reported and in each case it appears to have required an energetic external stimulus. For example, studies by Tyurin *et al* observe atomic hydrogen release from Pd only when the film is bombarded with high energy electrons. The other significant case of atomic hydrogen release is seen by Lisowski when the Pd film undergoes a phase transition [11], [12].

In light of these studies and theoretical modelling, it has been assumed that there is a high energy barrier for the release of atomic hydrogen such that it may only occur in the presence of an additional stimulus. However, previous SHP work using Pd leaf [7], [8] has clearly demonstrated that a significant quantity of atomic hydrogen is released from the surface of the leaf opposite the plasma, after which it is detected by diffusion into adjacent Si material. Similar results are presented in this work. It is stressed that the hydrogen containing plasma used in SHP is present only on one side of the metal whereas release of atomic hydrogen is observed from the *opposite* surface. This is a surprising and potentially important result.

2.2 Palladium-Silver Foil In order to allow for the separation of the plasma and sample chambers it is necessary to develop a "shield" that is capable of withstanding atmospheric pressure. The 100 nm palladium leaf used in earlier work [7] could not support this difference. Furthermore, such thin leaves are susceptible to pinholes, which could interfere with the process. These pinholes were seen to develop after the leaf was exposed to plasma under a range of temperatures; these have the potential to cause the process to fail by exposing the sample to the plasma and thus causing surface damage.

Literature suggests that pinholes may be caused by the phase change of Pd when exposed to H below certain temperatures. The change in lattice dimension from the α phase to the hydrogen rich β phase causes internal stresses, which could cause pinholes to develop. To counteract this phenomenon, Pd-Ag alloys have been used in hydrogen purification work [13], [14]. The alloying suppresses the phase change curve, thus reducing internal stresses.

In this work, a Pd/Ag (ratio 75:25) foil, 10 μm thick, is used to observe a potential decrease in the formation of pinholes and to investigate whether the increased thickness, relative to the 100 nm thick Pd leaf previously used, affects the levels of atomic hydrogen released and hence the effectiveness of the passivation technique. This work contributes to evaluating the possibility of modifying the current SHP process to an in-line technique by having shields capable of withstanding pressure differences whilst giving effective passivation.

2.3 Poisoning The concept of poisoning catalysts is well known and is important, for example, in the gas purifying industry [15]. Poisoning occurs when impurities are adsorbed onto the surface of the catalyst thus reducing or preventing its catalytic activity. In the case of Pd, the catalytic property is the dissociation of molecular hydrogen into atomic hydrogen. This occurs when molecular hydrogen reaches the surface of the metal, once it is dissociated atomic hydrogen is absorbed into the bulk of the material [11], [16].

Sulphur is well known to poison Pd [17]–[19]. It has been shown that when Pd is exposed to molecular hydrogen such that the hydrogen passes through the material (eg when

Pd is used as a membrane), the presence of S on the surface of Pd stops the dissociation process and hence the penetration of atomic hydrogen into Pd [20]. However, it is also known that this poisoning effect has little or no impact on the permeation of hydrogen into Pd when it is exposed to an atomic hydrogen source. This is the case in SHP as an atomic hydrogen generating plasma is used [8]. This indicates that poisoning the surface of Pd only affects the sticking and dissociation probability of molecular hydrogen and does not impact the adsorption of pre-dissociated atomic hydrogen. This concept has previously been observed by Livshits *et al* in their work observing the “superpermeability” of Pd membranes when exposed to a S containing H gas compared to a pure H gas [21]–[23]. They witnessed a higher level of hydrogen released from a poisoned Pd membrane, but did not distinguish whether this increase was of molecular or atomic hydrogen.

A second effect of S poisoning is that, in addition to decreasing the dissociation of molecular hydrogen, it also slows the re-association of atomic hydrogen into its molecular form on both top and bottom Pd shield surfaces. After atomic hydrogen has diffused through a metal, it is assumed to be released from the surface by recombinative desorption. This is when the atomic hydrogen first pairs to form molecular hydrogen before it can escape the metallic surface. It is the authors’ hypothesis that poisoning will therefore slow the release and formation of molecular hydrogen from the material whilst leaving the capture of atomic hydrogen unchanged.

It is hypothesized that, in an SHP setup, S poisoning of the metallic shield will have two results. Firstly, a higher concentration of H in the bulk of the metal will be seen. Secondly, the amount of molecular hydrogen released from the metal will decrease. Both these effects should promote the release of atomic hydrogen. This should result in the observation of a greater atomic hydrogen release flux from a poisoned membrane than from a clean membrane in an otherwise similar configuration.

3 Method

3.1 Hydrogenation using SHP The setup shown in Figure 2 is used to introduce atomic hydrogen into samples. An aluminium holder encases a metallic membrane and is positioned between the sample and the shower head. The standard parameters for SHP applied to Si substrates are: 100W RF power, 650 mTorr chamber pressure, 100 sccm ammonia gas with varied base plate temperature and times depending on experimental purpose. The ammonia gas is used as the source of atomic hydrogen. Before the leaf is appropriate for use it must be “prepared”. This involves the removal of any native oxide and, where appropriate, sulphur poisoning which is outlined further in the following section. All samples are processed using a “prepared” 99.9% pure, 10 cm x 10 cm, 100 nm palladium leaf, unless otherwise stated.

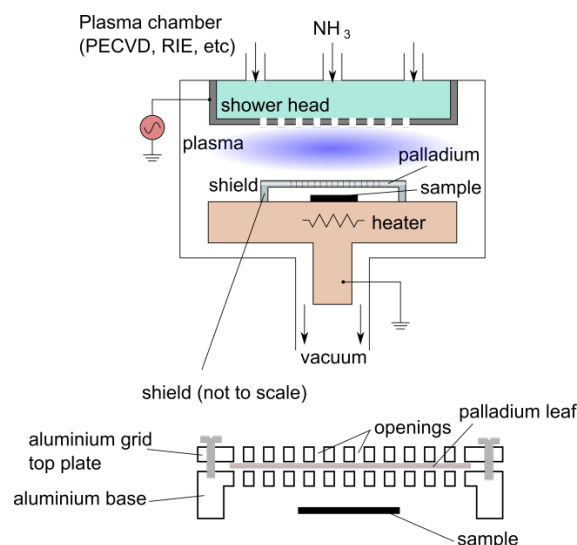


Figure 2 Schematic representation of SHP setup used in this paper. Pd leaf or Pd-Ag foil is held between two 2 mm thick aluminium grids with 5 mm lattice openings. The grids are themselves held in place above the sample by an aluminium holder.

3.2 Surface preparation and poisoning Previous works [7], [8], confirmed that surface preparation of the leaf plays a critical role in the amount of atomic hydrogen released during SHP. It was concluded that a pre-existing layer of palladium oxide present on the surface of the leaf was a barrier to the diffusion of hydrogen.

For the results presented in this paper, the palladium leaves and palladium-silver foils used were prepared using the PECVD plasma. It was found that subjecting the metal to the hydrogen containing plasma for at least 30 minutes at 250 °C was enough to remove the palladium oxide layer on both surfaces. Only one side of the metal needed to be exposed to the plasma since hydrogen diffusing through the Pd, and reaching the other surface, was sufficient to remove the oxide on this side.

After this preparation, some of the metallic shields were poisoned with sulphur to investigate its effect on the atomic hydrogen concentration in the silicon samples. Hydrogen sulphide gas was produced using the following method. First, an enclosed space was created in a fume hood by using a large trough that could be covered by another, larger, up-turned trough. A 50 ml beaker containing $\leq 1 \text{ mm}^3$ of FeS mixed with 20 ml of 10% concentrated HCl acid was placed within the trough. The Pd leaf was kept in its aluminium holder to minimize the risk of ripping and both the leaf and holder were placed inside the enclosed space formed by the two troughs. The reaction of FeS with HCl releases HS, most of which is confined around the holder and leaf. The leaf and holder are left to be poisoned in the hydrogen sulphide gas for 10 minutes which results in coverage of the metal with sulphur atoms.

3.3 Detection of atomic H using boron deactivation in Si Atomic hydrogen is notoriously difficult to detect

and distinguish from molecular hydrogen so it is usually characterized through its interactions with other elements. The method used in this work is to measure the concentration of atomic hydrogen incorporated into silicon wafers using the boron deactivation method. This was done by using uniformly, highly boron doped, $0.175 \Omega\cdot\text{cm}$, p-type silicon wafers. When atomic hydrogen is introduced, it associates with boron so stopping its acceptor dopant behaviour, which in turn decreases the free carrier concentration of the deactivated region. The concentration of active and ionised dopant atoms is measured using capacitance-voltage techniques which thus provides a lower limit for the total amount of hydrogen present [24]. The technique has been shown to be very effective at temperatures below 200°C but becomes less so at higher temperatures due to subsequent de-trapping of hydrogen from the boron ions. Control experiments were performed where the silicon specimens were given the standard SHP treatment but without plasma ignition at temperatures up to 250°C and to anneals at atmospheric pressure in hydrogen at temperatures up to 250°C . Under these conditions it is expected that insignificant amounts of atomic hydrogen will be present and no boron deactivation was detected. This indicates that molecular hydrogen exposure under these conditions will not result in boron deactivation.

4 Results

4.1 Sulphur poisoning The effect of S poisoning the Pd leaf for use in the SHP processes was investigated. The boron deactivation method was used to characterize levels of atomic hydrogen introduced to Si samples, as presented in Figure 3.

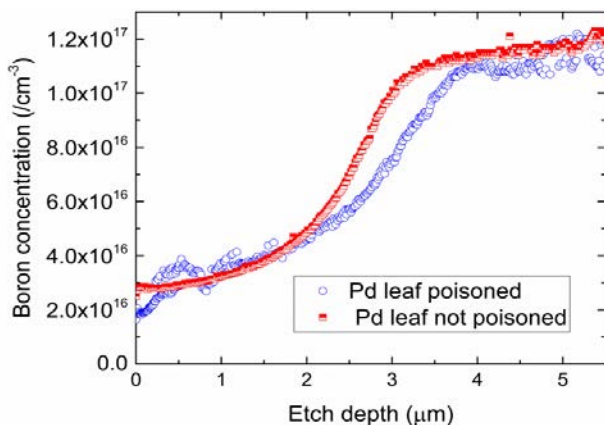


Figure 3 Boron concentration [cm^{-3}] versus depth measured using ECV. Samples exposed to ammonia plasma for 20 mins at 200°C , plasma power of 100W. Samples were processed using a prepared Pd leaf (red squares) and a prepared and poisoned Pd leaf (blue circles). Background doping concentration is $\sim 1.1 \times 10^{17} \text{cm}^{-3}$. The difference in measured ionised boron concentration and background concentration represents the amount of boron deactivated by atomic hydrogen.

It can be seen in Figure 3 that the poisoned leaf sample shows slightly more boron deactivated than the sample exposed under a clean leaf. This suggests a higher concentration of hydrogen is present in the silicon. Although the difference between the two samples is not very large, it is seen systematically on samples tested and certainly no reduction in atomic hydrogen due to S poisoning the leaf was ever recorded.

The effect of poisoning on the overall passivation performance of SHP was also explored by means of minority carrier lifetime measurements. SHP was performed on $200 \mu\text{m}$ thick, $1 \Omega\cdot\text{cm}$, n-type float zone Si samples with a 100 nm thermally grown SiO_2 sourced from Fraunhofer ISE. The sample was exposed to SHP on each side under a prepared and poisoned Pd leaf for 45 mins at 380°C with the standard PECVD parameters described in 3.1. The sample was lifetime tested before and after each process using the PCD Sinton lifetime tester. All lifetime measurements are reported at 10^{15}cm^{-3} minority carrier concentration. In order to achieve the best possible lifetime, field effect passivation was performed post SHP using the corona charging method described by Bonilla et al [25]. The results are shown in Figure 4.

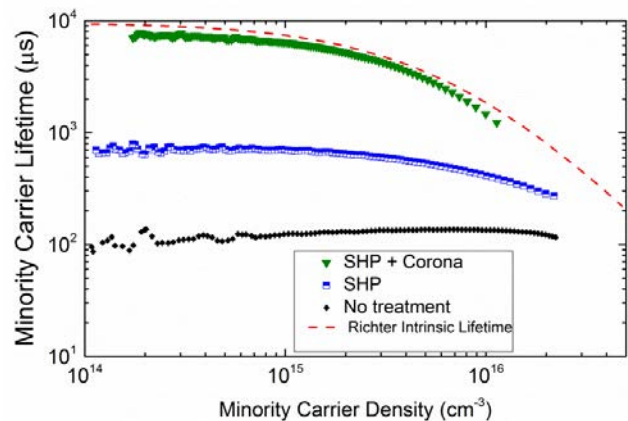


Figure 4 Minority carrier lifetime as a function of minority carrier density. Initial lifetime before any process is shown as black dots. SHP was performed using a prepared, poisoned Pd leaf for 45 mins at 380°C on both sides (blue squares). Corona charging was used to achieve maximum lifetime (green triangles). The Richter intrinsic lifetime limit is shown as a red dashed line for comparison [26].

It can be seen that SHP alone increased the sample lifetime from $12 \mu\text{s}$ to $1050 \mu\text{s}$. After corona charging, the lifetime rose to 6.3 ms , very close to the Richter intrinsic lifetime limit. Using the methodology developed by Bonilla [27] this is equivalent to an $\text{SRV} \leq 0.17 \text{ cm/s}$, a J_{0e} of 0.47 fA/cm^2 and an iV_{oc} of 740.1 mV . This is, as far as the authors are aware, the best surface passivation achieved with a single oxide layer.

It is interesting to compare these results, obtained with a poisoned leaf, to ones done without poisoning. The authors

previously published SHP results performed with an unpoisoned Pd leaf on wafers from the same batch of FZ 1 Ωcm n-type 100 nm SiO_2 -Si samples at 350 °C. They recorded a 4.5 ms lifetime post corona charging [7]. The difference between this result and the one shown in Figure 4 potentially shows a more significant effect of sulphur poisoning than was seen in the boron deactivation measurements presented in Figure 3. Since both experiments were done on the same batch of oxidized wafers it is likely that differences between their lifetimes post SHP reflect real differences in the SHP process rather than differences in the starting material or oxidation process.

The authors hypothesize that the discrepancy between the importance of sulphur poisoning, drawn from the boron deactivation results and the lifetime curves, may be due to the different temperatures at which the SHP was performed for these two tests. The boron deactivation samples, shown in Figure 3, were processed with SHP at 200 °C, whereas the lifetime samples, shown in Figure 4 and discussed in the text, were processed at 380 °C. This increase in temperature could impact the kinetics of the catalytic surface properties of the Pd. It is thought that there is an energy barrier to atomic hydrogen pairing on the Pd surface before its release as molecular hydrogen. Thus at higher temperatures the conversion of atomic to molecular hydrogen on the Pd surface will be faster than at lower temperatures. This might explain why S poisoning that largely stops the recombination process at any temperature would result in a bigger effect on atomic hydrogen emission at high temperatures compared to low temperatures.

4.3 Palladium-Silver Foil Pd-Ag foil (10 μm thickness, 75% Pd, 25% Ag) was used as the metal shield for SHP on 0.175 Ωcm , boron doped, p-type silicon. The samples were exposed to the hydrogen containing plasma under the prepared and poisoned Pd-Ag foil for 30 mins at 200 °C. Figure 5 shows the resulting ionised boron concentration using Pd-Ag foil and Pd (100nm thick) as a function of depth measured using ECV

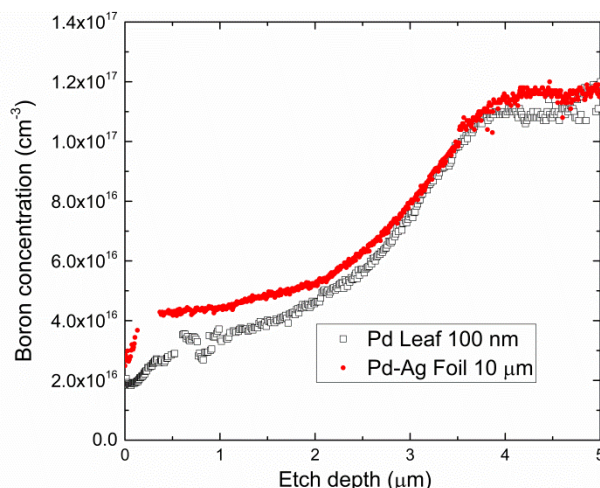


Figure 5 Ionised boron concentration as a function of depth measured using ECV. Samples were exposed to ammonia plasma for 30 mins at 200 °C, with a plasma power of 100W. Pd leaf shield results are shown as empty black squares. Pd-Ag foil shield is shown as red dots.

It can be seen that under the same conditions, the Pd leaf and Pd-Ag foil behave similarly. The offset of the curves could be due to a slight difference in bulk doping in the wafers or be an artefact in the eCV profiling. Both samples were exposed under a poisoned shield and show comparable levels of atomic hydrogen introduced.

This is in line with previous observations on the super-permeability of niobium, where the thickness of the membrane had a negligible impact upon the permeation of hydrogen [23]. In the present work, the experiment is different in that Pd-Ag foils are used, atomic hydrogen release is the parameter measured and the pressure and temperature are different, but the film thickness is confirmed not to play an important role.

Lastly, it is noted that the Pd-Ag foil was selected because it does not undergo a phase change when exposed to hydrogen and thus has no tendency to form pin holes or other defects. In addition, with a properly designed support, a Pd foil of this thickness should be able to withstand a pressure differential across it of the order of 1 atmosphere.

These results are an important step towards performing SHP with samples loaded under atmospheric or near atmospheric conditions, separated from the plasma chamber. It has been demonstrated that, when poisoned, foils that are thick enough to withstand a substantial pressure difference may be used for SHP.

6. Conclusions

In this paper, methods for increasing the levels of atomic hydrogen released through SHP were investigated. It was shown that SHP using a poisoned Pd leaf is very effective in passivating the interface between 100 nm SiO_2 and Si,

and a lifetime of 6.3 ms at $10^{15}/\text{cm}^3$ injection level was recorded. Sulphur poisoning was also seen to be effective in releasing slightly more atomic hydrogen than an unpoisoned leaf when tested on boron doped wafers. Atomic hydrogen was observed to be released from a 10 μm thick Pd-Ag foil. The concentration of atomic hydrogen seen with the Pd-Ag foil was comparable to that recorded using Pd leaf making Pd-Ag foil a promising shield to be used in future in-line SHP processes design.

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