Photo-degradation in Nanocrystalline Porous Silicon

S.C. Agarwal*

Department of Physics, Indian Institute of Technology, Kanpur, U.P., India

Abstract

We study the metastabilities created by light soaking (LS) in nanocrystalline porous silicon (nc-PSi), prepared by the electrochemical anodization technique. At first sight, these appear similar to the effects of LS observed in hydrogenated amorphous silicon (a-Si:H) prepared by PECVD (Plasma-enhanced chemical vapor deposition) method. In nc-PSi, the ESR (Electron Spin Resonance) and Raman results indicate the presence of a-Si:H. This suggests a-Si:H as a possible cause of photo-degradation in nc-PSi. A careful look, however, reveals the differences. Amongst these is the finding that a polymer coating on nc-PSi is found to stabilize it against LS, but does so only partially for a-Si:H. This and several other experiments seem to indicate that the LS in nc-PSi affects the surface, whereas it is generally regarded to be a bulk effect in a-Si:H. We discuss these and related puzzles and suggest their possible explanations.

Keywords: Light induced degradation, amorphous silicon, nanocrystalline silicon, porous silicon, Staebler Wronski effect, photodegradation

***Author for Correspondence** E-mail: sca@iitk.ac.in

INTRODUCTION

It is well known that exposure to visible light degrades hydrogenated amorphous silicon (a-Si:H) by creating dangling bonds and other defects. This degradation in a-Si:H was first reported [1] by Staebler and Wronski in 1977 and is popularly called the Staebler Wronski Effect (SWE). Since SWE affects the performance of the a-Si:H based devices, it has been studied quite intensively for the last more than 35 years or so. Several important insights have been obtained. For example, it is found that the recombination of photogenerated carriers produces energy that breaks the nearby bonds, giving rise to dangling bonds and other defects, resulting in SWE. Despite these advances, the fact that we are still unable to get rid of SWE, shows that our understanding of this phenomena is incomplete. It has been possible to make fairly stable a-Si:H with minimal SWE [2], by modifying the preparation conditions in a Plasma Enhanced CVD system for making a-Si:H, to make composite films consisting of a-Si:H and nanocrystals of Silicon (nc-Si:H).

While, the origin of this stability in nc-Si:H is still being discussed, it is interesting to note that the nanocrystalline silicon, prepared by electrochemical etching method (called porous silicon [nc-PSi]), also degrades upon prolonged exposure to visible light (Light Soaking, LS) [3]. At first sight, the degradation in nc-PSi looks quite similar to a-Si:H. Indeed, ESR, Raman etc. indicate the presence of a-Si:H in nc-PSi also. Thus one may be tempted to think that the a-Si:H present in nc-PSi is responsible for the observed degradation. This may well be true, but let us examine the results carefully, before finally accepting it. As we shall presently see, there are notable differences between the effect of LS in nc-PSi and in a-Si:H. One of them is the effect of coating them with a polymer. The photodegradation in polymer coated nc-PSi is almost completely gone, whereas in a-Si:H it is reduced only partially.

Another difference has to do with the effect of LS after exposure to various ambients. In a-Si:H the final electrical resistance of the sample does not depend on whether the sample is light soaked first and then exposed to the ambient atmosphere or vice versa [4]. On the other hand, in the case of nc-PSi the resistance value is found to depend on the order in which the two operations are performed.

If we assume that the nanocrystals themselves degrade, a comparison with nc-Si:H made by PECVD raises questions like, how can the nanocrystals stabilize a-Si:H, when they

(nanocrystals) themselves degrade upon LS? What is the mechanism of stabilization? Are the nanocrystals present in the PECVD film different from those obtained by the electrochemical method?

In this paper we examine these and related issues more closely, by studying the effect of light soaking (LS) in nanocrystalline porous silicon (nc-PSi) and comparing it with nc-Si:H. We find that in nc-PSi, LS affects mainly the surface, whereas it affects mainly bulk in a-Si:H. These differences are resolved by taking into account the boundary conditions at the amorphous/crystalline interface.

EXPERIMENT

Free standing nc-PSi layers (size~1 mm x 3 mm x 70 μm) were prepared by the electrochemical anodization of boron doped silicon wafers having resistivity ~1 ohm-cm. The layers were prepared as per the procedure illustrated in Ref [5]. Atomic force microscopy (AFM) showed crystallites of sizes 2–5 nm and larger in our nc-PSi [5]. The nc-PSi samples were mounted on Corning 7059 glass substrates using silver paint at the two ends of the sample. These also served as electrodes for the electrical measurements. The nc-PSi samples showed a symmetric, sub ohmic I-V behavior up to ±100 V. Raman micro-spectroscopy shows a peak at 480 cm^{-1} indicating the presence of a-Si:H in our nc-PSi samples [3].

However, some authors were able to fit their Raman data by choosing an appropriate distribution of nanocrystallites sizes without any contribution from a-Si:H [6], thus creating a doubt about the presence of a-Si:H in nc-PSi. In order to resolve this controversy we looked for the characteristic electron spin resonance (ESR) signal from the dangling bonds in a-Si:H. For this, nc-PSi flakes were sealed in helium atmosphere in a quartz tube. ESR was measured using an X–band (Varian E-3) spectrometer at a microwave power of 5 mW.

We found a broad asymmetric ESR signal (Figure 1) which could be fitted well with three signals, namely with g≈2.0058±0.0006 (Lorentzian, width≈7.0±0.5 G) corresponding to the dangling bonds from amorphous phase, and g⊥=2.0081±0.0006 (Gaussian, width≈2.0±0.5 G) and g$_{II}$=2.0028±0.0006 (Gaussian, width≈1.8±0.5 G), being the signatures of the dangling bonds present in nanocrystalline silicon. About 65% amorphous phase is present in the nc-PSi. Further, it is not possible to fit the observed ESR signal, without taking into account a large signal from a-Si:H. Thus, the presence of a-Si:H in nc-PSi is confirmed.
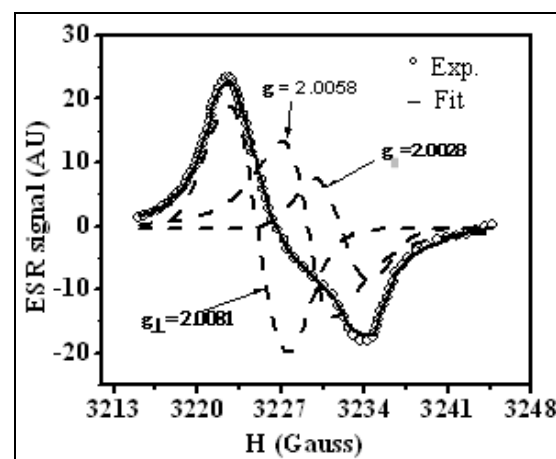

Fig. 1: *ESR Signal of nc-PSi (Circles) and its Fit (Continuous Curve) Using 3 Signals (Dashed Curves).*

In order to determine whether a-Si:H present in nc-PSi is responsible for the photodegradation, we made good quality undoped a-Si:H thin films containing different amounts of nanocrystals of silicon (nc-Si:H) by using the appropriate preparation conditions in a standard PECVD system. The details of preparation of the nc-Si:H films can be found in Ref [7]. Two thermally vacuum evaporated coplanar nichrome strips, separated by 0.5 mm, were used as electrodes. The a-Si:H samples were ohmic up to the highest applied bias of 100 V. Further, HRTEM and Raman measurements confirmed the presence of nanocrystals of size 3 nm and larger [7], along with the amorphous phase, in nc-Si:H.

For photoluminescence (PL), the sample was excited by an Argon ion laser (488 nm, 4 mW), and the emitted light was analyzed using a grating spectrometer and a CCD detector. In nc-PSi, the PL shows a single peak at ~800 nm with a full width at half maximum (FWHM) of ~130 nm. The peak fits well with the size distribution obtained from AFM, using quantum confinement. No PL could be

detected in nc-Si:H samples, although they also showed nanocrystals of similar size. Further, the optical gap in nc-PSi is larger, because of quantum confinement, whereas the gap in nc-Si:H is reported to be smaller [8].

All samples were annealed at 190^0C in dark for 1 h in vacuum, in order to remove the effect of previous light exposures, if any, and then were slowly cooled (rate ~0.5 K/min) to room temperature (state A), before starting any measurements. Light soaking (LS) was done in vacuum, using heat filtered white light from a 250 W tungsten halogen lamp, kept at a distance of about 15 cm from the sample.

RESULTS AND DISCUSSION
The effect of light exposure on an undoped a-Si:H samples is shown in Figure 2. The dark current (DC) and photocurrent (PC) decrease with the increasing exposure time. This is the familiar Staebler–Wronski effect (SWE) [1]. Further, as expected, annealing at 190 C for 1 h in vacuum restores the annealed state A.
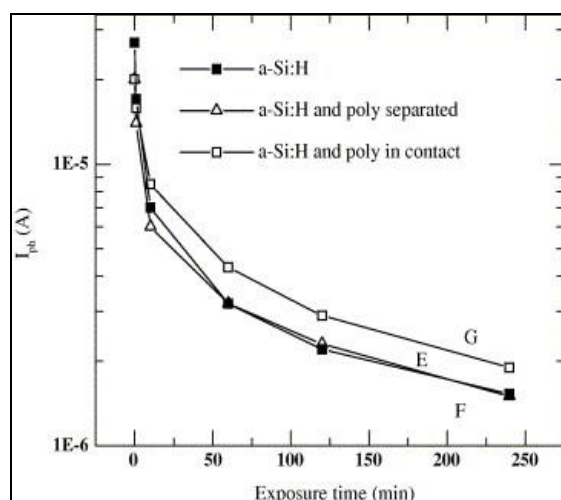
Fig. 2: Effect of Light Exposures on the Photocurrent of Uncoated (E) and Polymer Coated (G) a-Si:H Samples. Photocurrent when a-Si:H and Polymer are Physically Separated is Shown in (F).

Also, exposure to sub-gap light does not remove the SWE. This is indicative of atomic structural changes, involving movement of hydrogen upon LS, and may be explained, as done by Stutzmann *et al.* [9]. We now turn to nc-PSi. In Figure 3, we see that DC, PC, and PL increase for short light exposures but

decrease for longer exposures with a maximum at about 2700 s of exposure time. The new dark current persists for several hours even at room temperature.
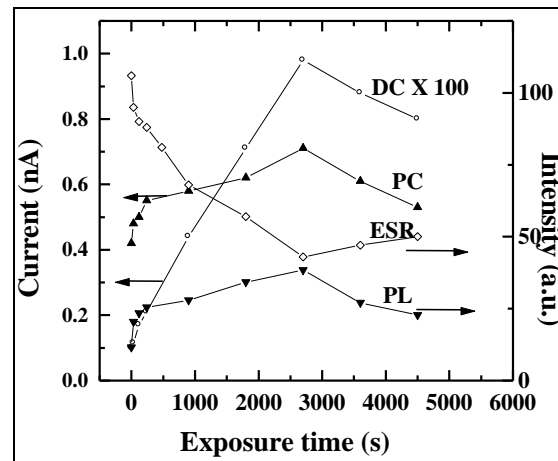
Fig. 3: Effect of Light Soaking on Dark Current (DC), Photocurrent (PC), Photoluminescence (PL), and Electron Spin Resonance (ESR) as a Function of Exposure Time. The Slopes of all Curves Change Sign at the Exposure Time of ~2700 s.

This excess dark current is called persistent photocurrent (PPC). Annealing at 190^0C (1 h), brings the sample to state A. Interestingly, ESR shows a minimum at about the same exposure duration (2700 s) at which DC, PC, and PL show a maximum. This indicates that in PL, the dangling bonds probably act as the nonradiative recombination centers. On the other hand, we see that for longer exposures ESR increases in PS with a decrease in DC, PC, and PL. So, the effect of long exposures is similar to the Staebler-Wronski effect (SWE) in a-Si:H [1]. Further, we find that exposure to infrared light (<1.2 eV) is unable to restore state A. This suggests that the metastable changes caused by LS cannot be purely electronic and might involve photo-structural changes in nc-PSi. The presence of the structure at 2700 s (Figure 3) can be understood if we assume two processes for the creation of the LS state. Process-I decreases the number of dangling bonds whereas Process-II gives an increase in the number of dangling bonds. We assume further that Process-I dominates for short exposures (<2700 s), but for longer exposures Process-II is more important. As an example we describe here a possible set of structural changes that

can fulfill these requirements. For process-I, the hydrogen present on the surface may connect two nearby Si dangling bonds, forming Si-H-Si.

Thus, it gives the desired decrease in the number of dangling bonds. Also, this process decays with time as the number of nearby Si reduces. Finally, the process-II takes over and as noted above we see the usual SWE type behavior. Hence, a mechanism similar to the one proposed for SWE might work for process-II [10].

In this case, hydrogen is believed to move from one Si-H bond into a weak Si-Si bond, creating one Si-H-Si metastable bond and one dangling bond [11]. In an attempt to test the validity of the presence of more than one processes, we measured the temperature dependence of LS, since it is unlikely that both the processes (I and II) have the same temperature dependence. We found that the peak in the PL versus exposure time shifts to lower exposure times as the temperature of LS and PL measurement increases [12].

This is consistent with the assumption of two processes as mentioned above. An increase in DC and PC after short exposures and a decrease after long exposures has also been found in doping modulated [13] and compensated [14] a-Si:H. However, the decrease in ESR described above, has not been reported for a-Si:H.

Now let us examine whether light soaking affects the bulk or the surface. The present view is that in a-Si:H SWE is a bulk effect. In this context, we recall our experiment [15], in which we exposed a-Si:H to cycles of light and wet nitrogen (Figure 4). The conductance (G) of the final state obtained is independent of the order of the exposures (cf. F and F′) are performed (upper part of Figure 4).

This will happen if the two operations are independent. It will be so, if LS affects the bulk only since moisture affects only the surface. However the lower part of Figure 4 shows that the surface photo-voltage (SPV) after cycle-I is different than cycle-II. This means that in SWE, surface is also affected,

although its contribution is too small to show up in G measurement.
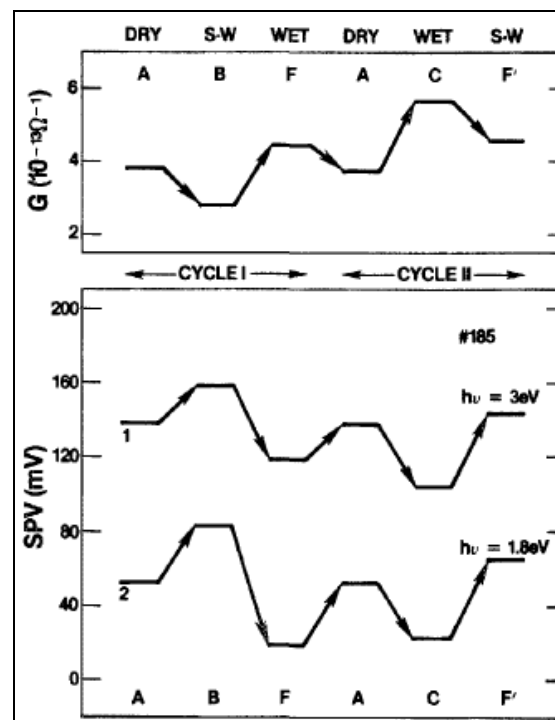
Fig. 4: *Conductance (G) and Surface Photovoltage (SPV) for a-Si:H, in Different States of the Cycle Experiment. Note that F=F′ for G but not for SPV.*
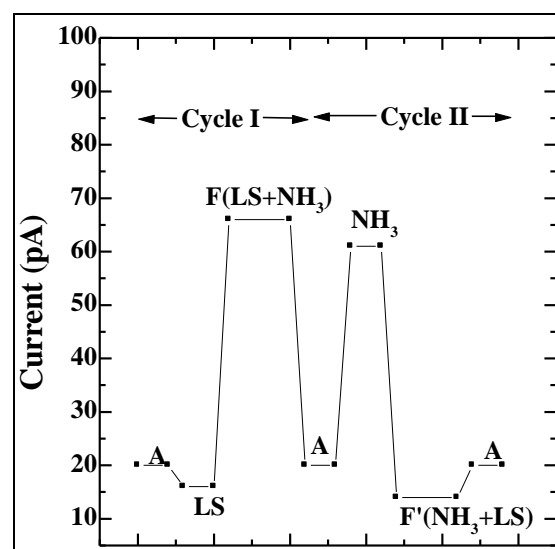
Fig. 5: *Effect of LS Followed by Ammonia Exposure (Cycle-I) and Effect of Ammonia Followed by LS (Cycle-II) on DC of a Free Standing nc-PSi. Note that F is not Equal to F′.*

Now we ask the same question for nc-PSi. Does LS in nc-PSi affect the surface or bulk or both? For this, we exposed nc-PSi to cycles of

LS and ammonia vapor [4]. Figure 5 (cycle-I) shows that the annealed state A goes to state LS after light soaking (8 h), with a lower DC. A subsequent exposure to ammonia results in the final state F, which has a DC value of 66 pA. Upon annealing, the state F returns to the initial state A. In cycle-II the order of LS and NH$_3$ exposure are reversed, i.e. the LS is done after exposure to ammonia (Figure 5), In this case, the dark current in the final state F′ is ~13 pA which is ~5 times is smaller than that in state F. This is contrary to the result for a-Si:H [15], where we found F=F′. Thus, the degradation observed in nc-PSi cannot be entirely a bulk effect and must involve surface, in contrast with a-Si:H.

Next, we coated our samples with a thin layer of polystyrene [16]. For a-Si:H, a few drops of polystyrene dissolved in toluene were coated on the sample and allowed to dry (half an hour). Care was taken to protect the film from strong room light while the polymer was applied to the a-Si:H samples and during the drying procedure. A thin layer of polystyrene polymer applied in this way on an annealed undoped a-Si:H sample decreases dark current by one order of magnitude.

Band bending at the surface of a-Si:H seems to be responsible for this decrease in dark current after polymer coating. The photocurrent of the annealed a-Si:H sample also decreases (by about a factor of 1.5) after applying the polymer. Curve E in Figure 2 shows the effect of light exposure on the photocurrent of an uncoated a-Si:H samples and curve G shows the effect after the a-Si:H is coated with polystyrene. We see that light soaking decreases the photocurrent of uncoated (curve E in Figure 2) as well as a polymer-coated (curve G in Figure 2) samples. Annealing at 190^{0}C in vacuum restores the initial state.

However, the decrease in photocurrent for the coated sample is smaller than that for the uncoated sample. This small decrease in the effect of LS in the polystyrene coated a-Si:H is caused by the changes at the a-Si:H/polymer interface and not simply by the reduction in the intensity of the light transmitted through the polymer. This is confirmed by curve F (Figure 2), which shows that the degradation is the same as the uncoated sample, when the a-Si:H and polymer are physically separated.

For estimating the effect of LS, we define a quantity $\beta=(I_{LS}-I_A)/I_A$, where I_{LS} is the dark current [17] measured 30 min after each exposure and I_A is the in dark current in the annealed state. This ratio β is plotted as a function of the length of the LS in Figure 6. Filled squares are the value of β before polystyrene coating and the open squares are the value of β after polymer coating. We see that the rate of decrease of β with the length of exposure reduces after polymer coating. Note that the value of β is negative, as expected, since SWE and PPC are in opposite directions. We see that the magnitude of β is smaller after coating with polystyrene.
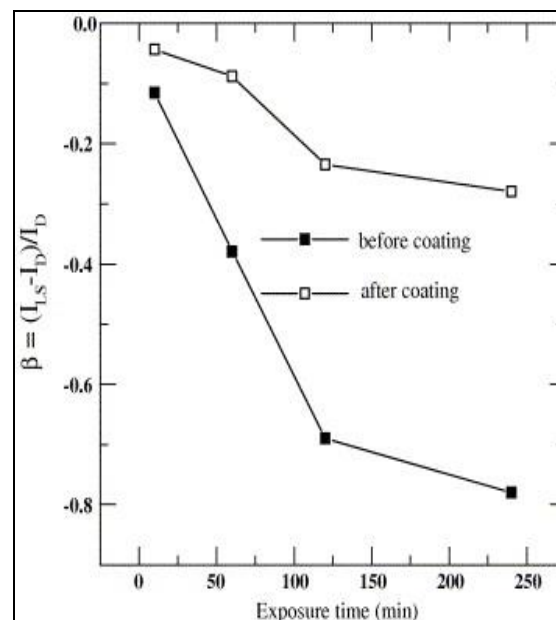
Fig. 6: *The Effect of Polystyrene Coating on β of a-Si:H Sample. β Decreases with the Duration of the LS After Polymer Coating. However, the Decrease is Reduced After Coating.*

Now, let us look at the effect of polystyrene coating on nc-PSi sample. For this, the nc-PSi sample was immersed in a solution of polystyrene dissolved in Toluene and sonicated briefly in an ultrasonic bath, to allow polystyrene to reach the inner pores. The observed PPC for a nc-PSi sample before coating (Figure 7 curve a) and after coating (Figure 7, curve b). We note that after coating the PPC is gone.
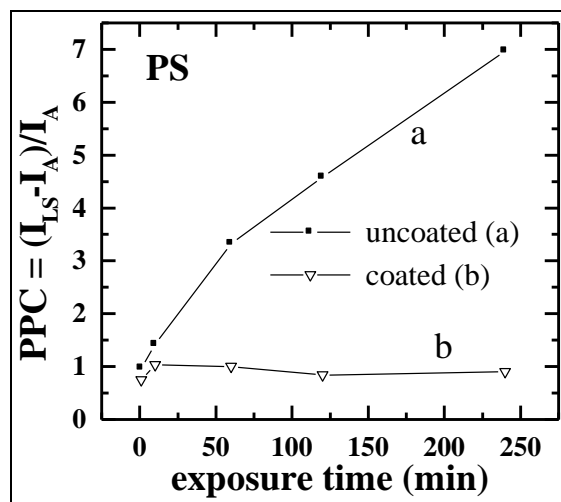
Fig. 7: The Effect of LS on PPC. For Uncoated nc-Si PPC Increases with the Length of Exposure (Filled Squares). The PPC is Considerably Reduced After Polystyrene Coating (Open Triangles).

FTIR studies show evidence of formation of new bonding arrangements at the polymer/nc-Si interface. Thus the polymer not only protects nc-PSi from ambients by providing a physical barrier, but also bonds chemically to nc-PSi and makes it stable [18]. Our polymer-coated sample was light soaked up to 300 h and it was found that the PL and ESR are stable against LS. Further, PPC was absent for the longest exposure (240 min) studied. Thus in the new bonding arrangements LS does not create any dangling bonds indicating better stability.

Since a coating on the surface alone is able to eliminate the photo-degradation (PPC) in nc-PSi, the light must be creating defects only on the surface of the nanocrystalline part which means in a-Si:H. So, we are forced to the conclusion that a-Si:H is responsible degradation in all cases considered here. This seems to be against the accepted view, that SWE is a bulk effect. The results can, however, be explained, if we go by the hypothesis that both surface as well as bulk are affected by LS. For example, in the ambient cycle experiment, if we measure conductance, we find that gas exposure and LS commute (bulk), whereas they don't, if we measure SPV. So, between surface and bulk, either one can appear dominant, depending on the situation. The conventional view is for the situations where bulk dominates. In

nanocrystalline silicon there is a very thin layer of a-Si:H on the on the surface of the nanocrystals. Since nanocrystals are stable, the degradation is in a-Si:H, which makes it look like a surface effect. This peculiarity may be the cause of the other differences, e.g. initial rise of PL decrease of ESR, etc., which are not found in a-Si:H, probably because they are surface related and are masked by the bulk, in practice. Finally, it has been found [2] that nc-Si:H, made by PECVD, is free of degradation [7], if the crystalline fraction is 25% or more (Figure 8).
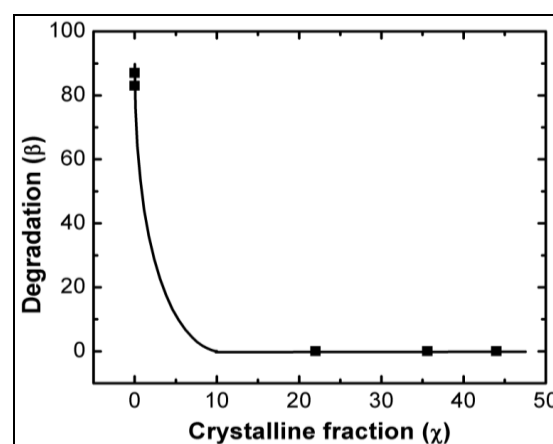
Fig. 8: Degradation (β), in a PECVD nc-Si:H as a Function of Crystalline Fraction (χ).

Regarding the other questions, e.g. differences in stability etc, one answer may be that the two preparation methods are different. In nc-PSi, one starts with a single crystal and etches it while in the case of nc-Si:H the nanocrystals are build up starting from molecules. There is no certainty that they will produce identical nanoparticles.

For the other possibility, we must first understand how the presence of nanocrystals stabilizes nc-Si:H. The carriers generated in the amorphous part go to nanocrystals and recombine there and not in the amorphous part. This is because the band gaps are such that at the interface the bands are bent [19]. This movement of charges has also been experimentally found [8]. Since the recombinations are essential for SWE, this saves the amorphous part from degradation. The crystalline part is assumed stable. Now if the boundary conditions at the amorphous/crystalline interface are such that they confine the carriers and do not allow

them to move freely between crystalline and amorphous phases, the degradation cannot be avoided. It seems to be so for the nc-Pi. On the other hand, if the traffic is allowed to move freely between the amorphous and crystalline phases, the carriers can go to the crystalline where they can recombine, for example, nc-Si:H. without causing any degradation. Also, the lack of quantum confinement of carriers in the nanocrystals will explain the absence of PL in nc-Si:H, and the smaller gap of nanocrystals in it, which incidentally, provides the necessary electric field to propel the photogenerated electron hole pairs, from the amorphous region to the nanocrystalline region efficiently [20], before they recombine in the amorphous phase.

SUMMARY AND CONCLUSIONS

Exposure to visible white light is found to create metastabilities in nc-Si:H and nc-PSi. Since all metastable states can be annealed out, but are stable against exposure to sub band gap (IR) light, it seems that a local structural rearrangement of bonds involving movement of hydrogen is responsible for the LS behavior in both cases. Both contain amorphous as well as nanocrystalline silicon. As expected, at first sight the LS effects in nc-PSi appear similar to those observed in nc-Si:H. A more careful look, however, reveals significant differences. For example, the decrease in ESR signal after short LS, as we observed in nc-PSi, has not been reported in a-Si:H, not even in nc-Si:H. Similarly, we find that in nc-PSi the metastable state F obtained by LS and ammonia exposures depends on the order in which these steps are performed. This is in contrast to the results in a-Si:H in which (F= F′). This might mean that LS affects the surface in nc-PSi to a much larger extent than in a-Si:H. Coating nc-PSi with a layer of polystyrene stabilizes it against LS. We have light soaked the polystyrene coated nc-PSi sample up to 300 h, without finding any noticeable degradation in PL. FTIR results show formation of new bonds at the polymer/nc-PSi interface. This suggests that these bond changes stabilize the surface of nc-PSi against LS and that light soaking is a surface effect in nanocrystalline porous silicon. On the other hand, the polymer coating on a-Si:H does not eliminate the Staebler Wronski Effect, although it does make it somewhat smaller. This also shows that although LS is mainly a bulk effect in a-Si:H, the surface also plays a part, which at times may be too small to be seen. These results can be explained by assuming that a-Si:H, responsible for the photo degradation of nc-PSi, is at the surface of the nanocrystals. This makes LS look like a surface effect in the present case, although it really affects the bulk also.

Now we address ourselves to the question raised in the Introduction: Why is it that the nanocrystals stop degradation in the PECVD films but not in nc-PSi? We shall consider here, several possible explanations. One possibility is that the nanocrystals in the two cases are not the same, because in the case of PECVD they are made by collecting small groups of atoms to form nanocrystals, whereas in the electrochemical case, we start with a single crystal and etch out some parts to obtain nanocrystals. It is not clear that they are going to be identical, in structure or properties. The nanocrystals in nc-PSi show PL but not in PECVD a-Si:H. The details of the LS do not match completely. One can probably check this possibility of obtaining different nanostructures when they are made by different methods, by running a computer simulation, which seems like a big job for a few nm size nanocrystals, Another possibility is that the degradation occurs only in a-Si:H and the nanocrystals are stable (likely). The boundary of nanocrystals in PECVD is such that the carriers from the a-Si:H can freely get into nanocrystals. Since the band gap of nanocrystals of Si is reportedly smaller than a-Si:H, the photocarriers recombine in the nc part, which is stable. Since most recombinations take place away from a-Si:H, there is very little degradation. In the case of nc-PSi, on the other hand, if the boundary conditions do not allow communication between the amorphous and crystalline parts, the recombinations will be in the amorphous phase, which degrades. The second explanation has the advantage that it explains the absence of PL in the samples made by PECVD and also the smaller gap because the carriers are not confined.

We examined the case of degradation of nano-crystalline silicon and reached the conclusion that the degradation must come from a-Si:H. The fact that it can be erased by surface coating, while SWE in a-Si:H is a bulk effect, can be explained by noting that SWE affects both surface and the bulk. Which of the two will dominate in a given situation depends on the experiment being done. We conclude that our experimental results are consistent with the hypothesis that the presence of a-Si:H is mainly responsible for the degradation in nc-PSi. This hypothesis is also shown to be consistent with the stability in nc-Si:H, arising from the presence of nanocrystals in a-Si:H and other issues.

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