Ultraviolet light induced annihilation of silicon dangling bonds in hydrogenated amorphous silicon nitride films

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We report results of electron paramagnetic resonance, photothermal deflection spectroscopy, and capacitance-voltage measurements on amorphous hydrogenated silicon nitride (a-SiN$_x$:H) thin films exposed to ultraviolet (UV) illumination. It has been previously shown that exposure to UV light activates silicon dangling-bond defects, i.e., K^0 centers, in a-SiN$_x$:H thin films. Here, we demonstrate that the initially UV-activated K^0 center can be irreversibly annihilated at long illumination times. Because this effect seems to scale with H content of the measured films, we propose that hydrogen may be passivating the K^0 defects during the extended UV exposure. We also show that films subjected to long UV exposures trap charge as efficiently as those having much larger K^0 concentrations. A few possibilities to explain this effect are discussed. © *1995 American Institute of Physics.*

I. INTRODUCTION

Amorphous hydrogenated silicon nitride (a-SiN$_x$:H) is widely used in the electronics community as a charge storage medium in polysilicon-nitride-oxide-semiconductor memories[1,2] as well as the primary dielectric in thin-film transistors for use in flat panel displays.[3,4] An understanding of the role of defects in this amorphous insulator is crucial to understanding and controlling the charge traps in this material. Recently, a number of theoretical[5,6] and experimental studies[7–24] have been performed. As a consequence, evidence has accumulated which identifies the charge trapping centers with silicon dangling-bond defects.[7,9–11,17] The singly occupied silicon dangling-bond defect in either stoichiometric or N-rich a-SiN$_x$:H is called the K^0 center ($\cdot$Si$\equiv$N$_3$).[14]

Interesting features of the K^0 center in N-rich alloys have been reviewed elsewhere.[17] We briefly mention a few here:

(1) it can be activated[8–12,14–18] and reversibly photobleached[15,19] by ultraviolet (UV) illumination (the relative rates of activation and photobleaching depend on the photon energy; activation dominates for photon energies greater than 3.5 eV, while photobleaching dominates for photon energies less than 3.5 eV;
(2) it is believed to be responsible for the enhancement of sub-band-gap optical absorption in both unilluminated[20] and illuminated[19,21,22] nitride films;
(3) based on a series of charge injection and UV sequences it has been proposed that the UV light activates the K^0 center by simply changing the spin state and charge state of pre-existing negatively charged ($^-$Si$\equiv$N$_3$,K^-) and positively charged ($^+$Si$\equiv$N$_3$,K^+) silicon sites.[9–11;17,18,21,23,24]

It has been suggested that all these observations are consistent with a negative correlation energy model of the K center in the N-rich a-SiN$_x$:H films,[9,11,16,17,23–25] i.e., a negative U.[26–28] In the negative U model, the charged diamagnetic sites are more stable than the paramagnetic neutral silicon site (K^0). As a consequence, the diamagnetic positively charged (K^+) and negatively charged silicon sites (K^-) will serve as the electron and hole charge trapping centers in unilluminated silicon nitride thin films, respectively. Recently, Fritzche and Nakayama[13,22] and others[29,30] have proposed that the charged K^+ and K^- centers may also arise from potential fluctuations. In this case the Si dangling bonds have a positive U; however, the magnitude of the fluctuations exceeds the U.

In this paper we show that the initially UV-activated K^0 center can eventually be irreversibly annihilated by room-temperature UV illumination. Note that this is a very different phenomenon than photobleaching; photobleaching is reversible. This irreversible decay occurs in hydrogenated plasma enhanced chemical vapor deposited (PECVD) a-SiN$_{1.6}$:H at 250 °C thin films subjected to UV illumination for times as short as 10 min. All measurement techniques employed in this study, electron paramagnetic resonance (EPR), photothermal deflection spectroscopy (PDS), and capacitance vs voltage (CV) measurements indicate a similar trend during UV exposure. Our results seem to suggest that the irreversible process might be related to the film's hydrogen concentration. Based on past work, we would have expected that the concentration of charge traps in the a-SiN$_x$:H films should become smaller with prolonged UV exposure since the K^0 center is annihilated. However, our charge injection experiments indicate that this is not the case. A few possibilities to explain this result are discussed.

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II. EXPERIMENTAL PROCEDURES

The hydrogenated PECVD N-rich a-SiN$_x$:H ($x=1.6$) samples in this study are of device quality; they are used in a-Si:H thin film transistors as the primary gate dielectric. The optical energy gap of the films was measured to be 5.2 eV by optical absorption spectroscopy. They were deposited from undiluted NH_3 and SiH_4 gas mixtures on either fused quartz or crystalline Si substrates at temperatures of either $T_s=250$ °C or $T_s=400$ °C. The NH_3:SiH_4 ratio employed was 12:1. The films deposited at $T_s=250$ °C have a hydrogen concentration of 38 at. %; the films deposited at $T_s=400$ °C have a hydrogen concentration of 31 at. % as determined by nuclear reaction analysis. Stoichiometric low-pressure CVD (LPCVD) films were deposited on crystalline Si substrates at 820 °C using ammonia and dichlorosilane processing gases. The hydrogen concentration in these films is less than 2 at. %.

EPR measurements were performed on an X-and Varian E-line or a Bruker ESP-300 spectrometer at room temperature or at 77 K. Spin concentrations were measured by a comparison with a weak pitch standard. Care was taken to ensure that the resonance was not saturated. The absolute spin concentrations are accurate to about a factor of 2; the relative spin concentrations are accurate to about 10%. The sub-gap optical absorption measurements were made using transverse PDS with pentane as the deflection medium. Further details of the PDS measurements on a-SiN$_x$:H films are discussed elsewhere.[19,21]

One MHz CV measurements were made using either a 72BD Boonton Electronics bridge or a Princeton Applied Physics CV meter (model #402). Electrical contact for these measurements either employed a mercury probe, or the evaporation of semitransparent Al dots. Most of the UV illuminations were performed using broad-band, room temperature Hg lamps. The Hg lamps were either 1, 5, or 100 W. Some EPR measurements were performed by shining broad-band UV illumination *in situ* in the microwave resonant cavity at 77 K.

A corona discharge apparatus was used for the electron and hole injection sequences. The corona method for injecting charge into the nitride films was first used by Krick, Lenahan, and Kanicki.[10] Deposition of positive corona ions causes injection of electrons into the a-SiN$_{1.6}$:H films; negative corona ions causes injection of holes. The samples used for the charge injection experiments were deposited on crystalline Si substrates on which a 20 nm thermal oxide had been previously grown. The thermal oxide made the electrical measurements more reliable, since it blocked any charge injection that may occur at the moderate electric field employed during the CV measurements (1.5 MV/cm). The a-SiN$_{1.6}$:H films used in this part of our study were 370 nm thick as determined by ellipsometry.

III. RESULTS AND DISCUSSION

A. EPR results

Figure 1 plots the normalized K^0 center density versus UV-illumination time determined by EPR. The PECVD a-SiN$_{1.6}$:H films from which these data were obtained were

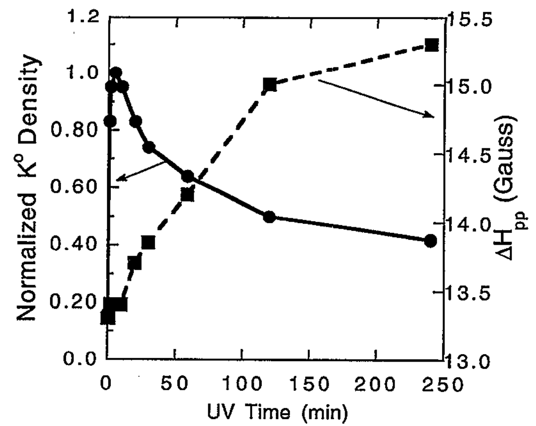

FIG. 1. Normalized K^0-center concentration and $\Delta H_{pp}(G)$ plotted as a function of room temperature, broad-band UV-illumination time showing both the activation and annihilation of the K^0 center. ΔH_{pp} is the peak-to-peak linewidth of the derivative of the absorption of the ESR spectrum. The a-SiN$_{1.6}$:H films were deposited at $T_s=250$ °C. The maximum spin density is 1.0×10^{18}/cm^3.

deposited at $T_s=250$ °C. The UV-light source was a 1 W broad-band Hg lamp, and the illumination was performed at room temperature. During the early stages of the UV exposure, the paramagnetic K^0 center is activated and its maximum concentration (after about 5 min) is 1.0×10^{18}/cm^3. The UV activation of the K^0 center has been observed by numerous investigators from several groups.[8–12,14–18] Note that we observe that longer UV-illumination times cause the K^0 center to decay. This observation has not been reported previously. Although not shown in the time scale of Fig. 1, illumination times in excess of 3000 min cause most (70%) of the UV-induced K^0 centers to be extinguished in these highly hydrogenated films.

Figure 1 also indicates that this decay is accompanied by a change in the EPR line shape; the line shape of the K^0 center begins to appreciably broaden as the illumination time increases. ΔH_{pp} is the peak-to-peak linewidth of the derivative of the absorption of the spectrum. Figure 2 illustrates the EPR spectra for films exposed to broad-band UV light at room temperature for 5 and 240 min. The EPR trace taken after the prolonged UV has been multiplied by 3.33 to make

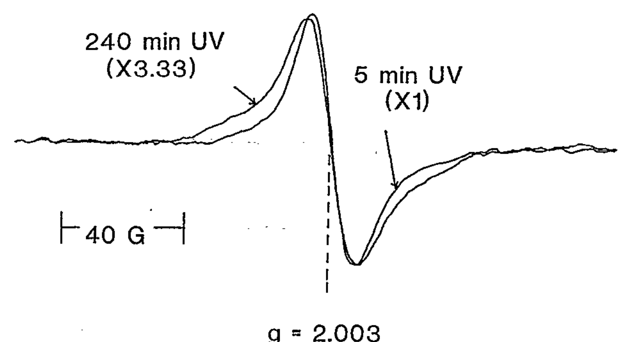

FIG. 2. EPR spectra of the K^0 center after the a-SiN$_{1.6}$:H films ($T_s=250$ °C) were UV illuminated for 5 and 240 min. The EPR spectrum taken after 240 min illumination was multiplied by 3.33.

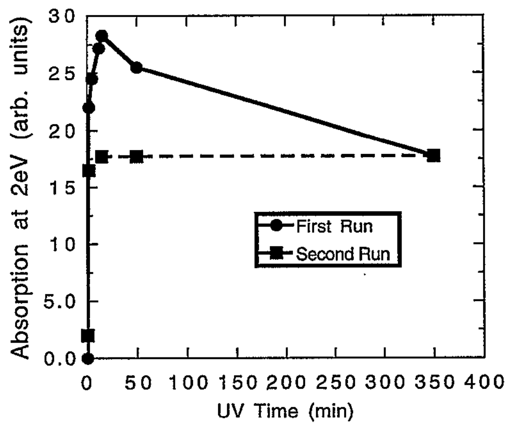

FIG. 3. PDS measurements of the subgap optical absorption taken on 2 eV of a-$SiN_{1.6}$:H films vs broad-band UV-illumination time showing the increase in the absorption followed by a decay with UV exposure time. The films were deposited at T_s=250 °C. Following UV illumination for 340 min, a second run was performed. The subgap absorption for the second run does not reach the intensity achieved during the first exposure, demonstrating the irreversibility of the process.

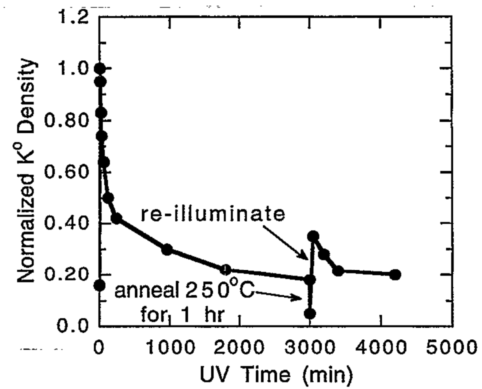

FIG. 4. Plot of the K^0-center concentration vs UV-illumination time for N-rich PECVD silicon nitride films deposited at T_s=250 °C. After illuminating for 3000 min the films were annealed at 250 °C in dry N_2 for 1 h, and then re-illuminated. The K^0-center, even after the anneal, is still irreversibly decayed.

its magnitude equal to the maximum (short time) signal. While the EPR line is noticeably broader, the g value of the defect remains the same during the short- or long-time UV exposure. The concentration of K^0 centers after a 5 min (240 min) UV exposure is $1.0\times10^{18}/cm^3$ ($0.42\times10^{18}/cm^3$).

B. PDS results

Recent PDS studies have been able to show that the UV light also leads to an enhancement of the subgap optical absorption in a-SiN_x:H films.[13,19,21,22] It appears as though the short time UV-enhanced optical absorption is related to transitions involving the UV-induced paramagnetic K^0 defects. If this is the case, then following longer UV exposure times, the subgap optical absorption should also decrease. Figure 3 illustrates our PDS measurements performed on samples deposited identically to those in Fig. 1; in this plot we show the UV-induced absorption at 2 eV versus broad-band UV-illumination time (at 300 K). Initially the UV-light enhances the optical absorption, but longer UV illumination times reduce it substantially. These data closely mimic the EPR results, providing further evidence that the increase in the subgap optical absorption involves a transition of the K^0 center.

Figure 3 also demonstrates the irreversible nature of this process. After an illumination time of 350 min, the UV light is turned off, and a second UV exposure is performed about 1 h later. During the second exposure, the subgap optical absorption rises to the level seen just before the UV light was turned off at 350 min; it does not reach the maximum absorption level achieved during the first UV illumination. Identical trend were also observed in the EPR measurements: after a dark period, a second UV exposure never causes the K^0 center concentration to reach the original maximum level achieved during the first exposure.

Thermal soaks do not restore the starting state of these films as illustrated in Fig. 4. In this particular experiment, the film was illuminated for 3000 min, followed by an anneal at 250 °C for 1 h, and then re-illuminated. A large fraction of the K^0 center is still irreversibly decayed even after the 250 °C anneal. The production of the K^0 centers has been irreversibly altered by the first rather long UV exposure.

C. *CV* results

It has also been demonstrated that UV illumination of N-rich a-SiN_x:H films lead to the creation of fixed positive charge[9,12] spatially located near the semiconductor/nitride interface. It is important to note that the positive charge concentration is typically two orders of magnitude smaller than the concentration of K^0 centers.[9,12] The mechanism behind this positive charge creation has been discussed elsewhere.[12] Studies show that the UV light causes the fixed positive charge and paramagnetic K^0 centers to appear on similar time scales. Figure 5 shows the creation and subsequent annihilation of the positive charge as a function of UV time for samples deposited identically to those used in Figs. 1–4. During UV exposure there is initially a rise in the positive space charge concentration followed by an irreversible decay. The UV activation and subsequent UV annihilation of the positive charge occur on reasonably similar time scales as the activation and annihilation of the K^0 centers (EPR) and the subgap optical absorption (PDS).

Figure 6 shows the relative concentration of the K^0 center in N-rich PECVD films deposited at T_s=250 and 400 °C and of a stoichiometric LPCVD film deposited at T_s=820 °C versus broad-band, room-temperature UV-illumination time. The creation and the irreversible decay kinetics of the K^0 center are very different in the three films explored. The maximum K^0 concentration occurs at much longer times in the 400 °C PECVD films as compared to the films deposited at 250 °C. No decline was observed in the K^0 concentration in the LPCVD films, even for illumination times as long as 3000 min (not shown). The results of Fig. 6 may suggest a link between the irreversible decay process and the films' hydrogen concentration; the decay rates are largest for the films with the largest hydrogen concentration. Note that the

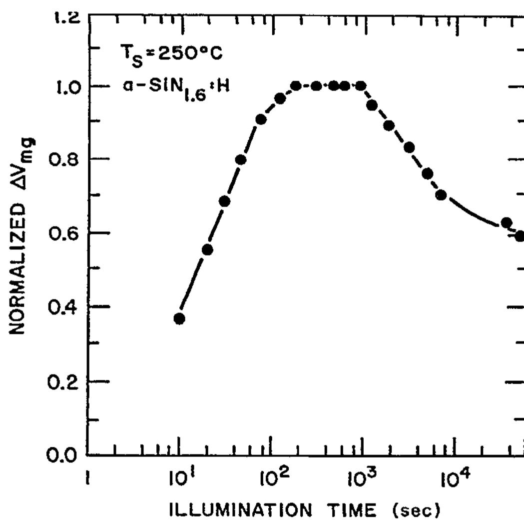

FIG. 5. Plot of the normalized positive space charge density vs UV-illumination time showing that the positive space charge is initially generated by the light, followed by an irreversible decay. The a-SiN$_{1.6}$:H films were deposited at T_s=250 °C. The maximum positive space charge density is $2.0\times10^{16}/\mathrm{cm}^3$.

maximum concentration of K^0 centers observed in all three films are similar; in the PECVD films deposited at T_s=250 and 400 °C these were 1.0×10^{18} and $1.2\times10^{18}/\mathrm{cm}^3$, respectively; for the LPCVD films it was $1.5\times10^{18}/\mathrm{cm}^3$. PDS measurements performed on the 400 °C PECVD films show similar trends to the EPR results. The decay of the subgap optical absorption is delayed and suppressed compared to the films deposited at T_s=250 °C. Note that these studies of different films show considerably different rates for the initial rise of K^0 with illumination. These differences may arise due to the marked difference in charge trapping and transport. The UV-creation differences cannot be explained by differences in the optical bandgap or Urbach energy since they are very similar in the LPCVD and N-rich PECVD films.[19] Because we wish to focus on the UV-induced decline in K^0, we will not discuss these issues in detail.

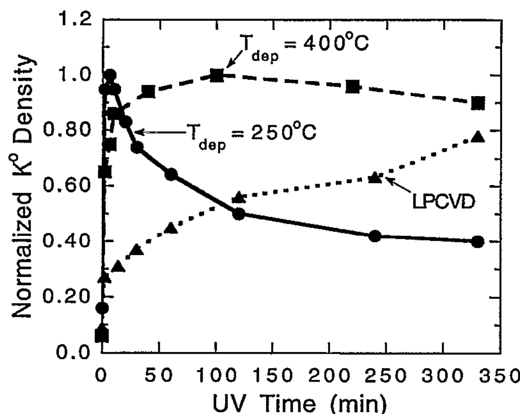

FIG. 6. Plot of the normalized K^0-center concentration vs room temperature UV-illumination time for three different a-SiN$_x$:H films. Two films are N-rich PECVD a-SiN$_{1.6}$:H deposited at T_s=250 and 400 °C, and the third film is an LPCVD film.

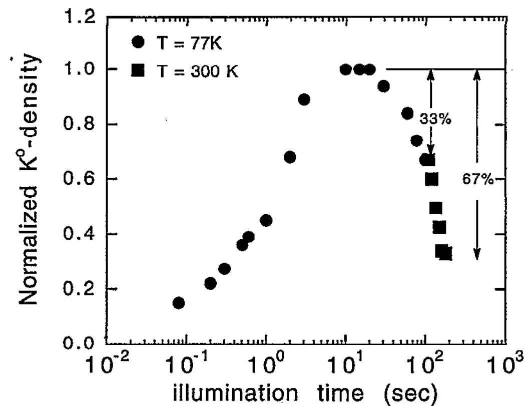

FIG. 7. K^0 spin density as a function of illumination time at 77 K followed by subsequent illumination at 300 K.

D. Possible annihilation mechanisms

Seager and Kanicki[21] effectively demonstrated that short time UV exposures do not lead to measurable changes in the Si-H or N-H linkages. However, experimentally we find that the K^0 annihilation rate may be related to the films' H concentration, suggesting that H may be involved in the process. For instance, UV light could crack trapped molecular H_2, possibly located at voids in the nitride matrix. The atomic H might subsequently diffuse to the K^0 center passivating it. The energy necessary to crack H_2 in vacuum is 4.52 eV,[31] which is certainly present in the spectrum of broad-band UV light. The dissociation energy for H_2 is expected to be smaller in an a-SiN$_x$:H matrix.

Partial support for a hydrogen-related process is provided by several experiments discussed below: infrared studies, low temperature EPR studies, and observation of a broadening of the EPR line shape. If the irreversible decay process is diffusion-limited or limited by cracking of a H bonded species, one might expect a temperature dependent K^0 decay rate. The EPR results illustrated in Fig. 7 indicate that this is the case; the UV-annihilation rate of the K^0 center is decreased by a factor of two when the UV illumination is performed at 77 K. The UV-annihilation of the K^0 center is smaller (33%) when the illumination is performed at 77 K. At room temperature, the illumination decreases the EPR signal by about 70%. This result suggests that the K^0 decay process could be diffusion limited. During this low temperature experiment, we were not able to observe paramagnetic H^0. If the UV light does crack H-related species, the steady state, unbonded atomic H density is very low due to fast recombination or dimerization. We do acknowledge, however that this is a very small temperature dependence for a diffusive process.

The broadening of the K^0 center EPR line shape during annihilation (see Figs. 1 and 2) could be a consequence of

(1) a structural change (bond angle distributions which will be discussed later) or
(2) a change in the hyperfine fields interacting with the unpaired spin. The interaction of the magnetic moment of

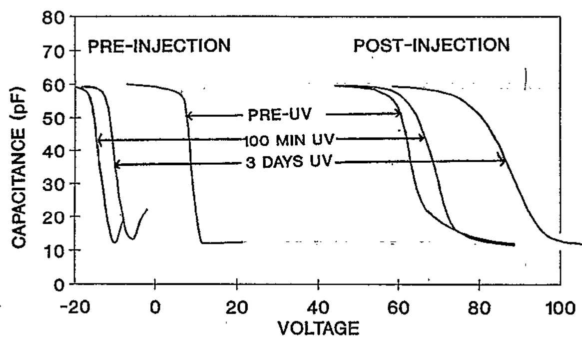

FIG. 8. CV curves of a-$SiN_{1.6}$:H films that were UV-illuminated for 0, 100, and 4320 min before and after electron injection. Positive corona ions were used to inject the electron; the positive field during the illumination was 5.5 MV/cm. The films were deposited at T_s=400 °C. The K^0-center concentration for the film exposed to the UV light has been irreversibly decayed by 57%.

the unpaired spin on the Si atom and the nuclear magnetic moment of H could cause this broadening.

It also appears that long illumination times lead to a small increase in the amplitude of the Si-H and N-H stretching modes. These data are shown in Fig. 2 of Ref. 9. This effect is not observed during short time UV exposures.[21] This increase was between 10% and 15% for the Si-H mode after 1024 min of broad-band UV illumination on N-rich films deposited at 250 °C. Following the 1024 min UV exposure, there was no detectable alteration in the Si-H mode for PECVD films deposited at 400 °C. The change in the N-H mode was about the same for both deposition temperatures, roughly 1%–2%. The film thickness also increased by 1%–2% by this long UV treatment. Similar infrared results on the effects of prolonged UV exposure were also reported by Brendel *et al.*[32]

A second possibility to explain the annihilation of the K^0 center is that the prolonged UV light exposure causes a structural change in the highly hydrogenated nitride films. This structural change could lead to changes in the K-center correlation energy, or perhaps increased potential fluctuations[22] via bond angle variations. For instance, suppose that the prolonged UV-light simply gives the dangling bonds a chance to go back to their positively and negatively charged configurations. This may occur if the activation energy for the net reaction

$$2K^0 \rightarrow K^+ + K^- \quad (1)$$

becomes smaller via structural relaxation during the prolonged UV exposure.

E. Charge trapping measurements

It is of interest to investigate the charge trapping ability of the a-$SiN_{1.6}$:H films following the prolonged UV exposure. The effect of injecting electrons into films UV illuminated for various times (0, 100, and 4320 min) is shown in Fig. 8. The a-$SiN_{1.6}$:H films used in this figure were deposited at T_s=400 °C, and the UV illumination was performed using a broad-band 1 W Hg lamp at room temperature. The nitride films were either

(1) not illuminated (K^0-center concentration $<3.7\times10^{16}/cm^3$),
(2) UV illuminated for 100 min (K^0-center concentration $=1.2\times10^{18}/cm^3$), or
(3) UV illuminated for three days (K^0-center concentration $=0.52\times10^{18}/cm^3$).

All films were subjected to identical charge injection conditions.

From Fig. 8 it appears as though the density of electron trapping centers in the a-$SiN_{1.6}$:H films is not reduced even though roughly 60% of the K^0 centers are annihilated in the films exposed to UV light for three days. One could argue that the density of electron traps is somewhat greater in the films exposed to UV light. Essentially identical results were also obtained by hole injection experiments, i.e., the UV-illumination time does not, to first order, affect the concentration of hole traps in the a-$SiN_{1.6}$:H films (not shown). The reason why the density of traps is the same between the unilluminated and the 100 min. UV-illuminated samples has been explained elsewhere.[17] Simply put, short-time UV exposures convert the K^+ and K^- centers to K^0 centers, leaving the density of trap sites unchanged, i.e., UV-light changes the spin state and charge state of the defects, but not the density.

The results of these trapping experiments are surprising. We would have expected significantly less electron and hole trapping to occur in the samples exposed to UV light for longer periods of time. To explain this anomaly we offer two possibilities. First we suggest that prolonged UV exposure creates new defects which also trap charge, making up for the UV-induced loss of the K^0 center. Some support for this new defect creation model may be found by noting that similar phenomena are also observed in chalcogenide glasses (another negative U system). In the chalcogenide glasses it is believed that the light creates electron/hole pairs which become trapped at pre-existing defects; however, new defect centers are believed to be created by prolonged light exposure.[33]

The second possibility, as discussed earlier, is that the prolonged UV exposure causes a structural relaxation that leads to the deactivation of the K^0 centers to its K^+ and K^- precursors. This would not change the nature nor the density of charge traps. Some support for this possibility is found by noting that crude etch back experiments indicate that the charge centroids for films injected with electrons (or holes) are roughly independent of UV-exposure time. This result could contend that the same centers are responsible for the trapping of the electrons (holes). Our etch back experiments indicate the charge centroid for electrons (holes) is roughly 65 nm (80 nm) from the $SiO_2/SiN_{1.6}$:H interface.

From the above discussions, and given all of the uncertainties of the annihilation process, it becomes quite clear that further studies are needed for a better understanding of the annihilation mechanism and the relationship between the K center and the charge trapping in a-SiN_x:H thin films.

IV. CONCLUSIONS

We have employed electron paramagnetic resonance, photothermal deflection spectroscopy, and capacitance-voltage measurements to demonstrate the simultaneous UV activation and UV annihilation of Si dangling-bond defects, subgap optical absorption, and positive charge, respectively, in a-$SiN_{1.6}$:H films. These experiments show a similar time evolution for all of the above quantities. While short-time UV exposures appear to activate Si dangling bonds (subgap absorption, and positive charge) due to charge conversion of K^+ and K^- sites,[9–11,17,24] K^0 annihilation predominates at longer exposure times. Our results imply that the annihilation of the Si dangling bond may be related to the film's hydrogen concentration; the decay is most rapid for the films with the largest density of hydrogen. For the annihilation mechanism a number of possibilities exist such as a H cracking mechanism or a structural relaxation. The irreversible decay rate of the K^0 center is reduced when the illumination is performed at low temperatures (77 K), suggesting a diffusion-limited process. Last, the density of charge trapping centers in the a-$SiN_{1.6}$:H films is not strongly affected by the annihilation of the Si dangling-bond defects via prolonged UV exposure. The idea of a simple Si dangling bond being the charge trapping center is somewhat difficult to reconcile with our prolonged UV results; perhaps another charge trapping center is involved in this particular case.

ACKNOWLEDGMENTS

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