ROOM TEMPERATURE REACTIONS INVOLVING SILICON DANGLING BOND CENTERS AND MOLECULAR HYDROGEN IN AMORPHOUS SiO_2 THIN FILMS ON SILICON

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Abstract

Exposing thin films of amorphous SiO_2 to molecular hydrogen at room temperature converts some silicon dangling bond defects, E′ centers, into two hydrogen coupled complexes. We argue that these reactions may play important roles in radiation and hot carrier instabilities in metal/oxide/silicon devices.

I. INTRODUCTION

The damaging effects of ionizing radiation on metal/oxide/silicon (MOS) field effect transistors (MOSFETs) have been intensively investigated for nearly thirty years [1-15]. These investigations have been of real and growing technological importance, as the MOSFET has become the fundamental building block of modern microelectronics technology. The utilization of this technology in satellites and deep space exploration has long required the development of radiation tolerant or "radiation hard" MOS devices. With the advent of ultra large scale integrated (ULSI) circuitry, a fundamental understanding of radiation damage becomes a topic of more widespread technological importance. ULSI processing can involve ionizing radiation; examples would be synchrotron and electron beam lithography. Perhaps most significantly, the short channel MOSFETs of ULSI technology may be susceptible to a variety of hot carrier instabilities. These hot carrier instabilities are created when hot holes or hot electrons are injected from the silicon into the oxide. It is clear that the structural changes at the Si/SiO_2 interface caused by hot hole injection and ionizing radiation are the same [16,17].

Radiation damage in MOSFETs involves the capture of holes in the oxide and the creation of interface traps at the Si/SiO_2 interface. It has been convincingly established that the radiation damage at the Si/SiO_2 interface involves, to first order at least, the interaction of radiation induced holes with the oxide [9], not direct interaction of energetic photons and damage sites.

For many years it has been widely suspected that hydrogen plays an important role in determining the radiation response of MOS devices [4-6,10-15]. This suspicion is well justified. Early studies of MOS device processing had demonstrated that hydrogen exposure at elevated temperature could profoundly affect device performance. Later studies indicate that the radiation response of MOS devices is strongly influenced by the presence of hydrogen during processing [1-3].

A number of studies of the time, temperature, and electric field dependence of the radiation damage process at the Si/SiO_2 interface can be explained in terms of the migration of some form of radiolytic hydrogen to the interface and a reaction of this hydrogen to form dangling bonds at the Si/SiO_2 interface [4-6,10-12]. These dangling bond centers, P_b centers[16], would then be responsible for radiation induced interface traps.

With the very clear evidence that holes in the oxide dominate the damage process and the somewhat more circumstantial evidence linking hydrogen to interface traps, it is reasonable to suspect a link between holes, hydrogen, and the radiation damage process at the Si/SiO_2 interface.

Since it is well established that much of the interface trap generation process proceeds in a period of tens of seconds to a few minutes, any defect/hydrogen interaction relevant to the Si/SiO_2 interface trap generation process must also take place, at least in part, within a period of a few minutes at room temperature.

We have identified two reactions involving E′ centers and molecular hydrogen which can occur in a few minutes at room temperature when SiO_2 films are exposed to molecular hydrogen. (The E′ center is an unpaired electron residing in an sp^3 orbital of a silicon bonded to three oxygens [18]; in thermal SiO_2 films on silicon it is a hole trapped in an oxygen vacancy [16]. The E′ center dominates hole trapping in thermal SiO_2 films on silicon [16]. It is the primary cause of positive charge buildup in irradiated MOS devices [16].) These results are consistent with possible roles for these reactions in the interface trap generation process in MOS devices.

II. BACKGROUND

About twelve years ago, McLean [10] developed a fairly detailed model to explain interface trap formation at the Si/SiO_2 interface in terms of hole/hydrogen interactions in the oxide, the subsequent drift of protons to the Si/SiO_2 interface, and the eventual creation of silicon dangling bonds at that interface. The McLean model is consistent with many earlier observations [4-6] and predictions of his model are strongly supported by recent studies of radiation damage kinetics [11,12].

Several authors have proposed modifications of McLean's model. Griscom [19] has proposed a model in which molecular hydrogen is cracked at point defects in the MOS oxide to release atomic hydrogen. (He suggested non-bridging oxygens as the most likely cracking defect site but did not rule out E′ centers for ultra-dry oxides.) In the Griscom model, Si/SiO_2 interface trap generation takes place after the "cracking" event when the released atomic hydrogen reacts at the Si/SiO_2 interface to form P_b centers. Recently, Shaneyfelt etal. [20] have proposed a model in which the protons of the McLean model are liberated at the site of hole capture.

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The experimental evidence regarding hydrogen's role in MOS interface trap formation is compelling, but it consists primarily of electrical measurements. Although very detailed atomic scale models have been proposed to explain precisely how the radiation induced interface traps are created, there is very little direct experimental evidence regarding the atomic scale structures involved in the reactions of hydrogen and radiation damage centers in SiO_2 films on silicon.

Some tantalizing evidence regarding hydrogen reactions with SiO_2 damage centers was provided in a pioneering study by Triplett, Takahashi, and coworkers [21,22]. Triplett, Takahashi, and coworkers [21,22] were first to observe E′/hydrogen complexes in thin SiO_2 films on silicon. They subjected thermally grown oxides to very heavy irradiation (~2 x 10^{10} rads) and then briefly exposed the oxides to 10%H_2/90%N_2 forming gas at 110°C. They noted a substantial decrease in the central E′ line and the appearance of the 74G doublet signal corresponding to a density which closely matched the decrease in the central E′ intensity. The 74G doublet defect is an unpaired electron on a silicon backbonded to two oxygens and a hydrogen atom [23,24]. Triplett, Takahashi and coworkers suggested (we think correctly) that their observations might be relevant to Si/SiO_2 interface trap formation.

In this study we show that two hydrogen complexed E′ centers can be generated in irradiated SiO_2 films by briefly exposing these films to molecular hydrogen at room temperature. We observe some generation of the 74G doublet defects as well as the generation of larger numbers of a second hydrogen complexed E′ defect called the 10.4G doublet center.

Our study has utilized SIMOX (separation by implanted oxygen) buried oxides because, as we had shown earlier, [25,26] these oxides exhibit extremely high densities of E′ precursors. The high density of E′ centers in irradiated SIMOX buried oxides allows us to observe the hydrogen/E′ reactions with a level of sensitivity which would be difficult to achieve in conventional thermally grown oxides on silicon. (Similar studies on thermally grown oxides are presently under way.)

III. EXPERIMENTAL DETAILS

The 480 nm thick SIMOX buried oxides utilized in this study were prepared by implanting 1.8 x 10^{18} oxygens/cm^2 at an energy of 200 keV. The ion current density during deposition was 34 mA/cm^2. The temperature of the substrate during implantation was 640°C. The implant step was followed by a 5 hour anneal at 1315°C in an ambient of 99.5% Ar and 0.5% O_2. The silicon surface orientation was (100); the silicon is n-type with a resistivity of about 30Ω cm. A residual oxide layer formed by the high temperature anneal, as well as the superficial silicon layer, were removed by room temperature etches in HF (residual oxide) and KOH (silicon) prior to the study. After etching, the samples were cut into 3.5 mm x 20 mm rectangles and, with the oxides protected, subjected to a buffered HF etch at room temperature. This last etch removes mechanical damage from the edges of the samples.

The ESR measurements were conducted at room temperature on an X-band commercial spectrometer using a TE_{104} cavity and a calibrated weak pitch standard. The system allows an absolute accuracy in spin density of better than a factor of two and a relative accuracy in spin density of approximately ± 10%.

IV. RESULTS

A. Generation of Hydrogen Complexed Defects

Figure 1 shows a comparison of the ESR spectra of buried oxides exposed to a) about 210 Mrads (SiO_2) of gamma irradiation from a Co^{60} source and b) 40 hours of VUV irradiation. The gamma irradiated samples were not subjected to the KOH etch; they retained the ~1μm superficial silicon overlayer. The VUV irradiated samples were etched in KOH and did not retain the superficial silicon layer. The gamma irradiated samples and the VUV irradiated samples exhibit nearly identical ESR spectra. The strong signal with a zero crossing g-value of g=2.0005 corresponds to the normal E′ defect of figure 2a. (The g is defined as $g=h\nu/\beta H$, where h is Planck's constant, ν is microwave frequency, β is the Bohr magneton, and H is the field at resonance.) The two lines on either side of this large resonance correspond to the 74 Gauss doublet structure of figure 2b. The nuclear spin 1/2 of the hydrogen splits the E′ into two lines separated by 74 Gauss. This weak 74G doublet spectrum compromises 3-5% of the total E′ spins.

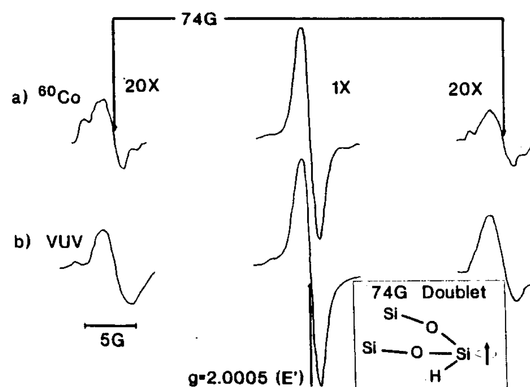

Figure 1: Comparison of effects of (a) C_0^{60} and (b) VUV irradiation on SIMOX buried oxides. The large central resonance at g = 2.0005 is due to simple E′ centers. The smaller "lines" on either side are due to hyperfine interaction in hydrogen complexed E′ centers called 74G doublets.

In figure 3a, we illustrate a wide scan ESR trace of buried oxides subjected to about 10 hours of vacuum ultraviolet radiation ($hc/\lambda \leq 10.2eV$). Also in figure 3, we illustrate ESR traces of the same SIMOX samples after b) ten minutes and c) one hour exposure to a 10%H_2/90%N_2 forming gas at room temperature. Note the large increase in the 74G doublet spectra and the appearance of two new lines on the shoulder of the central E′ signal. These two new lines have a separation of approximately 10.4 Gauss and correspond to the defect structure in figure 2c known as the 10.4G doublet center.

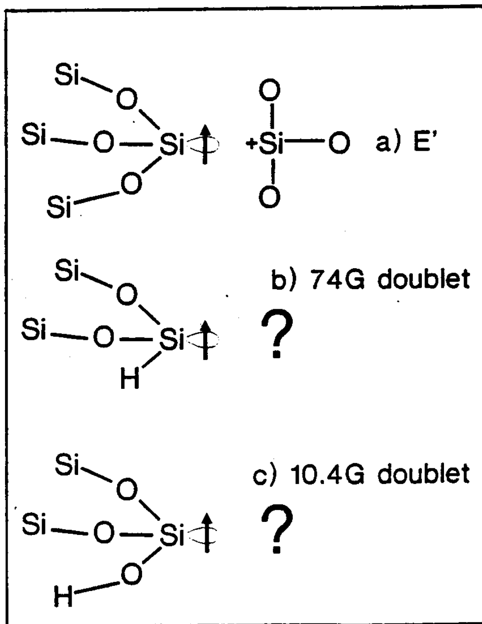

Figure 2: Schematic illustrations of defect structures. Shown are (a) normal E′ center, (b) 74G doublet, and (c) 10.4G doublet.

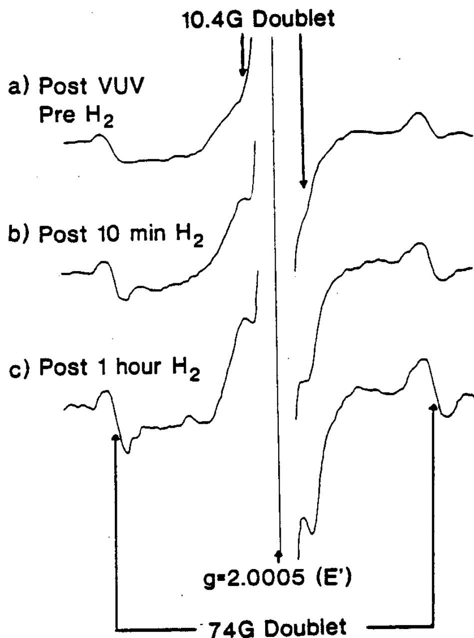

Figure 3: Wide scan ESR spectrum of VUV illuminated SIMOX oxides (a) before H_2 exposure, (b) after ten minutes H_2 exposure, and (c) after one hour H2 exposure. An additional hour of H_2 exposure did not significantly alter the spectra. Note the growth of the peaks separated by 74G and the appearance of the peaks separated by 10.4G.

In figure 4, we illustrate a narrow ESR trace with spectrometer settings optimized to fully resolve the 10.4G doublet. The fully resolved lineshape strongly supports the atomic structure of figure 2c proposed by Tsai and Griscom [24]. However, the doublet splitting appears to be slightly greater than 10.4G.

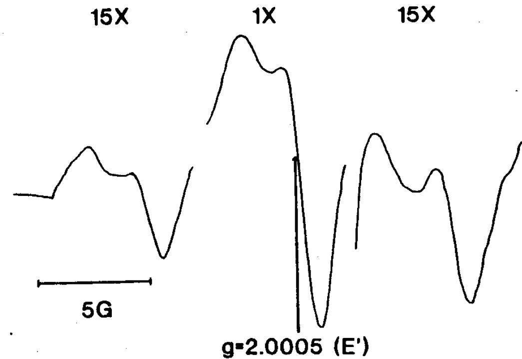

Figure 4: Narrow scan ESR trace of the samples from trace 2c. The spectrometer settings are set to optimize resolution of the 10.4G doublet lineshape. Note that the doublet patterns closely match the shape of the central E′.

In figure 5, we plot E′, 74G doublet, and 10.4G doublet defect densities as a function of hydrogen exposure time. Ten minutes of hydrogen exposure results in a 25-30% decrease in normal E′ density. (By "normal" we mean E′ centers which are not hydrogen complexed. However, in using this terminology we do not distinguish between $(0_3 \equiv Si \cdot {+Si} \equiv 0_3)$ and $(0_3 \equiv Si \cdot)$ E′ centers.) This decrease in normal E′ density is accompanied by a large increase in 10.4G doublet density and a smaller increase in 74G doublet density. An additional 50 minutes H_2 exposure results in a substantial increase in 74G doublet density with only a slight increase in 10.4G doublet density and a slight decrease in normal E′ density. Another additional hour of H_2 exposure results in no significant changes in defect density (less than experimental error).

It is interesting to note that the number of defects associated with the sum of the newly created 10.4G and 74G doublet signals are approximately equal to the decrease in normal E′ defects. The total number of spins is preserved. We thus tentatively conclude that we are observing a transformation of normal E′ centers (2a) into two hydrogen complexed E′ centers (2b and 2c). It may be worth noting that much of the hydrogen complexing process, especially the creation of 10.4G doublet centers, occurs in a time period which is roughly equal to the time required for the molecular hydrogen to diffuse across the oxide [19].

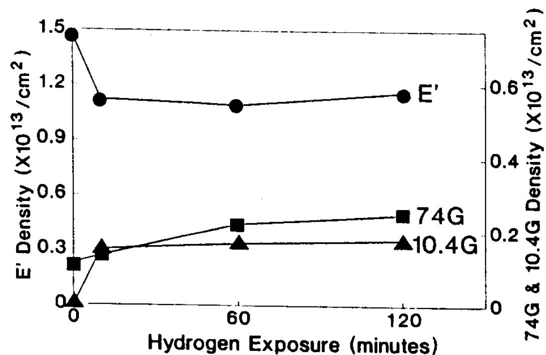

Figure 5: Plot of defect concentrations vs. H_2 exposure time for E′, 74G doublet, and 10.4G doublet. Note decrease in normal E′ density and increase in both 74G and 10.4G doublet densities.

B. Electronic Properties of Hydrogen Complexed Defects

To assess the electronic properties of the three E′ defects, we performed a series of charge injection experiments on VUV illuminated buried oxides which were subsequently soaked in hydrogen. (The charge injection techniques are described in detail in another publication [25].

First, using UV $(hc/\lambda \leq 5eV)$ illumination from a mercury-xenon lamp and oxides positively biased with corona ions, we photoinjected electrons into hydrogen soaked VUV illuminated oxides. Photoinjection of approximately 5×10^{13} electrons/cm^2 resulted in the annihilation of approximately 80% of the 74G doublet centers and approximately 50% of the 10.4G doublet centers. This same electron injection resulted in a 25-30% increase in normal E′ density.

Next, we subjected similarly prepared oxides to an equivalent time UV $(hc/\lambda \leq 5eV)$ exposure without bias. After roughly four minutes of UV exposure, we again observed annihilation of roughly 80% of the 74G doublet centers but only 20% annihilation of the 10.4G doublet centers. Normal E′ density was only increased by about 15%.

Finally, we subjected (again, similarly prepared) oxides to UV illumination $(hc/\lambda \leq 5eV)$ with negative bias. The negative bias should substantially impede the flow of electrons into the oxide. We found that this UV with negative bias very effectively annihilated 90% of the 74G doublet density and about 60% of the 10.4G doublet density. This time, however, the density of normal E′ centers was not affected.

The results of our positive, zero, and negative bias UV illumination, show that normal E′ density increased under UV illuminations both with positive and zero bias but not under illumination with negative bias, where electron photoinjection is impeded. This reveals that, in hydrogen soaked oxides, the E′ density increases as a result of electron injection. In addition, we see that both 74G and 10.4G doublet densities decrease under UV illumination, whether electrons are photoinjected or not. This result suggests that both 74G and 10.4G centers are responding directly to UV photons.

In order to evaluate these conclusions we injected electrons and holes into VUV illuminated oxides which were subsequently hydrogen soaked without exposing the oxide bulk to UV photons. We did this by again charging the oxide surfaces with corona ions, but then exposing the oxides to vacuum ultraviolet (VUV) $(hc/\lambda \approx 10.2eV)$ light from a notch filtered deuterium lamp. The oxide's optical absorption coefficient is extremely large for photons of this energy ~10^6/cm; thus, virtually all the photons are absorbed in the top 10nm of the 480nm oxide. Under positive corona bias, the bulk of the oxide is flooded with holes; under negative corona bias, the bulk of the oxide is flooded with electrons. In both cases the fluence of injected charge carriers was about 5 x 10^{13}/cm^2 and the average oxide field during the process was ~1 to 3MV/cm.

We found that electron injection with these non-penetrating photons generate a substantial 35% increase in normal E′ density. However, this substantial change in normal E′ density was accompanied by no significant change in either 74G or 10.4G defect densities.

Holes injected with these non-penetrating photons had a very similar effect. We observed a 35% increase in normal E′ density accompanied by little or no net change in either 10.4G or 74G defect densities.

The results of our charge injection and illumination studies are summarized in Table 1.

Post VUV, H_2 Soaked Oxides	E'	74 G	10.4 G
a) UV Exposure:			
+ Bias	+25-30%	-80%	-50%
Ø Bias	+15%	-80%	-20%
- Bias (no electron photoinjection)	unchanged	-90%	-60%
b) 10eV VUV Exposure (no bulk UV Exposure)			
+ Bias	+35%	unchanged	unchanged
- Bias	+35%	unchanged	unchanged

TABLE 1: Percentage change in defect concentrations of E', 74G, and 10.4G after exposure to either UV or VUV, with and without bias.

The combined results of the VUV and UV illumination measurements strongly suggest that the changes observed in the 10.4G and 74G signals with UV illumination are due to photons interacting with the defects. The results also strongly suggest that the normal E′ signal can be increased by electron capture at previously diamagnetic sites in hydrogen soaked oxides. This does not happen in SIMOX oxides which have not been exposed to hydrogen. (We reported earlier that [26,27], prior to hydrogen exposure, the injection of holes into the oxide consistently results in a substantial increase in E′ density, while the injection of electrons into the oxide consistently decreases E′ density.) These new results strongly suggest that the E′/hydrogen reactions involve substantial structural changes at the E′ site. Since the injection of electrons into the hydrogen soaked oxides <u>increases</u> E' amplitude, our results also suggest that the post irradiation hydrogen exposure can result in the creation of single silicon dangling bond sites $(O_3 \equiv Si \cdot)$ from sites which had initially

been holes trapped at oxygen vacancies $\left(O_3 \equiv Si \cdot + Si \equiv O_3\right)$. (Both of these centers would yield the normal E′ line shape - at least to first order.)

V. DISCUSSION

During the past fifteen years several models have been proposed to explain the interface state generation process at the Si/SiO_2 interface. Most models propose prominent roles in this process for hydrogen in one or more forms, or holes and hole traps, or some combination of hydrogen species and hole traps. We believe our work will help to identify the aspects of these models which are consistent with the actual damage process and help rule out other models.

Perhaps the most widely accepted Si/SiO_2 radiation damage model is that of McLean [10]. In McLean's model, radiation induced holes interact in the oxide to liberate protons. The protons drift to the Si/SiO_2 interface to create silicon "dangling bond" interface trap defects.

A somewhat less widely accepted model involves the transformation of trapped holes into interface traps. The Princeton group [28,29] has argued that there is a one-to-one or one to two correspondence between trapped holes and interface trap creation.

Several modifications of these ideas have been proposed in the past few years. Griscom [19] has proposed a model in which molecular hydrogen is "cracked" at defect sites in the oxide, a process which eventually leads to hydrogen reactions at the Si/SiO_2 interface creating P_b center interface traps. He proposed that these cracking sites would probably involve unpaired electrons on oxygen atoms; however, he thought E′ centers might also play a role in hydrogen cracking in "ultra-dry" oxides. The idea that molecular hydrogen might play a role in Si/SiO_2 interface damage is supported by a number of studies which indicate that a molecular hydrogen ambient can lead to a substantial increase in radiation induced interface trap generation [13,14,15,30].

Quite recently Edwards [30] has used molecular orbital calculations to argue that E′ center sites are not likely hydrogen cracking sites. His calculations indicate that E′/hydrogen reactions would have a quite large activation energy. As a result of these calculations, Stahlbush and Edwards [30] argued that "oxygen hole centers" would play dominating roles in the molecular hydrogen cracking process.

Recently, Shaneyfelt etal. [20] proposed a model in which protons are liberated at the site of hole capture. This could be a site near the Si/SiO_2 interface in a device which was positively biased during irradiation. It could be near the gate/SiO_2 interface in a device negatively biased during irradiation. Following proton liberation, the protons drift to the Si/SiO_2 interface and form interface traps as proposed by McLean.

The Shaneyfelt etal. model is of particular interest since it invokes the proton drift of the McLean model (which is supported by much experimental work) and appears to be, in part, consistent with the ideas of Griscom, which link oxide defect/hydrogen cracking reactions to interface trap formation. The Shaneyfelt etal. model also involves a connection between hole trapping events and interface trap generation. This aspect of the model is at least qualitatively consistent with the data of the Princeton group [28,29], though it is not consistent with the Princeton interpretation of that data.

Our UV and VUV illumination results quite clearly demonstrate that two reactions involving E′ centers and hydrogen can occur rapidly at room temperature. The creation of the 10.4G doublet centers appears to proceed in a time span roughly equal to the time required for the molecular hydrogen to diffuse across our oxides. The results, first of all, are not consistent with the recent calculations of Edwards [30], which appear to rule out E′ centers in hydrogen cracking. Our results suggest that if a "hydrogen cracking" step does play a significant role in the Si/SiO_2 interface trap formation process, E′ centers could indeed be the responsible defect. Since the recent work of Shaneyfelt etal. implicates the hole trap site in the radiation induced interface state generation process, and since E′ centers do dominate hole trapping in irradiated thermal oxides [16], our observations suggest that these E′/hydrogen reactions may be involved in the interface trap generation process.

Our results involving the electronic properties of the E′ centers after hydrogen soaking strongly suggest that the electronic properties of at least some of the centers are greatly altered by hydrogen reactions. These changes in E′ electronic properties may help to explain changes in oxide charge reported in studies of the effects of molecular hydrogen exposure on the electronic properties of irradiated devices [30].

Our results tend to confirm, in a qualitative sense at least, several ideas expressed in the pioneering study of Triplett, Takahashi [21,22] and coworkers; they suggested that the E′/hydrogen reaction they observed (the creation of 74G doublet centers) was likely connected to the radiation induced interface state creation process at the Si/SiO_2 interface. Their work also indicated changes in the electronic properties of E′ centers after hydrogen soaking.

A final point, possibly worth noting, is that, in the creation of the 10.4G doublet center, we observe the breaking of silicon-oxygen bonds near the paramagnetic silicon atom of the E′ defect. The 10.4G doublet almost certainly involves a paramagnetic silicon bonded to three oxygens. One of the oxygens is bonded to a hydrogen. That oxygen had previously been bonded to another silicon. A silicon oxygen bond breaking event would therefore be involved in this process. Silicon bond breaking events induced by hydrogen have long been invoked to explain interface trap creation. Our observation of 10.4G doublet formation represents a direct observation of such an event in SiO_2 on silicon, apparently for the first time.

VI. CONCLUSIONS

We observe two reactions involving E' centers and molecular hydrogen which take place rapidly at room temperature in SiO_2 films on silicon. Our observations directly contradict the conclusions of a recent theoretical study [30]. Our results suggest that an extension of the recent model of Shaneyfelt etal [20] to include molecular hydrogen/E' reactions may provide the most reasonable current explanation for the generation of interface traps at the Si/SiO_2 boundary. (The Shaneyfelt etal model is an extension of the earlier McLean proton drift model[10] in which the site of proton liberation is the site of hole capture. Earlier ESR work indentified the

dominant trapped hole defects to be E' centers[16].) This extension of the Shaneyfelt model would link the idea of molecular hydrogen/oxide defect reactions proposed by Griscom[19] with original proton drift ideas of McLean and the Shaneyfelt etal contribution that the proton liberation process is initiated at oxide hole trap sites.

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