Molecular Hydrogen, E' Center Hole Traps, and Radiation Induced Interface Traps in MOS Devices

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Abstract

We provide long sought direct atomic scale evidence for molecular hydrogen reactions at a specific point defect in irradiated thermally grown SiO_2 films on Si. Using electron spin resonance (ESR), we observe hydrogen interaction at E' centers in thermal oxides exposed to molecular hydrogen at room temperature. (The E' center is the dominant hole trap in thermally grown SiO_2.) The decrease in E' density occurs on a time scale similar to a comparable increase in density of interface traps. The similarity of the rate of E' decrease to the rate of interface trap increase and the approximate agreement between the number of E' centers and interface traps involved in the two reactions is very strong evidence that E' centers are involved in the interface trap formation process in radiation and hot carrier damaged thermally grown SiO_2 on Si.

I. INTRODUCTION

The deleterious effects of ionizing radiation and hot carrier damage on MOS devices have been studied for three decades. These studies have determined that one of the most harmful effects of ionizing radiation and hot carrier damage is the creation of interface traps at the Si/SiO_2 interface. Although the structural nature of radiation and hot carrier induced interface trap defects has been identified [1-8], the process by which interface traps form remains mysterious, a whodunnit with many suspects. The primary suspects include holes in the oxide, hydrogen (in various forms), oxide hole traps, and interface traps. Definitive evidence that holes in the oxide play an important role in radiation induced interface trap formation was provided by Winokur and Sokoloski [9]. Very strong evidence for the involvement of hydrogen has been provided by experimental studies of interface trap generation kinetics by groups at Harry Diamond Laboratories [9,10], the Naval Research Laboratory [11,12], and Sandia National Laboratories [13], to mention a few. A very recent isotopic substitution study by Saks and Rendell [12] unequivocally establishes a role for hydrogen in some form in the interface trap (D_{it}) formation process. A recent study by Kohler *et al.* [14] of the effects of a room temperature molecular hydrogen ambient during and after irradiation links H_2 to the post-irradiation generation of interface traps. All of these studies are generally consistent with the proton drift model proposed by McLean [15].

The clear evidence that oxide holes (and not direct photon interaction) dominate the radiation damage process, combined with strong evidence linking hydrogen drift to interface trap generation, leads one to suspect a link between oxide hole traps and hydrogen during radiation induced interface trap formation.

The suspicion of a link between hydrogen, hole traps, and interface traps is supported by recent investigations of Shaneyfelt *et al.* [13], Stahlbush *et al.* [16, 17, 18] and also Mrstik [19, 20] that considerably extended and refined the results of Kohler *et al.* [14]. All of these studies noted that during post irradiation exposure to an H_2 ambient, D_{it} increased as N_{ot} decreased. All of these studies suggest that the site of hole capture will somehow release a hydrogen species that will drift to the interface and react to form an interface trap.) The Stahlbush *et al.* and Mrstik studies both concluded, on the basis of purely electrical measurements, that H_2 was being cracked at a hole trap site.

The structure of the dominant hole trap site and the interface trap has been determined by electron spin resonance (ESR). The ESR studies of Lenahan and Dressendorfer established that the dominant oxide hole trap is the E' center [2-5]. The structure of the E' center involves an unpaired electron on a silicon backbonded to three oxygens. ESR work by the same group has also determined that the P_b center (an unpaired electron on a silicon backbonded to three other Si) is primarily responsible for radiation induced interface trap defects [2-5]. One would expect that if hole traps are involved in D_{it} creation, that E' centers would be involved and that the interface traps created during post-irradiation H_2 exposure would be the same defect created by irradiation, the P_b center.

It is very interesting therefore that although both Stahlbush *et al.* [16, 17, 18] and the Mrstik *et al.* [19, 20] eliminated E' centers from consideration, both studies still concluded that H_2 was being cracked at a hole trap site. Stahlbush *et al.* eliminated the E' center on the basis of theoretical calculations by Edwards [17, 18]. These calculations indicated that interactions between H_2 and E' centers were "unlikely at room temperature" and thus led Stahlbush *et al.* to suggest (without benefit of atomic scale data) that paramagnetic oxygen centers are the "cracking" sites [17, 18]. Mrstik *et al.* [19, 20] found that the room temperature D_{it} increase (during H_2 exposure) ended after only about 25% of the trapped positive charge had been used up. On the basis of this electrical evidence [19, 20], they too dismissed the E' center (predominantly responsible for the trapped positive charge) as a candidate for the H_2-cracking hole-trap site.

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Last year at the Nuclear and Space Radiation Effects Conference [21] we presented atomic scale ESR evidence that, in separation by implanted oxygen (SIMOX) buried oxides, E'/H_2 interactions can occur and in fact proceed rapidly (in minutes) at room temperature. (We had earlier determined that the E' center play a dominating role in SIMOX buried oxide charge trapping [22, 23], though the relationship between the E' centers and net charge is more complicated than in thermal oxides [24, 25, 26].) In irradiated SIMOX oxides exposed to H_2 (10% H_2 / 90% N_2) at room temperature [21, 27], we observed evidence for a direct conversion of E' centers to two hydrogen complexed E' defects, the 74 G doublet center and "10.4 G" doublet center. The E' center is an unpaired electron on a silicon backbonded to three oxygens. The 74 G doublet center structure is similar to the E'. The 74 G doublet center is an unpaired electron on a silicon backbonded to two oxygens and one hydrogen [28]. The "10.4 G" (we actually measure a splitting greater than 10.4 G) doublet center structure is also similar to that of the E' center. The "10.4 G" doublet is also an unpaired electron residing on a silicon backbonded to three oxygens, however one of the backbonded oxygens is, in turn, bonded to a hydrogen instead of another silicon. (This provisional model was proposed by Tsai and Griscom [28]; experimental evidence supporting this provisional structure has recently been provided [29].) The structure of the 74 G doublet center and the E' center can be seen in Figure 3. We suggested that these reactions might play a role in the D_{it} formation process [21, 26]. The first group to provide evidence that H_2 / E' reactions can take place in thermal oxides was Triplett *et al.* [30] and Takahashi *et al.* [31]. In their pioneering study, they observed a conversion of E' centers to 74 G doublet defects during exposure to H_2 at 110°C in extremely heavily irradiated (10^{10} rads (SiO_2)) oxides. These earlier studies however could neither address whether or not these reactions would occur in thermal oxides at room temperature nor whether or not these reactions had any direct relevance to the D_{it} formation process.

A review of the literature shows that hydrogen (in various forms) [10-14], mobile holes in the oxide [9], and trapped hole sites [13, 16-20] including E' centers have all been suggested as possible participants in the interface trap formation process. But it seems that the E' center has been ruled out as an important player in interface trap generation in nearly all of the recent literature [16-20]. Curiously, except for the isotopic substitution experiment of Saks and Rendell [12], there is very little direct and absolutely conclusive experimental atomic scale evidence for the reactions involved in the D_{it} creation process. Although the "kinetic" studies of Kohler [14], Stahlbush *et al.* [16, 17, 18], and Mrstik [19, 20], provide great insight, they can not provide direct unambiguous information regarding to the atomic scale processes involved. Another technique, ESR, can.

In this study, we employ ESR in an attempt to answer two questions. (1) Do room temperature E'/H_2 reactions take place in thermally grown SiO_2? and (2) Are E' centers involved in the interface trap formation process?

We believe that our ESR results answer both of these questions, question (1) definitively and (2) with high probability. With ESR we provide an apparently unexpected direct link between a prime suspect, H_2, and a seemingly innocent [17-20] bystander, the E' center, linking both H_2 and E' centers to interface traps and P_b centers. We find that E'/H_2 reactions do occur, in fact quite rapidly (in minutes), in thermally grown oxides [29] and we provide evidence that E' centers and P_b centers both play roles in the interface trap formation process. Finally, we provide a model for the whole process that features E' like hydrogen reaction sites. (The model is, of course, largely a composite of several ideas, most of which have long been in the literature.)

II. EXPERIMENTAL DETAILS

The soft oxides used in this study were grown in steam to 120 nm on (111) 200Ωcm n-type silicon substrates; the oxides were softened with an anneal in nitrogen at 1100°C. A poly-Si gate was removed in a $HF/HNO_3/H_2O$ etch before any measurements were taken.

Our ESR meaurements were taken at room temperature on an X-band spectrometer. The g-values and spin densities are determined using a TE_{104} "double" resonant cavity and a calibrated "weak pitch" spin standard. The spin densities obtained have a relative accuracy of ±10% and an absolute accuracy of a factor of two.

CV measurements were taken at room temperature using a 1 MHz Boonton capacitance meter and a mercury probe. Net space charge density was measured by the mid-gap CV shift. (Charge is assumed to be located at the Si/SiO_2 interface.) The density of interface traps (D_{it}) was measured from flat bands to midgap using the Terman technique; D_{it} values are estimated accurate to within ±10%.

The injection of holes into the oxide was accomplished by first positively charging the oxide surfaces with positive corona ions and then exposing the oxides to vacuum ultraviolet (VUV) light. Corona ions are created by applying a large voltage (10,000 V) to a very sharp needle a few centimeters above our sample. The corona ions drift to the surface of the oxide. The corona induced potential is then measured with a Kelvin probe electrostatic voltmeter. The corona ions have essentially thermal energy and thus do not damage the samples [32]. This method allows for a uniform-bias, transparent, hydrogen permeable "gate" over the large surface area (~1 cm^2) of the samples we use for ESR and CV measurements.)

With the surface of the oxides positively biased with corona ions, hole injection into the oxide was performed by briefly (~50 sec) irradiating the oxides with VUV light (hc/λ =10.2 eV). The 10.2 eV photons are absorbed in the top ≈10 nm of the oxide where they create electron hole pairs [9]. The holes are then driven across the oxide by the positive bias while the electrons are swept out. The hole injection process resulted in the creation of a large number of E' centers (~3 x $10^{12}/cm^2$) that closely corresponded to the net trapped positive charge.

Hydrogen exposures were performed with forming gas (10% H_2 / 90% N_2) at room temperature. A positive corona bias (described above) was applied to the oxides during exposure.

III. RESULTS

We first irradiated an oxide under positive bias (hole injection, described above) and then periodically exposed this oxide to H_2 (in 10% H_2 /90% N_2) at room temperature, also under positive bias. At each stage in the H_2 exposure, the density of E' centers (cm^{-2}) and D_{it} ($cm^{-2}eV^{-1}$) was monitored with ESR and CV measurements.

Prior to any H_2 exposure (time = 0, E' density = 100%), we injected holes into the oxide in order to create E' centers. We then positively bias the oxides with corona ions and expose them to forming gas. The percentage of remaining E' centers vs. H_2 exposure time is shown in Figure 1. Five minutes of room temperature H_2 exposure result in about a 10% decrease in E' density. The reaction saturates after about 30 minutes, consuming about 20 to 25% of the total E' density. (We also see a net decrease in positive charge density of the same order of magnitude as the decrease in E' density.) The fact that we observe a reaction between E' and H_2 at room temperature contradicts the results of Edward's calculations [17, 18]. The fact that we see about a 25% decrease in E' density is consistent with the data of Mrstik [19] who sees a 25% decrease in trapped positive charge, but inconsistent with Mrstik *et al.*'s [19, 20] conclusions. We observe rapid E'/H_2 reactions at room temperature in a wide variety of oxides studied.

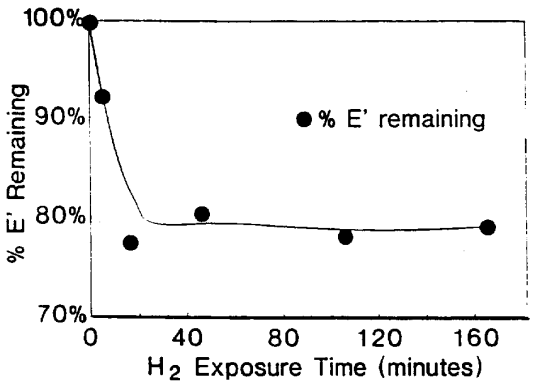

Figure 1: Percentage of remaining E' centers vs. H_2 exposure time under positive bias. The first point (at time = 0, E' = 100%) was taken immediately after irradiation.

In Figure 2a, we compare the absolute change in E' density with the change in D_{it} vs. room temperature hydrogen exposure time. The initial hole injection creates approximately 3 x 10^{12} / cm^2 E' centers, a similar density of trapped charge, and few interface traps (1x10^{11}/cm^2). Again, the oxide was positively charged during H_2 exposure. As E' density decreases in 2a, D_{it} increases in 2b. The saturated value of the change in E' is within a factor of two of the saturated change in D_{it}, within experimental error. (This close correspondance in absolute numbers may be fortuitous.) The response time of these two curves are roughly similar; within the (moderate) range experimental error they are nearly the same. The result strongly suggests a link between E' centers, H_2, and interface trap formation.

Control experiments (no figure) show that both prior radiation damage (now, presumably the E' center) and hydrogen is necessary for the reaction. In one control experiment, we exposed a non-irradiated device to H_2 exposure with positive bias and observed a negligible changes in both D_{it} and E'. In another control experiment we expose an irradiated device to an air ambient with positive bias and again saw no change in either E' or D_{it}. These control experiments are completely consistent with previous results [15, 16, 19] regarding D_{it} response and show that both oxide radiation damage (presumably involving E' centers) and hydrogen are required for the reaction to go.

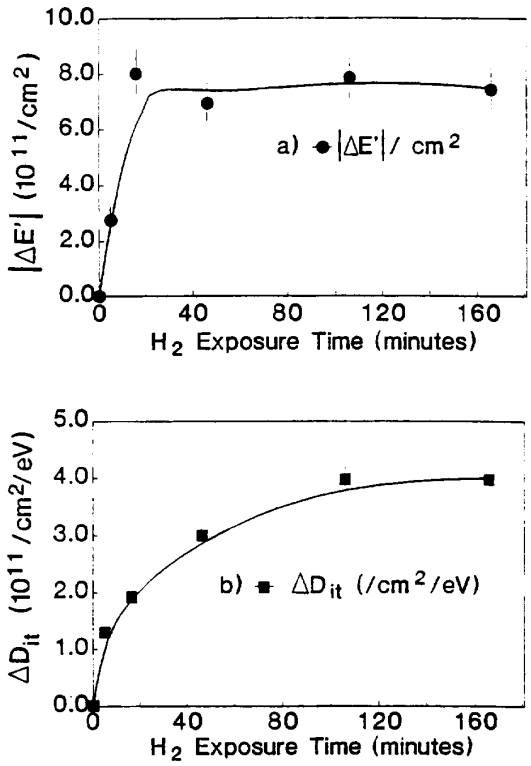

Figure 2: Shown are (a) Absolute change in E' concentration and (b) change in D_{it} vs. H_2 exposure time.

Shown in Figure 3 is the 74G doublet generated in a thermal oxide. The oxide was irradiated in VUV ($hc/\lambda < 10.2$ eV) for an extended period without bias and then exposed to molecular hydrogen for 70 minutes at room temperature. (The 74G doublet is known to be an unpaired spin on a silicon backbonded to two oxygens and one hydrogen [28].) Therefore, at least one of the H_2 induced E' conversion

reactions that we observed in SIMOX also occurs in thermally grown oxides. (Since hydrogen doublets are split in two by the hydrogen hyperfine interaction, and since the spin densities are an order of magnitude lower that for standard E' signals, these defects are very hard to observe through ESR.)

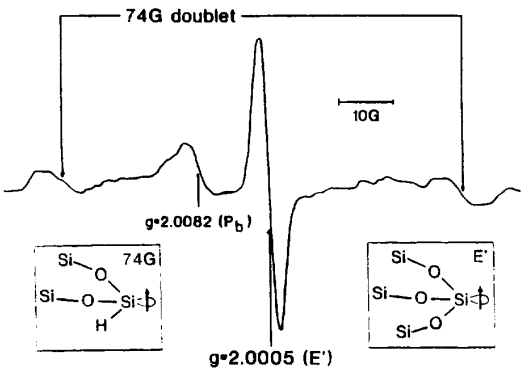

Figure 3: Shown is the 74G doublet in an irradiated thermal oxide after 2 hours exposure to H_2 at room temperature.

IV. DISCUSSION

Do E'/H_2 reactions take place at room temperature in thermally grown SiO_2? Our study answers this question with an unequivocal yes. We observe a rapid room temperature reaction of H_2 at E' sites in several very different thermally grown oxides. In addition, we observe the conversion of E' centers to 74 G doublet centers (hydrogen complexed E' centers) in thermally grown oxides exposed to a room temperature H_2 ambient. The fact that we observe an E' / H_2 reaction is somewhat surprising in that both theoretical [17, 18] and experimental [19] evidence indicate that this reaction is very unlikely. Our observations directly contradict the theoretical predictions of Edwards [17, 18] whose calculations had (seemingly) demonstrated that the room temperature reaction of H_2 with E' centers in thermal oxides were all but impossible. Our results are also inconsistent with the conclusions of Mrstik *et al.* [19, 20], but are actually completely consistent with his data. The primary objection of Mrstik *et al.* [19, 20] to the involvement of E' was that the observed H_2 ambient-induced interface trap generation saturated after only about 25% of the N_{ot} had been removed. We observe that interface trap generation saturates after only about 25% of the E' sites have been removed. Both Stahlbush *et al.* [17, 18) and Mrstik *et al.* [19, 20] postulate that hole traps are involved in the interface trap formation process. The simularity between the percentage of E' centers involved in our study and the percentage of hole traps involved in the Mrstik study is strong evidence that E' centers (known to be the dominant hole trap [2-5]) are those hole traps.

Are E' centers involved in the interface trap generation process? Our study provides a compelling, if not incontroveratable answer to this question as well. We find it very likely that H_2 reactions at E' like centers are relevant to the interface trap formation process in SiO_2 on Si. During post irradiation exposure to H_2 at room temperature, we observe a decrease in E' density occuring on a very similar time scale as an decrease in D_{it}. Both D_{it} and E' changes saturate at values within a factor of two of one another (although this may be fortuitous). Note that we find (as others have) that both radiation induced oxide charge (now, presumably E' centers) and H_2 are required for the interface trap formation process. The similar hydrogen reaction rates, in particular, provide strong evidence that, not only do E'/H_2 reactions occur, they are very likely to be involved in the interface trap formation process. There is other evidence that H_2 reacts at a trapped hole site [16-20]; if this is true, this trapped hole is very likely to be the E' center and not the broken bond hole trap (BBHT) as others have suggested [18]. (We observe no evidence of BBHTs (an ESR active defect) in our study either before or after irradiation or after hydrogen exposure.)

Our results are consistent with a model in which radiation creates holes which then drift through the oxide releasing atomic hydrogen. (Earlier ESR work established that ionizing radiation creates atomic hydrogen in the oxide [33].) This radiolytic atomic hydrogen quickly dimerizes to form H_2. We propose that the H_2 then reacts at radiation generated trapped hole centers (E' like defects). This reaction releases some form of hydrogen which makes its way to the interface where perhaps a bond breaking event of some sort occurs. This end result in an interface trap. Disclaimer: Many models (for example the hydrogen models of McLean [15] and Griscom [34] and the injection model of Lai [35]) have been proposed by many authors to explain the radiation induced interface trap formation process. Radiation damage models are reviewed extensively by Winokur [36]. Our model borrows many ideas from many of those models.

V. SUMMARY

We provide long sought direct experimental atomic scale evidence for a point defect that reacts with H_2 in thermal oxides at room temperature. This point defect is the E' center. We have shown that it is very likely that these E'/H_2 reactions are involved in the interface trap formation process. We also show that E' centers are likely to play roles in post irradiation interface trap formation. We feel that this new found link between H_2, E' centers, D_{it}, and interface traps represents a substantial contribution to the experimental evidence needed to construct a holistic picture of the radiation induced interface trap formation process.

Note added in proof: A study by Li *et al.*. [37] has very recently come to our attention. The Li *et al.*. study invloved the annealing of E' centers in a variey of annealing ambients at a variety of temperatures. They too saw E' density decrease (a greater percentage decrease than we saw, however) with room temperature H_2 exposure. They did not discuss the relevance of their observations to MOS device radiation damage, gate oxide electronic properties, or interface state formation.

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