

Radiation Effects in Low Dielectric Constant Methyl–Silsequioxane Films

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Abstract—The use of state-of-the-art microelectronic devices in space radiation environments faces new challenges with the adoption of low dielectric constant (low- k) materials as inter-level dielectrics. This is demonstrated in a preliminary study of methyl–silsequioxane low- k films. We report radiation damage, induced by a 2-keV low-current-density ($\sim 2 \times 10^6 \text{ s}^{-1} \text{ cm}^{-2}$) positron beam, and observed by positron annihilation spectroscopy.

Index Terms—Dielectric, electron radiation, ionizing radiation, low dielectric constant (low- k), polymer.

I. INTRODUCTION

THE PERFORMANCE in state-of-the-art microelectronic devices can no longer be improved predominantly through miniaturization of the device components as in the past. To realize future progress, the semiconductor industry has been forced to undergo an historic evolution, adopting the use of new materials such as Cu interconnects, low dielectric constant (low- k ; $k < 3.0$) interlayer dielectrics (ILD), metal diffusion barriers, high- k materials for capacitors and gate dielectrics, and so on [1]. Following the Cu technology, the implementation of the low- k dielectrics holds the key for future advances. The quest for the successful low- k ILD-candidate is focused mainly on silicates and polymers (organic, inorganic, and their hybrids) with no prior use in the semiconductor industry. Spin-on and sol–gel fabrication methods are replacing the currently used chemical vapor deposition techniques. This enables the development of methods for incorporating porosity ($k \approx 1$) in the dielectric matrix, which is the only alternative to lowering further the dielectric constant (to $k \sim 2.1$ and below). Low- k dielectrics are entering production at the 130-nm technology node, whereas the implementation of their porous variants is expected at the 90-nm generation [1].

The ever-increasing trend of using commercial off-the-shelf (COTS) microcircuits in satellite and spacecraft systems, and

the inevitability of low- k dielectrics replacing the SiO_2 in COTS parts, necessitate an evaluation of the performance of such materials in space environments. Their properties can be critical for the device performance in a high-radiation environment regardless of the circuit architecture.

Materials behavior under irradiation can be assessed using test structures and blanket low- k films. This can provide valuable information, which does not depend on the maturity of the low- k technology, or the device design, as would be the testing of actual microcircuits. To this end, we have studied porous methyl–silsequioxane (MSSQ) films. MSSQ is among the most studied ILD candidates with a high commercialization potential.

We have used positron annihilation spectroscopy (PAS) [2], [3] to detect the radiation damage. This technique is extremely sensitive to open-volume defects (from vacancies to large pores) due to the propensity of a positron to “seek” defects, in contrast to common (electron, x-ray, neutron) scattering techniques. PAS is an established tool for the characterization of free-volume in bulk polymers [4], [5]. Recently, PAS has gained industrial recognition [6]–[11] and is currently utilized by International SEMATECH as a porosity characterization tool for low- k dielectrics. The effects reported in this work were first noted in such porosity characterization studies. Despite the widely accepted nondestructive nature of PAS [2], [3], radiation effects were noted in previous studies of polymers [12], [13].

II. EXPERIMENTAL

A. Positronium as a Probe for Porous Dielectrics

Positron beams with tunable energy (0–70 keV) [2] have a depth-profiling capability and are used for studying thin (microns) films. In analogy with electrons, positrons injected in a solid lose their initial energy predominantly through ionization. For incident energies in excess of 1 keV, the positron and electron implantation profiles are virtually identical [14], [15]. Therefore, a material exposed to a positron and an electron beam with a given energy receives equal radiation doses.

A positron reaches thermal equilibrium with the material within 1–2% of its lifetime, the remainder of which is spent in a state of thermal diffusion. During this time, a positron probes the surrounding electronic environment until it eventually annihilates with an electron, giving off radiation (typically two 511-keV photons) that leaves the material without further interaction. These photons carry information about the electron momentum (measured through the Doppler shift or deviation from the 180° angle) and electron density (measured through the positron lifetime).

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In dielectric materials, thermalized and low-energy (~ 1 eV) positrons can form positronium (Ps), which is a hydrogen-like bound state with an electron. Like hydrogen, Ps forms in two states: *para*-Ps (*p*-Ps; $\uparrow\downarrow$ electron-positron spin configuration) and *ortho*-Ps (*o*-Ps; $\uparrow\uparrow$). Their annihilation characteristics are drastically different. The annihilation in the $\uparrow\downarrow$ state is fast ($\sim 8 \times 10^9$ s $^{-1}$) and occurs predominantly via two photons (2γ). The 2γ -channel is forbidden for parallel spins ($\uparrow\uparrow$)—because angular momentum cannot be conserved—leading to a dominant higher order 3γ -annihilation process, with a significantly smaller rate ($\sim 7 \times 10^6$ s $^{-1}$). The resulting *p*-Ps and *o*-Ps lifetimes in vacuum are 0.125 ns and 142 ns, respectively [16]. The *p*-Ps lifetime changes little in solids, whereas the *o*-Ps lifetime is shortened by the so-called “pick-off” (2γ) annihilation with molecular electrons of opposite spin to that of the positron. This pick-off lifetime, which spans two orders of magnitude (1–142 ns), and the *o*-Ps decay mechanism (2γ or 3γ) make the Ps a unique probe for porous materials [4]–[11]. However, the presence of unpaired electrons at the walls of a pore (e.g., from free radicals or broken bonds) can alter dramatically the annihilation signatures of *o*-Ps trapped in that pore [17]–[19]. Such a scenario enhances strongly the pick-off annihilation rate.

B. Samples and Experimental Details

Thin (~ 0.7 μm) mesoporous MSSQ films were fabricated by the sacrificial porogen technique [20]. Si wafers were spin-coated with a solution containing low-molecular-weight MSSQ resin and volatile polymer additive (pore generator, or porogen). The samples were treated thermally in steps first (at 200 °C) to vitrify the MSSQ without affecting the porogen, and then (at 450 °C) to decompose the porogen, which leaves behind voids embedded in the MSSQ network. The studied films, designated as A and B, contained approximately equal porosity fraction ($\sim 20\%$), but were produced using different resins and porogens. Both films had a density of ~ 1.05 g/cm 3 .

MSSQ is a resin with the empirical formula $T = \text{CH}_3\text{SiO}_{3/2}$. Several structural representations of silsesquioxanes are possible. The two most common are a ladder type chain and a cubic structure with eight silicon atoms placed at the vertices of the cube (denoted as T_8), to which the CH_3 radicals are attached. In a polymerized MSSQ, the structure is more complicated but contains elements of these two basic types incorporated in a random network.

A monoenergetic low-current (~ 30 fA; $\sim 2 \times 10^5$ e $^+$ /s) positron beam with a diameter of approximately 3.5 mm (a flux of $\sim 2 \times 10^6$ s $^{-1}$ cm $^{-2}$) was used to simultaneously irradiate the samples and to probe the porosity for up to 50 hours. Depth-profiles of the *o*-Ps annihilation signatures were taken by means of varying the beam energy; however, no depth-dependent effects were noticed. For the measurements reported here, the beam energy was set to 2 keV in order to maximize the *o*-Ps signal from the MSSQ films. The *o*-Ps lifetime and intensity and its 3γ -annihilation signal were monitored as a function of the exposure time.

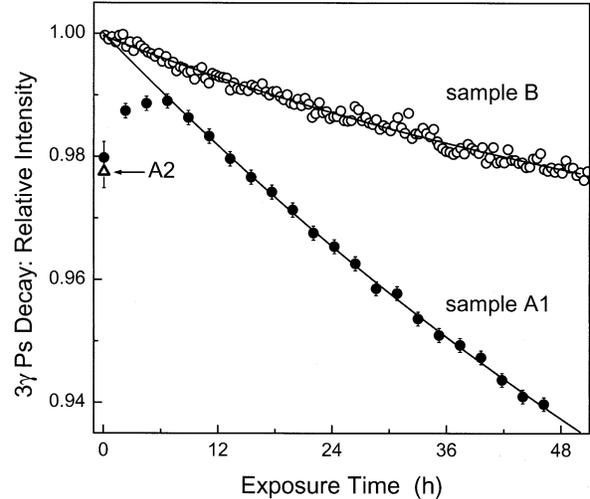


Fig. 1. The relative intensity of the three-photon *o*-Ps decay signal in mesoporous MSSQ films A and B (open circles). A1 (solid circles) and A2 (open triangle) are identical pieces of sample A; A2 was measured at the end of the irradiation exposure of A1 to assess possible vacuum-related effects.

III. RESULTS

Fig. 1 shows the 3γ -signal in the two films (A and B) as a function of the irradiation time. After several hours of exposure, the 3γ -signal decreases in both films. The dependence can be approximated by an exponential decay with different decay constants. The intensities of the 3γ -signals are normalized to the respective values of these fits at $t = 0$. In order to distinguish the radiation- and vacuum-induced changes, sample A was split into two identical pieces (A1 and A2). Both specimens were placed simultaneously in vacuum, but only one (A1) was irradiated. The results from the brief measurements of sample A1 at the beginning of the irradiation and sample A2 at the end of the A1 exposure time agree within the statistical error, which rules out any detectable vacuum-related effects. (Note that these measurements had to be fast because the measurement is based on the positron beam, which is also used for the irradiation.) The initial signal increase, which occurs in sample A but not in sample B is not well understood at this time.

The spot, illuminated by the positron beam for 50 hours, can be seen in a lateral scan along the sample surface, intersecting the initial beam position. The scan of sample B (Fig. 2) exhibits a decrease to ~ 0.98 in the relative 3γ -intensity, as seen in Fig. 1. The full-width at half-maximum (FWHM) of 4.8 ± 0.2 mm was derived from a Gaussian fit (line). The FWHM is consistent with the beam cross section of ~ 3.5 mm. Note that the fitted value in Fig. 2 results from the convolution of the size of the irradiated spot (equal to the beam size), and the size of the lateral scan probe (the beam). Therefore, the calculated FWHM value is $3.5 \text{ mm} \times \sqrt{2} = 4.9 \text{ mm}$.

Fig. 3 shows the results from the measurements of the long *o*-Ps lifetime in film B, using a detection scheme [7], which is insensitive to the short lifetimes originating from the annihilation of positrons, *p*-Ps and pick-off *o*-Ps. The equivalent pore size [8] is given on the right scale. The constant lifetime values (bottom plot) during the irradiation period show that the mean size of the pores in the film is unaffected by the irradiation. The

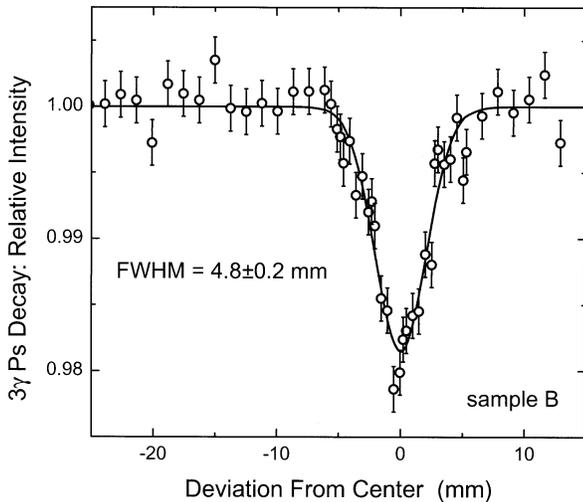


Fig. 2. A lateral scan of the relative intensity of the three-photon o -Ps decay signal, intersecting the irradiated spot on sample B. The full-width at half-maximum of the feature is consistent with the beam size.

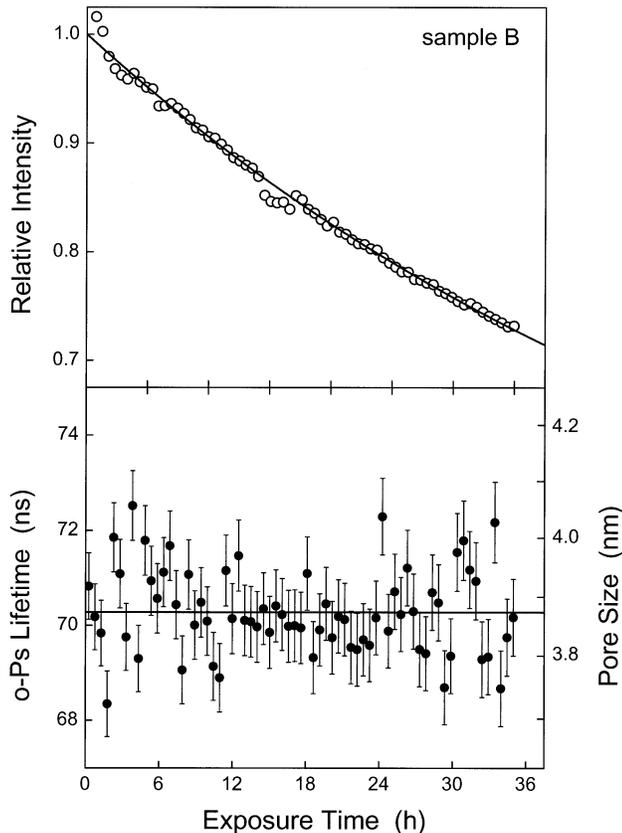


Fig. 3. (Top) The relative intensity of the long o -Ps lifetime in sample B, measuring the density of pores, whose electronic environment remains unaffected by the positron irradiation. (Bottom) The o -Ps lifetime as a function of the irradiation time remains unchanged, ruling out changes in the mean pore size.

intensity of the long lifetime decays as a consequence of the accumulated radiation dose. This indicates that with the increase of the exposure time, an increasing fraction of o -Ps in the pores undergoes pick-off annihilation (shorter lifetime) at the expense of the characteristic ~ 70 -ns lifetime. The intensity values are normalized to the $t = 0$ value from the exponential fit.

IV. DISCUSSION

PAS has been used in the past to study defects induced by γ , x-ray, neutron, electron and ion radiation (e.g., see [21]–[23]). However, effects due to the positron irradiation were not noticed until recently [12], [13], and the technique is widely regarded as nondestructive. Typically, PAS uses 10–30- μ Ci radioactive β^+ sources in bulk studies. Positron beams achieve 1–5- μ Ci equivalent intensities in the samples. Thus, for the majority of materials, the radiation dose accumulated in a measurement is insufficient to cause any measurable effects. Only a few exceptions were noted in several-hours-long positron lifetime experiments. All of them were carried out on polymer samples. Positron-induced radiation damage to any other type of material has not been reported to date.

The exceptional defect sensitivity of PAS can be utilized for the early detection of radiation damage. Here, a positron beam is used to simultaneously irradiate the sample and to detect the radiation damage. The purpose of this work is to demonstrate the effects of radiation, rather than to conduct a detailed investigation of its nature. The latter would require more information about the material.

A. Positron and Positronium Kinetics

The accumulation of radiation damage is a kinetic process, governed by the dose rate, the bond dissociation and recombination rates, and the sample temperature. In this context, a discussion of the kinetics of the positron irradiation is helpful. Chemical bonds are broken by ionization during the positron thermalization process. Thermal equilibrium with the sample is achieved in 2–5 ps (1–2% of the positron lifetime). Positrons spend the remainder of their lifetime either diffusing or becoming localized in the MSSQ (the repulsive dipole potential at a pore surface confines the positrons to the solid), or exist as Ps. Ps can be formed in the solid or at the pore surface in a region limited by the positron diffusion length (20–30 nm in these MSSQ films). Subsequently, Ps may remain in the solid, or they may diffuse and become localized in a pore. (The Ps diffusion length in organic and inorganic polymers is ~ 1 nm [24].) Only Ps trapped in pores contribute to the results presented in this work; all other scenarios need not be considered. Thus, the small relevant diffusion lengths hint to a high probability for a Ps atom to detect a broken bond created during the thermalization of its own positron.

This scenario, however, is not sufficient to explain the monotonically decreasing intensities (Figs. 1 and 3) without the existence of an energy barrier for bond reconstruction. The low positron current density, combined with the short Ps lifetime, set a negligible probability for the coexistence of two Ps atoms at the same time. Hence, the cumulative nature of the observed effects implies that Ps detects damage caused by a prior positron injection. Therefore, broken bonds cannot recombine at room temperature for times compatible with the duration of the experiments. Future studies are needed to assess the annealing behavior at a higher temperature.

B. Radiation Exposure

In thin film studies, for which positron beams are used, the induced radiation dose can exceed significantly that of a bulk

source. For a reference, the positrons from the β^+ decay of ^{22}Na have a mean energy of approximately 200 keV and penetrate a polymer sample to a 200–300- μm depth. For a comparison, a beam with a 100 times smaller energy (2 keV) is implanted to a ~ 100 -nm mean depth. For an identical spot size (typically 3–5 mm in diameter in both cases) and similar intensity, this translates into an order of magnitude larger dose rate incurred by a positron beam exposure.

The maximum accumulated dose in these studies is many times smaller than the dose acquired in a single image obtained by a scanning electron microscope (SEM). For an order-of-magnitude estimate, consider a 100×100 - μm^2 area of the sample, irradiated by electrons with a ~ 10 -pA current for ~ 20 s. For 2-keV electrons (identical energy deposition to that of the 2-keV positrons), the dose rate is 10^6 times greater (10^3 times larger current; 10^3 times smaller volume), whereas the exposure time is 10^4 times shorter. For this example, the dose by an SEM is some 100 times larger than that of the maximum achieved dose in these experiments.

C. Damage Detection

The signature of *o*-Ps annihilating in the pores provided the most conclusive confirmation of the radiation damage. The separation of this signal from the annihilation of positrons, *p*-Ps, and pick-off *o*-Ps is straightforward for materials with nanometer-sized pores. The 3γ *o*-Ps self-annihilation measured at the beginning of the experiments (at a negligible radiation dose) was $\sim 46\%$ for sample A and $\sim 34\%$ for sample B, whereas the contribution from the MSSQ matrix was only 2–3%. The normalized intensity in Fig. 1 ignores these material-related differences. The separation of the 70-ns *o*-Ps lifetime from the other lifetimes is also trivial [7]. Thus, the shown results are representative of the electronic environment at the pore walls, which is probed by the trapped in the pores *o*-Ps. The radiation damage, which occurs in the MSSQ matrix, is not evident from these data, but it is implied by virtue of the identical bond configuration in the bulk.

The evidence relating the positron beam with the radiation damage is contained in Figs. 1 and 2. The results from the measurements of A1 and A2 at $t = 0$ (Fig. 1) rule out any film changes, which could be associated with the transition from air to vacuum, or attributed to a prolonged vacuum exposure. Further confirmation is obtained from Fig. 2, where the irradiated spot is cross sectioned, and its FWHM is consistent with the beam size.

Structural changes in the films are ruled out by the results in Fig. 3. The long *o*-Ps lifetime (bottom) remains constant throughout the duration of the experiment. This excludes the possibility for changes in film thickness due to a gradual decrease in the pore size. Further, optical observations rule out a $\sim 30\%$ change in film thickness, which would be implied by the decreased intensity [Fig. 3(a)], if interpreted as a sign of collapsing pores. Therefore, we conclude that the mechanical structure of the films and the morphology of the porosity remain unchanged.

We attribute the radiation damage to broken bonds near the pore walls. This changes the electronic environment, leading

to enhanced pick-off annihilation. The mechanism of this enhanced pick-off process can be Ps interaction with paramagnetic centers [17], or a chemical interaction [18]. In the former case, *o*-Ps undergoes a spin-flip due to the mixing of the $|0, 0\rangle$ *p*-Ps and $|1, 0\rangle$ *o*-Ps states, and subsequently, it annihilates with the characteristic fast rate of the $\uparrow\downarrow$ spin configuration. In the latter case, Ps (like hydrogen) bind to dangling bonds or free radicals, and it is unable to probe the open volume of the pore. The increased electron density at the wall enhances the probability for pick-off annihilation.

D. Relevance to Space Environments

The use of low-*k* COTS microcircuits in space applications will require assessment of their performance under electron, proton, and heavy ion radiation. The present results relate to electron radiation by virtue of the similarity of electron and positron ionization for energies greater than 1 keV. High-energy electrons, which slow down to \sim keV energies in the dielectric of a low-*k* COTS device, will cause similar damage as \sim keV electrons injected in a bare MSSQ film. Qualitatively similar damage can be created by other ionizing radiation.

The emphasis of this work was not on the positron-induced radiation damage, but rather on the detection method for broken bonds. Quantitative investigations of radiation effects in low-*k* materials require the use of radiation standards, such as x-rays and γ sources, and high-energy particle beams as in device tests. For that, PAS can be utilized in the detection of radiation damage in a material at low doses. The benefit of using Ps as a probe is in its sensitivity to the local electronic environment at the pore surface, and its strong interaction with unsaturated bonds. Using PAS in the prescribed way will enable the search for correlation between the materials properties and device performance. In a plausible scenario, the dielectric properties of the media can be altered due to the change in the polarization as a consequence of creating broken bonds or free radicals. This can affect the signal propagation delay, which, in turn, can alter the timing response of a device.

The accumulation of broken bonds in a low-*k* polymer ILD raises new concerns for using such microcircuits in space radiation environments. Electron beams have been used to polymerize MSSQ as an alternative to a thermal cure [25], [26]. Bonds are dissociated by energetic electrons and recombine to form cross-linked network in a process called bond rearrangement. However, other processes, which result in more oxide-like network or create byproducts, may occur simultaneously. Even polymerization by bond rearrangement must be balanced. On the one hand, a high degree of cross-linking is required for the dielectric to achieve sufficient hardness, needed to comply with the mechanical requirements of a package. On the other hand, excessive polymerization can render the dielectric brittle, thus, making it susceptible to fracture under residual stresses. In an earth-like environment, the radiation dose to a low-*k* material, accumulated during electron-beam cross-linking or other fabrication steps, will practically cease to increase in a finished product. In radiation environments, however, the degree of polymerization, and thereby the ILD's hardness, can increase continuously.

The emergence of state-of-the-art low- k devices, such as ASICs, SRAMs, and so on, can be used to enhance the capabilities of space missions. However, the lack of relevant information at the present hampers the evaluation of their performances.

V. CONCLUSION

We demonstrated a radiation-induced degradation of low- k methyl-silsesquioxane—one of the promising candidates to replace silica as interlayer dielectric in advanced integrated microelectronic circuits. The samples were irradiated by 2-keV positrons, which create similar damage to that of 2-keV electrons. The high sensitivity of a positronium atom (e^-e^+) to unpaired electrons (e.g., broken bonds or radicals) was used to detect the damage at low doses. This establishes the feasibility of using positron annihilation spectroscopy as a characterization tool for the assessment of material damage induced by photon (x-ray, γ), electron, proton, or ion radiation.

Some issues, concerning the utilization of low- k electronic components in satellites and spacecrafts, are discussed. These polymers can be susceptible to changes in their electrical and mechanical properties, which can lead to new failure modes. It must be stressed, however, that little information exists about the performance of this new class of materials beyond the specifications for commercial grade electronics. Future investigations are warranted.

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