Photocurrent spectroscopy of low-*k* dielectric materials: Barrier heights and trap densities

J. M. Atkin,[1] D. Song,[1] T. M. Shaw,[2] E. Cartier,[2] R. B. Laibowitz,[3] and T. F. Heinz[1,3,a)]
[1]*Department of Physics, Columbia University, New York, New York 10027, USA*
[2]*IBM T. J. Watson Research Center, Yorktown Heights, New York 10598, USA*
[3]*Department of Electrical Engineering, Columbia University, New York, New York 10027, USA*

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Measurements of photoinduced current have been performed on thin films of porous low-*k* dielectric materials comprised of carbon-doped oxides. The dielectric films were deposited on silicon surfaces and prepared with a thin gold counterelectrode. From the spectral dependence of the photoinduced current, barrier heights for the dielectric/silicon and dielectric/gold interface were deduced. Transient currents were also found to flow after the photoexcitation was abruptly stopped. An estimate of the density of shallow electron traps within the low-*k* material was obtained from the measurement of the net charge transported from this detrapping current. A density of traps in the range of 6×10^{16} traps/cm^3 was inferred for the low-*k* films, far exceeding that observed by the same technique for reference dielectric films of pure SiO_2. This behavior was also compatible with photocurrent *I-V* measurements on the low-*k* dielectric films and SiO_2 reference sample. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2907958]

I. INTRODUCTION

There is an important need within the semiconductor industry to understand electrical conduction mechanisms in low-*k* dielectric (LKD) films, particularly those associated with leakage and time-dependent dielectric breakdown. These properties are closely related to the overall reliability of low-*k* materials, a central problem for many industrial applications of these materials.[1,2] The key features determining the electronic transport properties in such insulating films are the position of the energy bands of the LKD, their disposition relative to the states in the electrodes, and the influence of traps both within the LKD film and at its interfaces.

Optical techniques, applied in conjunction with electrical measurements, are ideal for characterizing both interfacial barriers and trapping states in such films. Photocurrent spectroscopy, also termed internal photoemission spectroscopy or scanning internal photoemission, is an established approach for examining the band structure of insulators placed between two (metal or semiconductor) electrodes. The technique involves photoexcitation of charge carriers over the interfacial barrier into the conducting states of the insulator, where they can move to the collection electrode under the influence of an external electric field. The spectral dependence of the observed photocurrent yields the height of the interfacial barrier. This technique has been extensively used for high-purity SiO_2 films,[3–5] as well as other insulating and semiconducting films.[6] Photocurrent studies have also recently been reported on LKD systems by Shamuilia *et al.*[7] These authors obtained interfacial barriers for both carbon-doped oxide and polymer systems.

In the present investigation, we apply photocurrent measurements to examine several important properties of LKD films. The materials under study, which have a relative dielectric constant of $k=2.4$, are porous, amorphous films comprised of the elements Si, C, O, and H. This class of material has been designated in the literature by various names, including carbon-doped oxide, organosilicate glass, and SiCOH. For this material system, we obtain spectra of the photocurrent as a function of the energy of the exciting photons. Analysis of these data permits us to deduce barrier heights of the LKD films for interfaces with both silicon and gold electrodes.

In addition to the ability of photoexcitation experiments to examine barrier heights, the method can also be applied to probe properties associated with trapping and defect states in the dielectric films. These are of considerable importance for LKDs. There is evidence that trap states contribute to leakage current and thereby degrade both the performance and reliability of the insulating films.[8] In the early work of Williams,[5] the density of traps within SiO_2 films was examined by measurement of the current flow that could be induced after termination of photoexcitation with UV radiation by application of visible light. We have applied a related measurement technique in which we observe the spontaneous current flow after abruptly interrupting photoexcitation to determine the net amount of thermally detrapped charge. This measurement allows us to determine the density of shallow trap states in the film from which these charges originated. The LKD materials were found to have a trap density of 6×10^{16} cm^{-3}, far higher than observed in conventional SiO_2 dielectric films. This information is significant in analyzing the primary charge transport and breakdown mechanisms for LKD systems. As a complementary method of examining the role of trapped charge, we also present measurements on the current-voltage (*I-V*) characteristics of the photoinduced current. The voltage dependence of the current in the low-*k* films and its relation to that seen in pure SiO_2 can be correlated with trapped charges in the dielectric.

a)Electronic mail: tony.heinz@columbia.edu.

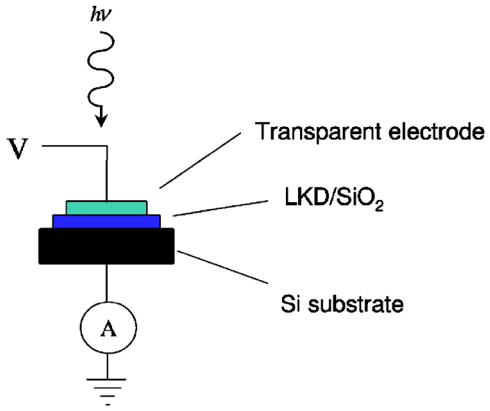

FIG. 1. (Color online) Schematic of layer structure samples, consisting of a silicon substrate, the LKD or SiO_2 film, and a transparent gold electrode. The light from the mercury lamp, either directly of after being spectrally filtered in a monochromator, is focused on the sample at normal incidence. A bias voltage V is applied between the gold electrode and the doped silicon substrate, as shown, and the leakage or photoinduced current is recorded.

II. SAMPLES AND EXPERIMENTAL TECHNIQUE

The low-k carbon-doped oxide films investigated in this study were deposited by industrial sources as blanket films (see ACKNOWLEDGMENTS). The films were porous, amorphous materials prepared by a plasma-enhanced chemical vapor deposition process described in detail by Gates *et al.*[9] The precursors have been previously reported by Grill *et al.*[10] The dielectric constant of the film was reduced by including an organic precursor in the deposition process, which was then removed by UV curing for 150 s, producing pores on the order of a few nanometers in diameter. This procedure can provide materials with dielectric constants as low as 2.0. The chemical composition of the final films has been previously studied using a variety of techniques including FTIR and x-ray diffraction,[11–13] and is dominated by the elements Si, C, O, and H.

In the present study, we report results for 130 nm thick LKD films with a dielectric constant of $k=2.4$. LKD film thickness was generally determined by establishing a step in the LKD film and measuring the step height with a profilometer. The steps could be established either by etching or scratching. The LKD films were deposited on p-type silicon wafers with resistivities of $1-10\ \Omega\,\mathrm{cm}$. The surface of the silicon wafers on which the films are deposited is covered by a native oxide of less than 1 nm thickness. The complete test structures for our measurements, shown in Fig. 1, were formed by the thermal deposition of a thin, transparent metallic electrode on top of the LKD film. For the investigations described in this paper, gold was chosen for the counterelectrode, with current flow occurring through the LKD film located between the Si substrate and the Au electrode. Gold is attractive because of its ability to form a clean interface, with little interdiffusion, with the LKD films. The gold electrodes covered an area of $0.05\ \mathrm{cm}^2$ and had a thickness of 10 nm. This thickness was chosen to ensure good electrical conductivity, while still absorbing less than 50% of the optical radiation (which must pass through the gold film to reach the LKD in the photoexcitation measurements.) To calibrate the spectral dependence of the photocurrent, we measured and accounted for the absorption of the incident light by the Au electrode. For the purposes of comparison, all measurements were also performed on samples of high-purity SiO_2 films that were thermally grown on similar p-type silicon substrates to a thickness of 860 nm.

The light sources for the photocurrent studies were 200 W mercury bulbs (Oriel N6291 and N6283). This source provided a continuous spectrum of light at wavelengths down to 200 nm. The radiation consisted of a broad continuum, with some discrete line emission features. The light was focused to a spot size of approximately $1\ \mathrm{mm}^2$ on the sample using a quartz lens. For the spectroscopic measurements, this radiation was spectrally narrowed to a range of wavelengths $\Delta\lambda \approx 10$ nm at an adjustable central wavelength λ by passing it though a UV-visible monochromator (ISA H20). In order to normalize our photocurrent signal to the intensity of the exciting radiation, we measured the variation in strength of this monochromatized radiation with a GaP photodiode (Thorlabs SM05PD7A). The current through the sample structure was measured with a picoammeter (HP 4140) at an adjustable bias supplied by the same instrument. In the dark, the leakage current through the samples at fields of $8\times10^5\ \mathrm{V/cm}$ was on the order of 1 pA and remained essentially constant throughout the measurements. When the light was switched on, transients in the current lasting approximately 1 s were observed before a steady-state value was reached. Under broadband illumination, the photocurrent was typically $\sim$100 times greater than the background leakage current. For the spectroscopic measurements with the monochromatized radiation, the photocurrents were considerably smaller and long integration times were used to obtain reliable data. Careful measurements of the dark current were also performed. This contribution was then subtracted out to obtain the photoinduced currents reported below. All measurements were performed with the samples held at a temperature of approximately 120 °C. This temperature was chosen to minimize effects of water uptake in the LKD, a process known to alter leakage currents.[2,14]

III. RESULTS AND DISCUSSION

Below we describe three related measurements that probe the behavior of the LKD sample through its photoinduced current. Our studies examine the photocurrent (a) as a function of excitation photon energy, (b) as a function of the bias voltage, (c) and in a transient regime after the photoexcitation is suddenly stopped. For each of these measurements, we present data for the LKD films and a comparison with a conventional high-purity SiO_2 dielectric film.

A. Photocurrent spectroscopy

Photocurrent spectra were obtained at an applied bias corresponding to electric field strength of approximately $8\times10^5\ \mathrm{V/cm}$ in both the LKD and SiO_2 samples. The photocurrent was measured for a range of excitation wavelengths between 200 and 500 nm. The observed photocurrents were normalized to account both for the variation in the incident light intensity and for the absorption within the Au electrode, as discussed in the previous section.

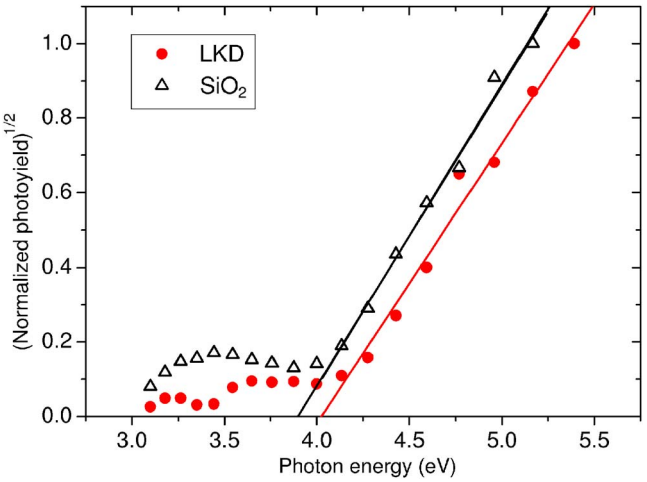

FIG. 2. (Color online) Photocurrent spectra for the response of the LKD (130 nm) and SiO_2 (860 nm) films for an applied bias of -8×10^5 V/cm (gold electrode at negative potential). The photocurrent data are normalized to correct both for the spectral variation of the light intensity and for the absorption of the gold electrode. The plot displays the square root of the normalized photocurrent $I^{1/2}$ as a function of the exciting photon energy $h\nu$. Fitting the data to Eq. (1), we deduce barrier heights of 3.9 ± 0.4 eV for SiO_2 and 4.0 ± 0.5 eV for the LKD, where the errors result from repeated measurements of different samples.

Significant photocurrents were observed for excitation of wavelengths less than approximately 300 nm. For the SiO_2 film, this photocurrent has been attributed to the injection of electrons over the interface barrier and their transport through the film,[3,15,16] Electrons originate either in the valence band of silicon or the conduction band of the counterelectrode, depending on the polarity of the applied electric field. Photocurrent could, of course, also arise from the injection of holes into and transport through the valence band of the insulator. For the case of SiO_2, this process is considered negligible in comparison to the electron transport, as the electron mobility in the insulator is known to be far higher than the hole mobility[4] and, more directly, because changing the material of the positive electrode does not affect the threshold for internal photoemission.[5] We assume that the hole transport can also be neglected for photoinduced processes in our LKD films and adopt a description based on electron injection and transport.

The results for the photocurrent spectra are shown in Fig. 2. Here, we display the square root of the normalized photocurrent as a function of the excitation photon energy. This scheme of presentation is chosen in accordance with a model[15,17] that predicts a spectral dependence of the photocurrent of

$$I \propto (h\nu - \phi_0)^2, \quad h\nu \geqslant \phi_0,$$

$$I = 0, \quad h\nu < \phi_0. \tag{1}$$

Here, $h\nu$ denotes the energy of the exciting photons, and ϕ_0 is the height of the barrier between the electrode Fermi level and the conduction band of the dielectric. Equation (1) is obtained by considering the electron momentum above the barrier, under the assumption that the density of states near the Fermi energy is independent of energy and that the emitter and collector of the photoexcited carriers have wide energy bands. The analysis further neglects the possible role of trap states in the dielectric, since all current is assumed to occur at energies above the barrier. In spite of these simplifications, this model has been shown to fit the experimental photocurrent spectra for a variety of materials quite well.[6]

According to the model, we expect to see a cutoff in the photocurrent once the photons have insufficient energy to excite electrons into the conduction band. Figure 2 shows that for photon energies 3.8 eV $< h\nu <$ 5.5 eV, the induced photocurrents for both the LKD and SiO_2 films obey the expected relation. (At somewhat lower photon energies, a small photoinduced current can still be observed. This response could be indicative of a trap-mediated current, which can be sustained with lower photon excitation energies than expected for current arising from excitation over the band edge.) For the principal photoinduced response in the linear regime of $I^{1/2}$ versus $h\nu$, analysis with Eq. (1) yields barrier heights of 4.0 ± 0.5 eV for the LKD film and 3.9 ± 0.4 eV for the SiO_2 reference film when the Si substrate was at positive potential with respect to the Au electrode. These barriers, therefore, correspond to electron injection over the Au/insulator barrier. Not shown are similar data taken with the opposite applied bias voltage. These measurements yield a barrier for electron injection from Si into the LKD of 4.1 ± 0.4 eV and a corresponding barrier for the Si/SiO_2 interface of 4.0 ± 0.3 eV.

Published results on the Au/SiO_2 barrier agree well with our value. Shamuilia *et al.*[7] report a value of 4.1 eV and no significant effect on the barrier under variation in the external electric field. Goodman[4] and Goodman and O'neill[18] obtain a value of 3.9 eV. This suggests that the barriers for both gold and silicon are very close, which is also seen in the LKD system. The value obtained here for Au/LKD is lower than that reported in Shamuilia *et al.* for a similar LKD with dielectric constant $k=3.0$,[7] but still within experimental uncertainty.

In their paper, Shamuilia *et al.* observed that the energy barriers at metal/LKD interfaces did not significantly vary when the composition of the metal electrode was changed. They noted a spread of only some 0.2 eV in barrier height for Al, Au, and TaN_x electrodes, which have significantly different work functions. This suggests that interfacial trapping states in the LKD play a significant role in electron dynamics, i.e., there may be some lower barrier process possible at the metal/LKD interface. Supporting this hypothesis, the authors presented results showing that the internal photoemission barrier was sensitive to the chemical composition of the LKD surface. We performed further measurements in order to investigate the influence of trapping states within LKDs.

B. Photoinduced *I-V* measurements

Current-voltage (*I-V*) measurements were performed under illumination of the samples by the full, unfiltered mercury lamp (all available wavelengths). A steady photocurrent was observed under these conditions that could be recorded as a function of the applied bias voltage V. These results are shown in Fig. 3 for both LKD and SiO_2 samples. We plot the

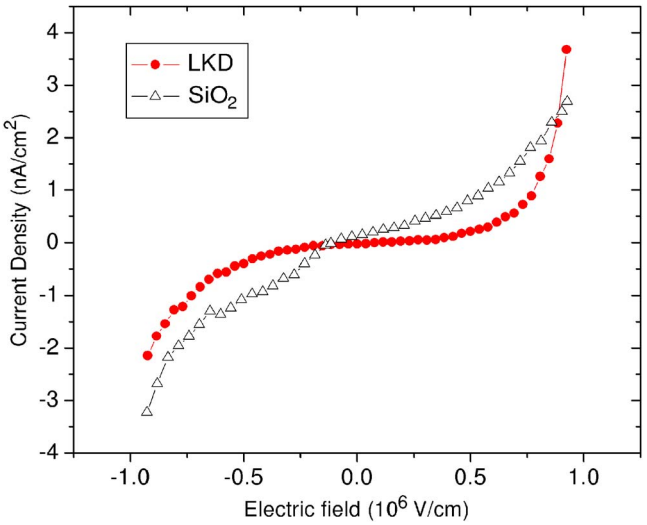

FIG. 3. (Color online) Photoinduced *I-V* curves for LKD and SiO_2 films, plotted as current density vs applied bias electric field. The bias is applied to the gold electrode, with a positive field corresponding to excitation of electrons over the Si/insulator interface and negative fields to excitation across the Au/insulator interface. (The solid lines are guides to the eye.)

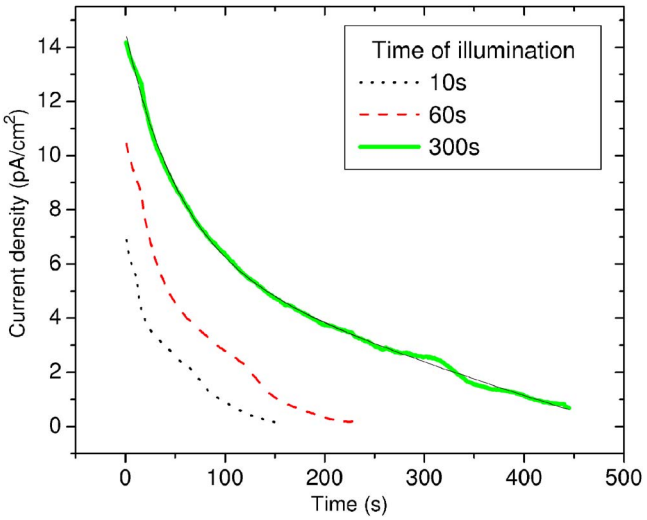

FIG. 4. (Color online) Temporal evolution of the current density after abrupt termination of illumination with broadband radiation for an SiO_2 film at an applied bias of -8×10^5 V/cm (silicon substrate at positive voltage). Results for three exposure times are shown. The solid black line is a fit for the 300 s data to a decay given as the sum of two exponential functions.

data as function of applied electric field (rather than applied voltage) to normalize for the different thicknesses of the films. For the LKD, the dark current is of the order of pA/cm^2, but for the SiO_2, it is almost a third of the total current. We observe relatively symmetric *I-V* curves for negative and positive bias fields. This result is compatible with the similar spectral response of the photocurrent for positive and negative potentials described in the previous section.

The shape of the photoinduced *I-V* response in SiO_2 has been previously discussed,[19,20] in particular by Williams.[5] The sharply reduced current at low bias voltages is attributed to the role of trapping sites that become charged and decrease the net electric field for charge transport. The more steeply rising portion of the *I-V* curves corresponds to an applied electric field great enough to overcome this trap-induced space charge. With this framework in mind, we can understand the qualitative difference between the photoinduced *I-V* characteristics in the SiO_2 and LKD films. The much smaller current in the LKD compared to the SiO_2 film for low and moderate bias fields is the result of greater charge trapping in the LKD film. The increased charge trapping induces greater screening of the applied field. An estimate of the trapped charge density can be obtained by taking the "turn on" voltage of the *I-V* curves to occur when all the traps have been filled and then calculating the charge that this corresponds to using $Q=CV$. The density of traps in SiO_2 found by this method is approximately 3×10^{15} traps/cm^3 for the SiO_2 film, while it is 9×10^{16} traps/cm^3 for the LKD. A similar conclusion is also reached by analysis of the photocurrent transients in Sec. C below.

C. Transient currents following photoexcitation: Detrapping dynamics

After abruptly terminating photoexcitation with the unfiltered light source, both the SiO_2 and LKD samples showed a slow relaxation back to the initial dark current values. This behavior is displayed for the SiO_2 and the LKD films in Figs. 4 and 5, respectively. The measurements were performed at an applied electric field of 8×10^5 V/cm, with the silicon substrate held at positive potential. We show the characteristic response for three different durations of the light exposure in the figures. The observed current transients are approximately exponential, with time constants on the order of 10 s and a magnitude that increases with light exposure up to a saturated response. We attribute the current transients to electrons that have become trapped while the photocurrent was flowing, but when the light exposure is stopped, are thermally ionized. These charges then move across the insulating layer under the applied bias field. Based on the photoinduced *I-V* response discussed in the previous section, we expect the LKD to display a higher density of electron traps than the SiO_2 film. The integral of the current transient gives the

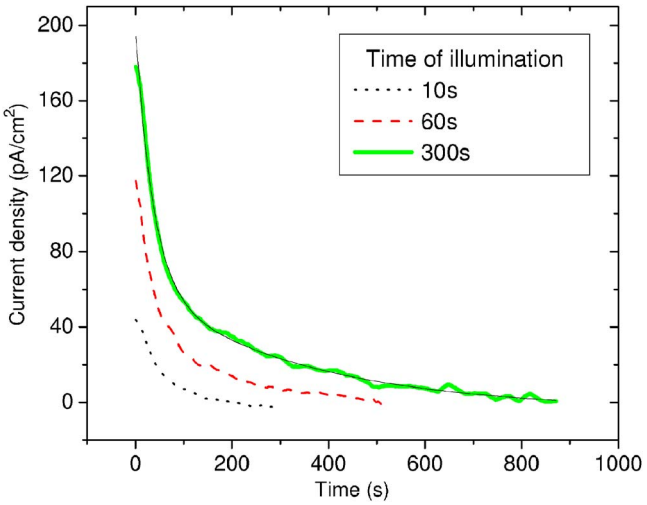

FIG. 5. (Color online) As in Fig. 4, but with a LKD film. Note the longer detrapping time and larger current compared to the behavior for the SiO_2 film.

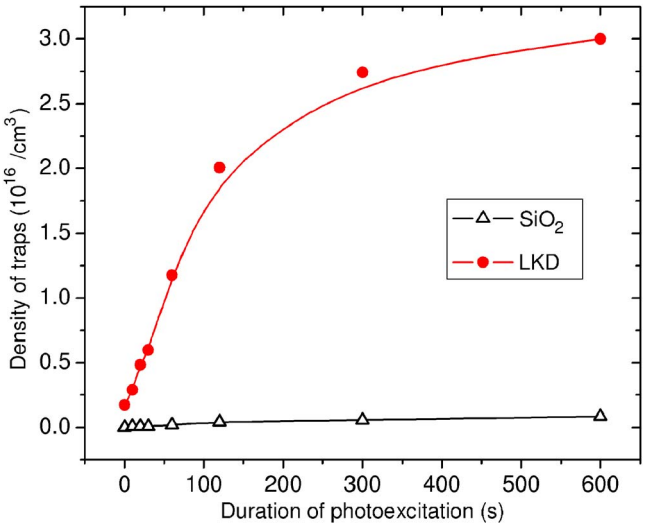

FIG. 6. (Color online) Inferred density of traps in the LKD and SiO_2 films based on the measured charge flow after stopping illumination, as in the data of Figs. 4 and 5. Both SiO_2 and LKD films display an increase in density of filled traps with increasing time of illumination up to a saturated level. (The solid lines are guides to the eye.)

charge transfer once the photocurrent is switched off. This charge/area corresponds to the sheet of charged trap states that are populated by the photocurrent and that then undergo spontaneous ionization.

The results of the calculation of charge transfer from detrapping are shown in Fig. 6 as a function of the duration of the light exposure time for both the SiO_2 and LKD films. The calculation of the concentration of traps assumes that each contains a single electron and that the sites are homogeneously distributed in the dielectric film.[21] The saturated value of the density of traps in the LKD is approximately 6×10^{16} traps/cm^3, which corresponds to a sheet density of traps of 8×10^{11} traps/cm^2. For the SiO_2 films, the saturated density of traps is approximately 1.6×10^{15} traps/cm^3, close to the value of 3×10^{14} traps/cm^3 of Williams, obtained using a related but somewhat distinct experimental approach.[5] This density corresponds to a sheet density of 1×10^{11} traps/cm^3. For the SiO_2 system, this value is actually comparable to the density of interface traps for high-quality SiO_2/Si.[22] The LKD material is seen to exhibit a much higher density of traps that the SiO_2 films. This difference can be attributed to the combined effect of the porosity of the films and the more complex chemical composition of the films. We note that the independent estimates for the density of charged traps obtained earlier from the photoinduced *I-V* characteristics yield values of comparable magnitude to the estimates from the detrapping dynamics: 9×10^{16} traps/cm^3 for the LKD material and 3×10^{15} traps/cm^3 for the SiO_2 film.

IV. CONCLUSIONS

Photocurrent studies have been used to investigate barriers and trapping dynamics within insulating films of porous LKD with a relative dielectric constant of $k=2.4$. Barrier heights for the Si/LKD/Au system were obtained, as were those for a reference Si/SiO_2/Au sample. The LKD film showed similar barrier height behavior to that obtained on carbon-doped glass with a higher dielectric constant, from an earlier publication.[7] The results for the SiO_2 films also agreed with previous measurements in the literature. From current transients, as well as the photoinduced *I-V* characteristics, we have estimated the trap density in the LKD films. It is found to be on the order of 10^{16} traps/cm^3, significantly greater than for SiO_2 films. In terms of charge transport and dielectric breakdown in the LKD materials, these trap states may contribute to a lower-barrier conduction mechanism for the dark leakage current.

Although these experiments provide valuable new information about the density of trap states in LKD films, several important questions still remain unanswered. In particular, it is important to establish the spatial distribution of the traps, i.e., whether they are homogeneously located throughout the dielectric film or predominantly at interfaces. This issue can be experimentally addressed by careful measurement of the photocurrent transients for well-controlled films of varying thicknesses. The role of these trap sites in influencing leakage currents and the time-dependent dielectric breakdown properties of these materials obviously is of central interest. This theme could be experimentally addressed by establishing the correlation between leakage and breakdown properties, on the one hand, and the density of trap states, on the other, for different chemical formulations of the dielectric, different degrees of porosity, and different materials processing protocols. An immediate possibility would be to investigate the influence of water uptake on the trap density, since this process is known to affect leakage currents and reliability in LKD materials.[14,23,24]

From a more fundamental perspective, one would ultimately hope to deduce energy-level structure, as well as the chemical and structural properties of the trap sites. While this endeavor will certainly require input from other experimental approaches and theory, important additional information might be extracted from an examination of the photocurrent response as a function of the sample temperature. The dependence of dynamics and magnitude of detrapping should reflect the depth of these trap states. Given the limited knowledge of these trap states and their important implications for the leakage current and time-dependent dielectric breakdown in LKD materials, we believe that further investigations are well justified.

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