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Low Temperature Cathodoluminescence in Disordered SiO_2

Amberly Evans, JR Dennison, Gregory Wilson, and Justin Dekany

***Abstract*—In recent charging studies, a discernible glow was detected emanating from sample surfaces undergoing electron beam bombardment that resulted from a luminescent effect termed cathodoluminescence. This suggests that some of the materials used as optical elements, structural components, and thermal control surfaces in the construction of space-based observatories might luminesce when exposed to sufficiently energetic charged particle fluxes from the space plasma environment. A central focus of our experiments was the temperature dependence of the luminescent behavior. Here, an overview of our experimental results is given, as well as a qualitative model to describe the luminescent behavior. We look at a simple model of band theory and the filling of localized or trapped states and the electron transitions from which the luminescence originates.**

***Index Terms*—luminescence, electron flux, space environment interactions, materials testing, low temperature**

I. Introduction

During charging studies using a low-flux intermediate-energy electron beam, thin film highly disordered silicon dioxide (SiO_2) samples exhibited a cathodoluminescent behavior. Similar behavior has been observed in other experiments at Utah State University (USU) for other materials including carbon composites and polymers. Previous studies have observed similar cathodoluminescence in both epoxy resin composites [1-3] and in disordered SiO_2 [4,5] however little information is available with regard to the temperature dependence of the cathodoluminescence. These results suggest that materials used in structural components, optical elements, and thermal control surfaces of spacecraft and space-based observatories could, when exposed to the space plasma environment, luminesce. If these visible, infrared and ultraviolet emissions are intense enough, they could potentially produce optical contamination detrimental to the performance of the observatory optical elements and sensors and act to limit their sensitivity and performance windows. As future observatory missions push the envelope into more extreme environments and use more complex and sensitive detectors, a fundamental understanding of the dependencies of luminescent intensity on time, temperature, incident electron flux and energy, and material structure becomes critical.

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II. Experimentation

Experiments were conducted in the main USU electron emission ultrahigh vacuum test chamber [6], modified for observations of low intensity UV/VIS/NIR glow over a broad range of sample temperatures [7]. Figure 1 provides a general schematic of the experimental system used. Experiments measured the electrical and optical response of sample materials bombarded with a beam of electrons.

A low energy electron gun [Staib, EK-5-51] delivered a well-characterized, low-flux beam (typically, ~50 pA/cm^2 to 1 μA/cm^2) over an energy range of 20 eV to 5 keV. For results shown in Figs. 2 and 3, beams of 5 keV at ~500 nA/cm^2 were used. It was necessary to use this relatively large current density, as compared with typical space environment fluxes, to produce sufficient emission to acquire spectra. A 5 keV at ~500 nA/cm^2 penetrating electron beam delivers ~$4\cdot10^4$ Gy/s dose rate to thin optical coatings. A dose of ~$6\cdot10^8$ Gy delivered in 10 hr—well in excess of the total cumulative time any sample was exposed in our tests—is still well below threshold damage values observed for cover glass materials (~$1\cdot10^{10}$ Gy) and for Corning 7940 fused silica (>$1\cdot10^{12}$ Gy) [8]. Therefore, the results of our studies are not likely to be attributable to radiation damage. The defocused electron

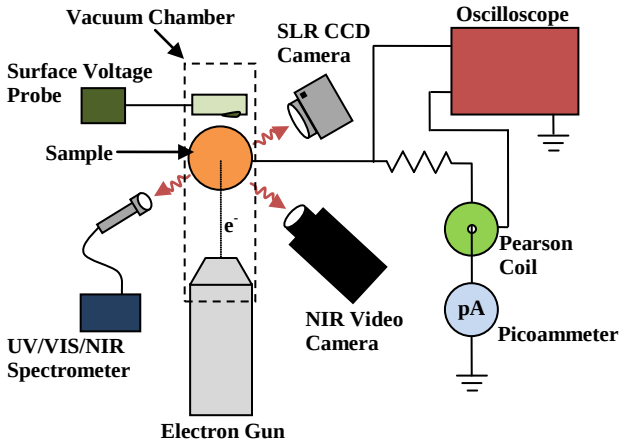

Fig. 1. Block diagram of instrumentation for collecting the pulse charging surface voltage, electrode current and cathodoluminescence data induced by electron beam bombardment. Instrumentation includes picoammeters, Pearson coils, and a storage oscilloscope for electrode current measurements and UV/VIS and IR spectrometers, an SLR CCD still camera, and a NIR video camera for optical measurements.

beam produced a beam profile at the sample with about ±30% uniformity over an ~3 cm diameter beam spot. Beam fluxes were stable to within ±5% and were measured before and after each experiment with a Faraday cup. The W filament of the electron gun also produced visible light that traveled down the bore of the electron gun and impinged on the sample; even at the low operating filament temperatures used for the low flux beam (~2500 K), the intensity of the filament spot on the sample was larger than the observed luminescent signals. To minimize this contaminating filament light, a blocking aperture was developed, which allowed an electron beam focused ~5º off axis and then electrostatically deflected back on to the sample while sending the filament light into an optical baffle. The baffle and the primarily specular nature of the reflected filament light that made it past the blocking aperture reduced the stray light contamination seen by the optical cameras to acceptable levels.

Two cameras and two fiber optic spectrometers were used to monitor low light visible and NIR intensity. Optical data were collected using UV/VIS (Stellarnet, 13LK-C-SR; ~200 nm to 1080 nm with ~ 1 nm resolution) and NIR (Stellarnet, RW-InGaAs-512; ~1000 nm to 1700 nm with ~ 3 nm resolution) spectrometers, an SLR CCD still camera (Cannon, EOS Rebel XT DS126071; ~400 nm to 700 nm, 30 s per frame), and a VIS/NIR image-intensified CCD video camera (Xybion, ISG-780-U-3; ~400 nm to 900 nm, 30 frames per s). Additional InSb (~1000 nm to 5500 nm) and InGaAs (~800 nm to 1700 nm) video cameras and filter combinations were used to monitor IR emissions; no IR in the 900 nm to 2500 nm range was observed for any of the experiments reported here. Prior to the luminescence studies, the spectral response and range were determined and the sensitivity of the instruments was calibrated with NIST traceable sources [7].

The sample carousel was thermally anchored to (but electrically isolated from) a thermal reservoir. In combination with resistive heaters and liquid N_2 cryogen, the samples were maintained over a range of temperatures from ~150 K to ~400 K with a stability of ±4 K maintained over typical 2 hr experiment durations. Temperatures were measured with platinum resistance thermometers with an accuracy of <0.5 K.

Samples (2.5 cm diameter) of optically smooth, thin film (~65 nm thick) disordered SiO_2 (fused silica) deposited on ~175 nm thick highly reflective, optically smooth metal (mostly Au) layers on a 2 mm thick fused quartz substrate. The samples were optically cleaned and underwent a ~12 hr vacuum bakeout at ~390 K and $<1\cdot10^{-3}$ Pa to eliminate adsorbed water and volatile contaminates. The samples were mounted on Cu pedestals on a multi-sample carousel, and were placed in an ultrahigh vacuum chamber (base pressure $<1\cdot10^{-6}$ Pa) for > 24 hrs to allow for outgassing before measurements were made. Though not the focus of this paper, each sample was wired appropriately for collecting electrical data during bombardment as well; these results are reported elsewhere [9].

III. RESULTS

The thin film SiO_2 samples exhibited readily observable luminescence, as shown in Fig. 2. The intensity scaled with electron beam intensity and responded to changes in beam position and profile on rapid time scales, as demonstrated by the image of a defocused and distorted electron beam incident on the sample shown in Fig. 2(d). A clear variation in the intensity and color as a function of temperature was seen with the SLR images Figs 2 (a-c); as temperature increased, the light emission from the samples became bluer and less red. This was confirmed with relative intensity measurements of the Red-Green-Blue bands of the CCD camera as a function of temperature, as in Fig. 3(a). Overall intensity of the glow in the visible range—as measured with the CCD video camera—increased from room temperature to the lowest measured temperatures at 163 K. These basic results were observed repeatedly over the course of our studies, suggesting that radiation damage was not a major cause of the changes.

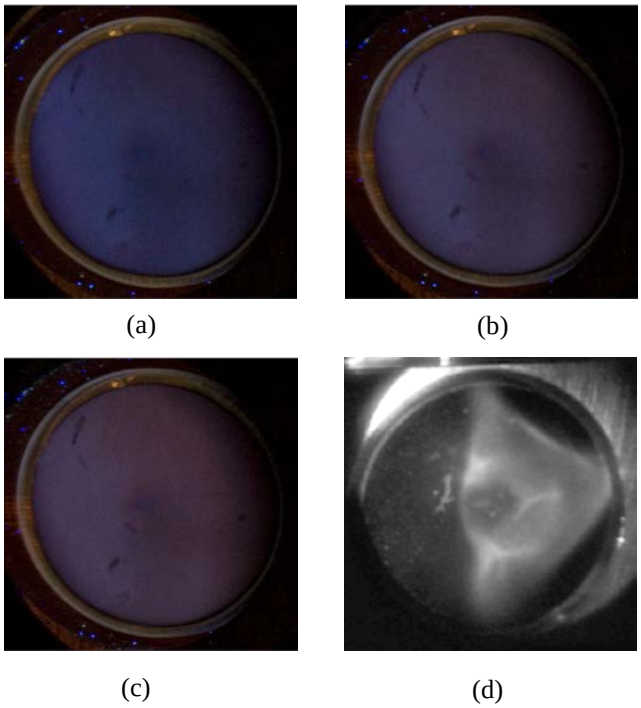

Fig. 2. Optical images of luminescent SiO_2 samples. SLR still images of the glowing thin film SiO_2 sample at (a) 273 K, (b) 193 K and (c) 163 K. Note that the lower temperature image has a very red tint to it. However, as the temperature continues to rise, and eventually reaches room temperature, the color begins to shift to blue. (d) CCD video camera frame showing the luminosity produced by a highly distorted, defocused electron beam.

The spectral dependence of the luminescence at room temperature is shown in Fig. 3(b). Three peaks were observed, centered at approximately 275 nm, 500 nm and 645 nm; an additional shoulder is observed at ~455 nm results. These peaks, which result from electron transitions from shallow trap states near the conduction band edge to deep trap states, were consistent with the results of Sahl and Trukhin for similar disordered SiO_2 samples at room temperature (see Fig. 5). Sahl identified the peak at 645 nm as a nonbridging oxygen hole center in the structure [4]. The peaks at 275 nm and 500 nm are results of an oxygen deficient center, as identified by Trukhin [5].

The variation in spectra intensity with temperature is shown for three representative temperatures in Fig. 3(b and c). The observed decrease in the red peak near 645 nm and the increase in the blue and UV peak near 455 nm and 275 nm,

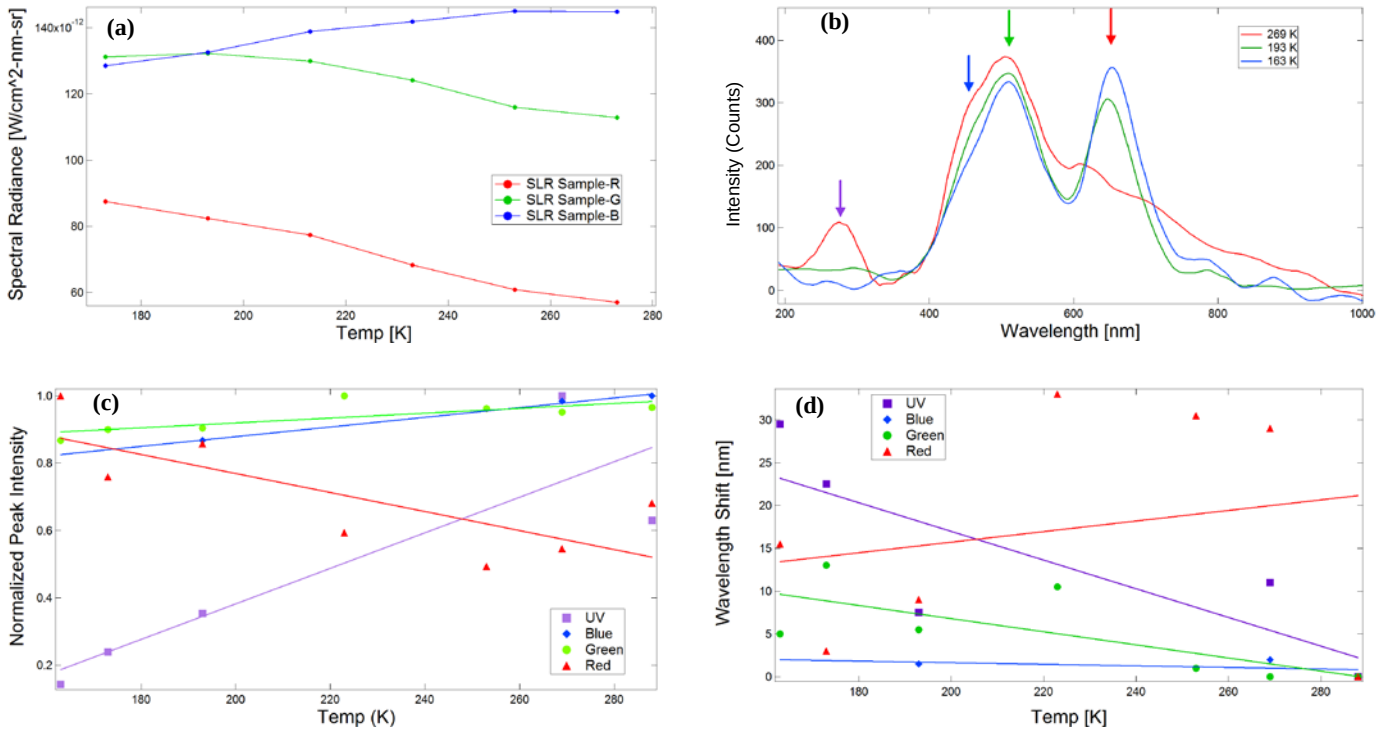

Fig. 3. **(a)** Variation with sample temperature of the Red-Green-Blue components of the SLR still CCD camera image average intensities across the glowing thin film disordered SiO_2 sample. **(b)** Three glow spectra at decreasing sample temperature, measured with the UV/VIS spectrometer. Four peaks are identified in the spectra. **(c)** Normalized peak amplitudes as a function of sample temperature of the red (~645 nm), green (~500 nm), blue (~455 nm) and UV (~275 nm) peaks. These are calculated as the amplitude of each peak (with baseline subtraction) normalized by the largest amplitude for that peak. **(d)** Shift of the peak wavelength as a function of sample temperature of the red, green, blue and UV peaks. Linear fits in (c) and (d) provide guides to the eye of the general trends.

respectively, are consistent with the qualitative SLR observations seen in Fig. 2. In Fig. 3(d), a downward shift in wavelength with increasing temperature is observed.

IV. Band Theory of Highly Disordered Insulators

The basic theory behind the observed luminescence phenomenon observed here is the band theory of highly disordered solids. In ordered band theory, the energy levels of individual atoms coalesce, forming a continuum of energy levels as the atoms are brought closer together thus creating an ordered solid structure. The number of levels created in a given band is the number of energy levels originally available, typically one per valence electron in the solid. Thus, as the number of atoms increases, so do the number of new energy levels, eventually creating a continuum of energy levels. These continua, called energy bands, are filled starting at the band of lowest energy (see Fig. 4a).

The extent to which these bands are filled is how solids are classified as conductors, semiconductors and insulators. Conductors have a partially filled energy band; electrons that occupy this band have high mobility, allowing electrons to move freely leading to high electrically conductive. Insulators have a fully filled valence band and a large band gap; even for large thermal energies, valence band electrons have an extremely low probability of being thermally excited into the conduction band. Thus, electrons in insulators have very little mobility, making these materials electrically insulating. Semiconductors are insulators with a small band gap so that, with sufficient thermal energy, electrons in the valence band have a small but significant probability of excitation into the higher bands, therefore leading to moderate conductivity (see Fig. 4a).

The Fermi energy of a material is the energy of the highest filled state at absolute zero; the Fermi level, ε_F, is the (weakly) temperature-dependent chemical potential defined as the energy for 50% probability filling a state. For intrinsic insulators and semiconductors with no defect states, the Fermi energy is at the middle of the band gap (see Fig. 4b). If, as in our experiments, electrons are added to a disordered material by, say, electron bombardment, these new electrons will start to fill energy levels above the Fermi energy, thus creating what is termed an effective Fermi level, ε_F^{eff}; this can be thought of as a charge- (and temperature-) dependent chemical potential.

SiO_2 is an insulating material that has a band gap of about 8.9 eV [5]. Hence thermal excitation from the valence to conduction band is highly improbable; the only likely source of such excitation is through collisions of the incident high energy electrons. Since visible light is in the 1.65 to 3.1 eV range, even if electrons in the valence band of SiO_2 were electronically excited to the conduction band, no visible light (only high energy UV radiation) would be emitted upon relaxation or recombination of the excited electron with an empty state in the valence band (termed a hole). Thus, there must be other states for the electrons to reside in within the band gap; there must be localized defect or "trap" states (see Fig. 4c).

The localized states in disordered SiO_2 are due to defects in the crystalline structure or chemical defects from possible substitutional dopants. These defects add energy states within

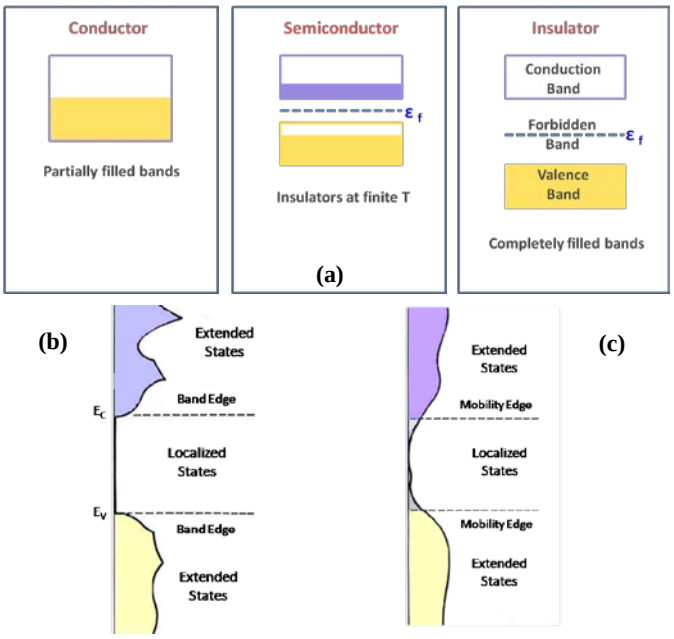

Fig. 4. **(a)** In a periodic solid, the classification of solids as conductors, insulators and semiconductors is determined by the extent of the band filling. Note the position of the Fermi energy, ε_F. **(b)** In ordered solids, extended states in the valence and conduction band are separated by a band gap. **(c)** In disordered solids, variations in the periodic order cause extended states to become localized in space. These occur in the forbidden band gap, forming localized trapped states, separated from extended states by a mobility edge [10].

the forbidden band (see Fig. 5). Now, when valence band electrons are excited into the conduction band by the high energy incident electron radiation and then relax, there are now 'closer' (in energy) trap states that the electrons can relax to. Some relaxation processes are photon emitting (radiative) processes. Therefore, if these radiative processes are in the appropriate energy range, visible or IR photons will be emitted. These light emitting defect structures, or chromophores, can be inherent in the material or can be induced by electron beam bombardment. Figure 5 illustrates how dose rates on the order of those of our experiments can create a laminating defect. Here, an oxygen vacancy is the defect precursor. When this defect experiences electron beam irradiation, it becomes a luminescing twofold coordinated silicon center [11].

V. Temperature Dependent Cathodoluminescence Model

A model with multiple bands of disordered states in the band gap has been developed to qualitatively explain the temperature dependence of the observed luminescence spectra [12-14]. A qualitative explanation of the temperature behavior of the cathodoluminescent intensity and spectra is presented here. The presence of four identifiable peaks in the spectra suggest that there are at least four bands of optically active states, as shown in Fig. 3, with energy separations determined by the energy of the emitted photons. Because transitions from an extended state in the conduction band to a localized disordered state are, in general, much more likely than between two localized disordered states with mean separations greater than the spatial extent of the localized states, we expect

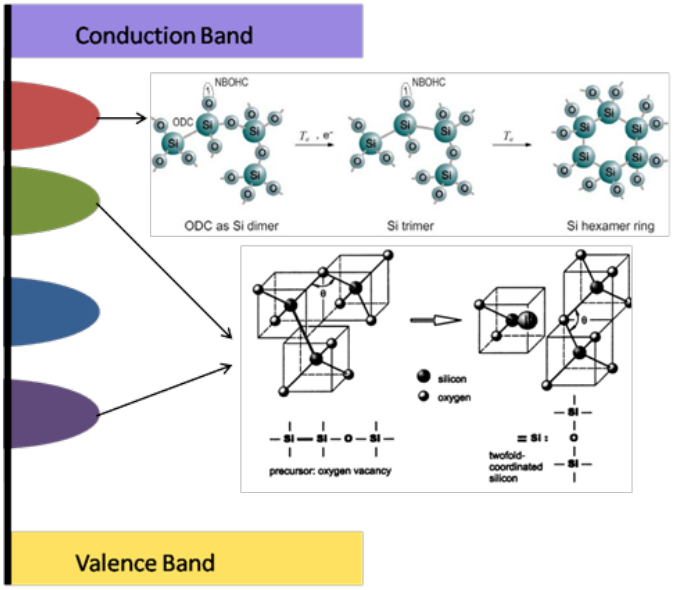

Fig. 5. The existence of at least four peaks in the disordered SiO_2 luminescence spectra (Fig. 3(b)) suggest at least four bands of localized defect or "deep trap" (DT) states. SiO_2 studies at room temperature have attributed a 645 nm peak to a nonbridging oxygen hole center [4] and 275 nm and 500 nm peaks to an oxygen deficient center [5].

the four bands to be at ~1.93, ~2.48, ~2.76, and ~4.97 eV below the conduction band edge.

To model the temperature dependent behavior of the peak intensities, we need to model the extent and occupancy of these disordered bands [12,13]. The intensity of a given band will be proportional to the number of excited electrons in the CB, the number of available, optically-active states in the disordered band, and a transition probability (or equivalent capture cross section). The occupancy of a given state is determined to first order by the effective Fermi level for a given density of trapped charges. A more detailed model will take into account the transition probabilities from one trap state to another, driven for the most part by thermal transitions.

A two band model with an effective Fermi level between peaks at ~275 nm and ~645 nm, as shown in Fig. 6, is used to illustrate the basic temperature behavior. In this model, the band generating red photons has available, optically-active states at an energy ε_{red}~1.93 eV below the conduction band edge. A similar band generating the ~275 nm photons is centered at ε_{uv}~4.97 eV below the conduction band edge. At different temperatures, the varying amounts of thermal energy control the thermal excitation of the electrons and the relative filling of the bands. These thermal excitations will fill available energy levels in some energy bands, while vacating levels in the other. This is the origin of the different photons being emitted.

At absolute zero (see Fig. 6(a)), no electrons will occupy levels above the effective Fermi level. Therefore, excited electrons can only relax into states in the red band, emitting lower energy red photons. At low temperatures with $0<k_BT<<(\varepsilon_{red}-\varepsilon_{uv})$ (see Fig. 6(b)), a significant number of electrons in the UV band (represented in blue in Fig. 6) are thermally excited to the red band (either directly or through an intermediate excitation into the conduction band). This creates empty states in the UV band that can then accept

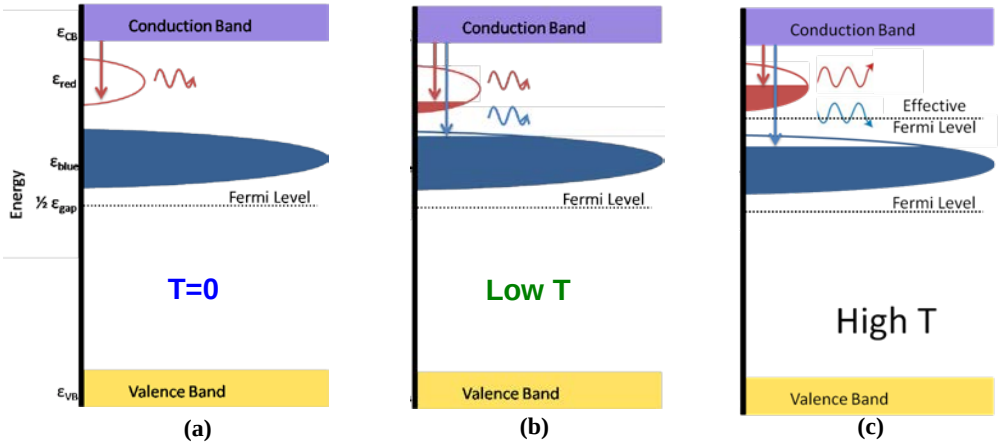

Fig. 6. Qualitative model of occupied deep trap densities of state as a function of temperature during cathodoluminescence. (a) At absolute zero, no electrons occupy levels above the effective Fermi level. Therefore, excited electrons can only relax to the red state, emitting red photons. (b) As small thermal energy is added to the system, some UV band electrons (represented in blue) are excited to the red band; thus, electronically excited electrons can now relax to either the red or UV band. (c) At higher temperatures, the same effect in (b) is enhanced further. Therefore, more UV photons can be emitted from the material.

electrons decaying from the conduction band and generating higher energy UV photons. Thus, significant numbers of electronically excited electrons can now relax from the conduction band into either the red or UV bands; the relative intensities of the red and UV emission depend on the relative number of unoccupied states in the two bands and the transition probabilities into these two bands from the conduction band. There is also a weaker dependence on the transition from red band states directly into UV band states and for recombination of electrons in the red and UV band with largely immobile holes in the valence band. At higher temperatures $0<<k_BT<(\varepsilon_{red}-\varepsilon_{uv})$ (see Fig. 6(c)), even more electrons from the UV band are thermally excited into the red band. At high enough T, if the decay time of electrons from sites in the red band into the UV band is longer than the trapping of electrons excited into the conduction band by incident high energy electrons, a population inversion can occur similar to that observed in a 3-level laser system.

This two band model predicts the wavelength shifts with temperature. With increasing temperature, peak wavelengths decrease for shorter UV, blue, and green wavelength peaks as these bands are thermally depopulated (see Fig. 3 (d)). However, according to the two peak model, the red peak wavelength should increase as temperature increases and the level of available disordered states increases. Thus, the two level model correctly predicts both the differences in intensity and the wavelength shifts observed for the red peak for states above the effective Fermi level and for the other UV, blue, and green peaks below the effective Fermi level.

VI. Conclusions

Low intensity cathodoluminescence was observed for disordered SiO_2 thin films under low intensity incident electron irradiation. The observed number and wavelengths of the observed peaks were consistent with previous room temperature observations. The intensity and peak positions of the primary peaks observed in the UV/VIS/NIR region were measured as a function of temperature from ~160 K to ~280 K. A model based on multilevel disordered peak density of states qualitatively explains the intensity versus temperature and wavelength versus temperature trends observed.

The next steps in this research involve measurements at lower T down to ~40 K and at higher (<400 K) temperatures. We intend to develop a quantitative model for intensity vs T and wavelength versus T behavior for all four of the observed luminosity peaks. In principle the luminosity measurements can be used to map the distribution and magnitude of the various densities of states for the different bands. Investigations of the luminescent intensity versus incident electron energy and beam current density, incident electron range, and film thickness are in progress. Through measurements of intensity versus electron beam exposure time and the decay time after turning the electron beam off we hope to study initial time dependence as traps fill to the effective Fermi level and as traps empty after excitation into the conduction band by the high energy electron irradiation has ceased.

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Greg Wilson is currently a graduate student at Utah State University in Logan, UT pursing an MS in physics. He received BS degrees in physics and mathematics from USU in 2011. He has worked with the Materials Physics Group for three years on electron emission and luminescence studies related to spacecraft charging. He also developed models for the penetration depth of electrons over a wide range of incident energies applicable to diverse materials and for emission and charging of multilayer materials.

Justin Dekany is currently a graduate student at Utah State University in Logan, UT pursing an MS in physics. He received BS degrees in physics from USU in 2010. He has worked with the Materials Physics Group for four years on electron transport measurements, electrostatic discharge tests, electron emission measurements, and luminescence studies related to spacecraft charging. He has been the Lab Manager for the Materials Physics Group for the last two years.

J. R. Dennison received the B.S. degree in physics from Appalachian State University, Boone, NC, in 1980, and the M.S. and Ph.D. degrees in physics from Virginia Polytechnic Institute and State University (Virginia Tech), Blacksburg, in 1983 and 1985, respectively. He was a Research Associate with the University of Missouri—Columbia before moving to Utah State University (USU), Logan, in 1988. He is currently a Professor of physics at USU, where he leads the Materials Physics Group. He has worked in the area of electron scattering for his entire career and has focused on the electron emission and resistivity of materials related to spacecraft charging for the last two decades.