Variations in Cathodoluminescent Intensity of Spacecraft Materials Exposed to Energetic Electron Bombardment

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Abstract—Many contemporary spacecraft materials exhibit cathodoluminescence when exposed to electron flux from the space plasma environment. A quantitative, physics-based model has been developed to predict the intensity of the total glow as a function of incident electron current density and energy, temperature, and intrinsic material properties. We present a comparative study of the absolute spectral radiance for more than 20 types of dielectric and composite materials based on this model which spans more than three orders of magnitude. Variations in intensity are contrasted for different electron environments, different sizes of samples and sample sets, different testing and analysis methods, and data acquired at different test facilities. Together, these results allow us to estimate the accuracy and precision to which laboratory studies may be able to determine the response of spacecraft materials in the actual space environment. It also provides guidance as to the distribution of emissions that may be expected for sets of similar flight hardware under similar environmental conditions.

Index Terms—cathodoluminescence, light emission, materials testing, electron flux, space environment effects

I. INTRODUCTION

Many highly disordered insulating materials used in spacecraft construction can exhibit electron-induced glow or cathodoluminescence when exposed to the space plasma environment [1-3]. Determinations of the absolute and relative cathodoluminescent intensity of spacecraft materials per incident electron flux are essential to predict and mitigate consequences for stray light contamination in space-based observatories [1-4] and optical detection [5]. They also provide important information about the defect structure and electron transport properties of these materials [6-10].

Previous studies have focused on how the relative spectral radiance of cathodoluminescent materials varies with changes in environmental conditions, including electron energy, current density, absorbed power density, and temperature. This has led to the development of a quantitative, physics-based model to predict the intensity of total glow as a function of incident electron current density and energy, temperature, and intrinsic material properties [1,4,9,11]. However, from a practical point of view, it is also important to develop an understanding of the variations of the cathodoluminescent intensity of diverse spacecraft materials exposed to energetic electron bombardment in both the space environment and space simulation studies. This is the focus of this paper.

We present results of ground-based measurements simulating space-induced cathodoluminescence from a wide variety of thin film dielectric, nanodielectric composite, and optical glass samples. Comparisons of the absolute spectral intensities per incident electron flux for these materials show three orders of magnitude variation. Variations in intensity are contrasted for different incident electron current densities and energies, different sample sizes and thicknesses, different testing and analysis methods, and data acquired at different test facilities. We also report on the variability of cathodoluminescence for a large set of similar epoxy samples exposed simultaneously to similar space-like monoenergetic electron flux conditions. The observed statistical fluctuations provide assessment of the instrumentation precision, the variation between samples, and the stochastic variations inherent to the material. The combined results of studies of numerous similar samples led to higher precision results that allow for quantification of additional materials properties.

II. MODEL OF CATHODOLUMINESCENT INTENSITY

The model developed for the observed electron-induced luminescence phenomenon is based on band theory of highly disordered insulating materials [4,9,12]. The observed luminescence occurs when an incident high energy, charged particle undergoes a series of inelastic collisions exciting valence band electrons into the conduction band. The excited electrons rapidly decay to localized (shallow trapped) states, with a mean binding energy $\varepsilon_T$ below the mobility edge [13].
A final electron transition, from the short-lived shallow trap states to longer-lived deep trap states is the origin of the excited photon. The model predicts that the overall luminescent intensity $I$, scales with incident current density $J_{inc}$, incident beam energy $E_{inc}$, temperature $T$, and emitted photon wavelength $\lambda$ as

$$I_{\gamma}(J_{\gamma}, E_{inc}, T, \lambda) \propto \left[ \frac{\partial (E_{inc}^0)}{\partial (E_{inc}^0)} \right]_{E_{inc}} \left[ 1 - e^{-\left(\varepsilon_{ST} / kT\right)} \right]$$

where $\varepsilon_{ST}$ is the electron charge and $L$ is the sample thickness. The dose rate $\dot{D}$ (absorbed power per unit mass) is given by

$$\dot{D}(J_{inc}, E_{inc}) = \frac{E_{inc} J_{inc}}{\eta_{em} R(E_{inc})} \left[ 1 - \eta(E_{inc}) \right]$$

where $\rho_m$ is the mass density of the material and $\eta(E_{inc})$ is the backscattered yield. $\dot{D}_{sat}$ is the material-dependent saturation dose rate. The exponential terms in Eq. (1) account for temperature dependence [6,14]. The two $\lambda$-dependent terms correct for photon absorption within the luminescent material and reflection from any underlying coating [1,15]. A more detailed discussion of the model is given in [1]. The present work focuses on the dependence of spectral radiance on $J_{inc}$, $E_{inc}$, and the range of the deposited electrons, $R(E_{inc})$. Fig. 1 shows the range and dose for select disordered materials as functions of incident energy.

The dependence of the spectral radiance on incident current density $J_{inc}$, in Eq.(1) is through the dependence on dose rate; $I_{\gamma}(J_{inc}) \propto \dot{D}(J_{inc}) \dot{D}_{sat} [\dot{D}(J_{inc}) + \dot{D}_{sat}] \propto J_{inc} \dot{D}_{sat} / \dot{D}_{sat}$. At low dose rates ($\dot{D} \ll \dot{D}_{sat}$), $I_{\gamma}$ is linearly proportional to $J_{inc}$. At higher current densities (i.e., $\dot{D} \gg \dot{D}_{sat}$), saturation occurs when trap states fill and limit the number of states electrons can decay into or when charge build up in the material modifies the landing energy and limits the electron flux reaching the sample. Then, $I_{\gamma}$ approaches a constant material-specific saturation intensity. Such saturation effects, at increasing current densities and fixed incident energies, have been reported for disordered SiO$_2$ [4], nanodielectric carbon-loaded polyimide [11], cyanate ester/graphite fiber composites [16], and bisphenol/amine epoxy [1,17].

The energy dependence of the spectral radiance is more complicated, due to the energy-dependent penetration depth or range, $R(E_{inc})$, in Eq. (2). For nonpenetrating radiation—where the energy-dependent penetration depth or range, $R(E_{inc})$, is less than the film thickness $L$—all incident power is absorbed in the material. At low incident power, $I_{\gamma}$ is linearly proportional to the incident energy and power density, $J_{inc} E_{inc} / q_e$. At higher incident power, $I_{\gamma}$ exhibits saturation effects for increasing energy and fixed current density. For penetrating radiation—where $R(E_{inc}) > L$—the absorbed power is reduced by a factor of $[L/R(E_{inc})]$ [18] leading to a similar dependence for $I_{\gamma}$.

An energy-dependent correction to the incident flux, $J_{inc}[1 - \eta(E_{inc})]$, is also included in Eq. (2) to account for quasi-elastic backscattered electrons that do not deposit substantial energy; $\eta(E_{inc})$ is the backscattered electron yield [15]. For the most part, this correction is small and weakly dependent on energy. For biased samples, or when excess charge is stored in the trap states, a surface voltage $V_s$ results...
and $E_{inc}$ is replaced everywhere in Eqs. (1) and (2) by the landing energy, $(E_{inc} - q_e V_s)$.

III. CATHODOLUMINESCENT VARIATIONS

A. Experimental Test Methods and Facilities

Cathodoluminescence tests used well-characterized, uniform, large-area, approximately normal-incidence, monochromatic electron beams to irradiate tests samples. The samples were in high vacuum conditions, held at fixed temperatures that could be controlled to cryogenic levels. Results presented here used two optical detectors: (i) an image-intensified CCD video camera (Xybion, ISG-780-U-3; ~400-900 nm bandwidth and 830 nm peak wavelength), and (ii) a single lens reflex CCD still camera (Cannon, EOS Rebel XT DS126071; ~390-650 nm bandwidth and 553 nm peak wavelength) [1]. The cameras were calibrated on an absolute scale using NIST traceable optical coatings [1]. Values for optical glasses converted from Treadaway’s measurements [2] are shown above, with circled numbers corresponding to: 1-Schott BaK4; 2-Schott BK7; 3-Schott LaK1O; 4-Schott SF10; 5-Schott SF11; 6-Schott SK7; 7-Schott SK14; 8-Schott UBK7; 9-Schott BK7-G14; 10-SiO$_2$ (Z-cut quartz); 11-SiO$_2$ (fused silica); 12-α-Al$_2$O$_3$ (sapphire).

Measurements were conducted at two independent facilities: the Environment Effects Laboratory at NASA Marshall Space Flight Center (MSFC) and the Space Environment Effects Materials (SEEM) test facility at Utah State University (USU). USU testing used a ~1 m$^3$ ultrahigh vacuum chamber (~10$^{-6}$ Pa) equipped with a high energy electron gun with a focused beam (5-30 keV at 0.1-1000 nA/cm$^2$ flux) [19]. Temperatures from ~50 K to ~350 K were used for cathodoluminescence tests [20]. Samples from ~1 cm to ~3 cm diameter were tested. Additional NIR-IR and mid-IR cameras and detectors, UV/Vis/NIR spectrometers, and electron emission measurement capabilities were used for some measurements [1].

MSFC testing used a larger ~2 m x 1 m diameter ultrahigh vacuum chamber (~10$^{-6}$ Pa) able to accommodate large (up to 41 x 41 cm) flight samples. It is equipped with a high energy electron flood gun (1-100 keV at 1-100 nA/cm$^2$ flux) with in situ Faraday cups positioned on either side of the large sample to monitor the incident beam current (see Fig. 2(a)). Temperatures down to ~120 K were attained by mounting samples on an electrically-isolated liquid nitrogen reservoir.

Measurements at the two test facilities are identified separately in Fig. 3. Measurements for similar materials (bisphenol/amine and intermediate conductivity carbon-loaded polyimide samples) show agreement to within a factor of ~2-3. Comparison can be made for disordered SiO$_2$ optical glass measured at USU [1,4,6,14] and previously by Treadaway [2], which agree to within ~25%. Thus, this is reasonable agreement given the differences in sample materials, temperatures, test methods, instrumentation, and electron bombardment conditions for the different tests.

![Cathodoluminescence data diagram](image_url)
However, it also serves as an indication of the uncertainties associated with ground-based testing.

B. Variations with Material

Figure 3 show a comparison of measured absolute cathodoluminescent spectral radiances for different spacecraft materials, which spans more than three orders of magnitude. These different results for keV electron bombardment with varied current have been scaled linearly to 10 µW/cm² incident electron power densities, which is representative of severe space environments. The materials tested include: polyimide films, neat urethane and bisphenol/amine epoxy films; bulk and thin optical coatings of disordered SiO₂; several grades of commercially available high-conductivity carbon-loaded polyimide nanodielectric composites; cyanate ester and urethane epoxy resins in graphite fiber and fiberglass composites; and multilayer dielectric/conductor composites. In addition, measurements of the total cathodoluminescence of various optical glasses by Treadaway [2], using 2.5 MeV electrons, have been scaled to the same incident power density.

In general terms, we found that the relative cathodoluminescence of organic materials for a given electron flux ranks from lowest to highest intensity for polyimide nanodielectric composites, bulk polyimide, bulk epoxy materials, and epoxy resin composites. Polyimide and carbon-loaded polyimide exhibit relatively weak cathodoluminescent intensities, with the intensity decreasing with carbon content. The cathodoluminescence of optical glasses span ~30X at the low intensity portion of the range, for borosilicate glasses up to crystalline quartz and sapphire. As a point of comparison, note that these absolute spectral intensities for isolated samples exposed to electron fluxes comparable to solar wind intensities span the intensity of the zodiacal background in the visible and near infrared wavelength range (vertical dashed line of Fig. 3) [21]; this relative comparison of intensities to the zodiacal background is most important for space-based observatories, where the zodiacal background can be a significant source of external light contamination [1]. Also note that these measured intensities are greater than the scaled CCD video camera detection threshold.

Figure 4 shows three cathodoluminescence spectra typical of spacecraft materials which are all similar in the visible region, and exhibit multiple peaks indicative of multiple types of defects. The cyanate ester [16] and SiO₂ [4] spectra were acquired at USU; the epoxy spectra is from a different facility [8].

C. Variation with Energy and Penetration Depth

The energy dependence of cathodoluminescence scales as a saturation function of the dose rate (see Eq. (1)) For nonpenetrating radiation, the dose rate (see Eq. (2)) scales linearly with energy with a small—usually negligible—correction term for the energy dependence of the electron backscatter yield. Figure 5 shows spectral radiance versus incident energy curves for two bulk materials—cyanate ester/graphite fiber composite (red circle) [16], and bisphenol/amine epoxy (blue triangles) [17]—where the sample is thick enough that all the incident electrons are deposited. These increase linearly with energy at low incident power density, but reach saturation limits at higher power densities (~10³ µW/cm² for the cyanate ester and ~230 µW/cm² (~70%) for bisphenol materials). The bisphenol data fit by the linear curve in Fig. 5 (blue triangles) does not show appreciable saturation, while other data (blue inverted triangles) shows significant saturation effects. Saturation of bisphenol/amine epoxy is also evident in Fig. 6 at lower power densities. The saturated spectral radiance curve (blue curve, with a saturation power density of 360 µW/cm² (~30%) taken after the electron beam is turned on and after the sample has reached equilibrium deviates noticeably above ~50 µW/cm² from the linear spectral radiance curve (green curve) taken immediately after the electron beam is turned on and before saturation is reached.

For thin films, such as the ~60 nm SiO₂ optical coating in Fig. 5 green curve), incident electrons above a threshold energy (~1.2 keV; see Fig.1) penetrated through the material. For energies above the penetration energy, dose rate power deposition decreases with increasing energy, so the spectral radiance also decreases. Radiance from thicker ~80 µm SiO₂ layers increased linearly at lower incident energies and exhibited saturation effects at higher electron power (see Fig. 5) [6,14].
With composite materials, such as the carbon-loaded polyimide, the relationship between energy and cathodoluminescent intensity is more complicated [22]. The black curve in Fig. 5. is a linear superposition with ~88% of a penetrating curve (10 nm thickness, with ~350 eV penetration energy) that models thin polyimide layers above carbon particles and ~12% of a nonpenetrating curve (2 μm thickness, with a penetration energy equal to the incident energy of ~10 keV) that models thick polyimide regions between the carbon particles. The relative surface areas of thin (~19%) and (~81%) thick polyimide regions determined with scanning electron microscopy are consistent with these estimates [23]. Even such a simple bimodal distribution of polyimide thicknesses predicts the relatively flat energy dependence of the spectral radiance curve observed at higher energies. A similar effect is seen qualitatively in Fig. 2(f) of the cyanate/graphite fiber composite sample. Thicker regions of epoxy are lighter, while the thinner epoxy layers over graphite fibers are more intense and brighter blue in color.

D. Variations with Analysis Methods

Analysis of bisphenol/amine epoxy data taken at MSFC emphasizes the variation in results using three different analysis procedures [17], as explained in subsequent paragraphs. Use of these different procedures to determine the absolute spectral radiance per incident electron power density as a function of incident electron energy, as shown in Fig. 6, produced results that differed by more than an order of magnitude.

The “edge region analysis” procedure defined a large square annular region that enclosed all 36 ~1 mm diameter “glue dot” regions of bulk epoxy as well as an ~220X larger area of surrounding carbon-loaded polyimide (see Fig. 2(b)). As expected and seen in Fig. 3, the estimated spectral radiance was much closer to that of carbon-loaded polyimide than bulk bisphenol/amine epoxy.

The “single dot analysis” procedure eliminated most of the contribution from carbon-loaded polyimide contamination by analyzing only a small region around a single epoxy dot. This procedure gives more accurate intensity results, but has a large statistical uncertainty since only ~15 pixels per frame comprising the single dot could be analyzed.

The most accurate results are obtained using the “multiple dot average” procedure. Data for all 36 epoxy dots individually are analyzed simultaneously using the single dot analysis; the statistical uncertainty is reduced by using the average value and associated standard deviation. As is readily apparent from Fig. 6, the multiple dot analysis results in much greater accuracy. Slopes of the linear fits in Fig. 6, or of the spectral radiance per incident electron power density in [W/cm²-nm-sr] per [μW/cm²], measure the conversion efficiency of high energy electron power to luminescent photon power. The slope of 2.16 x 10⁻² [W/(W-nm-sr)] (±2%) from the statistical analysis procedure (green) is ~55% larger than from the single dot analysis (red) and ~32X larger than from using edge region analysis (black). The multiple dot analysis result is the closest to the results measured at USU for a larger bulk sample, as shown in Fig. 2.

Fig. 6. Comparison of different methods to determine the absolute spectral radiance as a function of incident electron power density for bulk bisphenol/amine epoxy samples. The slope of the linear fit, or conversion efficiency of high energy electron power to luminescent photon power, of 2.16 x 10⁻² [W/(W-nm-sr)] (±2%) determined from the statistical analysis of 36 “glue dot” (green) is ~35% larger than from analysis of a single “glue dot” (red) and ~32X larger than from using edge region analysis (black). The range of variations of the 36 sample data set is indicated by the ~±5% standard deviation (error bars) and ~±60% minimum to maximum range (green triangles). Errors for the spectral radiance and slope values for the linear fit of the single “glue dot” are ~5X larger than for the analysis of all dots. Comparison for the multiple sample data set of green (unsaturated) and blue equilibrium (saturated) curves show time-dependent saturation effect at higher dose rates. The fit for the saturated (blue) curve from Eq. (1) is of the form I_y = D/ (D + D_sat), with a proportionality constant of 9 x 10⁻²[W/(cm²-nm-sr)] (±20%) and D_sat=1.5 kGy/s (±30%) or P_sat=360 μW/cm².

E. Variation of Similar Samples

Analysis of multiple dots characterizes the variation between samples. The range of variations of the 36 sample data set is indicated by the ~±5% standard deviation (error bars) and ~±60% minimum to maximum range (triangles) shown in Fig. 6. The multiple dot analysis also results in much greater precision. Errors for the spectral radiance and slope values for the linear fit of the single dot procedure are ~5X larger than for the analysis with the multiple dot procedure.

When many samples are statistically analyzed, precise measurements can be made at low intensities and additional small effects can become apparent. It is evident from comparison of the curves and associated uncertainties in Fig. 6 that the saturation effects discussed in Sec. III.C at low dose rates would not be observable without the higher precision obtained with the multiple dot procedure. That is, the observed differences for the green unsaturated curve and blue saturated curve are greater than the uncertainties in the red single dot curve. The uncertainty in D_sat was also reduce by more than a factor of 2.

IV. CONCLUSION

Cathodoluminescence is an important space environment-induced phenomenon to understand, especially in applications where extremely sensitive space-based optical detection is necessary. Measurements of the absolute spectral intensities per incident electron flux have been presented that confirm a quantitative, physics-based model to predict the intensity of the total glow as a function of incident electron current density and energy, temperature, and sample thicknesses and composition. Comparisons for these materials show three orders of magnitude variation. For bulk nonpenetrating materials, spectral radiance increases with increasing incident
electron power and flux. In thin films where electron penetration is possible, a linear relation is seen at low energies, but once penetration occurs intensity decreases with increasing energies. Composite materials, where both penetrating and nonpenetrating electrons effects were present, required a combination of these two effects in the model. Saturation effects at higher doses were observed and accurately modeled, for both penetrating and nonpenetrating electrons.

Statistical analysis of the observed statistical fluctuations of cathodoluminescence for a large set of similar epoxy samples exposed simultaneously to similar space-like monoenergetic electron flux conditions provided measures of both the instrumentation precision and the stochastic variations inherent to the material. The statistical analysis of the combined results of studies of numerous similar samples led to higher precision and accuracy results that allow for quantification of additional more subtle effects. Together, these results allow us to estimate the accuracy and precision to which laboratory studies may be able to determine the response of spacecraft materials in the actual space environment. It also provides guidance as to the distribution of emissions that may be expected for sets of similar flight hardware under similar environmental conditions.

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REFERENCES


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