4-2011

Electron Induced Luminescence of Insulating Polymeric Materials

Doug Ball

Electron Induced Luminescence of Insulating Polymeric Materials

Doug Ball

Department of Physics
4900 Project
Research Mentors: J.R. Dennison and Alec Sim
April , 2011

Electron Induced Luminescence of Insulating Polymeric Materials

U.R.C.O. Grant Report, Spring 2011
Douglas Ball, Undergraduate, Physics Department
Alec Sim, Graduate Mentor
JR Dennison, Faculty Mentor

I. Abstract

The study of luminescence and electron transport in disordered insulating materials provides detailed information about the material structure and interaction of incident electrons within a material. Electron induced luminescence of insulating polymeric materials has been observed in tests by the USU Materials Physics group. Conduction electrons can transition between extended states in the valence and conduction band and a distribution of localized trapped states within the band gap. Electron transport and luminescence is governed by the distribution of states and transition rates between them. This study investigates the exponentially decaying signatures of both luminescence and sample current of M55J under electron bombardment and relates their origins and relative intensities to a proposed theory based on quantum band structure models.

II. Methods

Electron bombardment tests were done by the Materials Physics Group to simulate the space environment effects on the James Webb Space Telescope (JWST) insulating materials. This was done by placing 1 cm diameter samples of several carbon composite materials in an ultrahigh vacuum (UHV) chamber (see Fig. 1) and accelerating electrons at the sample using a high energy electron diffraction gun under the following conditions:

- Low Pressure: 10^{-8} Torr
- Low Temperature: 130 K (Liquid-Nitrogen cooled)
- Electron Energy: 7- 22 keV
- Current Flux Density: 5 nA/cm^2
- Exposure time on sample: 3600 seconds

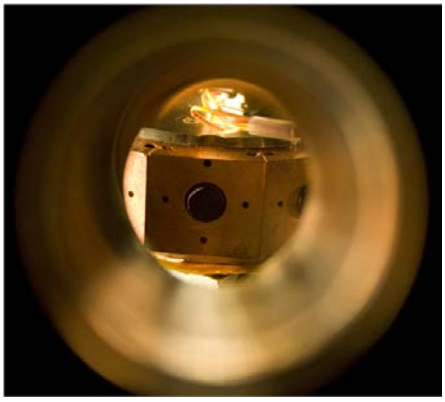

Figure 1 SLR image looking down port in ultra high vacuum chamber showing sample mounted in carousel.

	Instrument	Range	Sample Rate
Electron Detection	Electrometer	> 0.1 pA	17ms
	Oscilloscope	> 0.1 µA	< 10 ns
Photon Detection	SLR Camera	300-700 nm	34 s
	CCD Video Camera	300-1000 nm	33 ms
	InGaAs NIR Video Camera	800-1700 nm	17 ms

Table 1 Detectors used including the range and sample rate.

The objective was to detect both electron emissions and photon emissions from each sample while it was exposed to flux from the keV electron gun. To do so, two detectors were used for electron detection and three cameras were used for photon detection. Table 1 lists the range of detectors and sample rate.

This study focused on using a Xybion charged couple device (CCD) video camera with a detectable wavelength range of 300 to 1000 nm. This study also focused on a single hour long run from a single M55J epoxy/

carbon fiber composite sample, one of more than a dozen samples to be studied. From the video camera images taken at 30 frames per second, the hour long video was spliced into JPEG images for each frame (see Fig 2a). Each of more than 10^5 frames for the entire run was then processed with a MathCAD algorithm that averaged the intensity values of all pixels over the entire sample per frame. Values for the electric current signature were taken with an electrometer which had a tiny electrode wire on the rear of the sample and was connected to a data acquisition program using LabView.

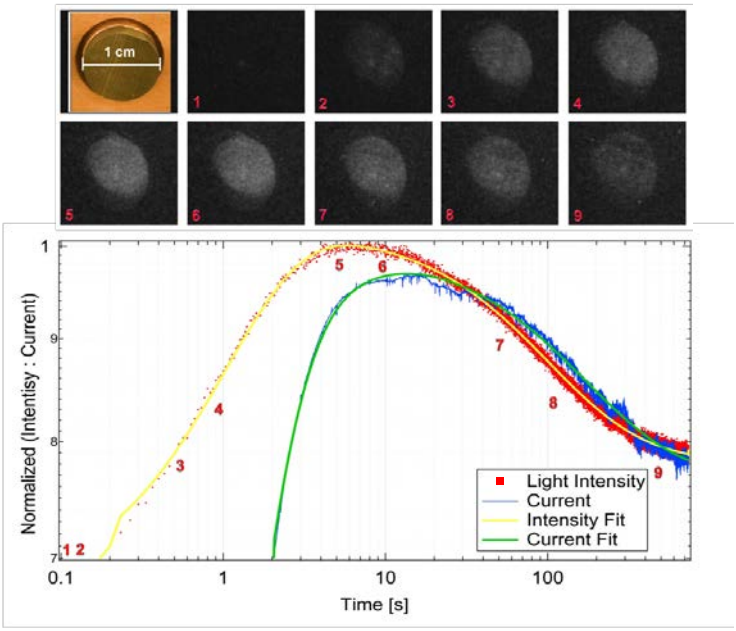

Figure 2 (a) Top left most pictures shows sample under lighted condition in chamber. Other images are Xybion video images of frames showing a log time lapse of the sample glowing. Red numbers correspond to the time markers seen below the red curve of light intensity in the plot below. **(b)** intensity (red curve) normalized to max intensity and current (blue curve) normalized to agree with the intensity plotted on a semi-log graph to show in better detail their correlation. The same exponential model (see equation 1) was fit to both curves.

III. **Results**

On both a digital Canon Single Lens Reflex (SLR) still camera's images (Fig. 2a) and the Xybion CCD video camera's footage, luminescence and its decay were very apparent. The analysis done so far has been limited to only the first 750 seconds of data collected for current and light intensity after the beam was turned on. This limit is due to the vast amounts of data and early stages in developing analysis routines. These values were tabulated with their time stamps and plotted against elapsed time as seen in Fig. 2b. The light intensity curve was normalized with its maximum intensity, and the electric current was also normalized to agree with the light intensity curve. Both signatures appear proportional and, at time greater than 20 seconds, nearly follow the same curve.

IV. **Analysis**

From Fig. 2, it is apparent that the light intensity curve is proportional to the sample current curve at time greater than 20 s. Equation 1, from Griseri's paper on recombination-induced luminescence in epoxy resin[4], indeed fits both the sample current and light intensity signatures with slightly different parameters.

$$J_{norm}(t) = J_1 e^{-t/\tau_1} + J_2 e^{-t/\tau_2} + J_3(1+\alpha t)^{-m} \qquad (1)$$

Each of the three terms of Eq. 1 describes distinct processes for electron migration within the material and the correlated light intensity (See Fig. 3). From Eq. 1, J_1, J_2, J_3 are fitting parameters characterizing the relative contributions of the three processes, whereas τ_1 and τ_2 are short term decay times, and t is the independent elapsed time. The exponential terms e^{-t/τ_1} and e^{-t/τ_2} describe the first two processes of when the current and light intensity both increase exponentially followed by a gradual exponential decay. The third term, $(1+\alpha t)^{-m}$, describes a power law decay of power m leading to a much longer decay time than the exponential terms.

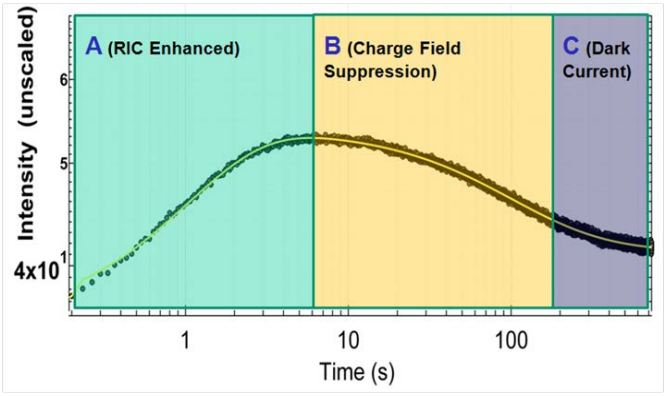

Figure 3 Light intensity profile on a log-log scale showing the different processes by section. Each process correlates to it respective term in equation 1.

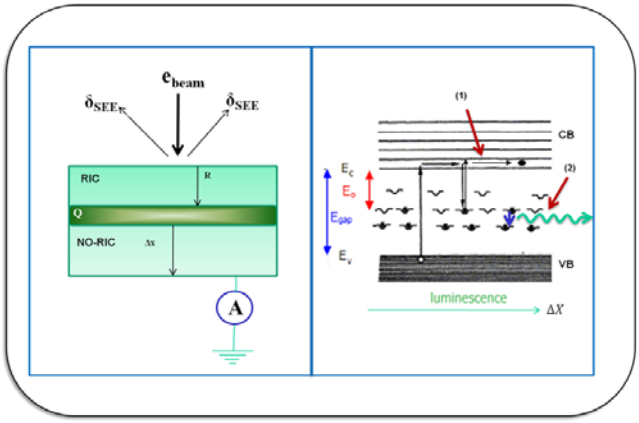

Figure 4(a) Cross section of sample with normal incidence electron beam. Shows the incident electron beam, charge distribution at range R, secondary electron emission δ_{SEE}, RIC enhanced region and dark current region. The ammeter at the rear electrode measures the current. **Figure 4(b)** Schematic of energy band structure. Shows the conduction band (CB) at E_c the valence band (VB) at E_v, trapped states, conduction resulting from trapping and de-trapping (1) and emission of light due to de-trapping and recapture at a lower state (2). E_0 is the Fermi energy. Δx is the spatial coordinate.

The first process and first exponential term of Eq. 1 is characterized with a known mechanism called radiation induced conductivity $(RIC)^2$ which creates a higher conductivity in the upper region of the sample where the electrons first penetrate (see figures 3(a) and 4(a)). RIC is a result of incoming radiation that deposits enough energy into the material that trapped electrons can overcome the band gap and be excited into the conduction band. This induced conductivity allows more electrons to migrate within the top region of the sample spreading out away from each other with the net charge distribution migrating toward the top of sample as they would naturally do in a fully conductive sample. It is not yet fully understood as to what mechanism most drives the current; however, the exponential increase in current is at least partially driven as a result of the electric field increase as charges accumulate in the upper region. This top layer of negative charges as a consequence yields a bottom mirrored layer of a positively charged region in the conductive plate under the insulating sample that connects to the grounded wire leading to the electrometer. It is also not yet clear as to why the onset of light emission occurs before the onset of current as seen in Fig. 2(b).

Electrons remain in the conduction band in the top region of the sample for only a very short duration until they relax back down to lower energy states, which for many polymers results in trapped states between the valence and conduction bands. Electron charges can drop in energy states from the conduction band to a trapped state, or from a trap state near the conduction band to a lower energy trap state and thereby give off energy in the form of a photon (Fig. 4b). The number of photons being emitted is proportional to the transitions of electrons between trap states of the energy band gap between the valence and conduction bands that are inherit in the polymeric material. This cyclic process of electrons being excited and decaying to trap states and then transitioning between trap states to give off light continues as long as RIC continues via incoming electrons. This light emission increases exponentially following the current since there are more electrons filling the trap states. It is still uncertain as to what percentage of the electrons dropping to a trap state from the conduction band and what percentage of electrons going from a trap to a trap is emitting light.

The second process is a result of the electrons that have accumulated enough in the material to suppress the incoming beam of electrons (Fig. 3-B). The number of available electrons in trap states is dependent on the amount of electrons that enter the material from the electron beam. As a result both the current and therefore the photon emission begin to dissipate.

The third process and thus the third term of Eq. 1 are a result of the RIC effect being suppressed from the reduced number of incoming electrons, which in turn permits the electron distribution near the top of the sample to migrate toward the rear. This current at this time is considered dark current with the light emission being a function of the number of thermally assisted trapped charges.

The measured current is modeled by both the dark current and RIC enhanced charge movement as

$$J_3(t) = \left(\sigma_{DC}(F,T,t) + \sigma_{RIC}\left(F,T,\dot{D},t\right)\right)F \qquad (2)$$

where, σ_{DC} and σ_{RIC} are the dark current and radiation induced conductivity.[2] F is the electric field between the top charge distribution and the rear of the sample. Ḋ is the dose rate defined as the total energy deposited in a material by the incident radiation per unit mass per unit time.[1] Knowing τ_1 and τ_2 from Eq. 1, we can calculate the dark current and Radiation Induced Conductivity using the relation:

$$\tau_i = {}^{\varepsilon_0\varepsilon_r}/_{\sigma_i} \qquad (3)$$

Knowing the conductivity of the material allows one to calculate the current density J using Eq. 2. A model like Eq. 1 allows an accurate prediction of how the charge penetrating a polymeric material will behave within the material as well as its accompanying luminescence. Application can be made to use this in developing composite insulating polymers that yield lesser or even more luminescence/current depending on the use of the material.

Further analysis will be done to the entire run as well as a number of other runs of different materials being tested. Current progress in analysis is being made with other materials, and the MathCAD algorithm used in analysis of this experiment has been upgraded into a MatLAB program for quicker data processing.

V. **Personal Impact**

My specific contribution to this project included, sample preparation, loading samples into the chamber, setting up part of the instrumentation used, writing an analysis program in MathCAD to rip through the large amounts of video data and plot it, controlling collection of video data during experiment, developing part of the LabView program used in data acquisition, learning to use the software program IGOR to plot and model plots from video and electric current data taken, and then to interpret analyzed data. Though I am not a great scientist, I have learned from and been in observance of some of the greatest to me. It was a privilege to witness this proposed theoretical model develop from the early stages of the lab up through this analysis and have the assistance of Alec Sim and JR Dennison, who both taught me most of the background in electron transport theory needed to begin to address the question of what was occurring with the luminescence phenomena.

I shall make mention of a few of the most profitable principles learned from working on this study. First, I knew next to nothing when I started this project on how to program in LabView, MathCAD, or even how to use IGOR to create plots. A number of weeks out of the summer I spent learning how to program in LabView writing a program that controlled optical filter wheels and communicated with a PC over a LAN connection as well as communicated with the main acquisition program. This was an ideal hands-on introduction to constructing and troubleshooting programs in LabView. Furthermore, Josh Hodges, a recent graduate student within our group showed me how to use MathCad beyond its normal numerical analysis skills by teaching me the basics to MathCad programming. Using this, and with his guidance along the way, I developed a program that would sift through each of the frames from the video data, and determine the average light intensity of a specified region. This could be done for video taken to observe light intensity, which analyzed over 100,000 video frames for every hour of footage. This program was also used to extract the electric current data of the sample taken by the electrometer. I also spent a great number of hours with Alec Sim, a former PhD student of our group, who taught me how to bring data into a program known as IGOR to plot curves and fit models to the curves. Each of these programming skills is becoming a necessity to work in a physics research field and being more fluent in them has given me an advantage in that regard.

Through this experience I gained a better eye in searching a great number of journal articles that would give me an understanding to what mechanisms played a role in the luminescence observed. Also, I assisted in a poster that was presented at the 11^{th} International Spacecraft charging Technology Conference, and then also put together my own poster and presented at an American Physical Society professional conference, both being my first few times presenting at a professional scientific meeting.

Some of the greater things learned in working with this group on this study, were that of learning to always being inquisitive, working hard with integrity while having fun, and taking the initiative. I think that these are contagious characteristics from JR's personal work, and what he expects us to be in his group. I should also acknowledge that Alec Sim worked a great number of hours discussing with me the possible physical explanations of the electron induced luminescence, of to which I owe a great amount of gratitude to see how this PhD theorist would sort through a problem like this.

VI. **References**

1. Douglas Ball, Alec Sim, Josh Hodges, Ryan Hoffman and JR Dennison, "Electron Induced Luminescence of Insulating Polymeric Insulators," Poster, American Physical Society Four Corners Section Meeting, Weber State University, Ogden, UT, October 15-16, 2010

2. Jennifer A. Roth, Ryan Hoffmann, JR Dennison, and Jonathan R. Tippetts, "Effects of Radiation Induced Conductivity on Electrostatic Discharge in Insulating Materials," Paper Number: AIAA-2009-3527, *Proceedings of the 1st AIAA Atmospheric and Space Environments Conference*, 2009.

3. Alec Sim and JR Dennison, "Charge Dynamics in Disordered Insulating Materials," American Physical Society Four Corner Section Meeting, Colorado School of Mines, Golden, CO, October 23-24, 2009.

4. V. Griseri, "Photoluminescence, recombination induced luminescence and electroluminescence in epoxy resin," J. Phys. D: Appl. Phys., Vol. 34, 2001