4-27-2020

Optical Relaxation of Defects in Kapton Caused by Irradiation

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Physics 4900 Project
Report April 27, 2020

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Abstract

Radiation can create atomic-scale defect states in polymers, leading to changes in their optical, electrical and mechanical properties. Recent studies of polymers have shown that these defect states are sensitive to oxygen or air exposure. It is believed that air cause the number of defect states to decrease and the polymers to revert to their original states. However, the time scale of this regression is not known. This experiment quantified the time that it takes one polymer to recover and the extent of said recovery; polymide (PI). In order to study the regression, optical transmission data were taken using a StellarNet UV/VIS Spectrometer. Optical data were collected at different intervals of time and then compared in order to quantify the time frame of the regression. Failure to account for this time-dependent recovery in radiation studies may result in inaccurate results and has called into question previous studies involving radiation effects in polymers where exposure times were not recorded.

Introduction

The goal of the proposed work is to obtain information on how ionizing electron radiation from the space environment, over time, affect materials commonly bombarded by radiation onboard spacecraft. Radiation is expected to significantly affect UV/VIS/IR optical transmission of the samples. Changes in transmission and reflection will be the primary indicators in comparing sample materials.

Satellites, shuttles, stations, and other space vehicles often utilize very sensitive optical lenses, coatings, and filters. Many of these optical parts can become no longer serviceable once the spacecraft has been in space for a prolonged amount of time due to the irradiation they receive while in space. Knowing how irradiation exposure time and intensity affects important components of a spacecraft are imperative to a design team. This information is of great value in the design and manufacture of sensors and other components used on spacecraft.

Theory

When light is shone on a material, it will either be reflected, transmitted, or absorbed (see Fig. 1) at different rates depending on its energy [1]. Transmission and reflection spectra, like those shown in Fig. 2, provide information about which energy photons are absorbed.

To measure transmission spectra, samples are placed in between the two metal plates (see Fig. 3(a)), one connected with fiber optic cables to a Deuterium-Tungsten Halogen light source (215 nm to 2500 nm). The other fiber optic cable is connected to two StellarNet spectrometers (RED-Wave NIR-25 and the Black Comet C-SR-25), measuring a wavelength range of ~240 nm to 1700 nm with 0.2 nm to 1 nm resolution. For reflectivity spectra, the basic setup is the same; however, there is a splice-bushing needed to use both spectrometers simultaneously. Instead of using the optical stands, reflective data are taken with a fiber optic probe for specular reflectivity (see Fig. 3(c)) or an integrating sphere for diffuse reflectivity (see Fig. 3(b)).

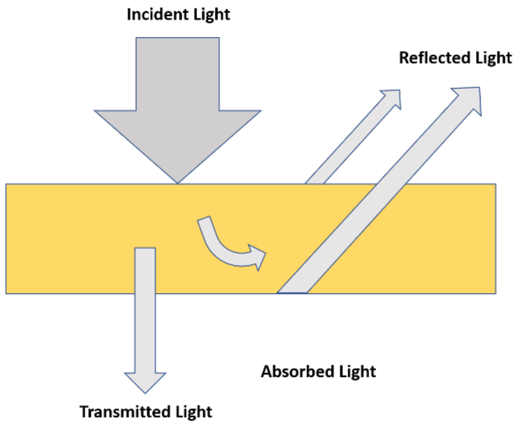


Figure 1: Absorbed, Transmitted, and Reflected Light

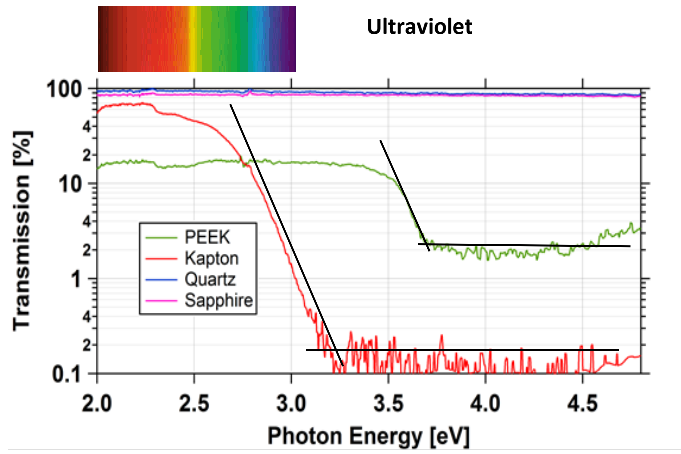


Figure 2: Transmission spectra from four spacecraft materials (PEEK, Kapton, quartz, and sapphire). The band gap energy is determined by the intercept of the black lines with the horizontal black line indicating the background noise intensity, while the slope of line is proportional to the energy density of defect states

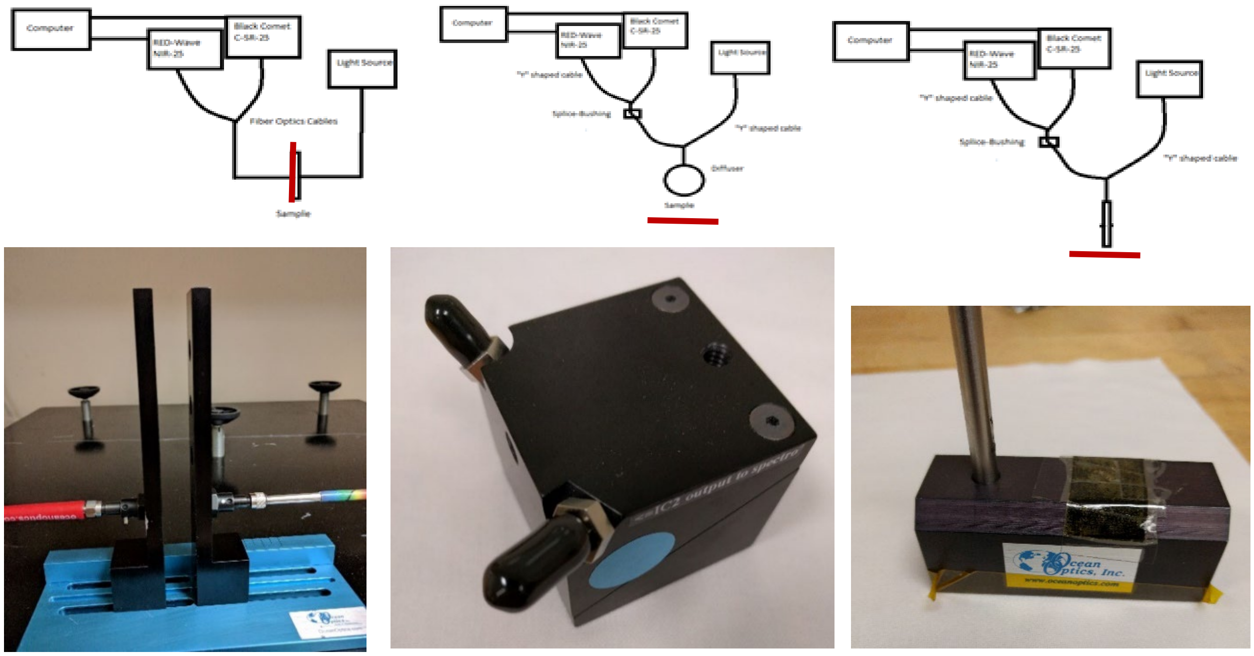
The data from the spectrometers are acquired by a computer and are then analyzed. Custom *Mathcad* analysis software is used to perform data averaging and background subtraction, calculate the transmission/reflection coefficients as functions of photon energy, and create spectral graphs. Typical transmission spectra are shown in Fig. 2. These can be used to determine broad spectral features such as optical cut off edges, optical band gaps, and optical absorption and sharp spectral features due to atomic absorption or emission [2].

Research Objective

When a highly disordered polymer is exposed to in ionizing radiation additional defect states are created through processes including atomic displacement, band breaking or rotation, and crosslinking. Recent work has suggested that some of these defect states created by irradiation are sensitive to atmospheric exposure (more specifically oxygen, OH^{-}, or other reactive ions) [3]. It has been noted that exposure to oxygen, or other atmospheric gases, can relax these defects and potentially recover. The time it takes for this relaxation to occur is unknown and the objective of this research. Based on other projects done [4] the most relaxation is estimated to happen within two hours of the sample being taken out of the irradiation chamber and exposed to oxygen. The objective of this research was to determine the time frame and the extensiveness of the relaxation in Kapton.

Methods/Procedure

Two Kapton samples were irradiated in an electron gun chamber. The chamber was pumped down to 8×10^{-8} mPa and got about 0.85 MGy of radiation over the course of 3 days getting around 3.58 Gy/s. This radiation dosage was chosen because it mimics the dosage that a spacecraft in a geosynchronous orbit would receive over one year. Two one-inch rounds of Kapton were mounted on a sample holder made for the electron gun, stacked on each other; the Kapton sample closest to the electron gun labeled Kapton 1 and the sample furthest from the electron gun labeled Kapton 2. Due to the placement of the Kapton rounds, Kapton 1 received more irradiation than Kapton 2.



(a) (b) (c)

Figure 3: Optical spectroscopy instrumentation. (Top) Instrumentation block diagrams and sample configuration. (Bottom) Photographs of detectors. (a) Transmission setup, (b) Diffuse reflectivity with integrating sphere, (c) Specular reflectivity with probe

This happens because of the electron path going through the Kapton. Less electrons hit the second sample because they are being absorbed by the first sample.

In this study, 5 samples were looked at optically throughout the experiment; two irradiated Kapton, two unirradiated Kapton, and a glass witness sample. Once the two Kapton samples had been irradiated, transmission data was taken of the two repeatedly. Transmission was also taken of a witness sample made of borosilicate; this witness sample was not irradiated and acted as a control in the study. Transmission was taken of the two one-inch rounds of Kapton that had been baked out alongside the irradiated rounds but had been kept in a Food Saver vacuum instead of being irradiated. These two samples, Unirradiated 1 and Unirradiated 2, were also considered a control. The witness and the two unirradiated samples had transmission data taken right after bake-out, and right before the irradiated samples were taken out of the chamber.

For the first 30 minutes the irradiated samples were out of the chamber, transmission was taken on Kapton 1 only every two minutes. The reasoning behind this is that Kapton 1 had supposedly gotten more irradiation and if most of the relaxation would have happened in the first two hours, we thought it wise to look closely at the sample in this time frame. Only taking transmission of 1 sample instead of 5 allowed the transmission to be taken more quickly and closer together. After 30 minutes, data was taken of all 5 samples every 5 minutes apart, then 10 minutes apart, doubling the time in between each set taken every so often. This allowed us to closely monitor change in the beginning right after irradiation while still looking at the longer-term relaxation, if any.

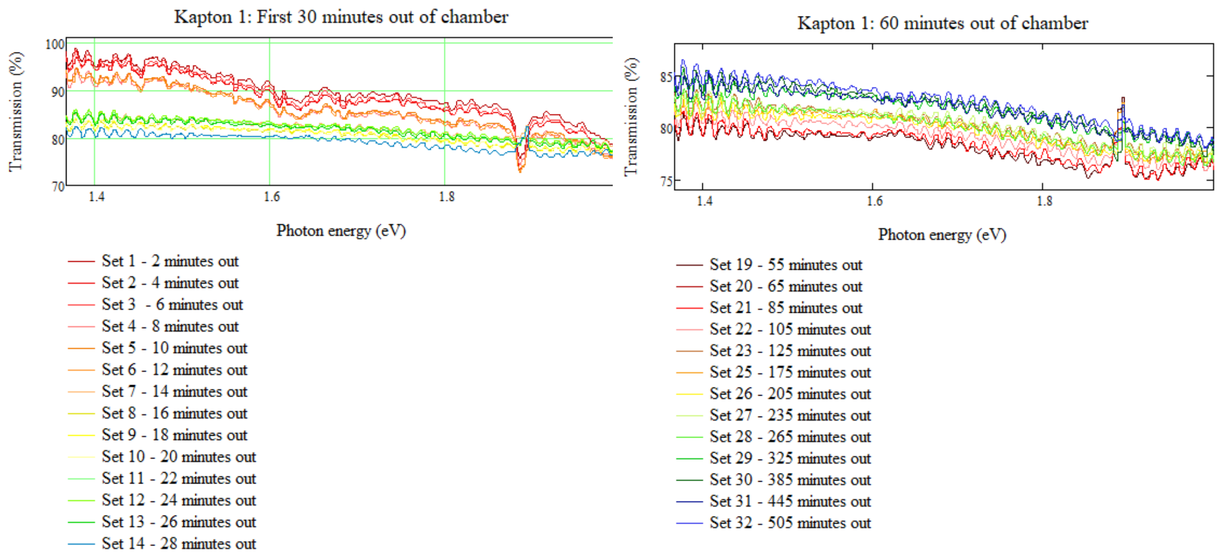


Figure 4: Kapton 1 Data. First 30 minutes, then 60-minute graphs

I decided, once I had done some error analysis, that I should only focus on the low eV range: 1.367 eV to 2 eV. The ranges beyond these created to much error, probably due to the equipment used. This range seemed to be the most accurate.

Results/Data and Analysis

A. All the results were graphed.

The Kapton 1 data was split into two graphs: the data taken in the first 30 minutes the samples was out of the chamber and from 60 minutes after the samples were taken out of the chamber. This allowed a closer look at the samples which were most likely to relax due to oxygen; the data taken in the first 30 minutes. The graphs are arranged by color. The earlier runs are in red, orange, and yellow, where the older runs are done in green, and blue. This gradient allows us to see the data in order of time.

Easily we notice that in the first 30 minutes, the transmission of Kapton 1 seems to start higher, at 1.4 eV the transmission is about 95%, but as time goes on, the transmission at 1.4 eV lowers to about 80%. In the second graph, 60 minutes out of chamber, the transmission seems to increase from 80% to 85%.

Unirradiated 1 and 2 don't seem to have a pattern as had been expected. This makes sense because the unirradiated samples should not be changing due to oxygen exposure.

The witness sample is the same as unirradiated 1 and 2 holding no pattern.

B. Relative Transmission and Error Calculations

First, I looked at the relative change of the witness sample using the equation

$$\frac{Set\# - Before}{Before} \tag{1}$$

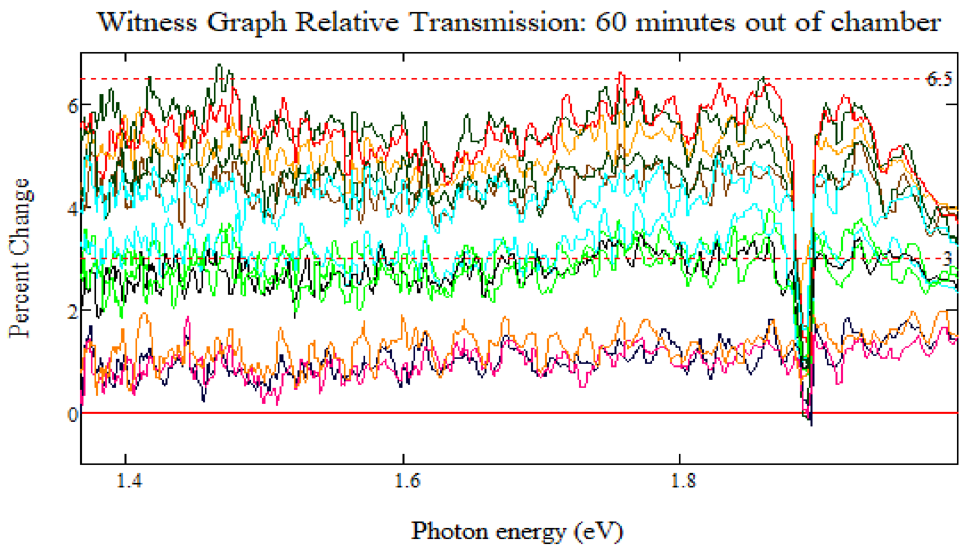


Figure 5: Relative Transmission of Witness Sample

where Before is a set of data taken before the experiment started.

From this I concluded that the instrument error was around 6.5%, each of the data sets from the witness sample fell within 6.5% of each other while no pattern of time emerged. I compared this to the relative transmission of the Unirradiated 1 data and found that error was confirmed.

With this error in mind, I plotted each Kapton 1 graph overlaying the error and the unirradiated data.

As can be seen in the picture, the two red lines indicate the error of the Kapton data, the actual data set would fall directly in the middle of the two. The grey lines indicate the error of the Unirradiated data. This allows us to see what data, of all the data collected, can show something useful. As seen in the last graph, run 8, the red and the grey lines lay on top of one another, but in the first graph, run 1, the Kapton data has a higher percentage than the Unirradiated sample. From this I infer that the Kapton sample gained defects by the irradiation causing the transmission to increase. Over 15 minutes, which samples 1-8 include, the transmission of the irradiated Kapton returned to the previous transmission of a Kapton sample not irradiated. We can see again that the transmission decreases as time goes on, presumably as the defects are repaired by oxygen, in a relative transmission Kapton graph with respect to the Unirradiated sample.

From this graph we can infer, using the same color scheme as before; the colors of the rainbow indicating time, red as the earliest and green as the latest, that the transmission of the initial data set was about 15% (at 1.4 eV) higher than the unirradiated transmission data at the same point. Over time, the Kapton data reverted back within error of the unirradiated data. The data shown in green shades is the later data; this data falls in the range of Error. As shown in figure 9.

Data outside the low eV range indicated similar results as the last graph of figure 7; sets aligned with the unirradiated data.

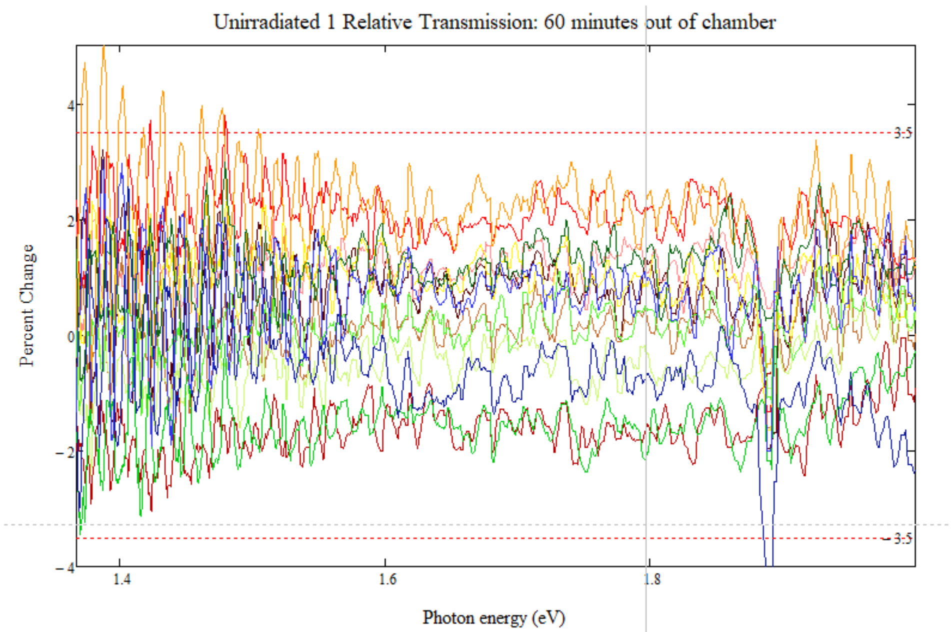


Figure 6: Relative Transmission of Unirradiated 1 compared to the before set

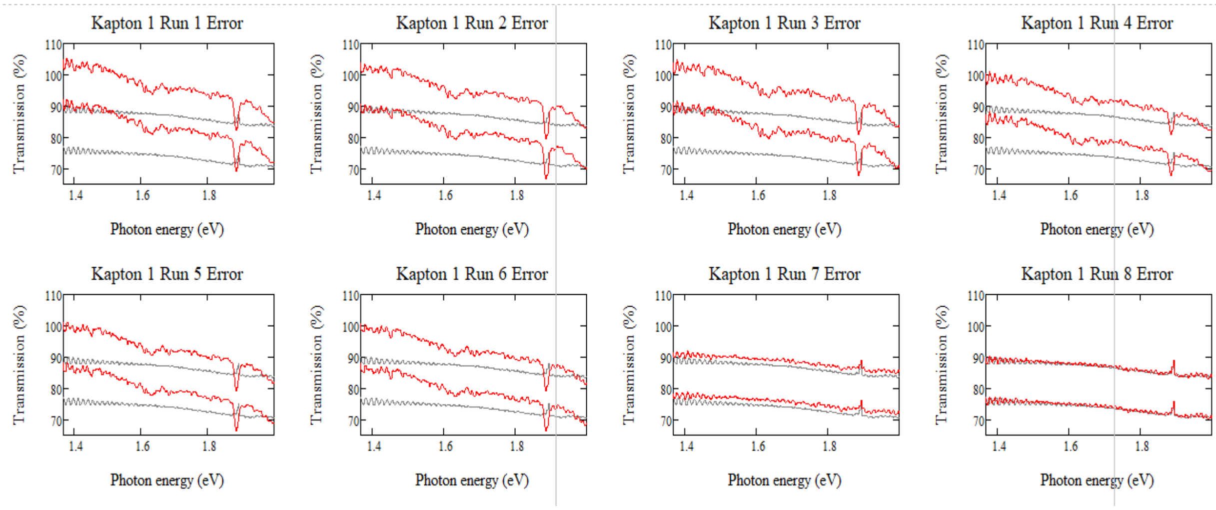


Figure 7: Kapton 1 with Error and respect to Unirradiated data

Conclusion and Future Work

From this project, I conclude that the optical defects in Kapton, created by irradiation, was repaired. The reparation seemed to take around 15 minutes, faster than our previously expected 2 hours.

However, the cause of the reparation is unknown. It is theorized that Oxygen could be the cause, but it could also be time. Another experiment will need to be done to confirm. One hypothesis could be that Argon does not repair the defects created.

Other eV ranges should also be looked at with more sensitive equipment. This will allow a more precise view of the full transmission spectrum of Kapton.

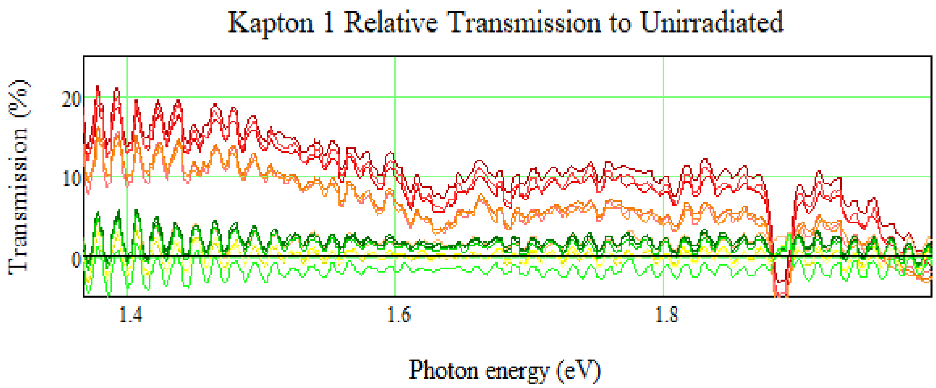


Figure 8: Relative Transmission of Kapton 1 compared to Unirradiated

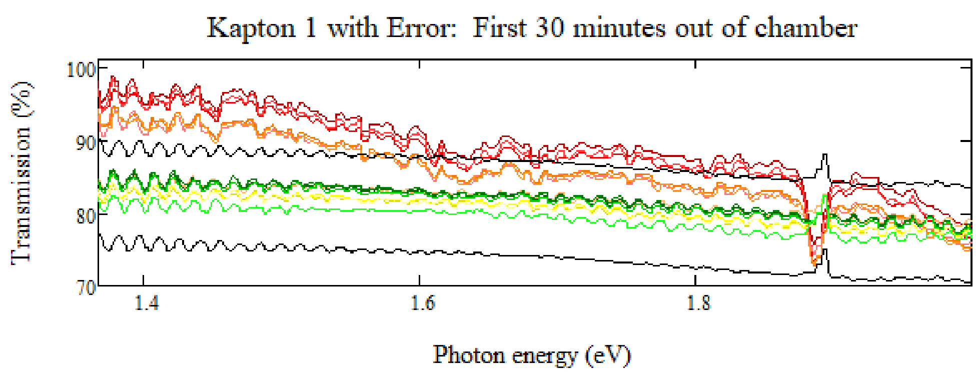


Figure 9: 1 Kapton 1 with Error in black

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[1] Amberly Evans and JR Dennison, "The Effects of Surface Modification on Spacecraft Charging Parameters," *IEEE Trans. on Plasma Sci.*, **40**(2), 291-297 (2012).

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[3] Alexandra Hughlett Nelson, Ashlan Keeler Swainston, and JR Dennison, "Relaxation of Radiation Effects in Polymers," Student Research Symposium, Utah State University, Logan, UT, April 9-10, 2019.

[4] Ashlan Keeler Swainston, and JR Dennison, "Optical Transmission of Radiated Optical Filters," American Physical Society Four Corner Section Meeting, University of Utah, Salt Lake City, UT, October 12-13, 2018.