Thermoluminescence of X- and UV-irradiated PEEK

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Statement of Purpose: Thermoluminescence (TL), also known as thermally stimulated luminescence (TSL), has been applied by many[1] to understand the mechanisms for emission of light or luminescence in polymers. Jahan et al.[2] used TSL to investigate aging of orthopedic polyethylene (ultra-high molecular weight polyethylene (UHMWPE)) following gamma-sterilization in air and subsequent storage in air, liquid N_2 or saline solution. TSL has been primarily attributed to recombination of trapped charges and/or radicals. Thermally stimulated chemiluminescence (TSCL) can also produce luminescence when oxidized species, trapped in the polymer matrix, undergo chemical reaction (in solid state) as a result of heating. In this study, we have detected thermoluminescence in as-received polyetherether PEEK and irradiated PEEK, which is, to our knowledge, the first time this has been done.

Methods: 10 mil. (0.254 mm) Victrex® PEEK film was used for this study. TSL samples, each of size 2x2 mm^2 and mass 1.4-1.6 mg were placed in TSL pans (a DSC pan without top) for testing. TSL measurements were performed using a commercial dosimeter (Harshaw QS 3500) in which the heating chamber was continuously purged with dry, filtered N_2. The samples were heated from 30°C to 300°C at a rate of 1°C /s, and the resulting TSL intensity (glow curve) was recorded for unirradiated PEEK, as well as following UV- and X-irradiations of the PEEK samples. UV-irradiation was performed with a broad-band ultra-violet-visible (UV-Vis) lamp (250 Watt, Oriel® 688 10 Arc Lamp). For X-irradiation, an X-ray source (Scientific America) operating at 50 kV and 45 mA was used. Some samples were heated before TSL measurements; these samples were preheated in air for 10 minutes at specific temperatures between 50°C and 340°C to check the effects of heat upon on the TSL output.

Results: As shown in Figure 1, the curve of unirradiated (as-received) PEEK exhibits a peak at 150°C; TSL peaks disappeared (pink line) after heating, suggesting that the species responsible for the TSL were quenched or annealed. The intensity of the glow curves decreased as a function of temperature in the interval 200-260°C, and disappeared when heated at or near 280°C (green line of figure 1). Although the primary 150°C glow peaks disappeared upon preheating at temperatures from 280°C to 340°C (near the melting temperature of PEEK), there were new peaks which appeared near 75°C (red line of Figure 1). Additional TSL was observed in x-irradiated PEEK near 100°C and 150°C (Figure 2), whereas UV-irradiation (not shown) did not produce TSL at 100°C, but increased near 150°C.

Conclusions: We report thermoluminescence in PEEK (as-received and irradiated), which is, to our knowledge, the first time this has been done. While the TSL is observed in as-received (unirradiated) PEEK, its intensity increases with UV- or X-irradiation. The primary thermoluminescence of PEEK decreases and disappears when heated through melting, but then new thermo-luminescence is detected at near 75°C of the TSL glow curve. Further analysis of TSL glow curves should shed light on the thermally stimulated chemical processes in PEEK.

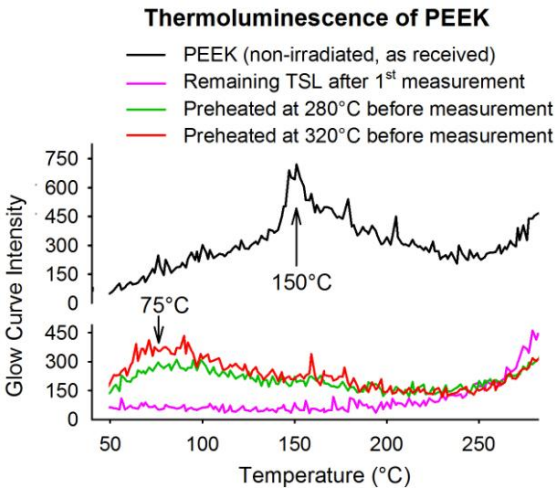

Figure 1. Thermoluminescence (TL) of PEEK. Shown are glow curves of as-received PEEK (black line), a second testing of the same sample (pink line), PEEK samples that were first pre-heated for 10 minutes at 280°C (green line) and 320°C (red line).

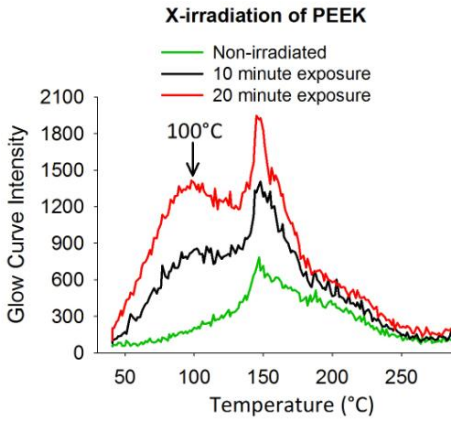

Figure 2. TL of PEEK which was X-irradiated for 10 minutes (black line) and 20 minutes (red line), as compared to as-received PEEK (green line).

References:

1. Partridge R. H. *The Radiation Chemistry of Macromolecules*. Vol. 1, Chap. 10. 1972.
2. Jahan *et al*. *J. Luminescence* 40/41, 242. 1988.