X-irradiated PEEK: Thermoluminescence observations with DSC correlation

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Statement of Purpose: Polyether ether ketone (PEEK) is a linear and highly aromatic semi-crystalline thermoplastic with a variety of implantable medical device applications. While PEEK is generally thought to be radiation resistant, we feel this characteristic needs more exploration. Past research of PEEK is mostly concentrated on the study of the effects of energetic radiation (such as γ-radiation and electron-beam) by using analysis techniques such as FTIR, DSC, and TEM. In this research work, we use the experimental techniques of TSL (thermally stimulated luminescence, also called thermoluminescence) and DSC (differential scanning calorimetry) to study the effects of X-irradiation on PEEK. Similar TSL research with PEEK has not been previously performed to our knowledge to study such effects of X-irradiation. We can advantageously perform X-irradiation treatments in-house and can therefore observe the effects immediately after radiation exposure. Moreover, TSL analysis in combination with DSC can give a better understanding about the material when exposed to X-irradiation.

Methods: PEEK-film (10 mil. (0.254mm)) was cut into appropriate size for TSL and DSC measurements. TSL measurements were carried out using a commercial dosimeter (Harshaw QS 3500). The samples PEEK materials were then heated from 40 to 350°C at a rate of 1°C /s. The resulting TSL with temperature (glow curves) was recorded as a function of temperature. Each of the glow curves were seen to be a composite of multiple curves, and were then deconvoluted into individual glow peaks using PeakFit™ software. The glow peak parameters of the individual peaks were then calculated using TSL Curvefit™ software from Los Almos National Laboratory. DSC measurements were performed with a Dupont 910001-901 differential scanning calorimeter. TSL and DSC were performed before and after radiation treatments.

Results: Before any radiation treatments, TSL data of PEEK shows a major peak at a temperature of about 150°C (glass transition temperature, T_g); this curve can be deconvoluted into six smaller curves which appear at temperatures of 75, 100, 125, 150, 180, and 210°C. After X-irradiation, some of these six smaller curves increase dramatically while others do not. In addition, some peaks decrease with time after X-ray while others remain increased. X-irradiation was performed at six different exposure times (from 10 minutes to 1 hour) to confirm these observations, whose effects were amplified with radiation treatment time. The calculated thermal activation energy associated with these radiation-induced glow peaks ranges between 0.89 eV and 3.2 eV, and the reaction mechanism is estimated to follow a kinetic order of 1.5. From DSC measurements, we were able to determine the corresponding glass transition, crystallization, and melting temperatures (T_m). There was a large exothermic crystallization peak at about 180°C, indicating an increased tendency to crystallize. The glass transition temperature was observed at 150°C and correlated with the 150°C thermoluminescence observations; this and melting temperature did not change significantly with radiation exposure.

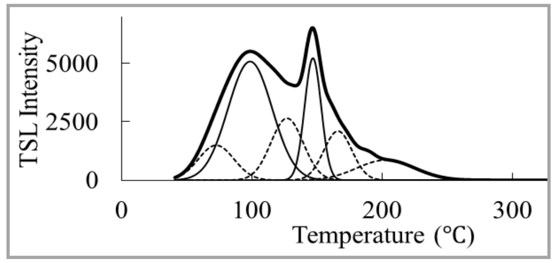

Figure 1. Thermoluminescence of X-irradiated PEEK.

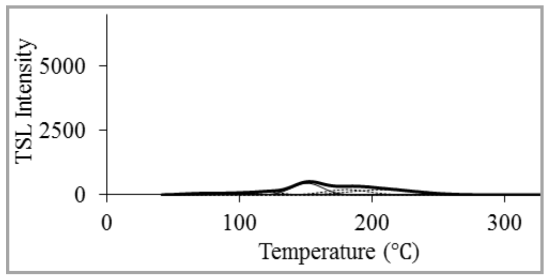

Figure 2. Thermoluminescence of non-irradiated PEEK, same y-scale as to compare with figure 1.

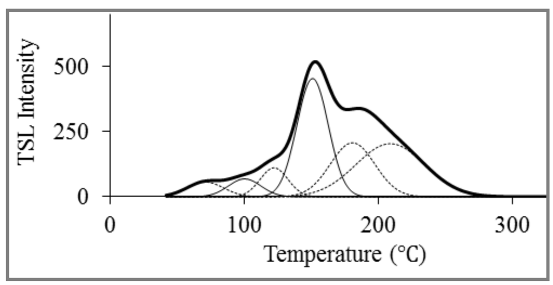

Figure 3. Same as figure 2 (non-irradiated PEEK), zoomed in (y-scale x0.10).

Conclusions: We found that there are some indications that thermoluminescence can be used to further evaluate PEEK, especially in regards to changes in the material resulting from radiation exposure. Through DSC, some possible correlations with TSL data were observed, such as glass transition temperature. Further work in order to correlate thermoluminescence observations with other experimental techniques such as for free radical identification or indications of oxidation would l increase the value of similar thermoluminescence analyses for studying radiation-induced changes in materials such as PEEK, where such radiation effects are otherwise unseen.