

Dynamic Mechanical Studies of Injection Molded and Gamma Sterilized PEEK and PEEK Composites

Kai Zhang, Michael C. Wallick, Dirk L. Pletcher, Hallie E. Brinkerhuff, Michael E. Hawkins.
Corporate Research, Zimmer Inc., Warsaw, IN 46580

Introduction: Poly (ether ether ketone) (PEEK) and PEEK composites have gained attention as high-performance thermoplastic materials for applications in orthopaedic and spinal areas. Implant examples include, but not limited to, the VerSys EPOCH full-coat hip stem (Zimmer Inc.) and the BAK™ Vista™ Interbody Fusion System (Zimmer Spine). During the processing of such implants and other medical devices such as instruments and provisional products, the PEEK and its composites need to be sterilized. Although the effects of irradiation sterilization on PEEK materials have been studied,¹ the effects of irradiation sterilization on PEEK composites, and especially on the dynamic mechanical properties of PEEK and PEEK composites, has not been fully investigated.

The goal of this study is to investigate the dynamic mechanical properties of PEEK and PEEK composites before and after gamma irradiation sterilization. The current study may provide a method to evaluate materials, and investigate their stability and dynamic performance for various potential clinical applications.

Materials and Methods: PEEK (GATONE 5300, Gharda Inc.), 30 wt% glass fiber-filled PEEK (PEEK-GF, GATONE 5330GF, Gharda Inc.) and 30 wt% carbon fiber-filled PEEK (PEEK-CF, GATONE 5330CF, Gharda Inc.) were injection molded to rectangular testing specimens with a dimension of 50 by 5 by 1 mm. The molded specimens were subjected to gamma sterilization with a dose range between 27.6 and 34.7 kGy. Following ASTM D5023-01 and E1640-04, a dynamic mechanical analyzer (DMA, TA Instruments Inc. Model 2980) was used to conduct frequency (0.1-100 Hz at 37°C) and temperature sweeps (35 – 200 °C at 1 Hz) with a 3-point bending setup. At least five specimens were tested for each material (PEEK, PEEK-GF and PEEK-CF) and condition (before and after sterilization). Storage modulus (E') and loss modulus (E'') were measured, and the glass transition temperature (T_g) was determined by the peak position of E'' during temperature sweeps. Statistical analyses of data were performed using a student t -test with level of significance of $p < 0.05$.

Results and Discussion: Typical DMA scans of PEEK-CF before and after gamma sterilization are plotted in Figure 1. The results generalized in Table I show: (1) Before sterilization, as compared to the T_g of PEEK, the T_g of PEEK-GF and PEEK-CF are not dramatically different. (2) After sterilization, the storage moduli of PEEK, PEEK-GF and PEEK-CF composites at 1Hz and 37°C during frequency sweeps are not statistically

different from those before sterilization. In addition, gamma sterilization does not significantly affect the T_g .

The results generated by DMA are likely related to the following two factors: (1) interactions between fibers and PEEK matrix before and after sterilization; and (2) competition between polymer chain scission and crosslinking after gamma sterilization.

This study suggested that the given dose of gamma irradiation does not change the dynamic mechanical properties of PEEK and composites. Future work will investigate the activation energies of the molded PEEK and composites using DMA.

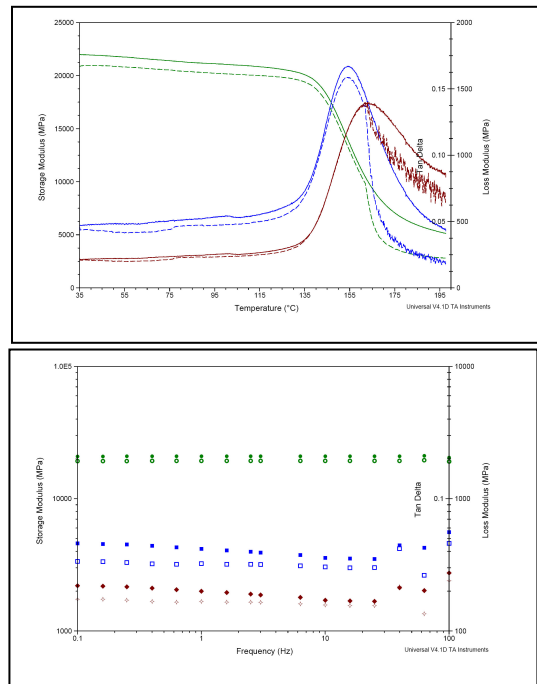


Figure 1. Representative DMA results of PEEK-CF for temperature (upper) and frequency sweeps, before (solid symbols) and after (hollow symbols) gamma sterilization.

Table I. Summary of DMA Results (Average \pm S.D.)

Sample	T_g (°C)	E' (MPa, 1 Hz, 37°C)
PEEK	155.6 \pm 0.3	4069 \pm 332
PEEK-GF	158.3 \pm 0.7	10681 \pm 386
PEEK-CF	154.9 \pm 0.5	21185 \pm 1421
Gamma PEEK	156.5 \pm 0.4	4141 \pm 164
Gamma PEEK-GF	153.4 \pm 3.3	10230 \pm 1490
Gamma PEEK-CF	153.9 \pm 0.5	21447 \pm 1326

Reference

1. Vaughan AS and Stevens GC. Polymer. 2001;42:8891.