Degradation of Carbon Fiber Composites by Repeated Sterilization

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Statement of Purpose: This study evaluated the effect of high cycles of repeated steam autoclave sterilization on the flexural strength of carbon fiber reinforced composite materials. Elevated sterilization temperature and steam exposure is a harsh environment causing complex and synergistic degradation modes, and the effects of this exposure are important to understand not only for material selection but also to identify or even prolong the expected service life of a part. This entails consideration of continuous damage accumulation by many contributing factors such as hygrothermal (combined moisture and temperature) effects, polymer oxidation, matrix cracking, and microstructural changes. It is generally understood that polymeric composite properties are significantly affected by the elements of this study – high temperature exposure, thermal cycling, and moisture absorption. The degree to which composites are affected depends on the matrix, reinforcement, and fiber-matrix interface [1]. This study stemmed from a need to identify suitable composite materials for a surgical instrument subjected to repeated hospital steam autoclave sterilization. Initial interest focused on the high performance thermoplastic composites PEEK, PPS, and PEI because they have good environmental and moisture resistance [1-3], and also because PEEK and PPS are already used in medical devices. The objectives of this study include 1) learning more about the structural degradation of materials deemed suitable for steam sterilization following hygrothermal cycling, 2) identifying if material degradation plateaus at a certain point, and 3) assessing the effect of conditioning on composite part surface appearance.

Methods: The effects of repeated steam sterilization were evaluated for four continuous carbon fiber reinforced composite laminates chosen as candidate materials for a surgical instrument product design. The resin systems tested include polyetheretherketone (PEEK), polyphenylene sulfide (PPS), polyether imide (PEI) and epoxy. In this study, composite laminates were subjected to hygrothermal conditioning in a steam environment and the effects were evaluated mechanically and visually. Flexural strength and modulus, as well as changes in coupon weight were evaluated before and after cycling at 0, 200, 400, 600, 800, and 1000 cycle intervals. Photomicrographs were also acquired at these intervals to better understand the mechanism of degradation for each composite laminate.

Results: As shown in Figure 1, the flexural strength follows a significantly decreasing trend for all materials. However, the effect of cycling on flexural strength is least pronounced for C/Epoxy. There was no apparent plateau for any of the four materials tested. All materials also experienced a slight reduction in flexural modulus. After the initial reduction in flexural modulus, values remained steady from about 400 cycles to completion for all material groups. Overall, the average flexural modulus dropped to 90 % of baseline for C/PEEK; 93 % for C/PPS; 92 % for C/PEI; and 90 % for C/Epoxy. The typical exposure effects for all materials were primarily surface oxidation and transverse ply cracking, as observed in photomicrographs.

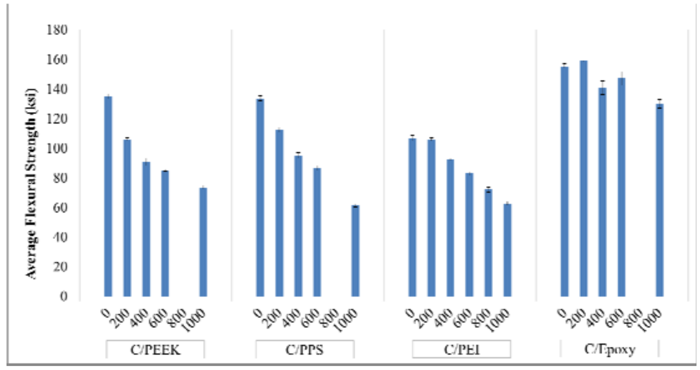

Figure 1. Flexural Strength at 200 Cycle Intervals

Conclusions: All materials experienced a significant reduction in flexural strength and a slight reduction in flexural modulus over the course of 1000 cycles. The typical exposure effects for all materials were primarily surface oxidation and transverse ply cracking. The visibly observable damage in cross-sectional images of aged C/PEEK and C/Epoxy was low. Thus it can be concluded that there are other unknown factors dominating the failure of these materials. The fact that modulus was hardly affected implies that the primary cause of strength reduction was matrix cracking and interfacial debonding rather than resin degradation. It is also worth mentioning that C/PEEK and C/PPS did not show noticeable surface degradation before failure, as opposed to C/PEI and C/Epoxy which were visibly cracked and discolored with increasing cycling. These C/PEEK and C/PPS woven composites are suitable candidates for surgical instrumentation if the service life is expected to be closer to 600 cycles and maintaining an aesthetic surface appearance is desired. C/Epoxy and C/PEI are suited to a longer service life, but are likely to be retired sooner due to color change and degraded surface appearance. This study shows that laminate materials cannot be expected to perform the same as unreinforced or short fiber reinforced materials deemed acceptable in hot/wet environments such as steam sterilization. Future studies could be used to evaluate the effects of specialty thermoplastic-sized carbon fibers, edge sealing, and stacking sequence on rate of hygrothermal degradation.

References:

1. Mensitieri G. J Appl Poly Sci. 1989 37:381-92.
2. Tai NH. Composites: Part B. 1999 30:849-65.
3. McKeen, L. The effect of sterilization on plastics and elastomers, 3rd Ed. Oxford: Elsevier, 2012:282-8