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Experimental Stress Analysis

Sterilization Decomposition Evaluation of Radiolucent Composite Materials for Use in Medicine

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Abstract: This study evaluates the resistance against multiple sterilization processes of radiolucent composite materials for use in medicine as an intraoperative imaging device material. The composite materials under investigation here are composed of carbon fibers, polyphenylene sulfide and polyetheretherketone matrix. The influence of multiple sterilization processes on changes in their mechanical and structural properties are determined. The radiolucency of the proposed composites is verified.

Keywords: Composite material, Sterilization Decomposition, Radiolucency

1. Introduction

Radiolucent composite materials have superior properties to insufficiently radiolucent metal alloys and unreinforced polymers with poor mechanical properties. Their use as medical device materials requires an understanding of the micromechanical properties that provisionally define their behaviour. Sterilization is a mandatory process for such materials used in a range of medical applications, e.g., intraoperative guides, screening equipment accessories and patient support systems. The steam or dry heat sterilization processes widely employed in medical practice can affect the micromechanical properties of polymeric composites, particularly in the interface region between the polymer matrix and the reinforcing fibers. However, the effect of sterilization processes on the properties of materials used in medical devices is often ignored [1]. The structural integrity and the overall performance of fiber reinforced polymer composites are strongly influenced by the stability of the fiber/polymer interfacial region. Absorption of moisture causes dilational

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expansion and induces stresses associated with moisture-induced expansion, which degrade the structural stability [2 - 7]. This may induce plastic deformation by plasticization or differential strains [2]. These effects may greatly alter the physical, chemical and mechanical properties of the material at different scales [8]. This results in a significant mismatch in moisture-induced volumetric expansion between the matrix and the fibers, and leads to the evolution of localized stress and strain fields in fibrous composites [8]. Several variables affect the performance of composite materials, including matrix, reinforcement, manufacturing method and reinforcement orientation. It is necessary to investigate both microscopic and macroscopic changes in mechanical and structural properties due to the sterilization processes that are employed. The aim of this study was to prepare an advanced composite material with suitable mechanical and radiolucent properties, together with structural stability, after repeated sterilization by widely-used techniques.

2. Materials and Methods

Composites based on carbon T300 fibers (plain weave fabrics, Toray, Japan) and/or polyetheretherketone (Porcher Industrie, France) and polyphenylenesulfide (TenCate, Holland) were prepared. T300/ polyetheretherketone (PEEK) was cured under a pressure of 0.08 MPa at 395 °C. T300/ polyphenylene sulfide (PPS) was cured under a pressure of 1.0 MPa at 310 °C.

The mechanical properties were measured before sterilization (A), after 1 sterilization process period (B1), and after 30 (B30) sterilization process periods. An autoclave (Sterident, Prodenta, CZ) for steam sterilization (134 °C, 304 kPa, 10 min) was used for this purpose. The ultimate strength in bending and the modulus of elasticity in bending in the direction of the fiber axis were determined with a four-point and three-point bending set-up using the Inspekt 100 HT material tester (Hagewald & Peschke, Germany), in accordance with ISO 14125.

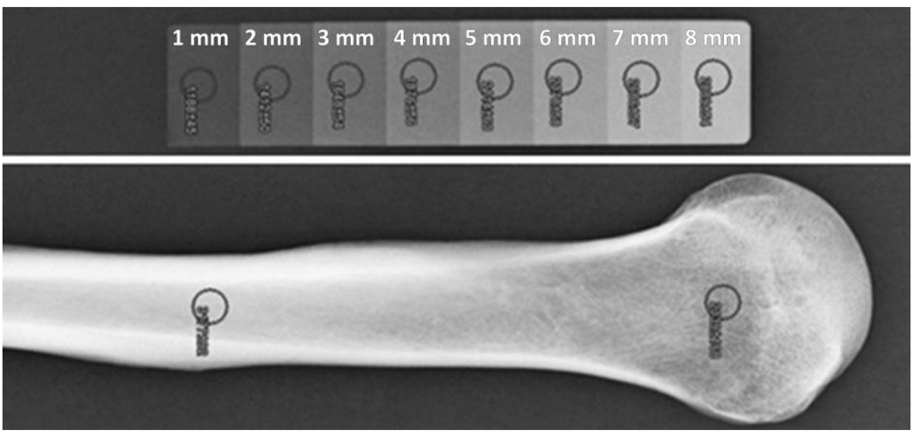

Fig. 1. An example of the optical densities of the X-ray film measurements

The intensity of an X-ray transmitted through the studied composites was measured (80 kV, 2 mA, 1000 ms). On the basis of the Beer-Lambert law, the transmissivity was calculated for each sample. Finally, the linear absorption coefficients (μ [mm^{-1}]) were calculated. Since the absorption coefficient is not influenced by sample thickness (it is influenced mainly by the material properties), it was possible to assess and compare the radiolucency of each studied material. The transmissivity was measured by Shado-o-BoxTM 4K (Rad-icon Imaging, USA), and the ISOVOLT 420/5 X-ray Tubehousing (Agfa NDT

Pantak Seifert, Germany) was used as an X-ray source. With a view to making a comparison with the real environment (radiolucency of human bone), a control material was added to the analyses that were performed. An aluminum control sample was used. An X-ray analysis of the Al sample was performed, and the transparency of its different thicknesses was compared with the transparency of the human humerus (see Fig. 1). A densitometer for single point measurements of the optical densities of the X-ray films (Densoquick 2, PEHA, Germany) was used for this purpose. Al control sample thicknesses up to 3 mm displayed suitable radiolucency, and the highest rate was displayed by a part 1 mm in thickness. In order to assess the influence of multiple sterilization processes on the inner structure of the composites, an image analysis of the polished sections was performed using NIS-Elements AR software, ver. 2.30 (Laboratory Imaging, Inc., Czech Republic).

3. Results and Discussion

The flexural properties after multiple sterilizations were tested and compared with those of the corresponding unsterilized samples (Figs. 2 and 3). The modulus of elasticity in bending is influenced by multiple sterilizations only in the case of T300/PEEK composite (PEEK). The inexpressive decrease in the modulus is equal to approx. 3 - 4% after 30 sterilization cycles. In the case of ultimate strength in bending, no decrease was observed. From this point of view, we can state that no weakening of the reinforcement-matrix bond occurred.

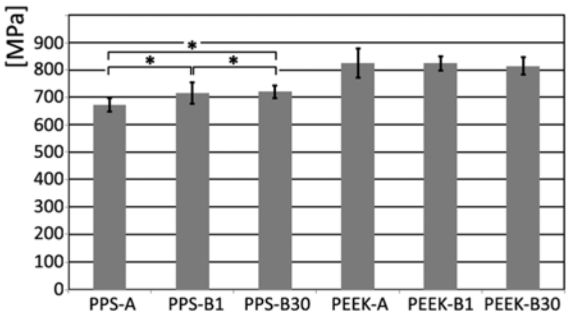

Fig. 2. The ultimate strength in bending (* denotes statistically significant differences, Newman-Keuls post-hoc test, $\alpha = 0.05$)

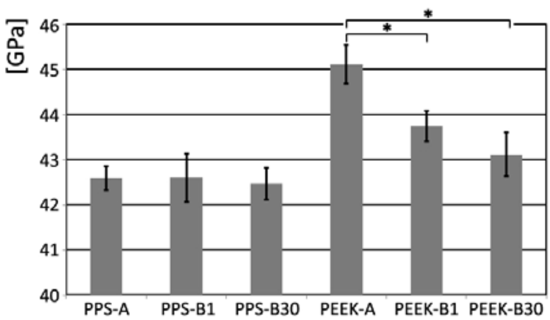

Fig. 3. The modulus of elasticity in bending (* denotes statistically significant differences, Mann-Whitney post-hoc test, $\alpha = 0.05$)

The influence of multiple sterilization processes on changes in the structural integrity of the composites was studied by an image analysis of cross sections. From the point of view of image analysis, we can state that no weakening of the reinforcement-matrix bond occurred (for illustration, see Figs. 4 and 5).

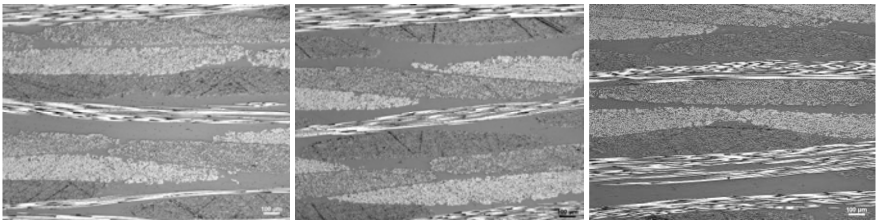

Fig. 4. Micrographs of polished sections of T300/PPS composites (from left: A, B1, B30)

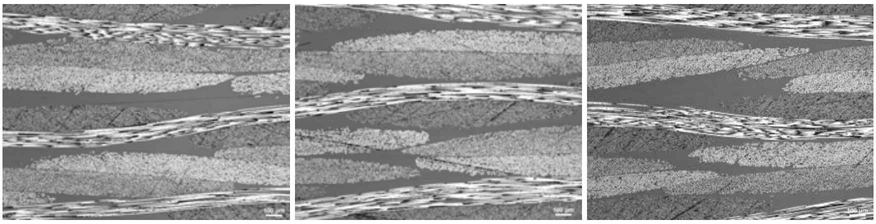

Fig. 5. Micrographs of polished sections of T300/PEEK composites (from left: A, B1, B30)

The X-ray analysis results are summarized in Fig. 6. Both composites are sufficiently radiolucent, and both displayed lower linear absorption coefficient values than the value for the Al control sample.

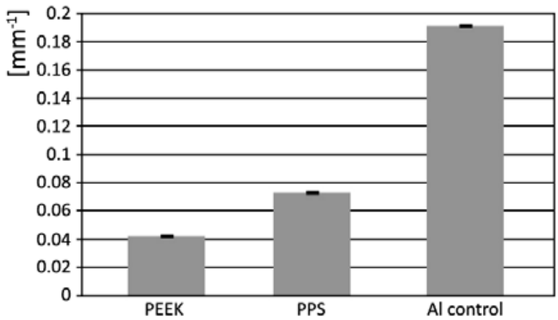

Fig. 6. Linear absorption (all values display statistically significant differences, Mann-Whitney post-hoc test, $\alpha = 0.05$)

4. Conclusions

On the basis of our analyses, we can state that both PEEK and PPS composites are good candidates for application as radiolucent materials providing resistance against sterilization decomposition. Before they are presented for application, it will be necessary to increase the number of applied sterilization processes and perform further analyses of physical properties.

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