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The Influence of Steam Sterilization Processes on the Micro- and Macromechanical Properties of Polyamide Fiber-Reinforced PDMS Composites for Medical Device Applications

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Abstract: The effect of multiple steam sterilization processes on the structural integrity of the polymer matrix composite polydimethylsiloxane (PDMS) reinforced with polyamide fibers (Aramid) is investigated by 3- and 4-point bending tests and by nanoindentation. The macromechanical properties of studied composite are strongly influenced by the sterilization technique that is applied. The nanoindentation tests help to explore microscopic changes in the reduced stiffness of the interphase region.

Keywords: Composite material; Sterilization; Nanoindentation

1. Introduction

Radiolucent composite materials have superior properties to insufficiently radiolucent metal alloys and unreinforced polymers with poor mechanical properties. Sterilization is a mandatory process for such materials used in a range of medical applications, e.g., intraoperative guides or screening equipment accessories. The steam sterilization processes widely employed in medical practice can affect the mechanical properties of polymeric composites. However, the effect

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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 dilatational expansion and induces stresses associated with moisture-induced expansion, which degrade the structural stability [2]. These effects may greatly alter the physical, chemical and mechanical properties of the material at different scales [3]. In this study, the effect of multiple steam sterilization processes on the structural integrity of the polymer matrix composite polydimethylsiloxane (PDMS) reinforced with polyamide fibers (Aramid) was investigated by 3- and 4-point bending tests and by nanoindentation.

2. Materials and methods

A composite material was prepared on the basis of Aramid balanced fabric (Aramid) reinforcement (HM 215 fibers, Hexcel, France) and PDMS matrix (Lukosil M130, Lučební závody Kolín, Czech Republic). The volume fraction of the fabric was kept at 55 vol. %. First, Aramide fabrics were impregnated with PDMS matrix precursor, after which they were cut into pieces of appropriate size for the curing mould after 24 hours (left in an air atmosphere at room temperature). The impregnated layers were placed into the curing mould (each layer has the same orientation of the warp, with ply direction (0°) and the fill weft, with ply direction (90°)). The green composite was cured under a pressure of 1.1 MPa at 225 °C in an air atmosphere for 4.5 hours and postcured under a pressure of 1.1 MPa at 250 °C for 4 hours in an air atmosphere. The preparation process was chosen on the basis of previous studies of composites based on PDMS matrix [4]. Finally, the cured composite was cut into test samples with dimensions of (3x8x50 mm).

The mechanical properties were measured before sterilization, after 1 sterilization process period, after 30 sterilization process periods, and after 100 sterilization process periods. An autoclave (Sterident, Prodenta, Czech Republic) for steam sterilization (134°C, 304 kPa, 10 min) was used for this purpose. After sterilization, a cross-section area (3x8 mm) of the specimens was polished according to the polishing protocol by Godara et al. [2]. The assessment of multiple sterilization cycles on the mechanical properties of the composites (reduced elastic modulus E_r [GPa]) was studied using the Hysitron TriboIndenter™ TI 950 nanomechanical instrument (Hysitron, USA). A Berkovich diamond fluid tip with tip angle 142.3° was used. For each tested composite, indents were applied on three 25x25 μm areas where fibers were perpendicular to the polished surface as a matrix of 5x5 indents with 5 μm separation (with 150 μN applied force, preload 1μN). Further, the ultimate strength in bending with a three-point bending set-up and the modulus of elasticity in bending with a four-point bending set-up were determined using the Inspekt 100 HT material tester (Hagewald & Peschke, Germany), in accordance with ISO 14125. The statistically significant differences were checked by nonparametric methods (the Mann-Whitney post hoc test) and the confidence intervals for the mean values were calculated at a significance level of α=0.05 (STATGRAPHICS Centurion XV, StatPoint, USA).

3. Results and discussion

The flexural properties after multiple sterilizations were tested and compared with those of the corresponding unsterilized samples (Fig. 1). The modulus of elasticity in bending is influenced by multiple sterilizations; the expressive statistically significant decrease in the modulus is equal to approx. 60% after 100 sterilization cycles. Also decrease in ultimate strength in bending can be observed; the expressive statistically significant decrease in the strength is equal to approx. 60% after 100 sterilization cycles.

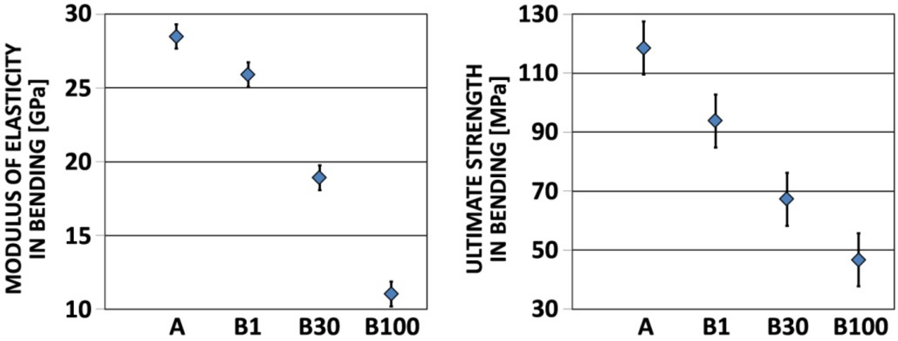

Fig. 1. Modulus of elasticity and ultimate strength in bending of Aramid/PDMS composite before (A), after 1 sterilization cycle (B1), after 30 sterilization cycles (B30) and after 100 sterilization cycles (B100). All values display statistically significant differences, $\alpha = 0.05$.

Representative reduced elastic modulus–contact depth relationships obtained from the indentation tests performed at a maximum peak load of 150 μN for the Aramid/PDMS specimen (0 and 100 sterilization cycles) are illustrated in Fig. 2. In this figure, the three different regions (fiber, interphase zone and bulk matrix) are schematically indicated according to the indentation depth values. It was possible to investigate the matrix/fiber interphase region. The zones identified as interphase regions show different mechanical property values after the application of the sterilization cycles. The 100 times steam-treated Aramid/PDMS specimens show a trend toward a reduced elastic modulus value, indicating a modification in the properties of the PDMS matrix in this region. This decrease can be estimated to be equal to approx. 40% in comparison with the untreated samples.

The mechanical performance of fiber-reinforced composites depends primarily on the mechanical properties of their basic constituents, the chemical stability of the matrix, and the effectiveness of the bond between matrix and fibers in transferring the stress across the interface [2]. Nanoindentation analyses verified the findings of an assessment of the impact of multiple sterilization processes on the inner structure of composites carried out at the macroscale by flexural tests. On the basis of the nanoindentation results we can support these findings that the decrease in both the modulus and the strength of the Aramid/PDMS composite may show not tight and hydrolytically unstable bond between matrix and reinforcement.

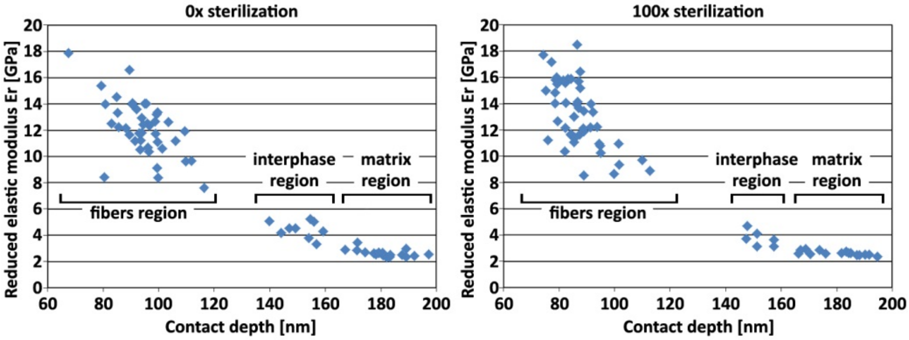

Fig. 2. Some representative indentation responses showing the reduced elastic modulus of the Aramid/PDMS composite sample (no sterilization treatment) and for the steam-treated specimens (after 100 sterilization cycles).

4. Conclusions

This paper has investigated the effect of 1, 30 and 100 steam sterilization cycles in an autoclave that is widely used in medical practice. The aim was to verify the possible influence on the mechanical performance of Aramid/PDMS composite. It has been shown that the mechanical properties are strongly influenced by steam sterilization. The decrease in the modulus of elasticity in bending and in the ultimate strength in bending was shown to be equal to approx. 60%. The results of nanoindentation analysis provided an assessment of the influence of multiple sterilization processes on the inner structure. The steam-treated specimens show a trend toward a reduced elastic modulus value in the fiber/matrix interphase region indicating weakening of the bond between fibers and matrix after application of multiple sterilization cycles.

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References

[1] Block S.S., *Disinfection, Sterilization, and Preservation* (Lea and Febiger, Philadelphia, 1991). ISBN 0-683-30740-1.

[2] Godara A., Raabe D., Green S., "The influence of sterilization processes on the micromechanical properties of carbon fiber-reinforced PEEK composites for bone implant applications," *Acta Biomaterialia*, **3**(2), pp. 209-220 (2007). ISSN 1742-7061.

[3] Zheng Q., Morgan R.J., "Synergistic thermal-moisture damage mechanisms of epoxies and their carbon fiber composites," *Journal of Composite Materials*, **27**(14), pp. 1465-1478 (1993). ISSN 0021-9983.

[4] Suchý T., Balík K., Černý M., Sochor M., Hulejová M., Pešáková V., Fenclová M., "A composite based on glass fibers and siloxane matrix as a bone replacement," *Ceramics-Silikáty*, **52**(1), pp. 29-36 (2007). ISSN 0862-5468.