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Cyclic stress-strain behavior of polymeric nonwoven structures for the use as artificial leaflet material for transcatheter heart valve prostheses

Abstract: Xenogenic leaflet material, bovine and porcine pericardium, is widely used for the fabrication of surgically implanted and transcatheter heart valve prostheses. As a biological material, long term durability of pericardium is limited due to calcification, degeneration and homogeneity. Therefore, polymeric materials represent a promising approach for a next generation of artificial heart valve leaflets with improved durability. Within the current study we analyzed the mechanical performance of polymeric structures based on elastomeric materials. Polymeric cast films were prepared and nonwovens were manufactured in an electrospinning process. Analysis of cyclic stress-strain behavior was performed, using a universal testing machine. The uniaxial cyclic tensile experiments of the elastomeric samples yielded a non-linear elastic response due to viscoelastic behavior with hysteresis. Equilibrium of stress-strain curves was found after a specific number of cycles, for cast films and nonwovens, respectively. In conclusion, preconditioning was found obligatory for the evaluation of the mechanical performance of polymeric materials for the use as artificial leaflet material for heart valve prostheses.

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1 Introduction

The clinical introduction of minimally invasive implantable heart valve prostheses in 2002 had a significant influence on the treatment strategy of severe symptomatic aortic valve stenosis [1]. Current devices are based on xenogenic leaflet material associated with a tendency to calcification resulting in limitations with regard to durability. Furthermore, homogeneity and processability issues are limiting the success of pericardium as leaflet material for heart valve prostheses.

Polymeric nanofiber matrices yield desirable properties and can be manufactured with high reproducibility. Polyurethanes, in particular, have been applied in view of their relatively low thrombogenicity and good blood compatibility characteristics [2]. Thermoplastic polyurethanes are randomly segmented copolymers composed of hard and soft segments forming a two-phase microstructure [4-6]. The presence of hard domains in segmented polyurethanes is very important regarding mechanical properties. At ambient conditions, soft domains are above glass transition temperature polyurethanes yield rubber-like behaviour [6].

Within the current work the viscoelastic responses of thermoplastic silicone-based polyurethane (TSPU) in uniaxial cyclic tensile tests was investigated. TSPU specimens were fabricated using electrospinning. The stress-strain behaviour was investigated and compared with TSPU cast films.

2 Materials and methods

2.1 Manufacturing of polymer samples

A 10% (w/w) solution of the TSPU was prepared in a 1:1 mixture of tetrahydrofuran and N,N-dimethylformamide. The elastomeric nonwoven specimens were manufactured using a needle electrospinning process (4Spin® C4S LAB2 of Contipro, Dolní Dobrouč, Czech Republic) with a rotating celindrical collector.

Cast films were prepared by solving 1.0 g polymer in 25 ml tetrahydrofuran and casting the solutions into Petri dishes. The films were dried for 48 h at room temperature.

Standard dumbbell-shaped samples according to DIN ISO 527 (type 1BB) were prepared by die cutting electrospun nonwoven specimens and cast film samples, respectively.

2.2 Uniaxial cyclic tensile testing

Analysis of cyclic stress-strain behavior was performed, using a universal testing machine (Zwicki ZN 2.5, Zwick GmbH & Co. KG, Ulm, Germany) and a 50 N load cell. Using a crosshead speed of 25 mm/min, samples were elongated up to 50 % nominal strain. After 25 cycles, samples were ultimately torn. During the cyclic tests the samples were immersed in physiological saline solution at a temperature of 37°C.

Tensile force as a function of elongation before and after cyclic loading/unloading was measured. Energy dissipation values of the samples were calculated on the basis of stress-strain curves for each cycle. The difference between the area below the loading (A_l) and unloading curve (A_u) is a measure of the conditioning progress (K) and was calculated as:

$$K = \frac{A_l - A_u}{A_l} \cdot 100 \qquad (1)$$

Furthermore, the remaining strain (ε_R) and the ultimate tensile strength (σ_M) were extracted before and after conditioning. Short time stress relaxation tests were performed using a strain of 50 %. Constant strain was applied for 1000 s before samples were unloaded and ultimately loaded up to the break [7]. A sample size of n = 3 was analyzed, respectively.

3 Results and discussion

3.1 Cyclic tensile tests

Examplary stress-strain curves obtained from the cast film (a) and nonwoven specimens of TSPU (b) are shown in **Figure 1**. The material behaviour during the first, second and 25th cycle is depicted, exemplarily. The first loading curves show an initially stiff response, more compliant behaviour at a strain of about 20 %. The unloading paths show a large hysteresis loop with a remaining strain, respectively. Both materials show pronounced hysteresis, based on stress softening due to the Mullins effect. Therefore, during second loading to a specific strain a material follows a stress-strain path closer to the previous unloading path than to the first loading path.

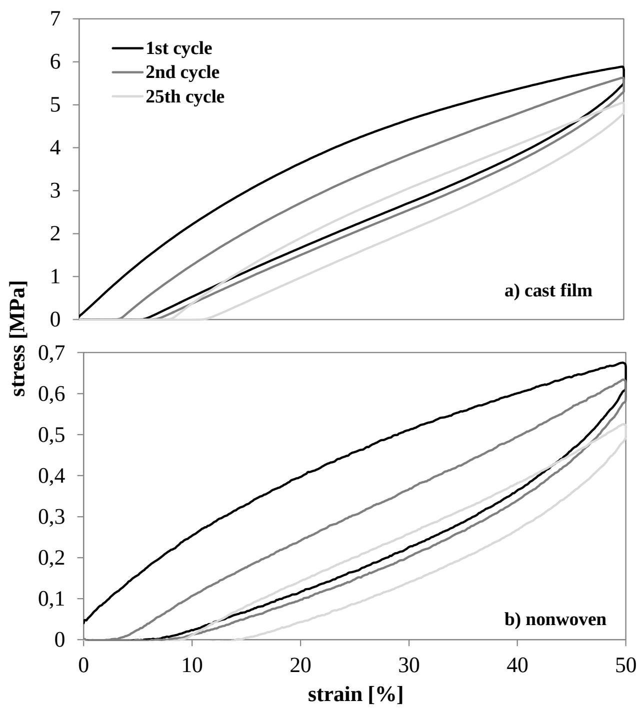

Figure 1: Representative stress-strain curves obtained in cyclic tensile testing. (a) cast film and electrospun nonwoven specimens of TSPU

Due to the specific microstructure of the analyzed samples, the maximum stress of the cast film is tenfold higher compared to the nonwoven material.

Figure 2 shows the remaining strain (ε_R) as a function of the number of cycles for nonwoven specimens and cast films of TSPU. The curves demonstrate that the amount of the remaining strain is greatest after the first cycle. A remaining strain of $\varepsilon_R < 1$ % was found after the third cycle for the nonwoven specimens and cast films of TSPU, respectively. Therefore, the microstructure of the nonwoven has no influence on the remaining strain.

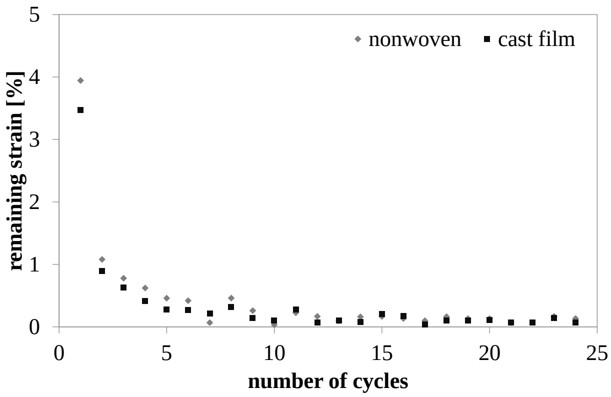

Figure 2: Remaining strain ε_R vs. number of cycles of electrospun nonwoven specimens compared to cast films of TSPU (n = 3).

Figure 3 depicts the decay of the mechanical conditioning as a function of the number of cycles. Most deformation is already completed after the first cycle. Equilibrium of the mechanical conditioning was observed after 5 cycles at $K_{equ., nonwoven}$ = 40 % and at $K_{equ., cast film}$ = 28 % for the nonwoven specimens and the cast films of TSPU, respectively. Prior tests with porcine pericardium yield a mechanical conditioning of $K_{equ., pericardium}$ = 0 %.

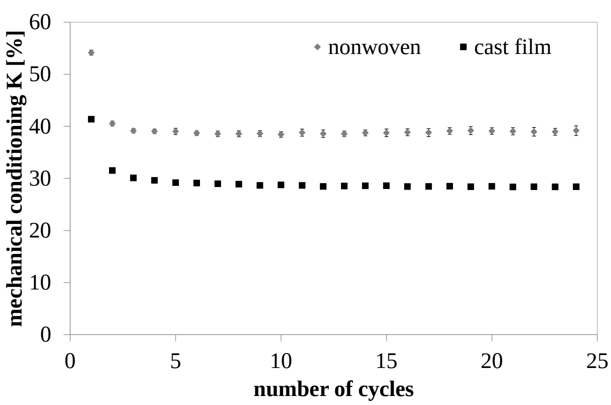

Figure 3: Mechanical conditioning behaviour of the electrospun nonwoven specimens compared to the cast films of TSPU (n = 3)

The mechanical properties of the electrospun nonwoven specimens and the cast films of TSPU without conditioning and after 25 loading/unloading cycles are summarized in **Table 1**. While the elongation at break of the nonwoven material was similar before and after conditioning, ε_R of the TSPU cast film showed a two-fold increase after conditioning.

Table 1: Mechanical properties without conditioning and after 25 cycles loading/unloading of electrospun nonwoven specimens compared to cast films of TSPU.

	without conditioning		**after conditioning**	
	ε_R [%]	**σ_M [MPa]**	**ε_R [%]**	**σ_M [MPa]**
nonwoven	430 ± 15	2.2 ± 0.5	440 ± 10	1.98 ± 0.21
cast film	600 ± 19	32.5 ± 1.4	1150 ± 15	38.5 ± 2.2

A slight increase of the ultimate tensile stress σ_M could be observed after preconditioning of the TSPU cast film. The nonwoven samples showed no changes in σ_M.

3.2 Stress relaxation tests

Figure 5 shows the stress relaxation as a function of time for both TSPU materials without conditioning and after 25 loading/unloading cycles. The data show, that the maximum stress of the TSPU cast film is ten times higher compared to the nonwoven specimens caused by the structure of the material.

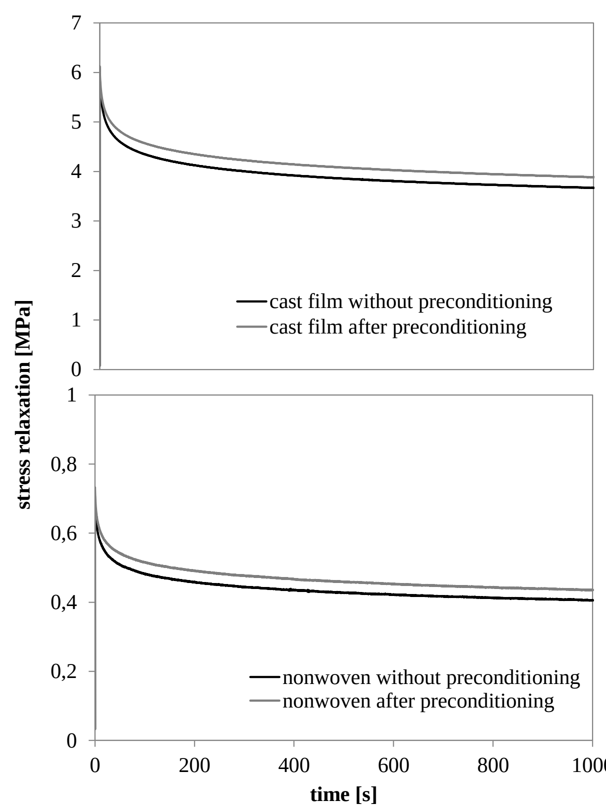

Figure 4: Stress relaxation behaviour of the electrospun nonwoven specimens compared to the cast films of TSPU without preconditioning and after 25 loading/unloading cycles, respectively

The relaxation of stress at relatively short times accurately follows the same way. After preconditioning the maximum stress is about 5 % higher compared to the relaxation curve without preconditioning. The relaxation equilibrium stress of both materials was reached after 1000 s. This variation could be reproduced for both materials.

4 Conclusion

A study on the mechanical behaviors of electrospun nonwoven specimens and cast films of TSPU showing stress strain behavior with pronounced hysteresis and softening of both analyzed specimens. Preconditioning has been completed after 5 cycles and stress relaxation equilibrated after 1000 s, respectively.

Our results indicate that polymeric nonwoven structures exhibit mechanical properties suitable for application in heart valve engineering.

Author's Statement

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