Effects of temperature and strain level on stress relaxation behaviors of polypropylene sutures

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Abstract An investigation has been conducted on stress relaxation behaviors of polypropylene sutures under different temperatures and strain levels in a temperature-controlled water bath. The study showed that the temperature and strain level significantly affected the stress relaxation behaviors of the sutures. High temperature resulted in fast stress relaxation. The stress relaxation data could be well described by two empirical formulas. For most of the test conditions, the stress relaxation tests caused limited permanent deformation may be illustrated by a power law. The tensile properties of the sutures were not affected adversely by the stress relaxation tests, indicating good properties of polypropylene sutures.

Introduction

Much work has been performed on polypropylene (PP) sutures in the areas of clinical applications, mechanical properties, structural analysis, and *in vitro* and *in vivo* studies [1 - 16]. However, relatively few researches could be found on their viscoelastical properties. To the authors' knowledge, no studies have been conducted on stress relaxation behaviors of these sutures. To try to understand the viscoelastic behaviors of polymeric biomaterials and their possible impacts to medical devices, we have performed a series of studies on creep, stress relaxation and fatigue properties of PP sutures. In a previous paper, we reported the experimental results on creep behaviors of PP sutures [17]. The

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R&D Division, Ethicon, a Johnson & Johnson Company, P. O. Box 151, Somerville, NJ 08876-0151 e-mail: mdeng@ethus.jnj.com present study summarizes our recent data on stress relaxation of the sutures. Specifically, the effects of temperature and strain levels on stress relaxation behaviors of PP sutures were evaluated. The tensile properties and permanent deformation of the stress relaxation-tested sutures were also examined.

Materials and methods

Materials

The experimental materials used in this study were PP sutures manufactured by Ethicon, Somerville, New Jersey. They were Size 2-0 blue monofilament sutures with a nominal diameter of 0.32 mm. For the purpose of this study, the specimens of 380-mm length were cut from the suture packages. Five samples were prepared for each test condition.

Stress relaxation test procedure

Stress relaxation is generally defined as load change in a material subjected to contant strain (elongation) at constant temperature. To perform a stress relaxation test, three key requirements are expected: (1) no shock during stretching, (2) applying constant strain, and (3) continuously monitoring change of load with time. It was found that programming of a stress relaxation test procedure could be easily realized using TestStar IIs MultiPurpose TestWare (MPT) software (MTS corporation, Eden Prairie, MN). For this study, the stress relaxation experiments were conducted on an MTS 858 Mini Bionix tester (MTS corporation, Eden Prairie, MN) with a 220-N load cell. The system was attached with a temperature-controlled water bath with deionized water. The suture was connected to MTS tester via suture grips and completely

immersed into the bath. The gauge length (test section of sutures) was 76 mm. This was measured between two center points of suture grips. Five temperatures of 10, 23, 37, 51 and 65 °C and three strain levels of 13, 26 and 33% were selected. The suture samples, while in grips, was placed into the water bath of testing temperature for about 10 minutes to reach equilibrium before a load (straining) was applied. Each stress relaxation experiment lasted 167 minutes (10,000 seconds). For each condition, at least three specimens were tested. The load, time and elongation were recorded, from which changes of load with time were monitored during stress relaxation tests.

Permanent deformation calculation

It is important to know whether or not stress relaxation tests would cause any plastic deformation or permanent deformation in the sutures. This was achieved by comparing the length of sutures before and after stress relaxation tests using the following formula.

$$\Delta L = \frac{L_{sr} - L_0}{L_g} \times 100\%$$

where ΔL is the percent permanent deformation; L_{sr} is the suture length after stress relaxation testing (measured after at least 7-day recovery at room temperature); L_0 is the suture length before stress relaxation testing; and L_g is the gauge length. These dimensions were measured by an engineering ruler. The data from this formula should be considered as maximum permanent deformation that a specimen may have in a stress relaxation test.

Tensile test

The tensile strength is one of the most important properties for sutures. The purpose of tensile test was to examine whether or not the tensile properties of the sutures would change after they had been subjected to stress relaxation test. To this end, the tensile tests were run on the stress relaxationtested suture samples at room temperature using an Instron 4501 tensiometer with a 500-N load cell after the sutures had a recovery time of at least 7 days at room temperature. A gauge length of 60 mm and an initial strain rate of 3.6%/sec were used. The tensile properties were calculated based on the original cross-section area of a specimen before stress relaxation test.

Data analysis

To determine statistical significance, the tensile data were analyzed by multiple comparison (Tukey-Kramer HSD) using JMP statistical software (SAS Institute, Cary, North Carolina). A p = 0.05 was selected. Whenever possible, the experimental data were presented as the mean \pm standard deviation.

Results and discussion

Stress relaxation behaviors

The stress relaxation experimental results are presented through Figs. 1 to 3. It is clear from these figures that the load in a suture specimen decreased with time, indicating significant stress relaxation in PP sutures. Generally, the stress relaxation process can be divided into three regions: (1) fast stress relaxation region; (2) slow stress relaxation region; and (3) the region between these two, or transition region. The fast stress relaxation region consisted mainly of elastic deformation and ended quickly. The suture responded to the applied strain viscoelastically in slow stress relaxation process. The stress in sutures decreased gradually in this region. The suture permanent deformation predominated in this



Fig. 1 Stress relaxation behaviors of PP sutures at different temperatures for strain level 13%.



Fig. 2 Stress relaxation behaviors of PP sutures at different temperatures for strain level 26%.



Fig. 3 Stress relaxation behaviors of PP sutures at different temperatures for strain level 33%.

region. It took only a few minutes for the transition region to complete.

The applied strain levels had a great impact on the stress relaxation behaviors of the sutures. The larger the strain, the higher the stress. When the strain was doubled, the stress was almost doubled. For example, the stress at the end of stress relaxation tests was about 50 MPa at 23 °C for a strain level of 13%; when elongation was increased to 26%, the corresponding stress was about 103 MPa; and when the strain was further increased to 33%, the resulted stress was about 127 MPa. So, there was more than 1-fold increase in stress when the strain was increased from 13 to 26%. Such results may suggest that the increase in stress was proportional to the increase in strain or an approximate linear relationship between them when the temperature was constant. Figure 4 indicated that the rate of stress relaxation propagation was affected slightly by strain levels.

The dependence of stress relaxation behaviors on temperature can be understood by studying Figs. 1 to 3. Similar to the strain level, the temperature had significant effects on the stress relaxation properties of the sutures. The higher the temperature, the lower the stress. This is because high temperature renders the sutures more complaint and accelerates the stress relaxation process. When temperature was increased from 10 to 65 °C, the stress in sutures was decreased more than 3-fold at the end of experiments for all three strain levels; and the higher the strain, the larger the reduction.

Modeling of stress relaxation

It is desirable to illustrate the stress relaxation behavior of a material using some analytical formula. If one can derive a formula based on the short-term stress relaxation experimental data, then it is possible that the long-term behavior could be predicted. This is important that in most actual cases we will not be able to run a month- or year-long stress relaxation



Fig. 4 Stress relaxation experimental results at 23 °C.

experiment, although we wish to know the long-term behaviors of the materials as permanent implants will be in body for years. To explore this, the representative stress relaxation data at 23 °C are replotted in Fig. 4 in a semi-logarithmic graph. It was found that the following two empirical formulas could be used to illustrate stress relaxation behaviors of the sutures.

$$S = a + b \ln t \tag{1}$$

$$S = ct^d \tag{2}$$

where S is the stress in sutures, t the stress relaxation time, and a, b, c and dthe constants. Although both equations illustrate well the stress relaxation data, Equation 1 is better for the data at low temperature and Equation 2 is better for the data at high temperature. Following this rule, one can curve-fit the experimental data with an $R^2 > 0.93$. The deviation of experimental data from the above equations increases slightly with the increases in strain and temperature, indicating that the sutures tended to behave more non-linear viscoelastically at high strain level and temperature. These two equations are very practical formulas as stress relaxation time will, in no cases, equal zero. The constants in above equations could be easily determined by curve fitting. Then, the dependence of stress relaxation behaviors on temperature and strain for PP sutures could be evaluated by examining the effects of temperature and strain on these constants.

Permanent creep deformation

The permanent deformation of the PP sutures following stress relaxation tests was summarized in Fig. 5. It is clear that the amount of permanent deformation increased with the increases in strain and temperature. The smallest permanent deformation was found to be about 1% at 10 °C for strain level 13%. However, at 65 °C and strain level 33%, about



Fig. 5 Effects of testing condition on permanent deformation.



Fig. 6 Effects of load and temperature on permanent deformation.

15% permanent deformation was observed. Fig. 6 illustrates the change of permanent deformation with temperature and strain level for 26 and 33%, which may be described by a power law. Furthermore, it was found that the temperature dependence of permanent deformation could be described by an Arrhenius-type equation showing below:

$$\Delta L = A e^{-\frac{L_a}{RT}} \tag{3}$$

where ΔL is permanent deformation, A constant, R gas constant, E_a active energy and T temperature. By curve-fitting the data, E_a was found to be 11.2 and 17.8 kJ/K-mole at strain levels 26 and 33%, respectively.

Tensile properties

The effects of stress relaxation tests on tensile properties of the PP sutures are presented in Figs. 7 to 9. The data points in these figures represent the averaged value of testing samples. It seems that the tensile properties of the sutures were affected only at high strain level and temperature. Statistical analysis showed that compared to the control the tensile strength was



Fig. 7 Tensile breaking strength of PP suture after stress relaxation tests.



Fig. 8 Tensile modulus of PP suture after stress relaxation tests.



Fig. 9 Tensile elongation of PP suture after stress relaxation tests.

not affected adversely by stress relaxation tests. But, the tensile strength tended to increase at the strain level 33%. Such results suggest that PP sutures would maintain their tensile strength after the stress relaxation tests. The increase in modulus and decrease in tensile breaking elongation indicate that the stress relaxation tests were further drawing and orienting the sutures. The above results may suggest that tensile modulus and elongation could be more sensitive to further drawing and orientation in comparison to tensile strength.

Conclusions

The effects of strain level and temperature on stress relaxation behaviors of Size 2-0 PP sutures were investigated in a temperature-controlled water bath. The results showed that the stress relaxation properties of the sutures depended significantly on the experimental conditions. The relationship between the stress and time could be well described by two empirical formulas. The stress relaxation tests of the sutures caused limited permanent deformation. The dependence of permanent deformation on temperature can be illustrated by an Arrhenius-type equation. Tensile test results indicated that stress relaxation experimental conditions used in this study did not affect adversely the tensile properties of the sutures.

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