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Characterization of Health-Care Products by Dynamic Viscoelastic Measurements

1. Introduction

One rapidly growing area of interest in thermal analysis is the characterization of materials/products used in the medical and health-care fields. Differential scanning calorimetry has been widely utilized to determine the purity of drugs while high sensitivity thermogravimetric analysis has been used to ascertain the moisture content of medicines. Dynamic mechanical spectroscopy (DMS) is becoming increasingly more utilized to characterize the mechanical properties of plastics and elastomeric components used in healthcare applications. This includes the analysis of components used in artificial joints, catheter tubes and, most recently, the characterization of fibers used to produce the mesh for angioplasty surgery. In the latter application, a mesh comprised of many fibers is inserted into a collapsed artery or vein which has been inflated using a small balloon. This provides immediate relief for heart-attack victims. The mesh then supports the arterial walls so that the heart can be stabilized. The mechanical properties of the fibers used in producing the mesh material are extremely important. The mesh must be able to swell properly and also retain a high stiffness in order to support the arterial walls and allow blood to flow freely to the heart.

In this study, two fibers used to generate the angioplasty mesh were characterized using dynamic mechanical spectroscopy to determine if detectable differences may be observed. The two fibers were produced using different means: one was heat-set while the other was not.

2. Experimental

In this particular study, the mechanical properties of two fibers (used to make angioplastic mesh) were characterized using the DMS200. The fibers were produced under different conditions in that one fiber specimen was heat-set while the other was not.

The following conditions were used to characterize the two fiber samples:

- Heating rate: 2°C /min
- Initial temperature: -10°C
- Deformation mode: combination auto-tension
- Sample length: 7mm
- Sample radius: 0.11mm
- Frequencies: 1, 2, 5, 10 and 20Hz
- Strain amplitude: 40µm
- Base force: 5g
- Initial force: 100g
- Tension proportional coefficient: 1.3

3. Results

Two different fibers were analyzed: one which was heat-set and one which had no heat-set treatment. Displayed in Figure 1 are the results obtained on the fiber which was heat-set. This plot shows the tensile storage modulus (E') and $\tan\delta$ as a function of sample temperature at frequencies of 1, 2, 5, 10 and 20Hz. The heat-set fiber has a modulus, E' , of 9.6GPa at room temperature. This fiber has a glass transition temperature of 74°C (at 1Hz) as reflected by the large peak in $\tan\delta$ at this temperature. The $\tan\delta$ peak temperature at T_g increases as the frequency increases since the glass transition is a second order, relaxational event. A second transition is observed at 171°C in the $\tan\delta$ response and this transition is due to the melting of the crystalline phase of the polymer. This transition temperature is independent of the frequency as melting is a first order, equilibrium event. A drop in the storage modulus is observed for both the glass transition and melting events.

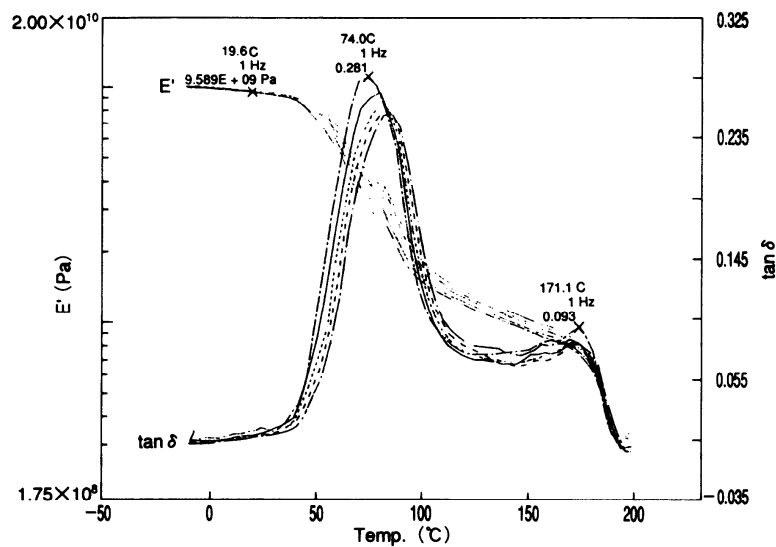


Figure 1 Dynamic viscoelasticity spectrum of the heat-set fiber

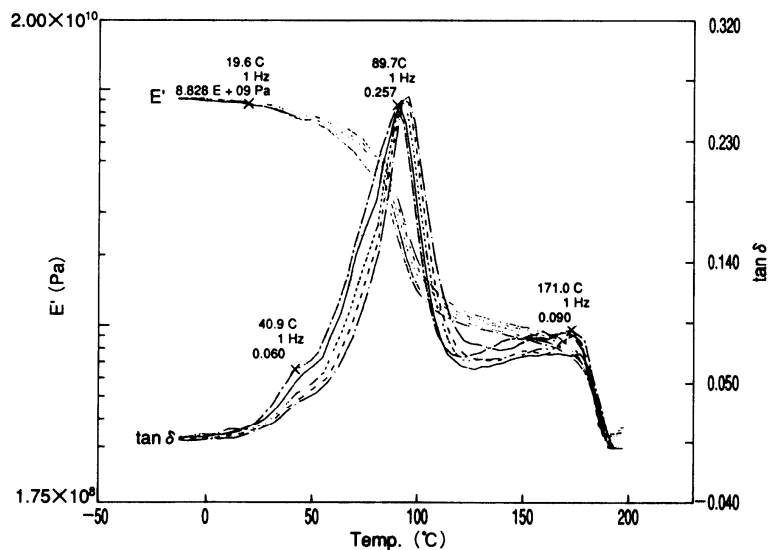


Figure 2 Dynamic viscoelasticity spectrum of the non-heat-set fiber

Displayed in Figure 2 are the results obtained on the fiber sample which was not heat-set. This fiber has a storage modulus of 8.8GPa at room temperature. This is slightly less than that obtained for the heat-set fiber. The glass transition event occurs at 90°C (at 1Hz) for the non-heat-set fiber. This temperature is significantly higher than that obtained for the heat-set fiber. A small shoulder is also observed in the tan delta response for the non-heat-set fiber at approximately 41°C. This shoulder is not obtained for the heat-set fiber. The melting point of the non-heat-set fiber occurs at 171°C and this is identical to that obtained for the heat-set specimen. These results indicate that the heat-setting treatment primarily affects the behavior of the amorphous phase of the fibers.

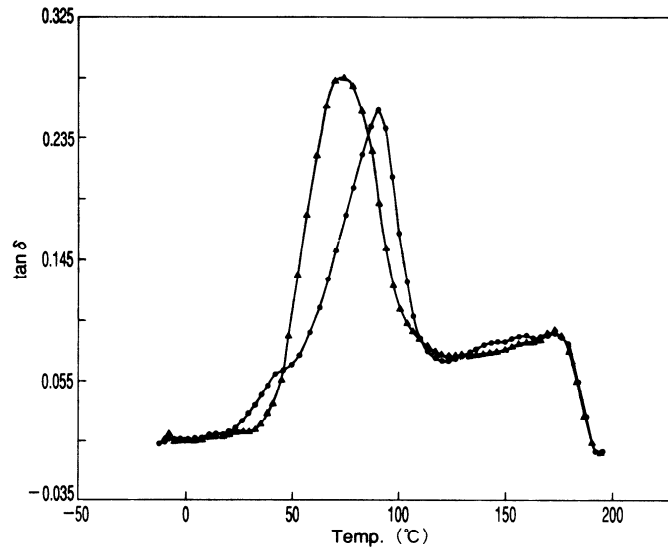


Figure 3 Comparison of the $\tan\delta$ curves of the two fibers
 ▲▲▲ : heat-set fiber
 ●●● : non-heat-set fiber

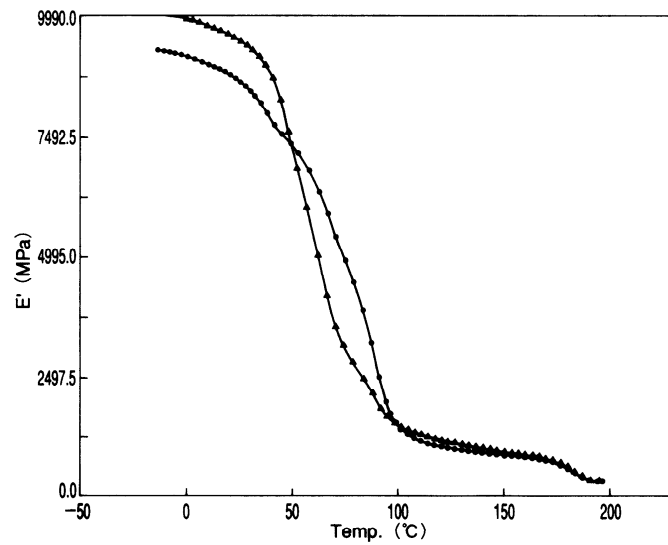


Figure 4 Comparison of the E' curves of the two fibers
 ▲▲▲ : heat-set fiber
 ●●● : non-heat-set fiber

A direct comparison of the viscoelastic properties of the two fibers is displayed in Figure 3. This figure shows the $\tan\delta$ behavior (at 1Hz) for the heat-set and non-heat-set fibers. There are dramatic differences in the behaviors in the region of the glass transition event. The differences in the modulus behavior between the two fibers are displayed in Figure 4. This figure shows E' as a function of sample temperature at 1Hz. The heat-set fiber has a significantly higher modulus below T_g . Above T_g , the two fibers have essentially the same response.

These results demonstrate that the heat-setting process affects the mechanical performance of the fibers. This should translate to actual performance differences when the fibers are used in the mesh during the angioplasty procedure.

4. Summary

The viscoelastic responses of two fibers (one heat-set the other non-heat set) were characterized using dynamic mechanical spectroscopy. The fibers are used to produce a mesh as part of the angioplasty procedure. The heat-set treatment appears to primarily affect the mechanical performance of the fibers in the amorphous phase. The fiber which was heat-set had a lower T_g (74°C versus 90°C) and a higher value of the storage modulus E' at room temperature (9.6GPa versus 8.8GPa). The melting points of the two fibers were identical (170°C).