Effect of storage temperature on the heat sealability of polypropylene film

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ABSTRACT: Storage temperature effects on the sealing properties of corona-treated polypropylene film were investigated. Samples of film stored at room temperature, 50°C, 75°C, 100°C, and 125°C for a period of 48 hours showed dramatic changes in both sealing temperature and sealing range, with sealability rapidly declining with increases in storage temperature. Differential scanning calorimetry was used to assess changes in crystallinity of the samples. Total crystallinity was found to increase with storage temperature. This in turn affected the film's sealability.

KEYWORDS: Closure, crystallinity, film, heat sealing, polypropylene, sealability, seals, temperature.

The crystallinity as well as other polymer film properties (softening point, polarity, etc.) determine the type of sealing apparatus utilized for producing a seal *(1)*. The type of sealing apparatus is determined by the softening behavior of the polymer. Bar sealing is the most widely used method for the fabrication of packages *(1–3)*. Temperature, pressure, and dwell time are the parameters influencing the seal strength of a polymer *(4–7)*. The quality of a seal is usually determined by a peel-strength measurement *(8)*.

The degree of crystallinity of polypropylene is known to be influenced by storage temperature *(9)* and duration *(10)*. Nath and Perlman *(9)* have reported an increase in polypropylene's crystal size with an increase in annealing temperature. Horrocks and D'Souza *(10)* showed that, for a storage temperature of 130°C, oriented polypropylene film's crystallinity increased with time.

In this study, we investigated the change in effect of storage temperature of polypropylene films on the sealing quality.

Experimental

Sample preparation

Unoriented, cast, corona-treated (on one side) polypropylene film supplied by Exxon Chemical Co. was utilized for experimentation purposes.

Initially, the film was cut into 2.54-cm x 22.86-cm strips. Samples of the film were stored at 50°C, 75°C, 100°C, and 125°C, using a Fisher Scientific Isotemp Oven Model 655G, for a period of 48 hours. Then the films were transferred to a desiccator (containing Drierite-Anhydrous $CaSO_4$ Hammond) for a minimum of 5 h and allowed to equilibrate to room temperature *(11)*. An Ono Sokki EG-225 digital linear gauge with an Ono Sokki ST-022 gauge stand was used to measure film thickness. Thickness of the films were 0.06699 mm ± 0.00223 mm.

Heat-sealing condition

A Theller Heatsealer Model EB (Precision Instruments West of Petaluma, CA) was used for the heat-sealing process. This apparatus contains a top and a bottom heating element bar. The dimension of the top bar was 13 cm x 0.9 cm, with the bottom bar dimension being 13 cm x 1.5 cm. Both heating elements were covered with 1.905 x 10^{-2}-mm-thick (0.75 mil) Mylar® film

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to prevent the corona-treated polypropylene film from sticking to the bars.

Samples of storage-temperature-exposed film were folded in half lengthwise and heat sealed to themselves, at the center location, perpendicular to the length, with the corona-treated side facing out. The sealing parameters (temperature, pressure, and dwell time) were regulated. At the completion of the heat-sealing cycle, the film was removed from the apparatus. The film then was placed on its side to stand so it could evenly equilibrate to room temperature. Prior to the seal-strength determination test, the film was placed in a desiccator for 24 h.

Determination of seal strength

A modified ASTM method *(12)* was used in determining the seal strength of the heat-sealed film. Instron Model 4201, with a load cell rated at 100 N static, was used to determine seal strength. Each end of the film was clutched by the Instron's pneumatic grips. The film was separated lengthwise, with the grips initially 6.25 cm apart. The speed of expansion was set at 100 mm/min. A 100% load range equivalent to 10 N of force was calibrated to the chart paper width.

Determination of crystallinity

A modified ASTM method *(13)* was utilized in determining the percent crystallinity of corona-treated polypropylene film. A sample of approximately 10 mg of the film was weighted with a Perkin-Elmer AD-4 Autobalance C655-001 (Norwalk, CT). The sample then was placed in a small metallic container and was sealed shut. The container with a sample of polymer film was then placed with a reference (empty) container in a DuPont Instruments 910 differential scanning calorimeter chamber. The tests were conducted in a controlled atmosphere of nitrogen. The initiation temperature of the tests was 20°C. From this temperature, the sample was heated to 200°C at a constant rate of 10°C/min. A linear sloped line was placed between the x-axis points of 35°C to 176°C. The areas of each exothermic region and endothermic region then were calculated in Joules per gram for the change in enthalpy of fusion. The following equation then was used to calculate the percent crystallinity of the polymer *(14, 15)*:

$$\text{Percent crystallinity} = X_c \times 100 = (\Delta H_f/\Delta H_f^*) \times 100$$

where

X_c = degree of crystallinity

ΔH_f = enthalpy of fusion of the unknown material

ΔH_f^* = enthalpy of fusion of the crystalline standard

ΔH_f^* = 207 J/g *(16)*.

The percent crystallinity of both of the endothermic regions was summed, and the percent crystallinity of the exothermic region was subtracted to obtain the total percent crystallinity. The percent crystallinity of the exothermic region was subtracted because this portion of percent crystallinity was created during the thermal analysis process *(17)*.

Results and discussion

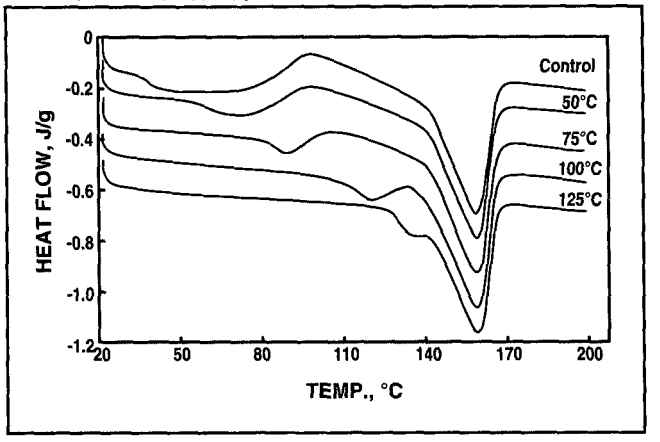
The thermograms showed an initial endothermic region followed by an exothermic region and a pronounced second endothermic region. Selikhova *et al.* *(17)* showed that the existence of an exothermic region above the glass transition temperature of a specific polypropylene thermogram is due to crystallization during the differential scanning calorimetry (DSC) process. The exothermic region is observed only during DSC runs which utilize a fast heating rate. Increase in the storage temperature resulted in a change in the DSC profile. The data in **Fig. 1** show that with an increase in storage temperature, the initial endothermic region migrated to higher temperatures and eventually became part of the pronounced second endothermic region, while the exothermic region disappeared. The behavior of the first peak is very much like the crystalline fraction of polymer, which will increase in melt temperature with an increase in crystallization temperature. Therefore, results suggest that the first endothermic peak may represent a crystalline fraction that is more sensitive to crystallization temperature and different in morphology than the crystalline fraction of the second endothermic peak.

The percent crystallinity of the polypropylene samples as a function of storage temperature is presented in **Fig. 2**. The regression analysis of the data points showed that there is a linear relationship between the storage temperature and percent crystallinity of the film during 48-hours of storage. An Arrhenius plot of the effect of storage temperature on the crystallinity change of the film is plotted in **Fig. 3**. From this plot, a rate of crystallization at room temperature can be extrapolated.

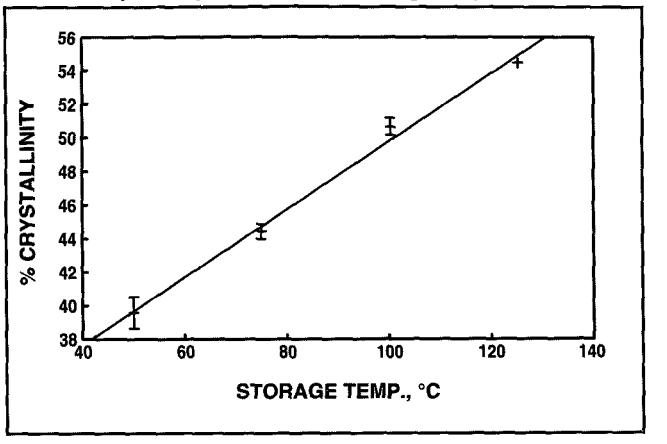
Figure 4 shows seal strength as a function of sealing temperature for film exposed to different storage temperatures for 48 hours. The seals were made at a dwell time of 1 s and a pressure of 30 psi. The data show that with an increase in storage temperature there is a corresponding increase in the minimum sealing temperature required to produce a given seal strength. Also, there is a consequent reduction in the heat-sealing temperature range available to produce a seal.

Since increasing the storage temperature will increase the percent crystallinity of polypropylene samples, the same data in seal strength were plotted as a function of storage temperature for various heat-sealing temperatures (**Fig. 5**). This shows more clearly the decrease in seal strength with increase in storage temperature and crystallinity. The plot of percent crystallinity versus the seal strength of the film is shown in **Fig. 6**. During the heat-sealing process, the interface disappears with interdiffusion of chain segments across the surface at temperatures greater than the glass transition temperature *(18–20)*. Subsequently, the mechanical strength at the polymer–

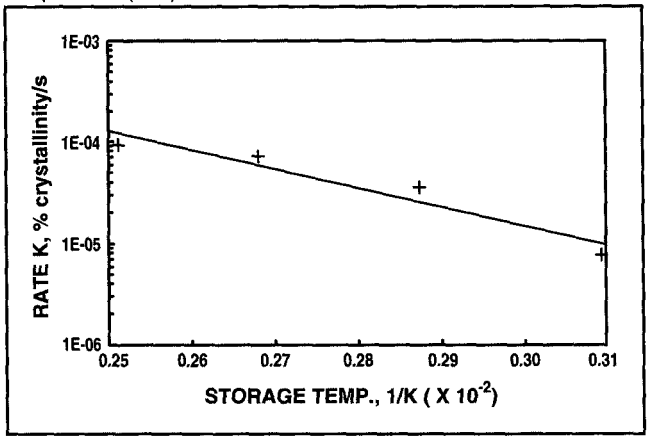
1. DSC profile of polypropylene film for various storage temperatures



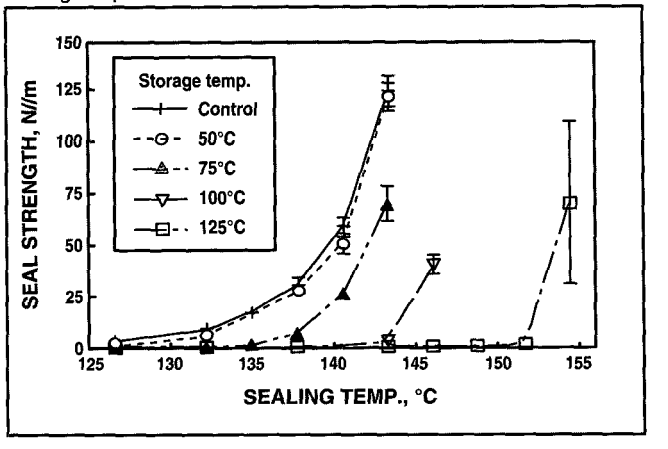
2. Percent crystallinity as a function of storage temperature



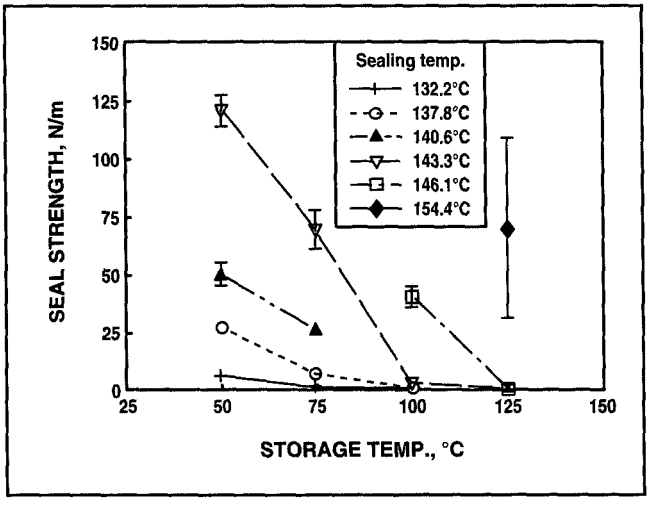
3. Arrhenius plot of crystallinity change as a function of storage temperature ($°K^{-1}$)



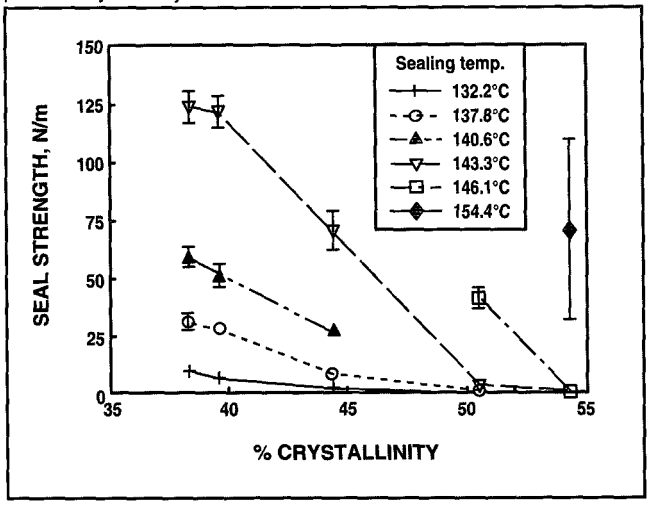
4. Seal strength of corona treated polypropylene film as a function of sealing temperature



5. Seal strength of corona treated polypropylene film as a function of storage temperature



6. Seal strength of corona treated polypropylene film as a function of percent crystallinity



polymer interface develops as the polymer film is cooled. However, increase in percent crystallinity will decrease the amount of interdiffusion of chain segments because it is unlikely the crystalline domain of the polymer chain would interdiffuse at the heat-sealing temperature.

Conclusion

Film exposed to elevated storage temperatures displayed a linear increase in percent crystallinity. This in turn decreased the film's sealability. There was a consequent reduction in the heat-sealing temperature range available to produce a seal. A shift and reformation in the crystalline melting point of the DSC thermograms were observed for film stored at elevated temperatures.

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