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EFFECT OF FILM PROCESSING ON CRYOGENIC PROPERTIES OF POLY(ETHYLENE TEREPHTHALATE)

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ABSTRACT

The effects of stretch temperature, heat-set temperature, and time on the cryogenic mechanical properties of PET film were studied by using a two-level factorial experiment. Amorphous PET sheet was oriented at either 85° or 95° C. The levels of heat-set temperature and time investigated were 190° and 210° C and 15 and 120 seconds, respectively. Film crystallinity was determined by the density-gradient technique. Tensile properties were measured in liquid nitrogen and in liquid hydrogen. Biaxial burst properties were obtained in liquid nitrogen. The number of flexural cycles to failure in liquid hydrogen and room-temperature gas porosities after 25 such cycles were also determined.

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SUMMARY

The effects of stretch temperature, heat-set temperature, and heat-set time on the cryogenic mechanical properties of poly(ethylene terephthalate) (or PET) film were studied by using a two-level factorial experiment. Squares of amorphous PET sheet were given a simultaneous biaxial 3X stretch in a laboratory film stretcher at either 85° or 95° C (358 or 368 K). The levels of heat-set temperature and time investigated were 190° and 210° C (463 and 483 K) and 15 and 120 seconds, respectively.

Crystallinity of the films was determined by the density-gradient technique. Tensile properties were measured at room temperature in liquid nitrogen (-196° C (77 K)) and in liquid hydrogen (-253° C (20 K)). Biaxial burst properties were obtained at -196° C (77 K). The number of flexural fatigue cycles to failure at -253° C (20 K) and room-temperature gas porosity after 25 cycles at -253° C (20 K) were also determined.

Processing variables significantly affected the stress-strain properties of PET film at cryogenic temperatures. These results and the encouraging values of flexibility indicate possible uses of specially processed PET films for cryogenic space applications.

INTRODUCTION

Filament-wound glass-reinforced plastics offer an attractive solution to the problem of providing lightweight, high-strength tanks for the storage of cryogenic propellants. Permeation, or leakage, of the cryogen through the tank walls is the major problem which has to be solved before the full potential of these reinforced plastics can be realized. The porosity problem is associated with both the filament reinforcement and the resin matrix.

An obvious solution to the porosity problem is to provide some type of liner within the tank to prevent permeation of the fluid. The liner material should be capable of being elastically strained a number of times from zero to at least 2.5 percent and back to zero again under biaxial loading conditions (ref. 1). In that study, both metallic and nonmetallic films were investigated as possible liner materials. The use of polymeric films would be preferred because not only are they light in weight but they might also serve as a means for expelling the fluid from the tank under zero-gravity conditions. If used in this manner, the polymeric films must be flexible as well as reversibly extensible.

The results of an extensive screening program performed by Beech Aircraft (ref. 2) showed that, although none of the currently available plastic film materials were suitable for use as liners or bladders, poly(ethylene terephthalate) (or PET) possesses limited cryogenic flexibility. There are a number of approaches to obtaining a satisfactory cryogenic polymeric film (e.g., continued screening of commercial materials, the synthesis of new or modified polymers (ref. 3), and the study of polymer processing variables). Heffelfinger and Schmidt (ref. 4) studied the effect of processing on room-temperature properties.

This investigation was conducted to determine if variation of processing conditions offered leads to significant improvement of cryogenic properties. Comparison with commercial PET was incidental to establishing these leads.

The effects of stretch temperature, heat-set temperature, and heat-set time on a number of properties of PET film were studied by using a two-level factorial experiment. Crystallinity of the films was determined by the density-gradient technique at room temperature. Tensile properties were measured at room temperature, in liquid nitrogen (-196° C (77 K)), and in liquid hydrogen (-253° C (20 K)). Biaxial burst measurements were made at -196° C (77 K). The flexibility at -253° C (20 K) and the room-temperature gas porosity of films which had been subjected to a given number of flex cycles at -253° C (20 K) were also determined.

MATERIALS AND PROCEDURE

The extruded amorphous sheet used in this study was provided by Dr. C. J. Heffelfinger of the E. I. du Pont Co. It was reported to possess no orientation and to have a number average molecular weight of 19 500. The sheet had a nominal thickness of 6 mils (0.015 cm).

Film Processing

The factors studied in this investigation were stretch temperature, heat-set temperature, and heat-set time. Two levels of each were selected to limit the number of experimental conditions to eight and form a 2^3 factorial experiment. This design permits independent estimates of the effects of all factors and their interactions with the use of a minimum number of film preparation conditions. The two levels of each factor were selected to be far enough apart to determine these effects in the presence of random experimental error. However, the levels should not be so far apart that their range includes the level of optimum or minimum response. If these factors affect the response and the levels are wisely selected, the results of a two-level factorial experiment will provide leads to further improvement of properties. Optimization of processing variables to attain specific design properties can follow.

Stretching temperatures of 85° and 95° C (358 and 368 K) were chosen to be sufficiently above the glass transition temperature of 69° C (342 K) (ref. 5) to stretch the amorphous PET without tearing. Heat-set temperatures of 190° and 210° C (463 and 483 K) were selected to be in the region which gives the minimum half-time for crystallization (ref. 6). Preliminary work had shown the biaxially stretched film changed little in density after 120 seconds at 190° C (463 K). Some heat setting is desirable to stabilize the film. Heat-set times of 15 and 120 seconds were used. Fifteen seconds is nearly the minimum time to reproducibly heat treat the film.

Films were also prepared at the center point of the design (i. e., stretched at 90° C (363 K) and set at 200° C (473 K) for 42 seconds (the geometric mean of the two levels of set time)). The difference between the response at this center point and the average for all eight conditions offers evidence of curvature of the response surface (which is a function of three independent variables). However, data at other experimental conditions are required to assess which factor is responsible for curvature.

Four-inch (10.16-cm) squares of the amorphous sheet were stretched in a commercially available biaxial film stretcher. The sheet was manually inserted into the stretching chamber, which was controlled at the selected temperature, and then automatically stretched to a 12-inch-(30.5-cm-) square film at a rate of 1000 percent per minute. The amorphous sheet was given a 15-second equilibration in the stretching chamber before and after stretching. The stretching machine is provided with a special device which quenched the film to room temperature and enabled the film to be easily removed free of wrinkles and/or folds. Heat setting was accomplished by clamping the film in a restraining frame and placing it in a circulating-air oven for the time and temperature specified. It was then removed from the oven and cooled to ambient temperature. Other films were stretched but not heat set.

Film Density

Density of the films was determined by the density-gradient technique (ref. 7). The liquid system chosen for the column used in this study was n-hexane and carbon tetrachloride. Glass floats of known density were used to calibrate the column. The tube was maintained at a constant temperature of 23° C (296 K). Two small pieces of each film were placed in the column and allowed to reach equilibrium. The position of the calibration floats and samples, relative to an arbitrary reference, were measured with a cathetometer.

Uniaxial Tensile Properties

Tensile properties of the films were evaluated in a universal testing machine with 1-inch-(2.54-cm-) wide specimens cut with the length perpendicular to the direction of extrusion of the amorphous film. Load and extent of grip separation were automatically recorded. An initial grip separation of 4 inches (10.16 cm) was used for all tests. The rates of grip separation for the room-temperature and cryogenic tests were 2.0 and 0.2 inch per minute (0.084 and 0.0084 cm/sec), respectively. Cryostats of the type described in reference 8 were employed for the liquid-nitrogen and liquid-hydrogen tests.

Biaxial Diaphragm Burst Test

Biaxial stress-strain properties of the films were measured at -196° C (77 K) in an apparatus similar to that described in reference 9. The test specimen was bolted between a circular flat plate and an 8-inch-(20.32-cm-) inside-diameter annulus. The bottom plate was provided with the means for introduction of the helium gas used for pressurization. All tests were performed with a constant gas flow rate to rupture. Vertical deflection of the film was measured with a linear potentiometer connected to a 0.9-inch-(2.2-cm-) diameter polytetrafluoroethylene disk which rested on the center of the film. The disk was counterbalanced to move in response to very little force from the film. The pressure underneath the film was recorded with a pressure transducer. The entire diaphragm clamping device was submerged in liquid nitrogen.

Twist-Flex Test

The equipment used to evaluate the flexibility of the polymer films is described elsewhere in considerable detail (ref. 2). The 4- by 11-inch (10.16- by 27.94-cm) sample was formed into a cylinder of approximately 4 inches (10.16 cm) in diameter by wrapping it around and clamping it to upper and lower cylindrical end plates. The clamped sample was immersed in liquid hydrogen and given a twisting motion by clockwise and counterclockwise rotation of the upper plate relative to the lower plate. The upper connecting rod mechanically followed a cam which lowered and raised the end plate as it was rotated. The sample was visually inspected for cracks or holes every 5 or 10 complete cycles.

Porosity Test

The porosity of films that had been subjected to 25 twist-flex cycles in liquid hydrogen was determined at room temperature in a gas collection apparatus. The device used to support the film exposed a 3- by 9-inch (7.6- by 22.86-cm) section of the film to a chamber maintained at 1 psig (6.89×10^3 N/m^2 gage) with helium gas. The volume of gas which passed through the film was collected in a burette by a water-displacement technique.

DISCUSSION OF RESULTS

The results obtained for each response measured on the PET films are described and include the following:

(1) Crystallinity

(2) Uniaxial tensile tests conducted in ambient air at 25° C (298 K), in liquid nitrogen at -196° C (77 K), and in liquid hydrogen at -253° C (20 K)

(3) Biaxial diaphragm burst tests in liquid nitrogen

(4) Fatigue failure by cycling on the twist-flex apparatus with the film immersed in liquid hydrogen

The data for each experimentally determined response were used to calculate the coefficients of the following regression model, except that b_D and b_{ABC} were zero for the tests with liquid nitrogen and additionally b_{AC} was missing for the tests with liquid hydrogen.

$$y = b_0 + b_A x_A + b_B x_B + b_C x_C + b_{AB} x_A x_B + b_{AC} x_A x_C + b_{BC} x_B x_C + b_{ABC} x_A x_B x_C + \frac{1}{3} b_D\left(x_A^2 + x_B^2 + x_C^2\right) \qquad (1)$$

where y is any response, x is -1 for the low level and +1 for the high level of each of the three factors designated by subscripts A, B, and C (stretch temperature, set temperature, and set time, respectively), D is the centerpoint, and b is the corresponding coefficient determined by regression analysis. These coefficients were then tested at a significance level of $\alpha = 0.05$ unless otherwise noted. Results shown on bar graphs represent only factors which have a significant effect, as determined according to appendixes A and B.

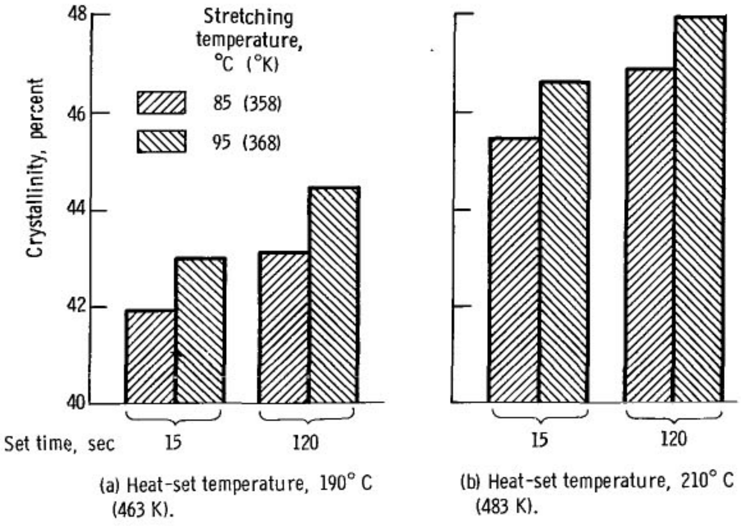
Crystallinity

In this report crystallinity of the films is considered to be a response to the processing conditions. Ideally, morphological properties of the film, such as crystallinity and orientation, should serve as factors upon which the mechanical property responses depend. However, it was first necessary to learn how to prepare films with a suitable range of crystallinity, and in this sense crystallinity is a response.

Crystallinity C can be estimated from the film specific gravity ρ. A value of 1.330 was measured for the amorphous PET; the calculated value of 1.455 was used for the perfect crystal (ref. 10).

$$C = \frac{\rho - 1.330}{1.455 - 1.330} = 8.00(\rho - 1.330)$$

Increasing the stretching temperature, the heat-setting temperature, and the setting time all increase the crystallinity of the film (fig. 1). The effect of the heat-setting tem-



(a) Heat-set temperature, 190° C (463 K).

(b) Heat-set temperature, 210° C (483 K).

Figure 1. - Crystallinity of PET film as function of stretching temperature and heat-set temperature and time.

perature is most pronounced, causing films heat-set at 210° C (483 K) to have 3.6 percent higher crystallinity than films heat-set at 190° C (463 K). The effect of each of the other factors is only about one-third as large. The range of crystallinity is from 41.9 percent for the low levels of all three factors to 48.0 percent for the high levels.

Films stretched at 85° and 95° C (358 and 368 K) and not heat-set are 15.8 and 20.9 percent crystalline, respectively. In both cases, this is less than half the crystallinity of heat-set films regardless of the conditions. Stretching, therefore, orients the polymer molecules and results in increased density. However, an increase in density of a larger magnitude occurs upon heat-setting at higher temperature. The molecules develop a more ordered and closely packed form when heated to 190° or 210° C (463 or 483 K) for short times, and films become over 40 percent crystalline. Heffelfinger and Schmidt (ref. 4) describe changes in the crystalline and amorphous regions during the stretching and heat-setting processes, as observed by X-ray diffraction, infrared, density, and stress-strain measurements at room temperature. The gauche form is transformed into trans during the stretching. The trans isomer crystallizes as a result of the alinement of adjacent molecules. Their evidence favors isomerization of gauche to trans to promote crystal growth during heat-setting.

The results on the film produced at the center point were included in the analysis and indicate that curvature of the crystallinity response over the range of these factors studied is not significant.

Uniaxial Tensile Properties

Tests in ambient air at 25° C (298 K). - Conditions promoting tensile strength are the lower stretching temperature of 85° C (358 K) and the higher heat-setting temperature of 210° C (483 K) (fig. 2). The stretch temperature - heat-set time is the only interaction that has a significant effect on this property. The breaking stress from two additional experiments at the center of the design averaged 27 400 psi (1.89×10^8 N/m^2). Analysis showed that there is significant curvature in the response over the ranges of the variables. Further, a film stretched at 85° C (358 K) but not heat-set required a stress of 33 000 psi (2.3×10^8 N/m^2) to break.

The higher levels of both temperatures caused greater strain at break (fig. 3); heat-setting time and all interactions were not significant. Overall, these results provided strong evidence of differences among the films produced by various processes and thus justified cryogenic testing.

Strain at the proportional limit. - This property is defined as the strain at which the stress-strain curve departs from linearity. It is of particular importance since a liner must expand with the tank as internal pressure is applied and then contract to its original

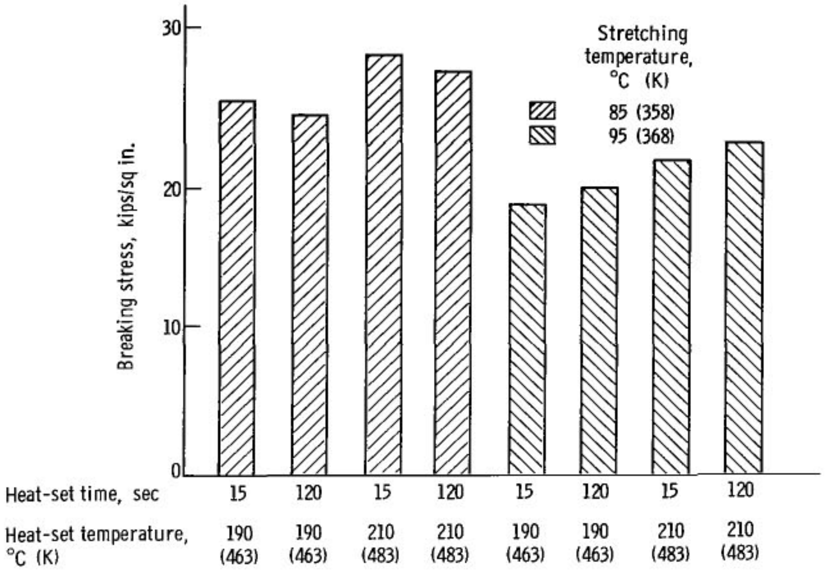


Figure 2. - Stress at break of PET film determined from uniaxial tensile test at 25° C (298 K) as function of stretching and heat-set temperatures and set time.

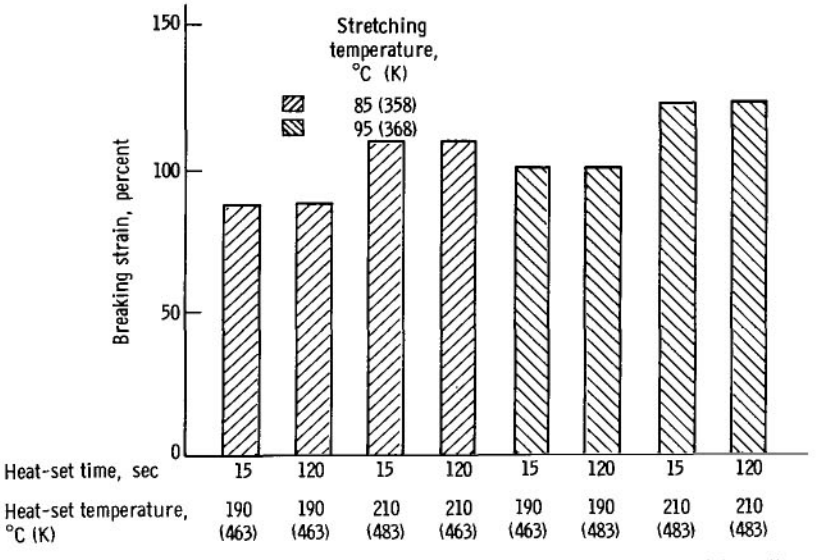


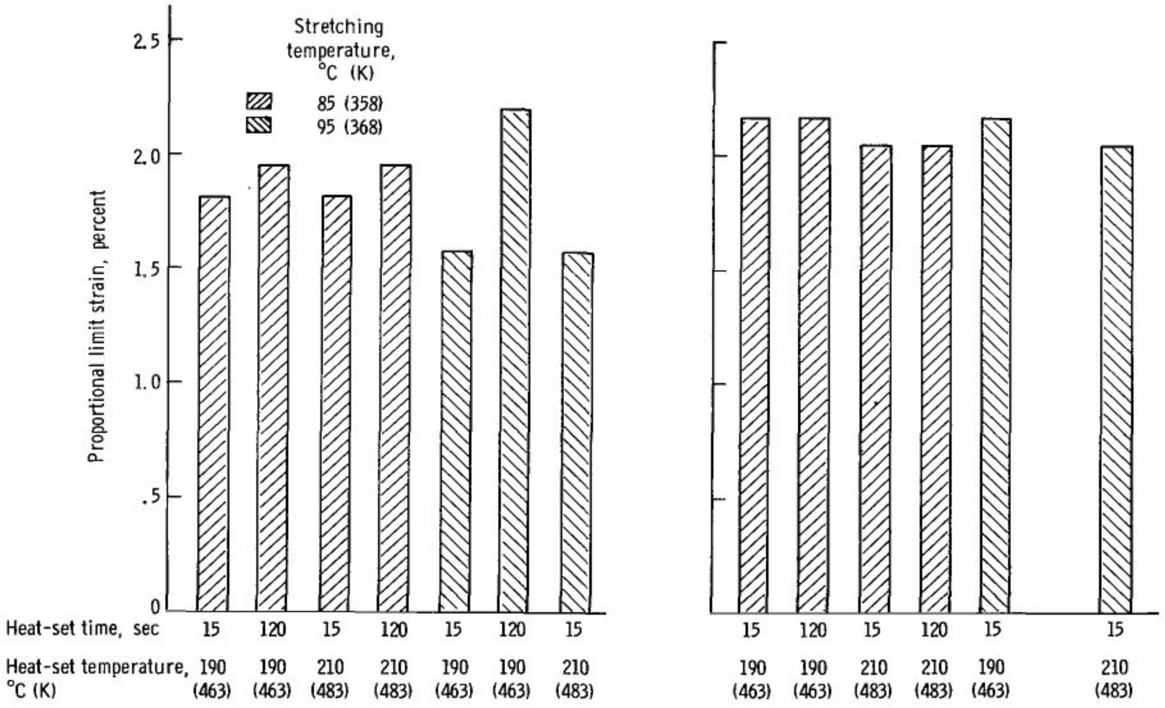
Figure 3. - Strain at break of PET film determined from uniaxial tensile tests at 25° C (298 K) as function of stretching and heat-set temperatures and heat-set time.

dimension. The strain at the proportional limit is more readily measured than the recoverable strain limit and yet serves as a guide to the usefulness of the film for liner application.

The heat-setting time and the stretch temperature - heat-setting time interaction are the factors affecting strain at the proportional limit in liquid nitrogen (table I). Films stretched at 95° C (368 K) and set for 120 seconds averaged 2.19 percent strain compared with 1.59 percent for a setting of 15 seconds (fig. 4). For films stretched at 85° C (358 K) the longer set time increased the strain by only 0.15 percent strain compared with 0.62 percent strain for the higher temperature. Thus, there is a significant interaction between these two factors in the manner in which they affect strain at the proportional limit. This response does not depend upon the heat-set temperature.

In liquid hydrogen the correlation of strain at the proportional limit with the treatment conditions was poor, and the only factor which appeared to be significant was the set temperature. The average strain was 2.1 percent, which is greater by 0.2 percent strain than the corresponding values obtained in liquid nitrogen.

Stress at the proportional limit. - This property is the stress at the same point of the stress-strain curve defined in the previous section. Stress at the proportional limit



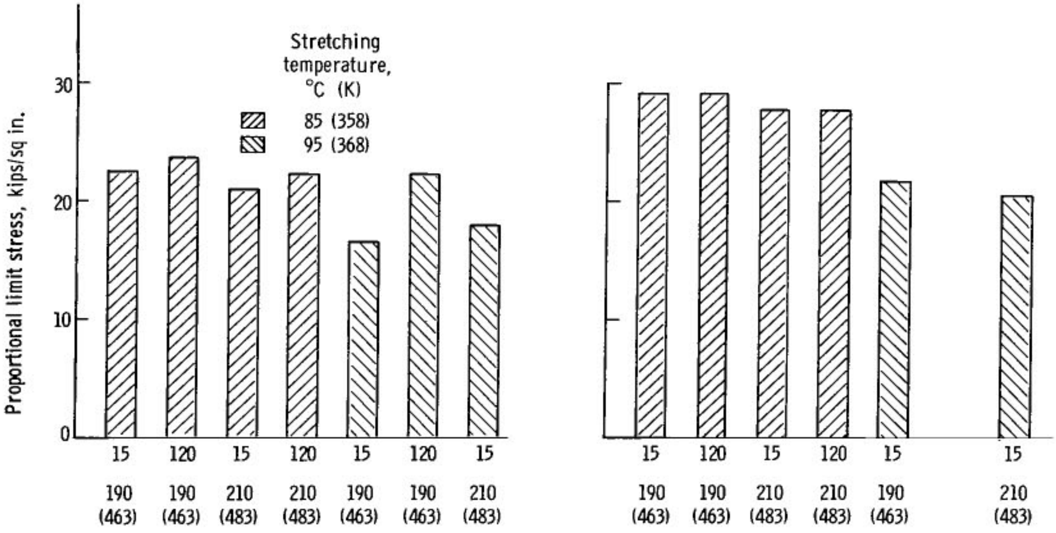
(a) Liquid nitrogen. (b) Liquid hydrogen.

Figure 4. - Strain at proportional limit determined from uniaxial tensile tests of PET film at cryogenic temperatures as function of stretching and heat-set temperatures and heat-set time.

decreased with increasing stretch temperature (fig. 5). The values were higher at the lower temperature of liquid hydrogen. When these results are combined with the previous strain values, in general, the modulus of elasticity is higher for films stretched at 85° C (358 K) than those stretched at 95° C (368 K). Heat-setting affects the modulus determined in liquid nitrogen, giving higher values for shorter times of setting as seen in table II.

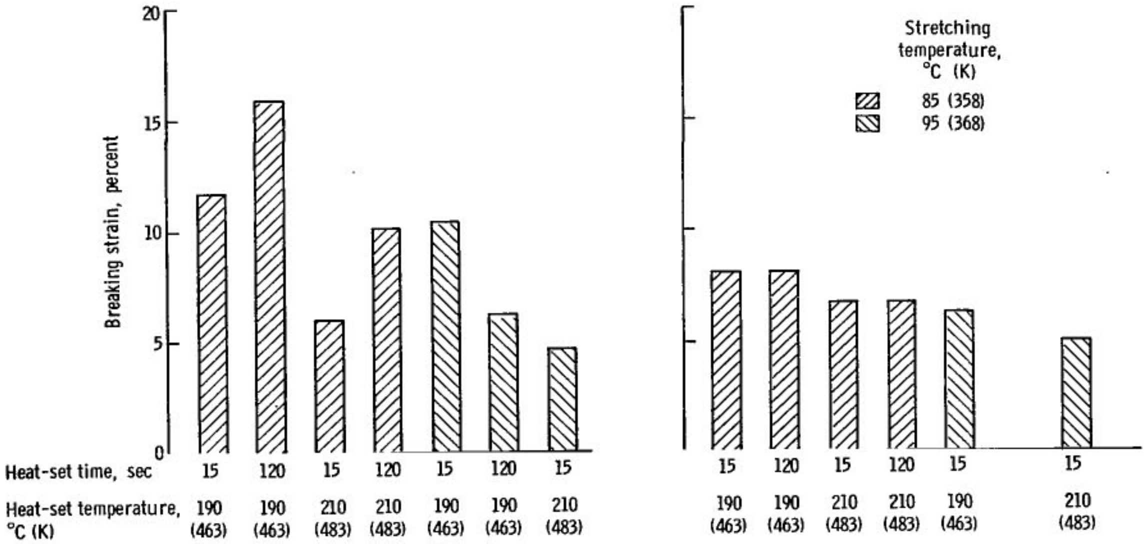
Strain at break. - The strain at which these PET films break is much greater than the proportional limit strain and also varied more with processing factors (fig. 6). In liquid nitrogen the stretching temperature and heat-setting temperature both have large effects; the greater ultimate strain of 15.4 percent was attained with both processing temperatures at the low levels. With high processing temperatures, only 5.0 percent strain was reached at break. These values are roughly inversely related to crystallinity. However, heat-setting time, which affects crystallinity to a limited extent, did not significantly change the ultimate strain (table I). Note also that the stretching temperature generally reduced the strain at break but was not significant with respect to the proportional limit strain (table I). The highest proportional limit strain was obtained by stretching at 95° C (368 K) and heating for 120 seconds (table III).

The strain at break in liquid hydrogen is lower than in liquid nitrogen. Stretching temperature also affects the strain at break; with experimentally determined values from table IV, 7.3 percent strain is obtained for 85° C (358 K) stretching and 5.0 percent for 95° C (368 K).



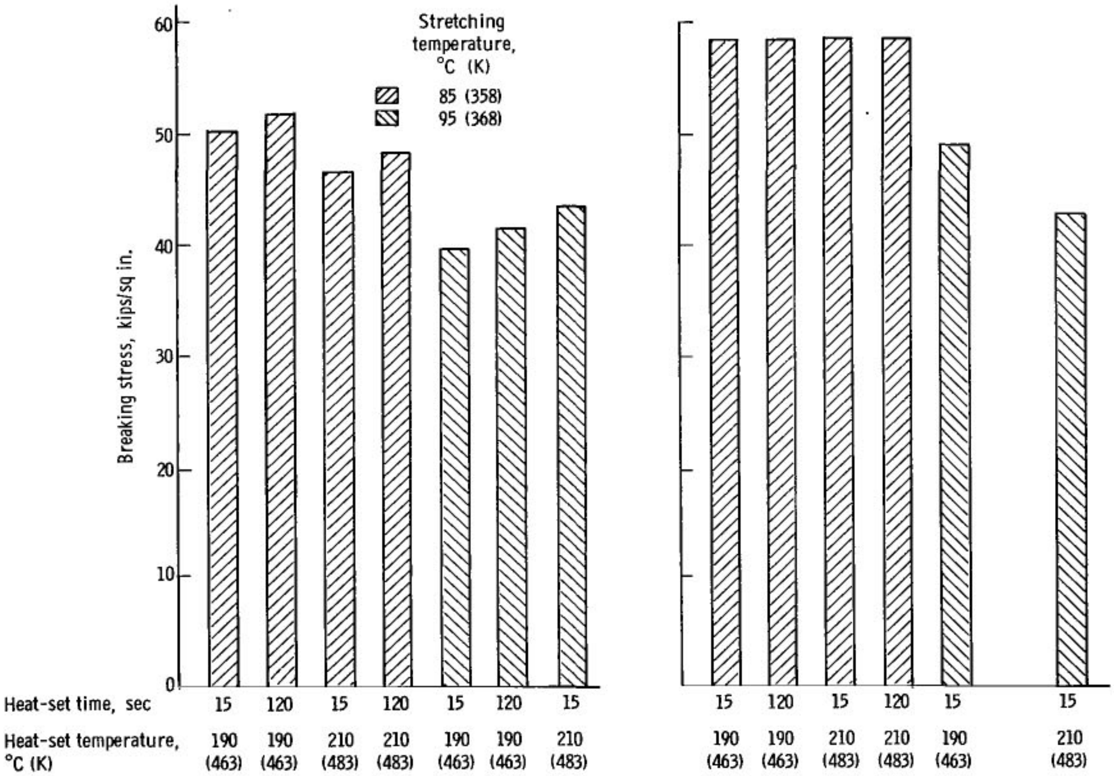
(a) Liquid nitrogen. (b) Liquid hydrogen.

Figure 5. - Stress at proportional limit determined from uniaxial tensile tests of PET film at cryogenic temperatures as function of stretching and heat-set temperatures and heat-set time.



(a) Liquid nitrogen. (b) Liquid hydrogen.

Figure 6. - Strain at break determined from uniaxial tensile tests of PET film at cryogenic temperatures as function of stretching and heat-set temperatures and heat-set time.



(a) Liquid nitrogen. (b) Liquid nitrogen.

Figure 7. - Stress at break determined from uniaxial tensile tests of PET film at cryogenic temperatures as function of stretching and heat-set temperatures and heat-set time.

Stress at break. - The breaking stress is affected mainly by the stretching temperature (table I and fig. 7). In both cryogens higher stress is reached with film stretched at 85° C (358 K), the lower temperature. Values in liquid hydrogen are about 15 percent higher than those obtained in liquid nitrogen. These results show the same pattern as for stress at the proportional limit, but values of the stress at break are over twice as high. Heat-setting temperature and time did not consistently affect the difference in stress at break between liquid hydrogen and liquid nitrogen.

Biaxial Burst Properties in Liquid Nitrogen

The results obtained for the various stress-strain properties reported in this section are based on single tests of each of the eight films prepared with the eight different processing treatments of the factorial experiment. Because of the lack of a good estimate of the reproducibility of films in this test, the significance of main effects was judged statistically by comparison with higher order (interaction) effects. This intepretation is, of course, conservative in reporting significant effects. True effects of smaller size would not be declared significant and would be "missed" by this procedure. However, examination of the results indicated duplicate tests are not necessary since smaller effects than those found are not of practical value.

Calculating stress and strain. - The engineering stress is calculated from the pressure difference across the film diaphragm by using the following equation:

$$\text{Stress} = \frac{\Delta PR}{2t} \tag{2}$$

where

ΔP pressure difference across film

R radius of curvature of deflected diaphragm

t original film thickness

The engineering strain is defined as the increase in length of a diameter line drawn on the circular film specimen divided by the original diameter

$$\text{Strain} = \frac{R\theta - 2r}{2r} \tag{3}$$

where

θ central angle of diameter arc formed by deflecting film

r original radius of free-film diaphragm

Since

$$\sin \frac{\theta}{2} = \frac{r}{R} \tag{4}$$

and

$$R = \frac{r^2 + h^2}{2h} \tag{5}$$

where h is the measured deflection perpendicular to the original plane of the film, strain is calculated from the measured values of r and h. A typical biaxial stress-strain curve for PET is shown in figure 8.

Strain at the proportional limit. - This property is defined by the point of departure of the calculated stress-strain curve from the linear relation which occurs at low strain. The high heat-setting temperature of 210° C (483 K) results in strains of 1.77 percent while heat-setting at 190° C (463 K) gives 1.44 percent strain at the proportional limit

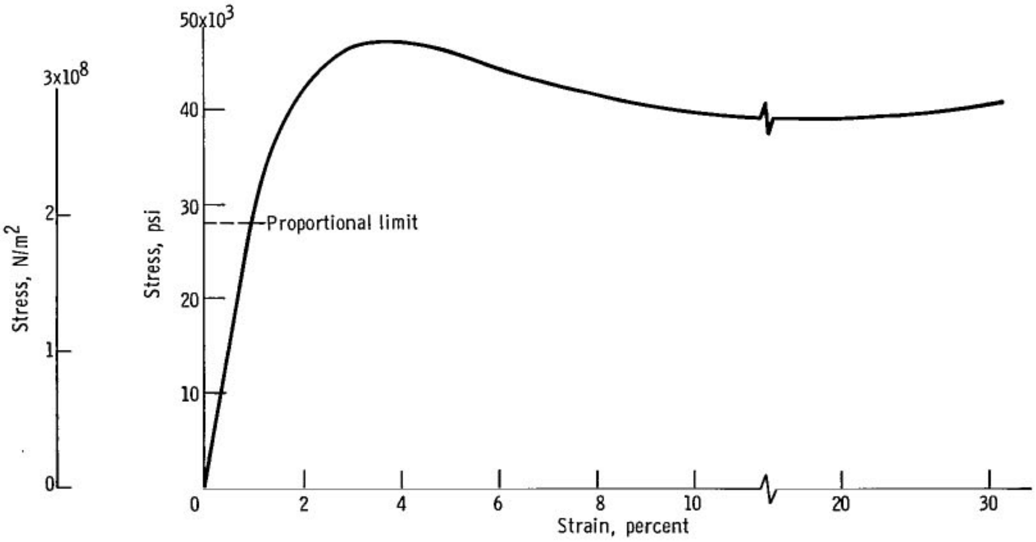
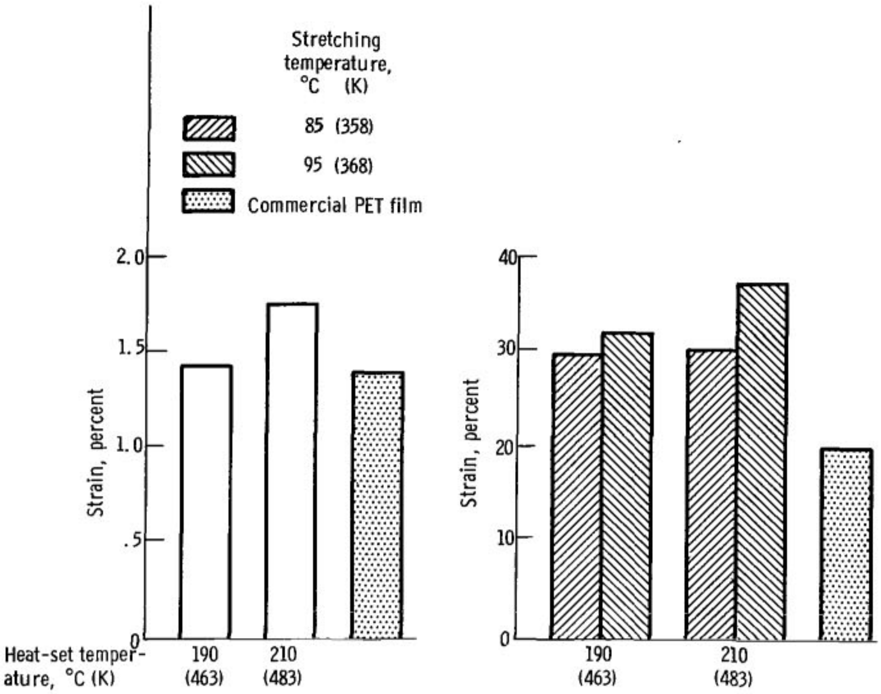


Figure 8. - Typical biaxial stress-strain curve for PET film.



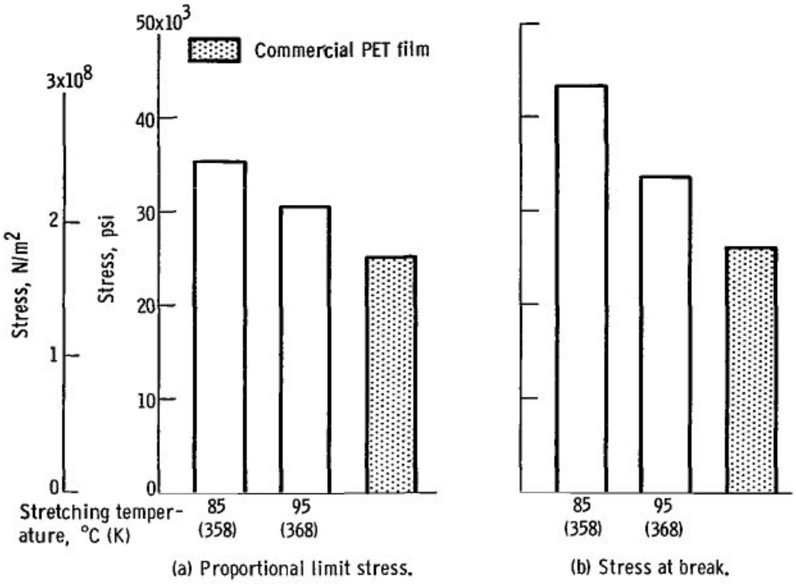
(a) Proportional limit strain. (b) Strain at break.

Figure 9. - Strain at proportional limit and at break, determined from biaxial burst tests of PET film in liquid nitrogen as function of stretch and heat-set temperatures.

(fig. 9). The difference, about one-third of a percent strain, is approximately a 25 percent increase in the value of this property, which is the same increase over the commercial PET film strain of 1.45 percent. A heat-setting temperature above 210° C (483 K) might result in further increasing the amount of strain at the proportional limit.

Strain at break. - The strain of the experimental films at break in liquid nitrogen are all far above the 17.4 percent value for the commercial PET (fig. 9). Both stretching temperature and heat-setting temperature affect this property. Films stretched at 95° C (368 K) give higher strain values than those stretched at 85° C (358 K). This effect is more pronounced if heat-setting is done at the higher temperature of 210° C (483 K). Films stretched and set at the higher temperatures averaged 37.5 percent strain, over twice the corresponding strain for the commercial film. As previously described in the section Crystallinity, higher film-processing temperatures result in high crystallinity. Thus, the most crystalline films attain the highest strain at break.

Stress at the proportional limit. - This is the stress corresponding to the strain at the proportional limit. This stress is almost 4000 psi (2.76×10^7 N/m^2) higher for films stretched at 85° C (358 K) than for those stretched at 95° C (368 K) (fig. 10). Other factors do not show significant effects on this property. The modulus of the film stretched



(a) Proportional limit stress. (b) Stress at break.

Figure 10. - Stress at proportional limit and at break determined from biaxial burst tests of PET film in liquid nitrogen as function of stretching temperature.

at 85° C (358 K) is also higher. These results are similar to those obtained in the uniaxial tensile test but are numerically higher in both stress and modulus. The biaxial modulus for the film stretched at 85° C (358 K) is 2.46×10^{6} psi (1.70×10^{10} N/m^2). These stress values are higher than those of commercial PET film.

Stress at break. - The average stress at break for films stretched at the lower temperature is over 10 000 psi (6.9×10^{7} N/m^2) greater than for those stretched at the higher temperature and 12 000 psi (8.28×10^{7} N/m^2) greater than for commercial PET (fig. 10).

Flexural Fatigue by Twist-Flex Test in Liquid Hydrogen

The results are given in table V and the large variation between duplicates (halves of the same stretched film) is evident. It was not possible to establish significant trends of the processing factors with these data. However, the average number of cycles to failure in liquid hydrogen for all 21 experimentally produced PET films tested exceed 50. In contrast, 0.5-mil (0.0127-mm) commerical PET film has been reported to withstand less than 10 cycles. (This unpublished data was obtained by R. F. Lark of Lewis on the same apparatus used in the present investigation.) Therefore, a significant and technically worthwhile cryogenic flexibility improvement has been demonstrated.

Porosity of Films Flexed in Liquid Hydrogen

Since the gas porosity of these films after flexing in liquid hydrogen varied over a wide range (table VI) of 0.01 to several hundred milliliters per minute, the analysis was based on the log of the flow rate through the film. Three films gave rates exceeding 100 milliliters per minute; such rapid rates could not be accurately determined in this apparatus. Therefore, in the analysis the uniform value of 100 was used for the porosity of such films. Because of the lack of duplicate data, the main effects of the three factors studied were compared with the combined interaction effects. The statistical "F-test" (table VII) shows that the main effects are not large enough to be established with significance. Since the interactions represent a possibly inflated estimate of error (e.g., each mean square is an estimate of the specific interaction plus error), the possibility of effects of these factors has not been eliminated by these data. A number of films which had negligible porosity after this severe treatment have been produced (table V).

SUMMARY OF RESULTS

In this study of the effects of stretching temperature, heat-set temperature and heat-set time on the cryogenic mechanical properties of poly(ethylene terephthalate) (or PET) film, the following results were obtained:

1. The crystallinity of the biaxially oriented PET film was higher for films heat-set at 210° C (483 K) than for films heat-set at 190° C (463 K). Crystallinity is also promoted by an increase in stretch temperature and set time, but to a lesser degree, for the range of this study.

2. The uniaxial strain at the proportional limit in liquid nitrogen of biaxially oriented PET film increases with an increasing set time, but there is an interaction with stretch temperature. The strain at break in both liquid nitrogen and liquid hydrogen show an increase with decreasing stretch temperature. A lower heat-set temperature increases the strain at break at liquid-nitrogen temperature.

3. The proportional limit and breaking stresses in both liquid nitrogen and liquid hydrogen are higher with the lower stretch temperature of 85° C (358 K).

4. Biaxial burst tests performed in liquid nitrogen showed that both proportional limit strain and strain at break increase with higher heat-set temperature. The breaking strain of the experimentally processed film of this investigation was almost twice that of the commerically available PET film.

5. Biaxial burst tests performed in liquid nitrogen showed that the proportional limit stress and stress at break decrease with increasing stretch temperature. These stresses measured on the film stretched at low temperature were about 40 percent higher

than for the commercially available PET film.

6. The average number of flexing cycles (twist-flex) to failure in liquid hydrogen was greater than 50 for the 21 samples tested. (Commercial PET film withstands less than 10 cycles.)

7. A number of films showed negligible porosity after 25 twist-flex cycles in liquid hydrogen, but no dependence on processing factors was established.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, May 23, 1968,
129-03-11-01-22.

APPENDIX A

REGRESSION ANALYSIS WITH INITIAL ERROR VARIANCE ESTIMATE OBTAINED FROM REPLICATION

The most general equation (model) that could be fitted to the larger experiments was given as equation (1) (p. 5) where the dependent variables were as follows:

x_A stretch temperature

x_B set temperature

x_C set time

and the quantity y is the particular observed response, such as density, breaking strain, log of flex cycles, and so forth. All the constants of that equation could be evaluated for the room-temperature experiments. The constants b_D and b_{ABC} could not be evaluated from the experiments in liquid nitrogen, and the constants b_D, b_{AC}, b_{ABC} could not be evaluated from the uniaxial experiments in liquid hydrogen.

The conditions investigated and the observed responses are summarized in table VIII and in tables III to VI and IX to XIII. Individual observations (rather than mean values) were used in fitting the regression constants. The regression procedure compared the magnitudes of the coefficients of the fullest possible model for the experiment with the estimate of error variance obtained from the residual variance. (Because of the design of the experiment and the choice of the model, the initial residual variance consisted only of replication error.) The t-test then was used to compute the descriptive significance level α (the probability that a constant as large in absolute value as that observed could have been drawn from a population having a population mean value of zero).

A stepwise statistical decision procedure was then used to simplify the model. It deleted coefficients, term by term, beginning with the least significant. All the retained coefficients were fitted, after each deletion. This process was continued until all the retained coefficients were significant at the 5 percent level, or better.

Results for the room-temperature tests are shown by table XIV. For each dependent variable there is listed first, the value of the coefficient in the full model; second, its descriptive significance level in the full model; and, third, the value of the coefficient that remained in the reduced model as a result of the stepwise deletions according to the statistical decision procedure. Values of the coefficient of multiple determination R^2 are also given for the full model and for the reduced model. This coefficient gives the fraction of variability of all the data that has been accounted for by the model.

Results for uniaxial tests in environments of liquid hydrogen and liquid nitrogen are given in table I using the same method of data reduction and presentation as is used for table XIV.

The reduced models were used to compute predicted values of responses for such combinations of the upper and lower levels of the independent variables that had been used in the experiments. These values are listed in table XV. Because the statistical decision procedure had designated certain effects as not clearly differentiated from experimental variability, the corresponding regression coefficients were set equal to zero. The resulting simplified equations sometimes resulted in equal predicted responses for differing levels of the independent variables.

APPENDIX B

ANALYSIS OF VARIANCE WITH ERROR VARIANCE OBTAINED FROM HIGHER-ORDER INTERACTIONS

Three types of experiments did not permit the estimation of error variance from replication. They were (1) the biaxial burst tests, where no replication was employed, (2) the porosity experiment, where a duplicate was used at only one test condition, and (3) the density measurements, where duplicates were obtained by dividing single specimens, so that the duplication measured only the unimportant variability within specimens, rather than the treatment-to-treatment variability.

Biaxial Burst Tests

The computation method of Yates (ref. 11) was used to analyze the results obtained from the biaxial burst test of the two-level factorial experiment. Coefficients for the response prediction equation and mean squares associated with each main effect and interaction are obtained by this procedure. In the absence of an estimate of reproducibility of duplicates, the interactions were used as estimates of error to establish the significance of the main effects as illustrated below. According to Yates' nomenclature the factor effects are designated by symbols as follows:

A	stretch temperature
B	heat-set temperature
C	heat-set time

The experiments are described by the corresponding lower-case letter when the high level of that factor was used. Absence of a letter means the lower level of the factor was used. Thus, experiment code ab means high level of factor A and B and low level of factor C. Results are shown by table XVI.

Porosity

The analysis of the porosity data proceeded in the same manner as the biaxial burst tests. Results are shown in table VII.

Density

The computer program used for the regression analysis was used to compute the mean squares. The mean squares for the four interactions were pooled to compute an estimate of error variance which was then used to test the significance of the curvature and main effects. Results are shown in table XVII.

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TABLE I. - RESPONSE MODELS FOR UNIAXIAL LOAD EXPERIMENTS IN LIQUID HYDROGEN AND LIQUID NITROGEN

Independent variables[a]	Proportional limit load			Breaking strain[b]			Breaking load			Proportional limit strain		
	Full model		Reduced model coefficient, lb	Full model		Reduced model coefficient, percent	Full model		Reduced model coefficient, lb	Full model		Reduced model coefficient, percent
	Coefficient, lb	Significance level, $1 - \alpha$		Coefficient, percent	Significance level, $1 - \alpha$		Coefficient, lb	Significance level, $1 - \alpha$		Coefficient, percent	Significance level, $1 - \alpha$	
Liquid hydrogen												
x_0	17.2026	-----	17.2522	6.40476	-----	0.257724	36.3170	-----	36.3649	2.04127	-----	2.08905
x_A	-2.62083	0.999	-2.55784	-.907500	0.935	-.034456	-4.43375	0.999	-4.40240	-.06500	0.772	--------
x_B	-.45814	.970	-.47203	-.538095	.748	-.026342	-.80625	.884	-1.06490	-.03016	.447	-.057619
x_C	-.12659	.592	--------	-.0527382	.117	--------	-.07929	.136	--------	-.02373	.548	--------
x_Ax_B	.00417	.014	--------	.167500	.276	--------	-.82125	.873	-1.14760	.03500	.489	--------
x_Bx_C	.08770	.435	--------	-.205952	.045	--------	.58500	.794	--------	.00484	.121	--------
Coefficient of multiple determination, R^2	0.889	-----	0.884	0.289	-----	0.157	0.793	-----	0.780	0.234	-----	0.1396
Liquid nitrogen												
x_0	14.9531	-----	14.8075	8.26443	-----	0.327104	31.7868	-----	31.9679	1.86076	-----	1.86919
x_A	-.68750	0.995	-.79389	-3.08244	0.998	-.109459	-2.58825	0.999	-2.40708	-.00486	0.081	--------
x_B	.30580	.823	--------	-3.19271	.998	-.115021	-.40834	.822	--------	.01257	.223	--------
x_C	1.24330	.999	1.20097	-.195982	.172	--------	.43259	.884	.56380	.18048	.999	.19039
x_Ax_B	.60268	.983	.48561	-.247917	.198	--------	1.16042	.999	1.26899	-.01882	.300	--------
x_Ax_C	.85268	.999	.77107	-.20786	.972	-.083264	.20759	.554	--------	.11173	.990	.119917
x_Bx_C	-.04688	.168	--------	-.665625	.514	--------	-.15625	.412	--------	-.00938	.159	--------
Coefficient of multiple determination, R^2	0.713	-----	0.696	0.356	-----	0.345	0.800	-----	0.781	0.487	-----	0.4841

[a] Stretching temperature, x_A; set temperature, x_B; set time, x_C.

[b] Breaking deformations.

TABLE II. - MODULUS AVERAGED OVER HEAT-SET TEMPERATURES

Test liquid	Stretch temperature		Heat-set time, sec	Modulus of elasticity	
	°C	K		psi	N/m^2
N_2	85	358	15	1.25×10^6	8.7×10^8
N_2	85	358	120	1.15	7.9
N_2	95	368	15	1.21	8.3
N_2	95	368	120	.87	6.0
H_2	85	358	15 and 120	1.35	9.3
H_2	95	368	15 and 120	1.00	6.9

TABLE III. - UNIAXIAL TENSILE TEST: STRAIN AT PROPORTIONAL LIMIT

Heat set			Liquid nitrogen		Liquid hydrogen	
Temperature		Time, sec	Stretch temperature			
°C	K		85° C (358 K)	95° C (368 K)	85° C (358 K)	95° C (368 K)
			Average strain, percent per number of tests averaged			
210	483	120	1.96/8	------	2.02/9	------
210	483	15	1.81/4	1.54/6	2.06/4	2.00/1
190	463	120	1.94/8	2.19/8	2.14/7	------
190	463	15	1.78/8	1.59/7	2.20/6	2.00/2

TABLE IV. - UNIAXIAL TENSILE TEST: STRAIN AT BREAK

Heat set			Liquid nitrogen		Liquid hydrogen	
Temperature		Time, sec	Stretch temperature, °C (K)			
°C	K		85 (358)	95 (368)	85 (358)	95 (368)
			Average strain, percent per number of tests averaged			
210	483	120	10.0/8	------	6.5/6	-----
210	483	15	6.8/8	5.0/6	6.8/5	5.2/3
190	463	120	17.5/8	6.5/13	8.0/7	-----
190	463	15	12.3/8	9.2/8	7.2/6	6.0/2

TABLE V. - TWIST-FLEX CYCLES TO FAILURE IN LIQUID HYDROGEN

Heat set			Stretching temperature, °C (K)					
Temperature		Time, sec	85 (358)		90 (363)		95 (368)	
°C	K		Crack-free cycles	Cycles to failure	Crack-free cycles	Cycles to failure	Crack-free cycles	Cycles to failure
---	---	---	75	85	--	--	80	90
---	---	---	---	---	--	--	[a]120	[a]130
190	463	15	90	100	--	--	80	90
		15	[a]25	[a]35	--	--	[a]25	[a]35
		120	100	110	--	--	80	90
		120	[a]30	[a]40	--	--	[a]30	[a]40
200	473	42	---	---	35	40	---	----
			---	---	40	50	---	----
			---	---	35	45	---	----
			---	---	35	45	---	----
210	483	15	125	130	--	--	30	35
		15	[a]10	[a]15	--	--	[a]5	[a]10
		120	35	40	--	--	60	75

[a] Duplicate, half of material above.

TABLE VI. - POROSITY OF PET AFTER 25 CYCLES OF TWIST-FLEX IN LIQUID HYDROGEN

[Experimental results in ml of helium/min through 3- by 9-in. (7.62- by 20.3-cm) test section of film at room temperature with 1-psi (6.9×10^{3}-N/m^{2}) pressure gradient.]

Heat set			Stretching temperature, °C (K)		
Temperature		Time, sec	85 (358)	90 (363)	95 (368)
°C	K		Porosity, ml of helium/min		
---	---	---	6.0	--	4.2
190	463	15	>100	--	.1
190	463	120	.1	--	.01
200	473	42	-------	0	------
210	483	15	1.68	--	100
210	483	15	3.43	--	------
210	483	120	>100	--	.06

TABLE VII. - SUMMARY OF STATISTIC ANALYSIS OF DATA ON POROSITY (LOG SCALE) AFTER 25 CYCLES OF TWIST-FLEX

Exponent code[a]	Response, y				Coefficient, b	Mean-square error	Observed F ratio	Effect
	(1)	(2)	(3)	(4)				
1	2.0	2.0	3.4	1.2	0.15	0.18		
a	.4	1.0	-2.2	5.2	.65	3.38	1.6	A
b	-1.0	1.0	1.4	-5.6	-.70	3.92	1.9	B
ab	2.0	-3.2	3.8	2.4	.30	.72		
c	-1.0	-1.6	-1.4	-5.6	.70	3.92	1.9	C
ac	2.0	3.0	-4.2	2.4	.30	.72 [b]2.10		
bc	-2.0	3.0	4.6	2.8	.35	.98		
abc	-1.2	.8	-2.2	-6.8	.85	5.78		
Total	1.2					19.6		

[a]Yates nomenclature.

[b]Pooled error.

TABLE VIII. - UNIAXIAL TESTS AT 25° C (298 K)

Heat-set time, sec	Heat-set temperature		Stretch temperature		Breaking stress, kips/sq in.	Breaking strain, percent
	°C	K	°C	K		
15	190	463	85	358	26.4	90
	190	463	95	368	18.6	103
	210	483	85	358	29.6	114
	210	483	95	368	22.2	127
120	190	463	85	358	25.9	78
	190	463	95	368	20.7	102
	210	483	85	358	28.7	111
	210	483	95	368	23.2	112
0	---	---	85	358	23.2	125
42	200	473	95	368	19.2	116
0	---	---	95	368	16.6	---

TABLE IX. - UNIAXIAL TENSILE TESTS AT 25° C (298 K) AND FILM SPECIFIC GRAVITY

Stretch temperature		Heat-set			Stress at break[a]				Strain at break,[a] percent		Specific gravity of film[a]		Crystallinity, percent
°C	K	Temperature		Time, sec	psi		N/m^2						
		°C	K										
85	358	210	483	15	29.0×10^3	30.3×10^3	2.00×10^8	2.09×10^8	108	121	1.3869	1.3869	45.5
85	358	210	483	120	27.6	29.8	1.90	2.05	102	120	1.3882	1.3879	46.4
85	358	190	463	15	25.8	27.0	1.78	1.86	87	94	1.3816	1.3828	41.8
85	358	↓	↓	120	25.6	26.2	1.77	1.81	75	80	1.3844	1.3842	43.4
95	368			15	17.6	19.7	1.21	1.36	90	116	1.3846	1.3847	43.7
95	368			120	19.4	22.0	1.34	1.52	93	111	1.3846	1.3849	43.8
95	368	210	483	15	23.0	21.4	1.59	1.48	136	118	1.3876	1.3879	46.2
95	368	210	483	120	23.0	23.4	1.59	1.62	108	116	1.3906	1.3906	48.5
95	368	---	---	0	16.6	--------	1.14	--------	---	---	1.3561	1.3562	20.9
85	358	---	---	0	22.5	23.8	1.55	1.64	122	128	1.3495	1.3500	15.8
90	363	200	473	42	18.9	19.6	1.30	1.35	111	122	1.3870	1.3874	45.8
90	363	200	473	42	--------	--------	--------	--------	---	---	1.3860	1.3859	44.8

[a] Two samples were tested at each condition.

TABLE X. - UNIAXIAL TENSILE TEST: STRESS AT PROPORTIONAL LIMIT

(a) U. S. Customary Units

Heat set		Liquid nitrogen		Liquid hydrogen	
Temperature, °C	Time, sec	Stretch temperature, °C			
		85	95	85	95
		Average stress, psi per number of tests averaged			
210	120	22 400/8	--------	27 700/9	-------
210	15	21 400/4	18 700/4	27 700/5	20 300/3
190	120	23 400/8	22 200/13	28 700/7	--------
190	15	22 200/8	16 000/7	29 300/6	21 700/2

(b) SI Units

Heat set		Liquid nitrogen		Liquid hydrogen	
Temperature, K	Time, sec	Stretch temperature, K			
		358	368	358	368
		Average stress, N/m^2 per number of tests averaged			
483	120	$(1.55\times10^8)/8$	-----------	$(1.91\times10^8)/9$	-----------
483	15	$(1.48\times10^8)/4$	$(1.29\times10^8)/4$	$(1.91\times10^8)/5$	$(1.40\times10^8)/3$
463	120	$(1.61\times10^8)/8$	$(1.53\times10^8)/13$	$(1.98\times10^8)/7$	-----------
463	15	$(1.53\times10^8)/8$	$(1.10\times10^8)/7$	$(2.02\times10^8)/6$	$(1.50\times10^8)/2$

TABLE XI. - UNIAXIAL TENSILE TEST: STRESS AT BREAK

(a) U. S. Customary Units

Heat set		Liquid nitrogen		Liquid hydrogen	
Temperature, °C	Time, sec	Stretch temperature, °C			
		85	95	85	95
		Average stress, psi per number of tests averaged			
210	120	46 900/8	--------	59 000/7	--------
210	15	46 800/8	42 100/6	57 600/5	42 600/4
190	120	51 900/8	41 700/14	57 300/7	--------
190	15	50 900/8	39 600/7	59 100/6	48 900/4

(b) SI Units

Heat set		Liquid nitrogen		Liquid hydrogen	
Temperature, K	Time, sec	Stretch temperature, K			
		358	368	358	368
		Average stress, N/m^2 per number of tests averaged			
483	120	$(3.23\times10^{8})/8$	------------	$(4.07\times10^{8})/7$	-----------
483	15	$(3.22\times10^{8})/8$	$(2.90\times10^{8})/6$	$(3.97\times10^{8})/5$	$(2.94\times10^{8})/4$
463	120	$(3.58\times10^{8})/8$	$(2.88\times10^{8})/14$	$(3.95\times10^{8})/7$	-----------
463	15	$(3.51\times10^{8})/8$	$(2.73\times10^{8})/7$	$(4.08\times10^{8})/6$	$(3.37\times10^{8})/4$

TABLE XII. - BIAXIAL BURST TESTS ON EXPERIMENTAL PET FILMS IN LIQUID NITROGEN

Stretching temperature		Heat set			Proportional limit			Break		
°C	K	Temperature		Time, sec	Stress		Strain, percent	Stress		Strain, percent
		°C	K		psi	N/m^2		psi	N/m^2	
85	358	190	463	15	34 000	2.35×10^{8}	1.86	42 500	2.93×10^{8}	28.2
		190	463	120	36 000	2.49	1.25	42 500	2.93	31.0
		210	483	15	37 500	2.59	1.86	40 500	2.80	31.0
		210	483	120	34 000	2.35	1.60	39 500	2.73	30.4
95	368	190	463	15	32 000	2.21	1.35	30 500	2.10	33.0
		190	463	120	28 000	1.86	1.30	31 000	2.14	31.2
		210	483	15	33 500	2.31	1.80	31 500	2.17	37.5
		210	483	120	33 000	2.28	1.80	31 000	2.14	37.4

TABLE XIII. - BIAXIAL BURST TESTS ON COMMERCIAL PET FILM IN LIQUID NITROGEN

Sample	Proportional limit			Break		
	Stress		Strain, percent	Stress		Strain, percent
	psi	N/m^2		psi	N/m^2	
1	24 000	1.65×10^8	1.52	30 500	2.10×10^8	7.6
2	28 000	1.93	1.52	28 000	1.93	22.4
3	24 000	1.65	1.50	27 500	1.90	17.0
4	22 500	1.55	1.25	27 500	1.90	20.9
5	22 500	1.55	1.20	31 000	2.14	9.3
6	27 000	1.86	1.50	28 000	1.93	27.8
7	26 200	1.81	1.55	30 000	2.10	10.1
8	25 000	1.72	1.20	31 000	2.14	8.1
9	28 500	1.97	1.75	27 500	1.90	20.5
10	26 000	1.79	1.55	27 200	1.88	20.5
Average	25 730	1.75×10^8	1.45	28 850	1.99×10^8	17.4

TABLE XIV. - RESPONSE MODELS FOR ROOM-TEMPERATURE EXPERIMENTS

Independent variables[a]	Breaking load			Breaking strain		
	Full model[b]		Reduced model coefficient, lb	Full model[b]		Reduced model coefficient, percent
	Coefficient, lb	Significance level, $1 - \alpha$		Coefficient, percent	Significance level, $1 - \alpha$	
x_0	19.2500	-----	19.2500	116.5000	-----	106.0000
x_A	-2.2625	0.999	-2.2625	6.3125	0.957	6.3125
x_B	1.0625	.999	1.0625	11.4375	.998	11.4375
x_C	.1375	.492	-------	-4.0625	.835	--------
$x_A x_B$	.0000	.000	-------	-2.9375	.698	--------
$x_A x_C$	.4000	.924	..4000	.0625	.017	--------
$x_B x_C$	-.1250	.454	-------	-.5625	.161	--------
$x_A x_B x_C$	-.0625	.237	-------	-2.9375	.698	--------
$\frac{1}{3}\left(x_A^2 + x_B^2 + x_C^2\right)$	-2.1500	.994	-2.1500	-11.8125	.823	--------
Coefficient of multiple determination, R^2	0.951		0.946	0.772		0.599

[a] Stretch temperature, x_A; set temperature, x_B; set time, x_C.
[b] Most general polynomial representing design of experiment, eq. (1).

TABLE XV. - RESPONSES PREDICTED FROM REDUCED MODELS

Dependent variable[a]	Stretch temperature, $^{\circ}C$ (K)								Figure
	85 (358)				95 (368)				
	Heat-set temperature, $^{\circ}C$ (K)								
	190 (463)		210 (483)		190 (463)		210 (483)		
	Heat-set time, sec								
	15	120	15	120	15	120	15	120	
Breaking stress (room temperature)	26.714	25.571	29.750	28.607	19.107	20.250	22.143	23.286	2
Break strain (room temperature)	88.25	88.25	111.13	111.13	100.88	100.88	123.75	123.75	3
Proportional load strain (liquid nitrogen)	1.7987	1.9397	1.7987	1.9397	1.5589	2.1795	1.5589	-------	4
Proportional load strain (liquid hydrogen)	2.1467	2.1467	2.0314	2.0314	2.1467	--------	2.0314	-------	--
Proportional load stress (liquid nitrogen	22.367	23.596	20.980	22.209	16.509	22.143	17.896	-------	5
Proportional load stress (liquid hydrogen)	28.974	28.974	27.626	27.626	21.666	--------	20.317	-------	--
Breaking strain (liquid nitrogen)	11.7075	15.8700	5.9575	10.1200	10.3975	6.2350	4.6475	-------	6
Breaking strain (liquid hydrogen)	7.9625	7.9625	6.6450	6.6450	6.2400	--------	4.9225	-------	--
Breaking stress (liquid nitrogen)	50.114	51.726	46.489	48.100	39.611	41.223	43.237	-------	7
Breaking stress (liquid hydrogen)	58.121	58.121	58.357	58.357	48.821	--------	42.500	-------	--

[a] The quantities opposite stress are stresses in kips/in.^{2} and the quantities opposite strain are percent strain.

TABLE XVI. - SUMMARY OF STATISTICAL ANALYSIS OF BIAXIAL BURST TEST DATA

Response and code	Exponent code	Response, y	Coefficient,[a] b_i	Mean-square error		Observed F ratio	Effect
Stress at proportional limit, S_p, psi	1	4.0	3.50	98.00		------	--
	a	2.0	-1.875	28.125		[b]7.6	A
$y_1 = \frac{S_p - 30\ 000}{1000}$	b	7.5	1.00	8.00		2.1	B
	ab	3.5	.625	3.125	[c]3.7	------	--
	c	6.0	-.75	4.50		1.5	C
	ac	-2.0	-.375	1.125		------	--
	bc	4.0	-.25	.50		------	--
	abc	3.0	1.125	10.125		------	--
Total		28.0		153.5			
Proportional limit strain, ϵ_p, percent	1	0.86	0.6025	2.90405		------	--
	a	.35	.0400	.0128		0.3	A
$y_2 = \epsilon_p - 1.0$	b	.86	.1625	.21125		[b]5.3	B
	ab	.80	.0750	.045	[c]0.04	------	--
	c	.25	.1150	.1058		2.6	C
	ac	.30	.1025	.08405		------	--
	bc	.60	.0500	.02		------	--
	abc	.80	.0375	.01125		------	--
Total		4.82		3.3942			
Ultimate stress, S_b, psi	1	12.5	6.125	300.125		------	--
	a	.5	-5.125	210.125		[b]164	A
$y_3 = \frac{S_b - 30\ 000}{1000}$	b	10.5	-.50	2.00		1.6	B
	ab	1.5	.75	4.50	[c]1.28	------	--
	c	12.5	.125	.125		.1	C
	ac	1.0	.125	.125		------	--
	bc	9.5	.25	.50		------	--
	abc	1.0	0	0		------	--
Total		49.0		517.5			
Ultimate strain, ϵ_b, percent	1	-1.8	2.4625	48.51125		------	--
	a	3.0	2.3125	42.78125		[b]11.6	A
$y_4 = \epsilon_b - 30$	b	1.0	1.6125	20.80125		[b]5.6	B
	ab	7.5	1.0675	9.03125	[c]3.7	------	--
	c	1.0	.0375	.01125		0	C
	ac	1.2	.5125	2.10125		------	--
	bc	.4	.2125	.36125		------	--
	abc	7.4	.6375	3.25125		------	--
Total		19.7		126.85			

[a] Coefficients of regression model in order of eq. (1).

[b] Significance at 0.90 level.

[c] Pooled error.

TABLE XVII. - SUMMARY OF REGRESSION ANALYSIS OF DENSITY RESPONSE

Source	Mean-square error		Observed F ratio
Stretch temperature, A	992.2		[a]9.2
Heat-set temperature, B	7569.0		[a]70.1
Heat-set time, C	961.0		[a]8.5
Interaction, A × B	6.2	[b]108	------
Interaction, A × C	2.2		------
Interaction, B × C	81.0		------
Interaction, A × B × C	342.2		------
Curvature	57.8		0.54

[a] Significant at 0.95 level.

[b] Pooled error.

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