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to

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STUDY OF FRACTURE AS RELATED TO POLYMER STRUCTURE

Under the Direction of

Lawrence J. Broutman Department of Mechanics

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I. INTRODUCTION

Ballistically impacted materials, particularly glassy polymers such as Plexiglas, fail in a complex manner with great amounts of internal crack initiation and propagation contributing to the total failure process. Crack initiation and propagation require or absorb large amounts of energy and so offer a means of dissipating projectile energy. Providing one understands the failure process, new materials or simply new combinations of existing materials can be rationally designed to better resist impact and fracture. This requires knowledge of the material structure and its influence on the fracture process, particularly the energy required to initiate and propagate a crack. The purpose of this research study is to answer this question. A complete solution to the above problem must eventually include dynamic effects such as the influence of crack velocity on crack propagation energy and mode of crack propagation.

II. PERSONNEL

This research program is under the direction of Dr. Lawrence J. Broutman in the Department of Mechanics. Mr. Takao Kobayashi and Mr. Michael Bein, graduate assistants, and Mr. Mike Laingor, laboratory technician have contributed to the program this quarter.

III. OBJECTIVES

The objective of this program is to study the fracture of glassy organic polymers so that a better understanding between the polymer structure and fracture properties can be gained. The fracture surface work and fracture surface characteristics will be studied. Polymer structural features to be studied include 1) chain stiffness, configuration and intermolecular bonding by studying homologous series of acrylates and methacrylates; 2) degree of crystallinity; 3) crosslinking; 4) prior molecular orientation. The ambient temperature will be varied so comparisons of polymers can be made in equivalent states.

IV. WORK ACCOMPLISHED

1. Design and Construction of Load Machine

The loading machine which was designed and built almost completely in the first quarter of this contract has been modified during the second quarter to improve the accuracy of measurements particularly at liquid nitrogen temperatures. The loading grips have been modified so that the entire specimen including a portion of the grip can be immersed in the liquid nitrogen container. The specimen is thus loaded such that the loading axis of the specimen is below the axis passing through the load cell and loading carriage. A moment is produced which can cause a vertical displacement of the loading grip. We have modified the machine by restraining the vertical displacement of the loading grip through a roller bearing so that only a horizontal displacement occurs along the axis between the load cell and load carriage. The grip itself has been stiffened and insulated to prevent a reduction in load cell temperature.

2. Design of Double Cantilever Cleavage Specimen

The design of the test specimen to measure the fracture surface work, γ , was discussed in the first quarterly report. Further analyses and comparisons with other methods have been

conducted this quarter and are presented in this section. In the first report we represented the force-deflection relationship





for the specimen shown in Figure 1 as

$$f = \frac{a\delta}{\ell^n}$$
(1)

although for a cantilevel beam with fixed ends and neglecting shear deflections this equation is

$$f = \frac{3EI\delta}{l^3}$$
(2)

where a=3EI and n=3. In the analysis of the first report (eq. 7) it was shown that the fracture surface work, γ , could be determined from

$$\frac{f\delta}{b_n} = \frac{2\gamma \ell}{n} \tag{3}$$

where b_n is crack width and n is experimentall; determined from experiment but is typically 2.6 to 2.7. The actual analysis of the data is discussed in the next section.

In addition to using the force-deflection relationship as shown in eq. (1), account can be taken of shear deflections and end rotations separately and then added to the bending deflections predicted by eq. (1). For example, the shear deflection, assuming the beam ends do not warp, is found from the equation: ⁽¹⁾

$$\delta_{\rm s} = \frac{1}{10} \frac{f\ell h^2}{EI} \frac{E}{G} \tag{4}$$

It is assumed that Poisson's ratio, μ , is 0.35 then

$$\delta_{\mathbf{g}} = \frac{0.27 \text{ flh}^2}{\text{EI}} \tag{5}$$

or

$$\delta_{g} = \frac{3.24 \text{ fl}}{\text{Ebh}} \tag{6}$$

The cantilever specimen in our experiment does not have fixed rigid ends but more likely an elastic support which causes additional deflections due to end rotations. This effect has been studied both analytically and experimentally by C'Donnell⁽²⁾ and he found that the following equation represents the deflection due to the end support effect:

$$\delta_{r} = \frac{16.67 f l^2}{\pi E h^2} + \frac{(1-v) f l}{E h}$$

or
$$\delta_r = \frac{fl}{E} \left(\frac{5.31 \ l}{h^2} + \frac{0.65}{h} \right)$$
 (7)

Now, the deflection measured in the experiment using the double cantilever cleavage specimen can be made equal to the sum of the bending, shear and end rotation deflections

$$\delta_{exp} = \delta_{b} + \delta_{s} + \delta_{r}$$
(8)

and the deflection, ${}^{\delta}_{b}$, due to the bending effects alone can be obtained from this equation. Furthermore this calculated bending deflection can then be compared to the theoretical bending deflection predicted by eq. 2.

The above comparisons have been made for Plexiglas specimens cleaved in liquid nitrogen (-196°C). The linear forcedeflection experimental measurement for a sample with h=0.6 inch, b=0.135 inch, $b_n=0.088$ inch and l=3.401 inches is shown in Figure 2. The comparison of the bending deflection calculated from eq. 8, to the experimental deflection (chosen from an arbitrary point on the force-deflection curve in Fig. 2) and to the theoretical bending deflection is shown in table 1. Results from two other samples are also shown in this table. It is evident that the shear and end rotation effects are a small portion of the total experimental deflection. Furthermore, the bending deflection obtained from subtracting the shear and end rotation from the total deflection agrees almost identically with the bending deflection calculated from $\frac{fl^3}{3EI}$ which indicates the accuracy of the equations 6 and 7. The choice for the elistic modulus will effect the comparison of the bending deflections and



<u>Table 1</u>

Comparison of Bending Deflections for Double Cantilever Cleavage Specimen

Specimen Number	Crack Length Inches	^δ exp (inches)	^ర s	<u>^</u> r_	^б ь	₀̂ _b -eq 2
1	3.401	.0758	.0018	.0023	.0717	.0719
2	3.449	.0971	.0023	.0030	.0918	.0938
3	3.400	.0955	.0022	.0033	.0900	.0900
4**	6.998	.2057	.0012	.0056	.1989	.1976

*arbitrarily chosen deflection from experimental force-deflection curve

**room temperature experiment E = 400,000 psi (h = .6, b = .125")

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a modulus value of 750,000 psi was chosen for Plexiglas at liquid nitrogen temperature.⁽³⁾ The significance of this analysis applied to calculation of fracture surface work will be discussed in the Data Analysis section.

In the design of a suitable double cantilever cleavage specimen the height of beam to length of beam can be chosen so as to minimize shear effects and end rotation effects. It can be observed from the presented equations that the bending deflection is dependent upon l^3 , the end rotation deflection upon l^2 and the shear deflection upon l so that as the beam lengthens the shear and end rotation deflections become less significant. The choice of the beam length or crack lengths can be made so as to be able to neglect the effects of shear and end rotations. Figure 3, which can be used for specimen design, indicates the percentage of shear deflection (does not include end rotation effects) present for various beam heights and lengths. Similar curves are now being constructed to include end rotation effects and will be presented in the next quarterly report.

Care must be exercised in choosing the dimensions of a double cleavage specimen particularly in choosing the height of the beam. This is important since the maximum tensile flexural stress in the beam will be dependent upon the beam height, h, as follows:

$$\sigma = \frac{6fl}{bh^2} \tag{9}$$

If the flexural stress becomes too great then the stress-strain relation for the particular polymer may become non-linear which



will invalidate the use of our simple elastic bending equations. Even more important, if the polymer tends to craze when subjected to a critical stress as does polystyrene, a high flexural stress will cause crazing of the beam which will accompany the growth of the crack. This has been observed in our experiments with polystyrene and will be discussed later. However, if one can control the crazing by appropriate choice of beam height then one can also calculate the energy absorbed in the crazing process. Figure 4 indicates the limiting loads which can be applied to uniform double cantilever cleavage specimens for various geometry specimens assuming that maximum bending stresses are not to exceed 2500 psi. These curves can then be an aid in judging the suitability of the test specimen.

3. Data Analysis

The analysis of the data this quarter proceeded as outlined in the first quarterly report. A computer program was prepared for data analysis which included a data plotting sub routine. Values for n and γ are provided including standard deviations in addition to the plots of log f/δ vs log ℓ and $f\delta/b_n$ vs ℓ . Typical plots for Plexiglas at room temperature are shown in Figs. 5 and 6. A line has been drawn in by hand according to the correct slope and intercept. In these analyses the experimental deflection was used and in most cases the exponent n ($f = \frac{a\delta}{\ell^n}$) was approximately 2.7. Ideally, it is desirable to obtain at least 10 data prints per sample so that a statistically significant straight line can be fit to these points. In cases where material is in short supply requiring small length specimens





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or in cases where crack propagation allows determination of only one point per sample, (e.g. Plexiglas in liquid nitrogen) γ can be simply calculated for each sample by assuming a value for n and then solving for γ from eq. 3. Several specimens can be evaluated in order to determine an average value of the fracture work. More detailed discussion of data analysis will be presented in the sections describing the experiments on the various materials investigated.

One point that should be clarified is the effect of using the experimental deflection or the bending deflection only on the value of the fracture surface work. This was investigated using the data which was already presented in table 1 for the Plexiglas samples measured at liquid nitrogen temperature. In one case, γ was calculated from eq. 3 using an approximate value of n=2.7 and the experimental deflection at inception of crack propagation. This calculated value of γ was compared to a value calculated by first finding $\delta_{\rm b}$ (table 1) and using n=3 in eq. 3. The results are shown in table 2. The values are within 5% which is excellent agreement considering an arbitrary value of n was chosen based on results from previous experiments.

4. Investigations with Polymethylmethacrylate (Plexiglas)

A. Room Temperature Experiments (T=75-&0°F)

Early in this quarter experiments were performed on Plexiglas cast sheets in order to measure the value of fracture surface work, γ . Since this value has already been determined by this investigator⁽⁴⁾ we used these measurements primarily to check the use of our recently constructed test machine. Also in previous experiments⁽⁴⁾ measurement of fracture surface worl for

Table 2

Comparison of Fracture Surface Work Calculated Using Experimental or Bending Deflections

Specimen Number	$\frac{\gamma_{exp}(\frac{\text{in-lb}}{\text{in}^2})^*}{\text{in}^2}$	$\frac{\gamma_{b}(\frac{\text{in-lb}}{\text{in}^{2}})^{**}}{\text{in}^{2}}$
1	7.85	8.24
2	7.87	8.27
3	6.22	6.54
*use **use	δ _{exp} and n=2.7 δ _b and n=3	

Plexiglas was made by growing a crack continuously through the sample separating the specimen ends at constant rates. In the experiments this guarter, the fracture surface work was measured by obtaining critical forces and deflections at various crack lengths. However, the crack was not grown continuously through the sample. The procedure was to separate the specimen ends at a constant rate until crack propagation occurred and the values of crack length, crack widt., force and deflection at this point are sufficient for the calculation of fracture surface work. Forcedeflection curves are shown for crack lengths of 6.58, 6.99 and 7.69 inches (Fig. 7). These curves are characterized by a deviation from non-linearity which represents crack growth in the specimen. As the crack lengthens or the beam length increases the compliance ($2\delta/f$) of the cleavage sample decreases. A maximum in the force deflection curve occurs as the crack lengthens faster than the rate of separation of the ends. We have chosen to use the force and deflection values at the onset of crack initiation. The crack length can be measured before the experiment and the crack is then observed with a telemicroscope to record the onset of propagation by making a pip in the force deflection curve. It was determined by inspection of the curves that this corresponds to the beginning of non-linearity in the curve. The position of the grack or crack length can also be accurately measured after the conclusion of the experiment for each point since the crack front has a characteristic fingernail-like appearance. For Plexiglas, as many points as desired can be obtained from a single specimen since the growth of the crack to



Table 3

Fracture Surface Work for Plexiglas*

Samp	le Dime	ensions (inches)	Temperature (°C)	n	$\gamma(erg/cm^2)$
h	b	(total length)			
0.6	.246	12	25	2.69	1.10x10 ⁵
0.6	.130	12	25	2.61	0.98x10 ⁵
0.6	.130	8	-196	2.70	12.7x10 ^{5**}
*Previous investigations ⁽⁴⁾ give a value $\gamma=1.25\times10^5$ erg/cm ² **Average of γ_{exp} for three samples shown in table 2					

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a new position can be accurately controlled. The fracture surfare work values for two Plexiglas samples are shown in table 3. The agreement between these current values and the value obtained in a previous investigation is excellent especially when you consider that different test methods were employed.

B. Liquid Nitrogen Measurements

In order to evaluate the fracture surface work of Plexiglas at -196°C a different experimental procedure must be used. After preparation of test specimens, cracks were introduced by separation of the specimen ends at room temperature until desired crack lengths were obtained. The samples were then immersed in the liquid nitrogen and remained in the dewar for at least one hour. One of the samples was then placed in the loading grips and the force deflection curve measured while the other samples also still remained in the dewar. The force deflection curve is shown in Fig. 2. Crack lengths were measured after the experiment by examination of the characteristic markings of the fracture surface.

In these experiments, it is only possible to make one measurement per sample since the crack propagates completely through the sample after initiation and the specimen does not arrest the crack growth. The average fracture surface work value for 3 specimens was 12.7×10^5 erg/cm² as shown in table 3. This is ten times greater than the value at 25°C. It is significant to note that this compares to a value of 10×10^5 erg/cm² determined by Berry⁽³⁾ through the use of edge notched tensile specimens.

5. Investigations with Polyvinylchloride

A polyvinyl chloride was obtained from Borden Monomer-Polymer Laboratories and molded in a press with the addition of 3pph of a tin stabilizer to prevent thermal degradation during the compression molding. The molding conditions were described in the first quarterly report.

Cleavage specimens were prepared in the standard manner using a 6 mil groove on each side of the sample. It was not possible to propagate a crack through this material at 25°C due to yielding of the material near the crack. However, two experiments were conducted in liquid nitrogen with two different specimens and the values of fracture surface work were 17.7×10^5 erg/cm² and 13.3×10^5 erg/cm² or an average value of 18×10^5 erg/cm² which is greater than the value determined for Plexiglas. Studies with the material will be continued to obtain additional data.

6. Investigations with Atactic Polystyrene

A. Room Temperature Studies

Two types of polystyrene have thus far been investigated. A commercially available sheet material and a material in pellet form (M.W. = 230,000) obtained from Borden Monomer and Polymers Laboratories. The material received from Borden was molded in accordance with the specifications given in the first quarterly report. The interpretation of the experiments with polystyrene are made complex by the crazing which can occur due to the high bending stresses alone or due to the stress intensification near the tip of the crack.

Specimens were prepared from 1/4 inch and 1/8 inch polystyrene sheets which were purchased from a local supplier. The specimen heights were 1.2 inches so each beam was 0.6 inch in height. The force-deflection curves differed from those of Plexiglas and were quite linear till propagation of the crack. The values of fracture surface work for the 1/4 inch and 1/8 inch thick specimens were 4.55×10^5 and 4.2×10^5 erg/cm², respectively. These values agree well with previously measured values.⁽⁴⁾ Calculation of the maximum flexural stresses for these specimens yields values of approximately 2600 psi which is below the typical crazing stress for polystyrenes.

Specimens were prepared from the plates molded of the polystyrene (M.W. = 230,000) pellets. Four specimens have been evaluated in a manner similar to the other materials described and the values for the specimens are 7.8×10^5 , 7.55×10^5 , 5.95×10^5 and 5×10^5 erg/cm². The geometry of these four samples are identical (b=1/8 inch) and each has a total height of only 0.5 inches so that the beam height is only 0.25 inches. Using values of load and crack length obtained from force-deflection curves, the maximum tensile stresses in these beams reaches 5000 psi. Crazing has been observed in these samples during crack propagation and the high values of fracture surface work may be a result of this crazing which accompanies crack growth. We are currently preparing additional samples of this material so that we can verify these results and give more attention to the crazing phenomenon by using specimens with different heights.

B. Liquid Nitrogen Studies

Polystyrene samples have been investigated at liquid nitrogen temperatures but values of fracture surface work have not yet been measured. An extensive amount of crazing occurs which forces the crack to immediately grow out of the notched plane. This behavior has not yet been resolved.

7. Investigation of γ -Irradiated Polystyrene

Six samples of polystyrene (M.W.=230,000) have been irradiated by a Cobalt 60 source located at IIT Research Institute. Irradiation dosages were 5×10^6 rads (2 specimens), 10×10^6 rads (2 specimens) and 25×10^6 rads (2 specimens). The specimens were completely machined before irradiation treatments. The specimen dimensions were b=.125", $\ell=6$ " and h=0.5". Wall and Brown⁽⁵⁾ have shown that a dose of 6 to 8 megarads of γ irradiation is sufficient to induce crosslinking in polystyrene. We are therefore investigating the effects of crosslinking on polystyrene on the fracture surface work and crack propagation modes. The results thus far obtsined are shown in table 4.

Although it appears that the γ irradiated specimens have lower values of surface work than the unirradiated polystyrene samples of the same geometry, the fact that the flexural stresses are too high make the interpretation of this data more complex. Additional data is required and we are currently having additional polystyrene samples irradiated up to 100 megarads. These samples have a total height of 1.2 inches so that lower stresses will be produced and more effective comparisons can be made.

Table 4

Effect of γ Irradiation on Fracture Surface Work of Polystyrene (M.W.=230,000)

Dosage	Specimen Number	<u>n</u>	(erg/cm^2)
25×10^6 rads	l (8 data points)	2.75	5.1×10 ⁵
10x10 ⁶ rads	l (4 data points)	2.76	6.5x10 ⁵
	2 (8 data points)	2.73	4.4×10^{5}
5x10 ⁶ rads	l (6 data points)	2.70	4.27x10 ⁵
	2 (4 data points)	2.76	5.00x10 ⁵

8. Investigations with Isotactic Polystyrene

Quantities of isotactic polystyrene have been obtained from Monsanto Chemical Co. (1/2 pound) and the Dow Chemical Co. (1 pound molecular weight = 575,000). This quarter, 3"x8" sheets (1/8 inch thickness) have been compression molded from the Dow material under conditions described in the first quarterly report. In order that we might study the effect of crystallinity on fracture an amorphous sheet was prepared (by quickly cooling from the molding temperature of 460°F) as well as two crystalline sheets. The crystalline sheets have been prepared by annealing for various times at 180°C. For example, full crystallinity (40 percent by weight) can be developed after 4 hours. We have found that the annealing can best be accomplished without removing the plate from the compression mold and by maintaining the pressure during annealing to prevent formation of air bubbles. With this method we have produced a plate having a density of 1.071 which corresponds to approximately 40 percent crystallinity. A plate has also been produced with a 2 hour anneal and the density was approximately 1.061. A comparison of the physical properties of a fully crystalline isotactic polystyrene with an atactic material is presented in table 5.

Two cleavage specimens have been prepared from the amorphous sheet with heights of .8 inch and 1 inch. Although all of the data points have not been analyzed yet, the 1 inch sample at a crack length of 1.73 inches gave a value of fracture surface work of 16.6×10^5 erg/cm². The .8 inch sample at a crack length 2.02 inches had a fracture surface work of 16.2×10^5 erg/cm². Crazing had accompanied the growth of the crack and this may account for these high values.

Table 5

Comparison of Physical Properties of Isotactic Crystalline Polystyrene and Atactic Polystyrene (Dow Chemical Co.)

Property	Crystalline Polystyrene	Atactic Polystyrene
Tensile Strength, PSI		
23°C 55 85	6600 6200 5600	5500 3800 2800
Modulus-Tensile, PSI x 10 ⁵		
23°C 55 85	5.3 6.3 3.5	4.9 5.3 2.9
Elongation-Percent		
23°C 55 85	1.2 1.0 1.0	1.4 1.1 1.6
<pre>Impact Strength-Izod ft. lbs/in. notch</pre>		
23°C 55 85	0.24 0.28 0.40	0.23 0.30 0.25
Melting Point, °C	235	-
Density at 25°C	1.07	1.05
Relative Crystallinity	25-40	0
Heat Distortion, °C		
Vicat Tensile (103 PSI)	190 185	100 92
Abrasion Resistance Taber, wt. Loss 2000 Rev, g.	0.46	0.70
Dielectric Constant, 10 ⁶ cps.	2.6	2.5
Dissipation Factor, 10 ⁶ cps.	2×10^{-4}	1×10^{-4}
Appear a nce	opaque-wnite	clear

The crystalline material, annealed for 2 hours at $180 \,^\circ$ C ($\rho \approx 1.06 \, \text{g/cm}^3$) was machined into 2 cleavage samples. The specimen heights were 1 and 1.2 inches. The data has not all been analyzed but for a crack length of 4.66 inches the 1.2 inch height sample has a fracture surface work value of only 1.91×10^5 erg/cm². This is approximately ten percent of the value determined for the amorphous polymer. Additional studies are being conducted to verify this result and the more crystalline polymer is also being studied although its brittleness makes it difficult to handle and machine. In addition, the fracture surfaces of these crystalline polymers will be studied in detail.

Studies have also been initiated to better describe the micro structure of these crystalline isotactic polymers. In order to study the structure, the polymer pellets have been heated between glass slides on a hot plate and pressure applied in order to produce a thin film. This film was then annealed for various times at 180°C (temperature for maximum rate of crystallization) and 210°C. The microstructure has been studied in a transmission microscope using crossed polaroids. The maltese cross patterns typical of spherulitic crystalline structures have been observed and hedrites can also be seen in the photomicrographs presented in Fig. 8. References to this hedrite type of morphological structure have been made by Geil. (6) The average diameters of the hedrites or spherulites ranged from 10 microns at 1 hour annealing (180°C) to 16 microns at 4 hours of annealing (180°C). After 1 hour of annealing at 210°C spherulitic structures could not be detected, but after 2 hours crystalline

structures having an average diameter of 10 microns were observed and after 4 hours the diameter increased to 20 microns. Studies are now being conducted to remove thin films from the bulk cleavage sample with use of a microtome in order to study the microstructure of the actual material.





(b) Annealed for 4 hours at 180°C (600 x)

Figure 8. Transmission Photomicrographs of Melt Crystallized Isotactic Polystyrene Films



Figure 8. (Continued)

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VI. FUTURE WORK

Cleavage experiments will be conducted with the acrylate and methacrylate polymers previously prepared. These experiments will be conducted at -190°C and techniques have to be developed for preparation of the specimens which are rubbery at room temperature.

Further experiments with atactic amorphous polystyrene will be conducted toffurther establish the role of crazing in the fracture process. Studies will continue with the irradiated samples and verification of crosslinking or degradation will be made by swelling, viscosity and molecules weight measurements. Additional measurements will be made on crystalline polystyrene and the microstructure of the material will be more closely studied.

The use of a wedge shaped cleavage bar will be studied and actual experiments with Plexiglas will be conducted.

VII. MAN HOURS OF EFFORT IN SECOND QUARTER

The man hours of effort expended during the second quarter can be summarized as follows:

Principal Prof. 1	Investigator L. J. Broutman	270	man	hours
Research Mr. T. Mr. S.	Assistants Kobayashi Sanu	370 80	man man	hours
Technician Mr. R. Mr. M.	N Weibel Laingor	80 80	man man	hours hours