EXPERIMENTAL METHODS TO STUDY PLASTIC BEHAVIOR OF POLYMER GLASSES AT LARGE DEFORMATIONS

by

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Abstract

Amorphous glassy polymers may undergo large deformations before the onset of ductile fracture. The initiation of ductile fracture in these materials is often characterized by the formation and development of shear bands. This paper describes attempts to develop experimental methods to study large deformations of solid glassy polymers and the initiation and development of such bands. The two main experimental methods considered here are plane strain compression and symmetric-double-simple shear. The advantages and disadvantages of the two methods are discussed.
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Chapter 1

Background

It is well documented in the literature [2, 3, 6] that when glassy amorphous polymers are loaded to failure either crazing or shear banding occurs. Our interest here is to study the mode of fracture involving the initiation and development of shear bands.

Several test methods are available to study the mechanical behavior of glassy polymers. Common tests include tension, torsion, plane strain compression, and simple shear. The advantages and disadvantages of these tests have been recently reviewed by G Sell, et al. [4]. This thesis attempts to develop test and observation methods to study large deformation of solid glassy polymers. Attention is confined to refining the plane strain compression test and the simple shear test.
Chapter 2
Experiments

2.1 Plane Strain Compression Tests

2.1.1 Material

All samples came from a single 1/2 inch thick sheet of commercial polycarbonate. The samples were rough cut and annealed at 145°C\(^1\) for 24 hours and then furnace cooled using the method of Redner and Nickola [5].

2.1.2 Specimens

The specimens were of the "sandwich" type employed by Anand and Spitzig [1] to minimize frictional forces at platen contact interfaces. The main specimens were made up of 0.5\(\times\)0.2\(\times\)2 inch pieces of polycarbonate and the top and bottom pieces were approximately 0.5\(\times\)0.145\(\times\)2 inches. Each dimension was uniform to within 0.007 inch. All pieces were polished to a 0.3 micron alumina finish on all six sides, and annealed at 90°C for 24 hours and then furnace cooled. The samples were then checked in cross polarized light and no residual strains were observed. The sandwiches consisted of the three pieces of polycarbonate divided from each other and from the tool steel of the test fixture by commercial PFTE tape.

\(^1\)The glass transition temperature for polycarbonate is \(\approx 150°C\)
2.1.3 Test Set-up

A plane strain subpress was placed in compression in a screw-driven Instron machine with samples in place. The plane strain test fixture (Fig. A-2) featured a vertically aligned R60 case hardened shaft running through recirculating ball bearing transfer plates allowing free vertical movement. "Sandwich" specimens (Fig. A-3) were compressed between 2×2 inch platens (Fig. A-4) and constrained in the lengthwise direction.

Large strain (20%) strain gages were affixed to the gage section of the specimen and, because of their limited range, were used only to calibrate the total strain versus stress graph obtained from the crosshead displacement versus applied load graph. One test was run at 0.02 inches/minute and the other three were run at 0.05 inches/minute.

After testing, thin sections were cut from the specimen gage lengths and polished to a 0.05 micron alumina finish and observed under an optical polarizing transmission microscope.

2.1.4 Results and Discussion

A test sample compressed without the two sandwich layers but with the teflon yielded the graph in figure A-5. Four other samples were tested in the sandwich configuration. Three of these failed by sliding of the three sandwich blocks upon one another nucleated by a ridge along the line of shear bands (Fig. A-7). This occurred just beyond the point of maximum stress (at point F in figure A-5). On the fourth test, the sample was unloaded prior to this slipping. A representative stress-strain curve for sandwich type specimens is given in figure A-8. A "slipped" test specimen and the one unloaded prior to slipping were observed under the optical microscope and are depicted in figure A-6.

Since extreme care was taken in these tests in calibration of the Instron and of the strain gage half bridge, and also in alignment of the sample in the test fixture and of the test fixture in the Instron, the observed failure mode of the compression specimens was
unexpected.

The observed deformation mode implies that once these shear bands have propagated across the sample, they have set up a slip plane which is the easiest way for the polymer to deform. Because of this slipping just after the formation of a strongly developed shear band, it is found that this test is not suitable for additional plane strain compression of polycarbonate specimens.

2.2 Symmetric-Double-Simple-Shear Tests

2.2.1 Material

Polycarbonate and high density polyethylene were both used in the symmetric-double-simple shear tests. The polycarbonate specimens were annealed as described previously. The high density polyethylene specimens were made from as-received material.

2.2.2 Test Set-up

One way to study the large deformation behavior of a material is to subject it to large simple shear. A test fixture developed by Professor Anand (Fig. A-9) for other experiments was pressed into service\(^2\). All tests were run at room temperature. Samples were machined to ±0.0005 inch tolerances (Fig. A-10) to be pinned exactly into place in the test fixture. This permitted the specimens to be deformed in approximate simple shear.

The simple shear tests were performed on a servohydraulic Instron machine. A ramp input of 0.04 inches/minute stroke movement was imposed and the stroke was plotted against the developed axial load. This could then be converted to a stress-strain graph.

---

\(^2\) It was later noted that this test fixture is very similar to that used by Sternstein, et al., and has similarities to that of G'sell, et al.
The high density polyethylene specimen was tested the same way as the polycarbonate specimens, but the specimen geometry was slightly changed (see figure A-11). It was machined to allow it to be clamped in the $e_2$ direction on the two symmetric parts of the sample. The polycarbonate specimen fractured after a shear strain of $\sim 20\%$ whereas the high density polyethylene specimen did not fracture even after a shear strain of $\sim 200\%$.

2.2.3 Putting on Marking Grids

In order to study the homogeneity of deformation in our "simple shear" tests, the gage sections of the shear specimens were gridded (Fig. A-12).

Various gridding techniques exist. Some alternatives include scribing, traditional printing techniques, or using photosensitive materials. Since sample geometry and sensitivity to surface finish is often a problem, a method using photosensitive materials was used. The gridding was done at the MIT Microelectronics Laboratory. Because of polycarbonate's solubility in some hydrocarbons (e.g. acetone and trichloroethylene), a protective 200Å layer of gold was sputtered on the samples after first cleaning them thoroughly with methanol. All areas except where the grid was desired were then masked with tape. A thin layer of Shipley B positive resist was then put on the sample either by coating the sample and spinning it on a centrifuge or by painting with a small paintbrush. Each layer of resist was on the order of one micron thick. The sample was then baked at $85^\circ$C for 20 minutes. It was found that this long bake time (as opposed to 3 minutes for electronic chips) was necessary because of the thermal conductivity properties of the relatively thick samples. The samples were coated with a second coat of photoresist and baked for an additional 30 minutes. They were then covered with a grid mask, exposed, and developed in Shipley developer and rinsed. Finally, the excess gold was etched off with Aqua Regia. A similar method is used in the production of electronic chips. This method produces very accurate spacing and widths of grid lines.

This method of applying a grid to the sample was developed to aid in studies of the
development and propagation of shear bands in the shear specimens. Its accuracy and spectrum of uses, however suggest its usefulness as a new experimental method. Its advantage lies in its use on hydrocarbon sensitive surfaces. Since the surfaces are protected by this method, an accurate pattern can be applied using conventional techniques that make use of hydrocarbons. Since the method uses existing LSI technology, very fine demarcations (on the order of angstroms) can be accurately spaced on a sample for reference during microscope studies. Finally, it causes no surface damage to the specimen.

2.2.4 Results and Discussion

Reduced gage shear samples of polycarbonate were subjected to shear. The first sample (Fig. A-13) fractured through the gripped section. This lead to the gage-section size relation [Appendix A]:

\[ h \ W_s \leq 0.07 \]

(2.1)

Where \( h \) and \( W_s \) are shown in figure A-16. In further samples \( h \) and \( W_s \) were progressively changed to try to minimize end effects and to obtain a deformation pattern as close to simple shear as possible. After testing many different gage geometries, it became clear that the characteristic end effects and mode of brittle fracture predominated (Fig. A-14) in all specimen geometries for polycarbonate. Specimen rupture occurred before shear bands were set up. G'Sell, et al. [4] have reported similar failure characteristics in their shear testing of the same material. Thus, this experimental method is also not suitable for the study of shear bands in polycarbonate.

A sample made of high density polyethylene was then tested. We were able to deform it in approximate simple shear with minimal end effects (see Figure A-15). This test set-up, then, is suitable for this material. Its usefulness for other polymers is discussed in a recent paper by G'Sell, et al. [4]. In conclusion, I make the following recommendations to further
refine this test method:

1. Eliminate pin and hole procedure for holding specimen. Utilize top to bottom clamping to increase effective grip area and to simplify machining of samples.

2. Use the ratios of G Sell, et al.:

\[ \frac{W_g}{L} \geq 0.44 \] (2.2)

\[ \frac{h}{L} > 15 \] (2.3)

along with the relation:

\[ h \ W_g \leq 0.07 \]

from appendix A to make a reasonably sized test fixture taking machining and polishing difficulties into account.
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Appendix A
Critical Gage Size

An upper bound analysis was carried out on an existing sample (see Figure A-16) to determine the maximum gage dimensions.

![Diagram of shear sample with dimensions labeled: W, H, L, d, F_s, F]

Figure A-1: Dimensions of Shear Sample

\[ F_{ub} = \frac{Y \times W \times d}{\sqrt{3}} \]  
(A.1)

where \( Y \) is the equivalent yield strength of the material. A factor of safety of 0.7 is figured in:

\[ \frac{0.7 \times Y \times W \times d}{\sqrt{3}} \]  
(A.2)

\[ F = \frac{0.7 \times Y \times W \times d}{0.5 \times \sqrt{3}} \]  
(A.3)
The shear force, \( F_s \), then is given by:

\[
F_s = \frac{0.5 \times 0.7 \times Y \times W \times d}{0.5 \times \sqrt{3}}
\]  \( (A.4) \)

The shear stress, \( \tau_s \), is:

\[
\tau_s = \frac{0.7 \times Y \times W \times d}{\sqrt{3}}
\]  \( (A.5) \)

\[
\tau_s \geq \frac{\sqrt{3}}{Y}
\]  \( (A.6) \)

\[
1 \leq \frac{0.7 \times W \times d}{W_s \times h}
\]  \( (A.7) \)

or,

\[
0.7d(W/W_s) = h.
\]  \( (A.8) \)

For our case, we have \( d = 0.4 \) and \( W = 0.25 \). We get, finally:

\[
h W_s = 0.07
\]  \( (A.9) \)
Figure A-2: Subpress used in plane strain compression tests.
Figure A-3: Compression sample configuration with dimensions.
Figure A-4: Compression sample in test fixture.
Figure A-5: Stress-strain graph for polycarbonate.
Figure A-6: Shear bands as seen through a microscope.
Figure A-7: Schematic of tested-specimen cross-section
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