MECHANICAL TESTING OF COMMON-USE POLYMERIC MATERIALS WITH AN IN-HOUSE-BUILT APPARATUS

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The polymer production or transformation industries employ a very significant percentage of chemical engineers. This justifies the presence of a variety of polymer science and engineering subjects in the chemical engineering undergraduate curricula. Topics on solid polymer mechanics, in particular, are often quite useful for future engineers. They establish the basic tools for evaluating whether a material is appropriate for an intended use, or to tune its performance by acting on the synthesis/processing conditions. These subjects are present in general polymer textbooks (*e.g.*, References 1 and 2). Because ChE student laboratories are not traditionally equipped with the machinery used for mechanical testing, however, introductory courses on this subject often suffer from not having an appropriate *applied* component. Therefore, students don't gain a hands-on understanding of the phenomena involved.

One of the most commonly used mechanical tests in industry is tensile testing, in which the stress exerted by the material is measured at a constant strain rate. In addition to providing direct measurements of relevant properties, the stress-strain curves constitute "fingerprints" of a material's mechanical characteristics. The stress-strain curves are also often used for quality control of either raw materials or final products.

Most mechanical testing machines can perform several standardized tests (tensile, compression, flexural, etc.) by using appropriate accessories. Such machines are designed for heavy loads, however, and are significantly expensive. In addition, in order to use such a "heavy duty" machine, one has to prepare a test specimen that will exhibit a measurable stress-strain behavior. Furthermore, the specimens must be cut or cast into a standard shape and dimension, which might not be easy for many materials of interest. Standard-shape polymeric specimens of known composition and molecular weight are commercially available, but at a cost.

There is also equipment available for *low* stress/strain measurements—suitable for testing specimens of smaller dimensions, such as thin films—but these are also high-priced (about $8,000 USD).

On the other hand, polymeric materials are readily available in the form of everyday-use items. Even informal observation of the behavior of these materials under mechanical solicitation may give the attentive student a wide variety of illustrations for important concepts in solid-polymer mechan-

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ics. An inspiring example was provided by J. Walker in the "Amateur Scientist" column of *Scientific American* magazine,[3] in which the molecular phenomena involved in the stretching of a polyethylene film are illustrated and discussed in a simple and captivating fashion.

Our challenge was to build, on a very tight budget, a tensile-testing machine that chemical engineering undergraduate students could use freely in the lab for testing polymeric specimens gathered from common-use objects selected and prepared by them. Some of the design criteria we adopted for this project were:

- *Keep costs as low as possible, without compromising the quality of the machine's measurements (i.e., reasonable accuracy and reproducibility).*
- *Keep in mind that the specimens should be easy to obtain and prepare. Using thin films for testing seemed to be a good idea. Many materials (plastic or otherwise) are commonly available in that form and can be easily cut into standard shapes. This option implies designing the machine for small loads and strains.*
- *Build a compact setup, so as to allow portability and use inside temperature-controlled chambers (e.g., refrigerators and ovens).*
- *Make it fully automated, allowing total control of its functionalities through a computer-based data-acquisition system.*
- *Make it operationally robust, safe, and intuitive, since students are supposed to operate it themselves.*

This paper describes the machine developed, as well as some of the experiments performed. This project has been successful in giving students a hands-on perspective on some key aspects of the mechanical behavior of polymeric materials.

SETUP OF THE TENSILE-TESTING MACHINE

The design of the testing machine comprised four key components: (1) a set of two grips, which firmly hold both ends of the test specimen; (2) a motor, which pulls one of the grips at a constant pre-set speed; (3) a force sensor, which measures continuously the force exerted on the material; and (4) a displacement sensor, which measures the distance traveled by the moving grip during the test. Figure 1 shows the original sketch of the machine's layout, comprising these components. Further details are discussed below.

We intended to mostly test polymeric materials, such as polyethylene, in the form of thin films. We looked at some of the standardized tests used in industry to have an idea of the sizes and shapes of the test specimens used. According to ASTM D882-02,[4] the plastic films being tested are cut into rectangular specimens (at least 150 mm in length and 5 to 25 mm in width). On the other hand, on ASTM D2370-98(2002),[5] which applies to organic coatings (*e.g.*, elastomeric paint films), the specimens are also rectangular in shape, but the length may be lower (at least 50 mm in length and 13 to 25 mm in width). We decided to use a short specimen length to minimize the maximum strain involved in the tests, and thus allow the use of a reasonably priced continuous-displacement transducer—*and* keep the machine's size small. Therefore, we adopted the latter standard. (Note that we actually also tested paint films with this machine.) Some crude preliminary tests done with film strips cut from supermarket plastic bags gave us the basic information for the specifications for the load sensor, the motor, and the displacement transducer.

To provide linear motion to a lead screw, we chose a permanent magnet-stepper motor that employs a rotor with an internal thread. One end of the screw pulls one of the grips. The other end is attached to the moving lead of the LVDT-type displacement transducer. This transducer was the most expensive component in the setup (about 35% of the total cost). Cheaper alternatives are available, but an LVDT offers high accuracy and reproducibility, as well as wear-free and

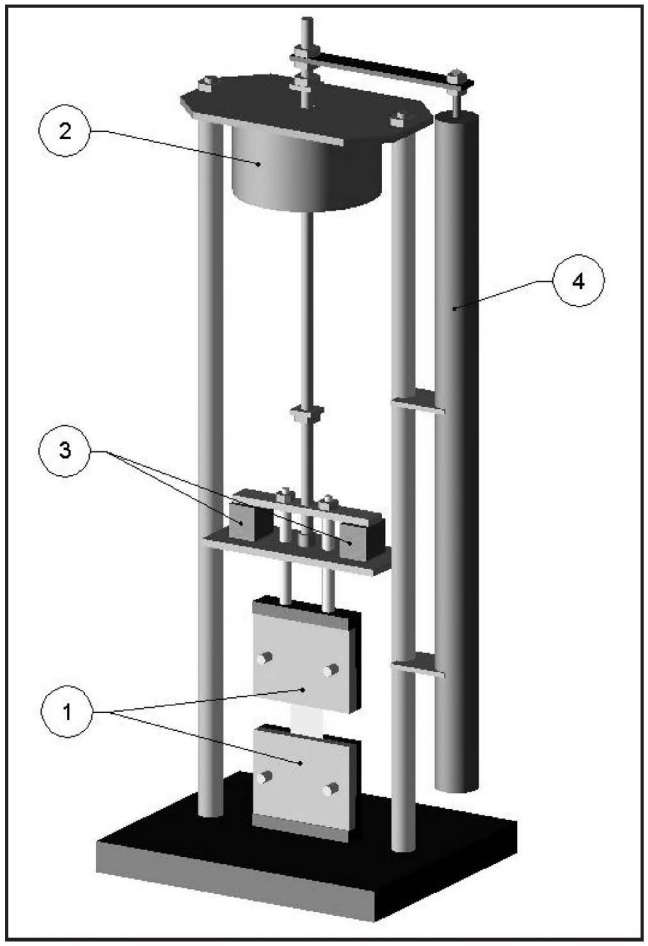

Figure 1. *A 3-D sketch of the tensile-testing machine, showing the major components: (1) grips; (2) motor; (3) force sensors; (4) displacement sensor.*

frictionless operation. Two force sensors, of piezoresistive type, are attached to the end of the lead screw. These sensors can measure only compression loads. The pulling grip is attached to a transversal bar that sits on top of the force sensors, thus transferring the tensile load, as is shown in Figure 1. Table 1 lists the main components and their costs. Figure 2 shows a photo of the actual unit, in its final working form.

The machine can measure loads up to 30 N. The maximum strain rate is 300 mm/min and the maximum linear displacement is approximately 140 mm, but this value can be increased by using a longer lead screw combined with longer lateral support bars. The machine is monitored and controlled with a desktop computer using a data-acquisition card (National Instruments PCI-6014). The program LabVIEW 7.1 (from National Instruments) was used to develop the software that fully controls the apparatus and analyzes the measured data. Figure 3 summarizes the information flow between the computer and the machine.

This machine was first implemented in an introductory course on solid polymer mechanics, which is an optional part of our chemical engineering undergraduate program. The students perform the tests themselves, on materials that they have gathered according to the instructor's suggestions. They analyze the results both qualitatively and quantitatively, computing different parameters from the measured data.

Some representative tests and results are described in the ensuing text. For the sake of conciseness, the discussion in this paper is kept on a qualitative level.

TENSILE AND TEAR TESTING HDPE FILMS

Students are asked to prepare test specimens from plastic shopping bags obtained at their favorite supermarkets. These are commonly made of high-density polyethylene (HDPE). Students can easily identify the polymer by noting the recycling symbol that is typically printed on the bags.

The specimens to be used on the stress-strain tests are cut into 60 x 15 mm rectangles and reinforced with adhesive tape at their extremities, covering 20 mm on each end (see Figure 4a). The grips hold the specimens by grabbing on these reinforced ends.

Special care must be taken in cutting these specimens. The cut should be perfectly straight and without indentations; im-

TABLE 1
Tensile-Testing Machine Components
(Not Including Data-Acquisition Board and Computer)

Component	Maker and Model	Specifications	Price (pre-tax, USD)
Stepper motor + lead screw	Mclennan L92411-P2	Max. linear force = 88 N	$263
Driver board for stepper motor	Eurocard		$48
Displacement transducer (LVDT)	Solartron DC50 920128	Range = ± 75 mm	$463
Force sensors (2) (piezoresistive)	Honeywell FSG	Max. load p/sensor = 15 N	2 × $75
Power supplies (2)	EMS B811 and Astec LPS23	12 V	$60 + $53
Holding structure and grips (carbon steel + polyacetal)	Local workshop		$250
Other components			$33
TOTAL			$1320

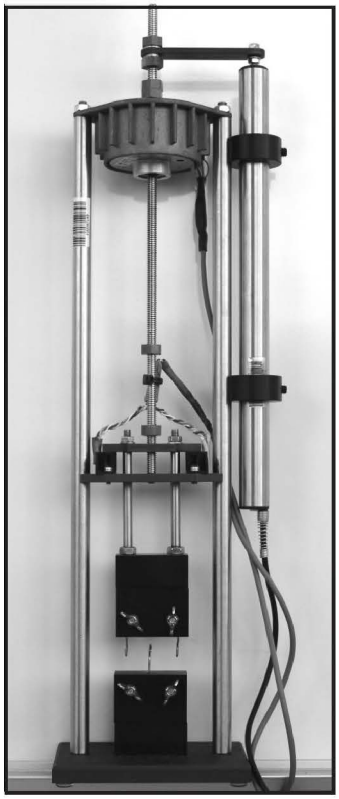

Figure 2. *Photograph of the tensile-testing machine (showing the holders used for the three-point bending test).*

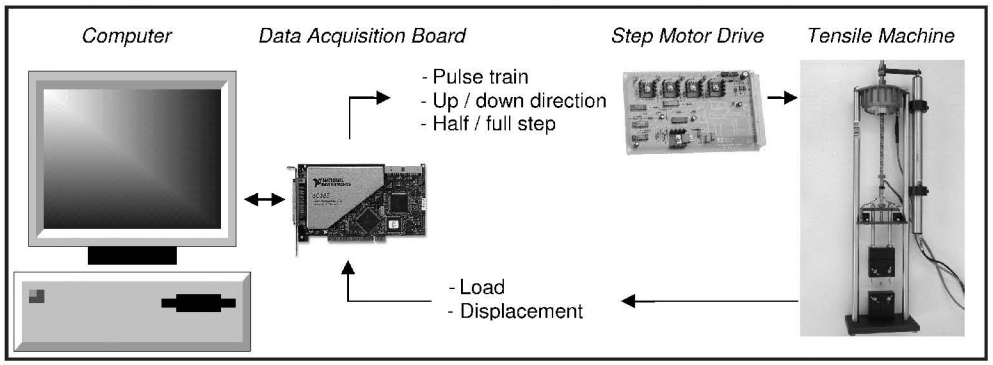

Figure 3. *Schematic representation of digital and analog signal flow between the machine and the data-acquisition system.*

perfections may cause the films to tear prematurely instead of reaching the ultimate rupture point. Sharp scissors or a fresh razor blade should be used. Students are asked to cut the specimens in two different ways: longitudinally and transversally in relation to the direction of the bags' "vertical" position (see Figure 5). Each specimen is labeled with a soft-tip marker so that the information on the specimen's cutting orientation is not lost.

Students also prepare specimens for tear-strength tests, which consist of 40 x 40 mm squares with an initial indentation (10 mm long) at the center of one of the sides (see Figure 4b). These indentations are done so that the tearing will propagate as intended: along the longitudinal or transversal directions mentioned before.

Before performing the stress-strain tests, students measure the thickness of each film, using a digital micrometer. This information is used to compute the initial cross-sectional area of the specimen, on which the loading stress will be based. Typical values are in the order of 10^{-2} mm.

Figure 6 shows representative results for two specimens cut along perpendicular directions as described before. The distinct behavior presented by the two is quite noticeable. The specimen cut along the bag's "longitudinal" direction (L specimen) shows a rapid increase in stress, followed by a short plateau and afterwards a gentler increase, up to rupture. On the other hand, the T specimen (cut along the transversal direction), after a similar initial raise goes through a very well-defined maximum in stress and then stabilizes on an essentially constant value, almost up to the final rupture. The stress for this specimen is always significantly lower than for the L specimen.

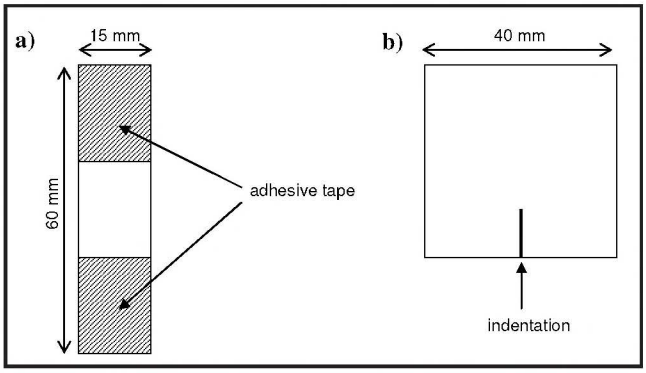

Figure 4. *Specimen cut from plastic bag films:* ***a)*** *for stress-strain tests and* ***b)*** *for tear-strength tests.*

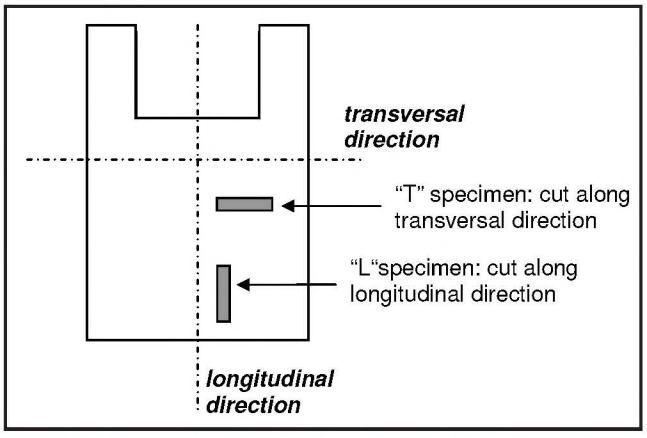

Figure 5. *Definition of the longitudinal and transversal directions on a plastic bag.*

Another fundamental difference can be observed when, prior to performing the tests, horizontal lines (perpendicular to the direction of elongation) are drawn with a soft-tip marker at different sections along the specimens. As the L specimen is elongated, one can see that the lines increase almost identically in thickness, up to rupture; this indicates that the material is being uniformly deformed. On the other hand, on the T specimen some lines become noticeably thicker as others remain almost unchanged; this indicates that the specimen is being stretched at the expense of deforming the material in limited regions. As elongation continues, the extent of the undrawn regions successively decreases until the entire specimen becomes uniformly stretched and rupture occurs. Some students recognize this as being an example of *cold drawing*—a phenomenon discussed in previous classes. It occurs on some semicrystalline polymers, like HDPE. The stress maximum corresponds to the yield point and the onset of necking.

But why is cold drawing not observed on the L specimen?

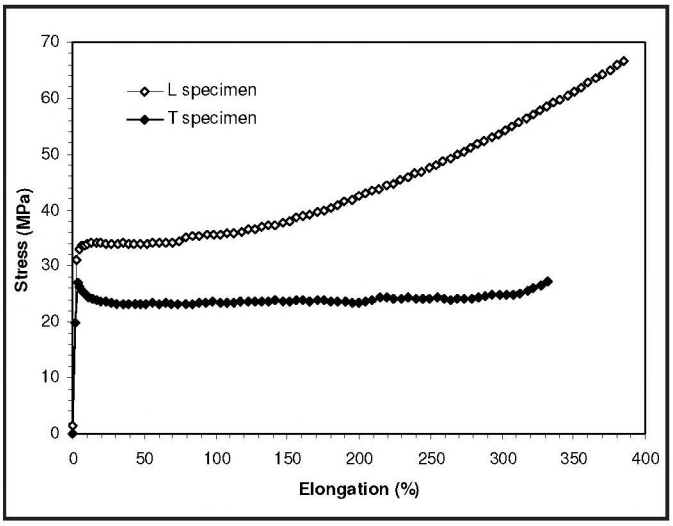

Figure 6. *Typical stress-strain curves obtained for high-density polyethylene films from a plastic shopping bag. The two specimens were cut along two perpendicular directions (see description of L and T specimens in Figure 4). The end point on each curve corresponds to rupture of the film. Operating temperature = 20 °C; strain rate = 200 mm/min; original film thickness = 0.016 mm. Note that the stress indicated is the "conventional" or "engineering" stress, i.e., the measured load divided by the initial cross-section of the specimen. Decreases in cross-section along the test are disregarded.*

This mechanical anisotropy is not usually expected by students, and they are encouraged to offer explanations. The tear-strength tests that are performed afterward supply extra material for the discussion.

The test is performed as schematized in Figure 7. The force exerted by the material is measured as the ends of the specimen are pulled at a constant rate and the tear propagates. Typical results from this test are shown in Figure 8.

When the tearing propagates along the longitudinal direction, the force is essentially constant and relatively low; in the end one observes that tearing originated a straight and clean cut. On the other hand, a much higher force is necessary to tear the specimen along the transversal direction; visually one can see that the material is stretched and distorted during the test and the final cut shows permanent deformation of the material at the edges.

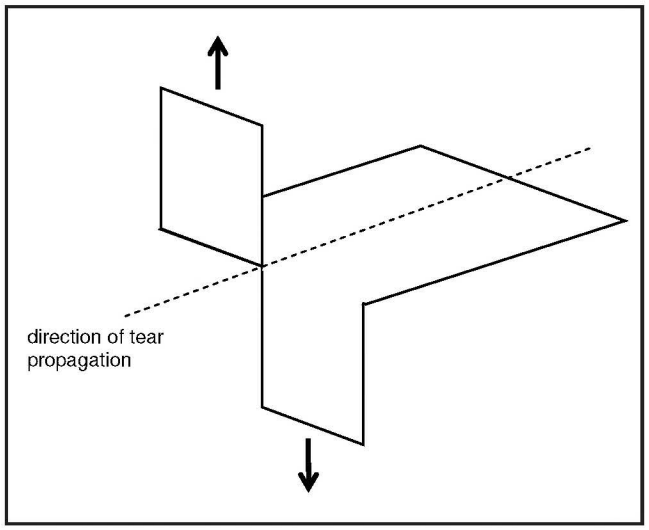

Figure 7. *Schematic representation of a tear-strength test.*

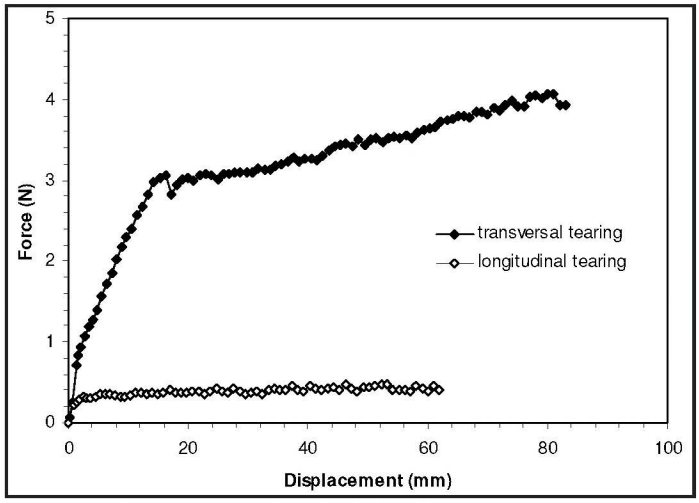

Figure 8. *Typical tear-strength curves obtained for polyethylene films cut from a plastic shopping bag. Tearing propagates along perpendicular directions on the two specimens (see description of transversal and longitudinal directions on Figure 4); same operating conditions as in Figure 5.*

After analyzing this second set of results, students often suggest that this anisotropy is associated to a particular molecular orientation of the polyethylene chains in the shopping bag. It becomes clear that this is the correct hypothesis after the instructor describes the manufacture process for HDPE bags, commonly known as *blown-film extrusion*. This process is described in several processing handbooks.[6, 7] It involves submitting the polymer to a sequence of transformations: melting, extrusion, blowing, drawing, cutting, and sealing. A continuously extruded thin-polymer tube is inflated by a jet of air blown into it. The blowup ratio (defined as the ratio between the diameters of the expanded film bubble and the die) controls the molecular orientation along the transversal direction. This ratio is usually 2 to 4. On the other hand, the drawdown ratio (the ratio between the speeds of the film at the nip rolls and at the die exit) determines the longitudinal orientation (called machine direction). A balance between these two parameters governs the final orientation within the film. Partial crystallization occurs as the material is cooled, thus conserving the molecular orientation imposed.

This flow-induced crystallization is actually a bit more complex than it would seem, due to the particular morphology that polymers exhibit upon crystallization. At a sufficiently high drawdown ratio, film-blown HDPE undergoes a so-called *row-nucleated crystallization*[8]: Extended molecular chains oriented along the machine direction form fibrillar structures that act as nuclei for the crystallization of the bulk material, in the form of radially grown lamellae. Figure 9 schematically illustrates these structures.

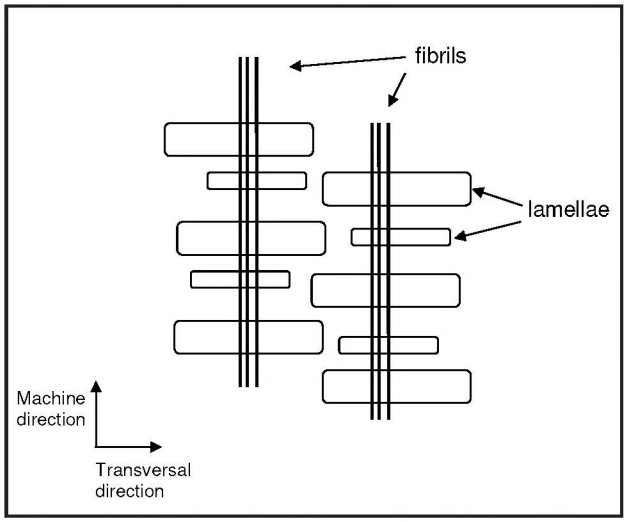

Figure 9. *Row-nucleated morphology of film-blown HDPE. The fibrils oriented along the machine direction act as nuclei for the growth of the lamellae (chain-folded crystalline structures).*

These long fibrils with perpendicular growths are often appropriately described as shish kebabs. They are responsible for the high tensile strength of the material along the machine direction (corresponding to the response of the L specimen in Figure 6).

Because, in the case of HDPE, there is no significant interconnection between the "kebabs" of adjacent fibrils, the tensile strength in the transversal direction is significantly lower. This transversal straining causes a noticeable yielding of the material, associated to local fibrillar reorientation toward the direction of the applied strain. The stress remains typically constant along this drawing process (see the curve for the T specimen in Figure 6). When the fibrillar rearrangement has extended throughout the entire material, the stress often rises slightly and rupture occurs shortly after. This cold-drawing phenomenon is characteristic of many semicrystalline polymers and is described in most polymer science textbooks. A recent paper by Zhang, *et al,*[9] provides an interesting discussion of the mechanical anisotropy and crystalline morphologies of different kinds of polyethylene-blown films.

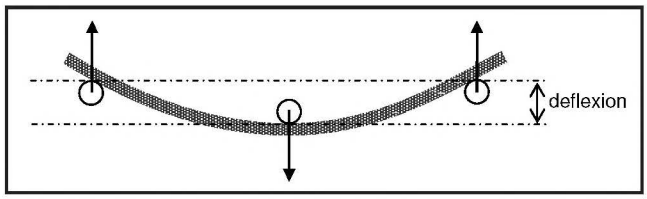

Figure 10. *Schematic representation of a three-point bending test. The two holders at the extremities are connected to the machine's upper grip and move at a constant rate. The holder at the center is attached to the lower grip and remains stationary. The two holders at the extremities are 30 mm apart. The specimen length is about 50 mm.*

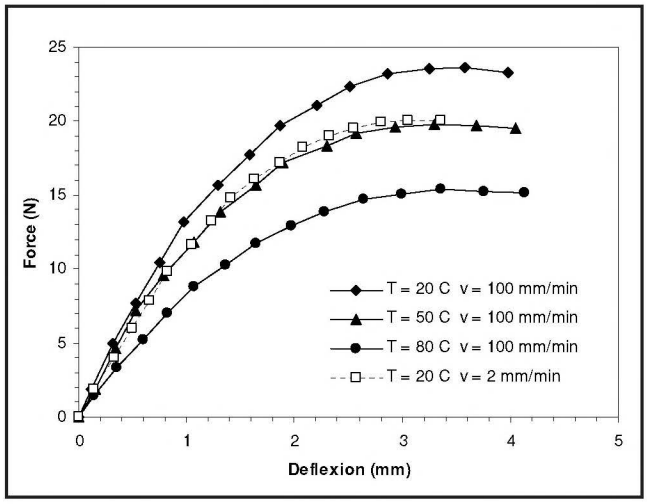

Figure 11. *Typical force vs. deflexion curves obtained for polystyrene coffee stirrers. The labels indicate the specimens' temperature in Celsius (T) and the deflexion rate (v). The end point on each curve corresponds to rupture of the material.*

From Figure 6 we see that the tensile strength (defined as the maximum stress measured during the test) is about 2.5 times higher when the material is strained along the direction that coincides with the fibrillar orientation (which we named the longitudinal direction). It makes a lot of sense that plastic bags are assembled so that normal use implies applying the stress along this direction.

The tear-strength test results (Figure 8) further confirm these findings. It is clear that it will be much easier to tear the material along the direction of the fibrils than along the transversal direction.

The "cherry on top" for this set of experiments comes when the instructor suggests that students take a piece of the plastic bag and heat it above a flame lighter (holding it with pincers and taking care to not actually burn the material). Immediately they see that the film starts to crumple and shrink. The temperature rise causes the gradual melting of the crystalline regions, loosening the mobility of the polymeric chains (the melting temperature of polyethylene is about 140 °C). This allows the originally extended chains to rearrange toward the more favorable coiled conformation. The result is the crumpling of the polyethylene film. Further heating would cause the total disappearance of the crystalline regions, resulting in a polymer melt.

FLEXURAL TESTING OF PS BARS

The machine is limited to tensile testing of thin films of relatively soft materials. Glassy polymers cannot be tested, since they involve much higher tensile stresses. Nonetheless, we have adapted the machine to perform a different kind of test on such materials: a three-point bending test at constant deflexion rate. We used polystyrene (PS) coffee stirrers, collected from a nearby vending machine, as test specimens. Figure 10 schematizes how the test is implemented: by using hard-wire hooks to attach the specimen to the grips. PS is glassy at room temperature (its glass-transition temperature, Tg, is about 100 °C).

Figure 11 shows some of the results obtained for different temperatures and deflexion rates. Since it is faster to place the rigid PS specimen on the support hooks than it was to attach a film to the grips, these tests did not involve placing the machine in a temperature-controlled chamber. Rather, the specimens were stabilized in an oven at the desired temperature. Prior to testing, each specimen was quickly transferred to the machine. The entire procedure (including performing the test) took no more than 30 seconds. In the figure one can see that the maximum deflexion is relatively low, as expected from a glassy polymer. For a deflexion rate of 100 mm/min, as the specimen temperature approaches the glass-transition temperature, its softness is significantly increased. Students

are asked to visually inspect the broken specimens. They notice that the ones tested at higher temperature show a visibly higher extension of *crazed* material (crazing, *i.e.*, the appearance of semi-opaque transversal bands in the neighborhood of the break surface, is a localized molecular-orientation phenomenon that occurs when some glassy polymers are close to rupture).

Note that the measured values of the deflexion at break are not reproducible and should not be considered. In the many tests performed, some discrepancies were obtained for this parameter. This was probably due to sample heterogeneity. The force-deflexion curves measured at lower deflexions were always quite reproducible.

When the test is repeated at room temperature, but at a lower deflexion rate (2 mm/min), the material's stiffness decreases, coincidentally giving a curve similar to the one previously obtained at 50 °C. This is a good illustration of time/temperature equivalence. In polymeric materials, molecular response is highly time and temperature dependent.

CONCLUSION

The in-house-built tensile-testing machine proved to be an economical tool for allowing students to test the mechanical behavior of different polymeric materials. The results can be analyzed both quantitatively and qualitatively. The fact that the test specimens can be obtained from everyday-use materials is not only an economic advantage but also an added factor of interest for students, since they can do the material selection and preparation themselves. The pedagogical benefits obtained from direct experimentation were confirmed by the interest and motivation shown by our students. Awareness that the machine was built in-house actually seemed to raise the students' curiosity toward comparing its components to the ones used in the commercial models.

The results shown here are representative of some important aspects of the mechanical behavior of solid polymers, such as the influence of processing conditions and the effects of temperature or strain rate. Students are encouraged to analyze their results and provide interpretations on a molecular level.

Other materials used successfully with this machine include rubber bands and films of elastomeric wall coating (EWC). The latter constitute a type of paint that, due its elastomeric character, is able to protect cracked walls from rain damage, since the film stretches to keep the gaps covered. It is an enriching exercise to analyze the mechanical response of EWC films under conditions such as low temperatures, aging (UV degradation), or water exposure (plasticization).

The tests performed with this device can be used either as an illustration of the concepts and phenomena previously discussed in class or, perhaps more interestingly, in a reversed approach. Indeed, the experimental observations are quite thought-provoking and motivate students to ponder and hypothesize on the reasons for the results obtained—thus paving the way for a structured discussion led by the instructor.

It must be remarked that the tensile and tearing tests described here for polymeric films are actually similar to some of the standard industrial practices, both in terms of specimen dimensions and operation parameters. It can be noted, as an example, that a data sheet for blown film obtained from ExxonMobil's HDPE resin HTA 001HD[10] (recommended for shopping bags, among other uses) reports an elongation at break of about 380% and a tensile strength (stress at break) of 56 MPa (it does not exhibit anisotropy for the particular processing conditions employed); this result is quite consistent with the values obtained with our shopping-bag material (of unknown origin).

In addition, a fairly good reproducibility is obtained with our in-house-built machine. The only problems are usually associated with discrepancies in the rupture points. For stress-strain tests, this is due to premature breaking caused by tears that initiated at imperfections in the specimen side cuttings, as mentioned before. These anomalous rupture behaviors can be easily detected visually and can be minimized by cutting the samples carefully. In the flexural tests, rupture discrepancies are probably associated to imperfections or inhomogeneities among samples.

Naturally, the machine presents limitations when compared to the models used industrially, namely in terms of measurement accuracies and limitations on load and strain ranges.

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