

POLYMER PROCESSING

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INTRODUCTION

The unique properties of polymers are often not apparent until they are encountered by chemical engineers on the job. The unique qualities of polymers most evident in their *processing*-- the fabrication of bulk polymer (resin) into a finished article-- which typically requires entirely different equipment from that required to process conventional liquids. Three characteristics of polymers will be examined in this experiment: mixing; non-Newtonian viscosity; and melt elasticity.

Because polymers are extremely viscous, the centrifugal or reciprocating pumps used for ordinary fluids are not effective at conveying polymers. Instead, some type of "screw pump", of which the extruder is a classic example, is used. Consider screwing a bolt into a nut; by turning the bolt, the nut can be run up and down the bolt threads. Similarly, if a screw is turned inside a tight-fitting but unthreaded cylinder, material between the screw threads (called "flights" in an extruder) and the cylinder walls will be conveyed along the length of the cylinder. The screw and barrel combination-- the heart of a single-screw extruder-- is shown schematically in Figure 1.

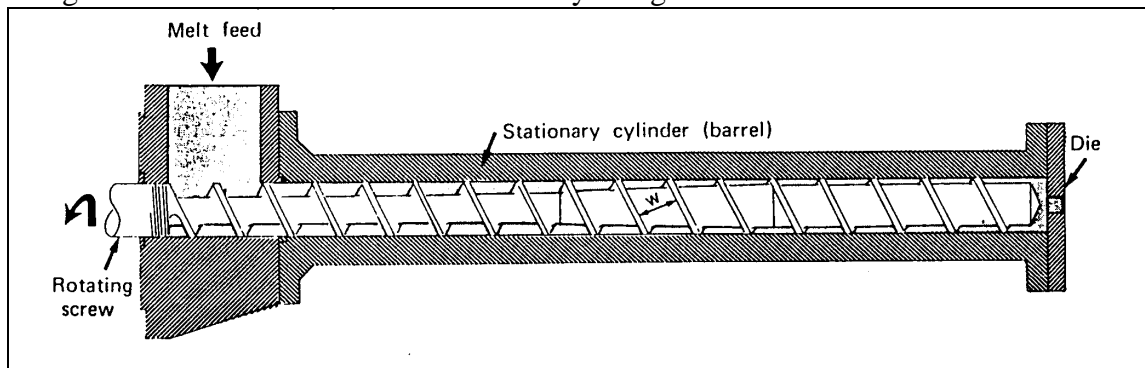


Figure 1. Schematic plan sectional view of a single-screw extruder barrel.

Many different screw designs are marketed, each claiming benefits in a particular application. Several simple examples are shown in Figure 2; the screw installed in the Killion is a basic design similar to (d). The AI will also show you a second screw, which

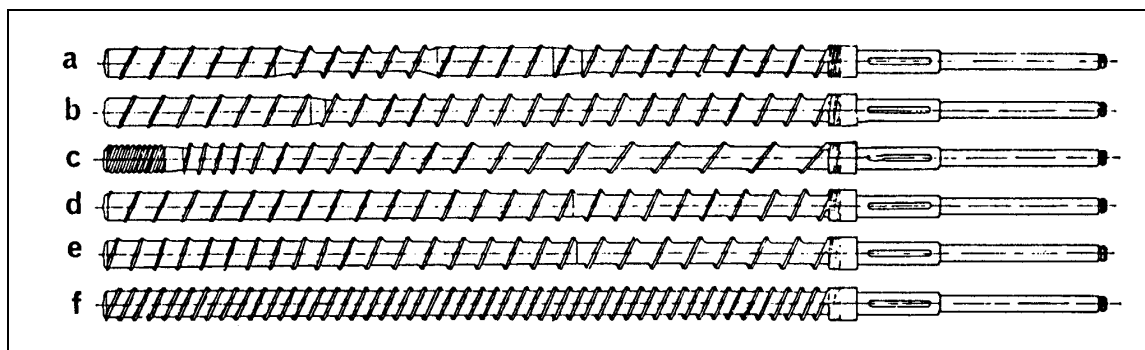


Figure 2. Six screw designs, with flow of melt from right to left. Design (d) is similar to that installed in the Killion; design (c) terminates in a mixing section, similar to that on the spare Killion screw.

incorporates a section designed to improve mixing. Screw design remains something of an art, and is certainly beyond the scope of this experiment, but be aware that the design of the screw can dramatically impact the residence time distribution (RTD) the polymer sees in the extruder.

Controlling the RTD a polymer sees is vital. Extruders are often used as mixers, which argues for a broad RTD (as in a continuous stirred tank reactor). On the other hand, most polymers will degrade if exposed to processing temperatures for too long, leading to a deterioration of mechanical properties as well as the production of appearance defects (“black specks”). This argues for a very narrow RTD (as in a plug flow reactor). The end result is that a compromise must be made, and the optimum shape of the RTD depends strongly on the particular polymer and mixing application in question.

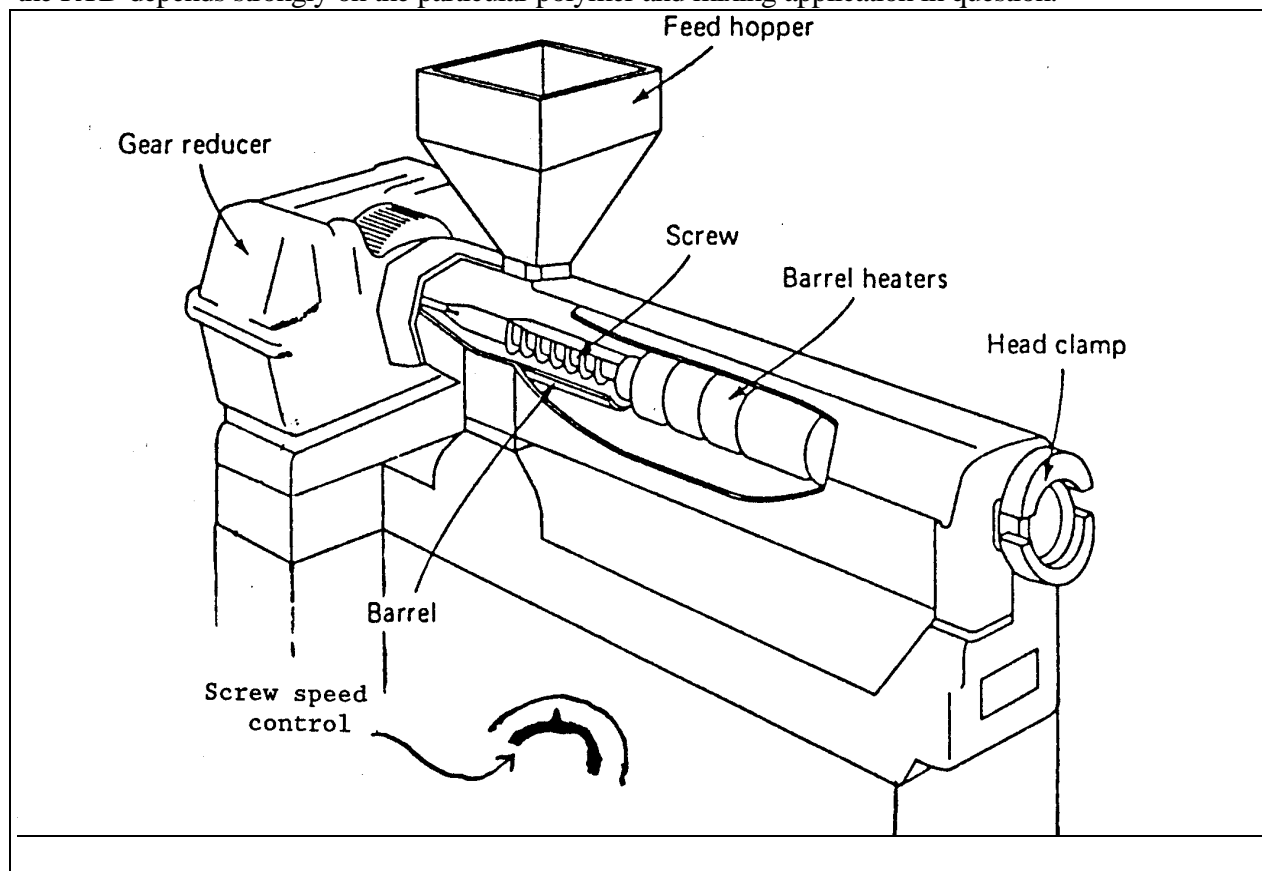


Figure 3. Schematic of a basic single-screw extruder. In the Killion, the die adapter is bolted onto the end of the barrel, rather than clamped.

A schematic diagram of an entire extruder is shown in Figure 3. This type of extruder is known as a “plasticating” extruder, meaning that its feed is solid polymer which is converted to a fluid (“plasticated”) in the extruder. (The less-common “melt” extruders operate with a molten polymer feed.) At the exit from the barrel is a die, which gives the extrudate a particular cross-section. Die designs also vary widely, depending on the shape desired for the extrudate. For this experiment, a “cylindrical” die is used, having a circular cross-section. This is the simplest type of die, and is typically used when the extruder is being used as a “compounder” (mixer). For example, it might be desired to blend two polymers, or to add an additive (colorant, stabilizer, *etc.*) to a polymer, and sell pellets of the compound to a customer who would in turn mold the pellets into a finished article. Automatic devices (“pelletizers”) which chop the extrudate into pellets are commonly employed. The red LLDPE which you will use as a residence-time tracer was formed in this manner. Other types of dies are available for profile extrusion (a product of specified cross-section, such as tubing), film extrusion (such as polyester

photo film backing), or blown film (such as garbage bags).

The polymer used for this experiment is linear low-density polyethylene, or LLDPE. LLDPE is a major commodity polymer; 12.9 billion pounds of low- and linear-low density polyethylenes (primarily the latter) were produced in the US in 1995 (*Chemical and Engineering News*, 6/24/96), or about 50 pounds per capita, much of which is exported. The principal use of LLDPE is as the base resin for thin but tough plastic films. LLDPE is a semicrystalline polymer, exhibiting a broad melting range terminating at the high end near 120°C, so all melt processing must take place above this temperature. “Linear” refers to the molecular architecture (no long branches are present in this material), while “low-density” refers to the fact that the material’s ability to crystallize is limited by the comonomer present, thus giving the material a lower room-temperature density. (LLDPE may be contrasted with high-density polyethylene, the material used to make milk bottles.)

The particular material we will be using is Tufin™ LLDPE HS-7028 Natural 7, a copolymer of ethylene and α -hexene generously provided by Union Carbide. (“Natural” means that no colorant has been added.) It is Carbide’s most popular resin for blown film. Some manufacturer's data on the material is attached for your information in Appendix A, as well as information on the molecular weight distribution (provided by Dr. Day-chyuan Lee, Union Carbide Corporation, Bound Brook, NJ). The density of the polymer at room temperature (where it is a semicrystalline solid), as given in this literature, is 0.918 g/cm³. The density of LLDPE in the melt can be satisfactorily described by:

$$\rho = 0.855[1+75 \times 10^{-5}(t-25)]^{-1}$$

where ρ = density (g/cm³) and t = temperature (°C).

A very useful reference for this experiment (and for polymer processing in general) is Z. Tadmor and C.G. Gogos, *Principles of Polymer Processing* (Wiley: New York, 1979), which is available in the Engineering Library (TP1087.T32).

EXPERIMENTAL

WEEK 1 - MIXING

The first lab period will be spent getting acquainted with the general operation and characteristics of the Killion 1" single-screw extruder around which this experiment is based. Although this extruder is now rather old, it has been retrofit with modern digital temperature controllers, and thus operates in essentially the same way a modern lab-scale extruder does.

There are four heated zones in the Killion, each of which has its own temperature controller and Type J thermocouple; the zones are labelled on the extruder. Zone 1 is the rear of the barrel (closest to the hopper). Zone 2 is the front of the barrel. Zone 3 is the die adapter, the part that both the pressure gauge and the die are mounted on. Zone 4 is the die itself. The controller (ECS 6414) and alarm module (ECS 6461) for each zone are mounted next to each other. The alarm module will activate if the temperature in a given zone exceeds a set value. The alarm settings are rather high and should not be activated during the course of normal use, but if they are (such as by disconnecting a thermocouple), the extruder must be shut down and Main Power turned off and on in order to rest the alarm. Both the alarm module and the controller module display the zone temperature in °C.

Only two features on each controller should be adjusted during the experiment. One is the "Process/Setpoint" toggle switch. When depressed, the digital readout displays the temperature setpoint (°C) rather than the actual temperature. The switch springs back automatically when released. The second control you will need to adjust is the potentiometer knob at the bottom of each controller faceplate. This knob is used to adjust the temperature setpoint; a setpoint of zero is obtained when the knob is turned fully counterclockwise. All setpoints should be set to zero when the extruder is turned on. The three screw adjustments labelled "Prop Band", "Reset", and "Rate" have (hopefully) been set to optimize the approach to setpoint; do *not* adjust these!

General procedures for operation of the Killion extruder are given at the end of the Experimental section: "Killion Startup", "Killion Shutdown", and "Tips on Killion Operation". Follow these general procedures, as well as the specific procedures described below. The die to be used for *all* measurements in this experiment has a length of 3½ inches, with a diameter of 1/8 inch.

Residence Time Distribution (RTD) Measurement

- 1) Follow the procedures described under "Killion Startup". Set the temperature to 160°C, and once operating, set the screw speed to 2.0 for measurements.
- 2) To determine the flowrate of polymer, all that is required is a stopwatch, a balance, and a pair of scissors. Use the scissors to cut the extrudate about 1" below the die exit; start the stopwatch at the same time. Remove from the quench bucket the LLDPE which was extruded prior to the cut, so the two extrudates do not stick together. After about three minutes, cut the extrudate again, at the same distance below the die. Remove the extrudate from the quench bucket. Using paper towels and/or the building compressed air, dry the extrudate. Weigh the extrudate on the top-loading balance. Record the elapsed time between cuts, extrudate mass, and pressure at the die inlet.

- 3) Obtain from the AI three pellets of red LLDPE to use as residence time distribution tracers (one for each measurement).
- 4) Allow the pellets in the hopper to be nearly exhausted. As soon as the first glimpse of the screw can be seen through the hopper, toss in the red LLDPE pellet, then immediately pour a load of clear LLDPE pellets after it to refill the hopper. Simultaneously with the addition of the red pellet, start the stopwatch. [Tip: the extrudate tends to pile up on itself, making it difficult to identify and section portions of the extrudate later to assess the state of radial mixing. This can be prevented by cutting the extrudate into shorter sections during the run, or by keeping the extrudate submerged in the quench bucket.]
- 5) Continually observe the extrudate for the first signs of color. Record the time at which the first hint of red is observed, the time at which maximum color is observed, and the appearance of the extrudate at a few time points thereafter (e.g., “orange”, “light yellow”, *etc.*; try to assign semiquantitative values to the color intensity). Allow the extruder to run for at least twice the time required to observe maximum color intensity, measured from the time the red pellet was added.
- 6) Use a razor blade to cut thin cross-sections of the extrudate at time points corresponding to those which you recorded your observations for in step 6. Tape the sections into your notebook; describe the state of radial mixing.
- 7) Repeat steps 2-6 for two other temperature/screw speed combinations: $220^{\circ}\text{C}/\text{ss}=2.0$ and $220^{\circ}\text{C}/\text{ss}=4.0$.
- 8) When your measurements are complete, follow the procedure under “Killion Shutdown”.

WEEK 2 - CAPILLARY RHEOMETRY

The purpose of these measurements is to determine the LLDPE's viscosity over a range of shear rates and temperatures. The relevant equations are described in the Analysis and Discussion section, which you should read *before* entering the lab. Polymers exhibit the phenomenon of “shear thinning”, meaning that the viscosity decreases with shear rate (rather than being a constant, as for a Newtonian fluid). The viscosity can vary by orders of magnitude over the accessible range of shear rates, so an understanding of the shear-thinning behavior of polymer melts is vital to understanding how they will process.

Capillary Rheometry Procedure

- 1) Follow the procedures described under “Killion Startup”.

- 2) Make flowrate measurements at three temperatures (160, 190, 220°C) and five screw speeds (0, 1.3, 2.0, 3.0, 4.0). Since the extruder heats up faster than it cools down, it is convenient to perform the measurements in ascending temperature. Also, the motor speed can be changed much more quickly than the temperature, so it is best to group the measurements into blocks of the same temperature, rather than the same flowrate.
- 3) Measure the flowrate as described in the procedure for week 1. (No colored pellets are needed here.) Record the times, masses, and pressures for each of the fifteen measurements. [Tip: in principle, the flowrate should be the same for a given screw speed setting regardless of the temperature. However, this is only approximately true, for two reasons. First, it can be difficult to precisely set the motor speed control, using only the large dial as an indicator. Second, the speed of the drive motor varies slightly under load, meaning that, for a given speed setting, the flowrate is higher at higher temperatures (where the viscosity is lower). The bottom line is that, to obtain accurate results, the flowrate must be measured for all fifteen conditions. If you are precise in setting the screw speed, you should notice that the flowrate increases monotonically (though weakly) with temperature.]
- 4) Record your visual observations of the extrudate for each of the fifteen conditions (e.g., “clear”, “hazy”, “ribbed”, etc.).
- 5) When your measurements are complete, follow the procedure under “Killion Shutdown”.

WEEK 3 - ELASTIC EFFECTS

Polymers also differ from Newtonian fluids in that they exhibit *elasticity*, which gives rise to some unusual phenomena. Again, please read the Analysis and Discussion section *before* coming to lab so that you'll be aware of what to look for. One particularly noticeable elastic effect is *die swell*, the tendency of a polymer melt to expand upon leaving a die. (You may have already noticed this effect in previous weeks.) The greatest difficulty in quantifying die swell is obtaining an accurate measure of what the extrudate diameter would be in the absence of other forces (principally gravity, which tends to decrease the diameter of the extrudate as it draws down under its own weight). A simple approach to solving this problem is taken here.

Elastic effects can also manifest themselves in certain flow instabilities not observed with Newtonian (or any inelastic) fluids. One of these, *sharkskin*, is manifested as roughness on the surface of the extrudate, even though the interior die walls are smooth. Small-amplitude sharkskin generally gives a matte or hazy appearance to the extrudate, while large-amplitude sharkskin results in clearly distinguishable ribs on the extrudate surface.

Die Swell and Sharkskin Procedure

- 1) Follow the procedures described under “Killion Startup”.

- 2) You will need to make measurements at three temperatures (160, 190, 220°C) and three screw speeds (0, 2.0, 4.0).
- 3) Fill a 1-liter plastic beaker about 2/3 full with cold tap water and hold it a couple inches below the die to quickly quench the extrudate. Use the scissors to cut the extrudate into short (about 4") lengths as it emerges from the die. Accumulate at least ten lengths before changing settings. Record the pressure gauge readings.
- 4) For all nine temperature/screw speed combinations, also measure the flowrate using the procedure described in week 1.
- 5) Measure the diameter of the extrudate using the caliper, taking care to avoid any regions of the extrudate which were distorted in the collection procedure, as well as any which are noticeably noncircular. You should obtain at least five diameter measurements at each temperature/screw speed combination to accumulate adequate statistics.
- 6) Record the visual appearance of the extrudate surface at each of the settings. For the higher screw speed settings and lower temperatures, you should notice that the hazy surface has a rather regular ripple. For each temperature/screw speed combination at which this ripple is clearly evident, measure the average wavelength of the sharkskin ripple using the calipers provided; rather than trying to measure individual ripples, count 10-20 ripples along the extrudate and measure the total length spanned by these ripples.
- 7) When your measurements are complete, follow the procedure under "Killion Shutdown".

KILLION STARTUP

- 1) Confirm with the AI that the extruder is plugged in (220V 3-phase, behind extruder), properly lubricated, and otherwise ready for use, and that the appropriate die is installed.
- 2) Unscrew the pressure gauge from the die adapter; use one wrench to secure the brass fitting mounted on the adapter, and one wrench to rotate the gauge. Remove any visible polymer from the brass fitting and the pressure gauge, *particularly* the inlet to the pressure gauge. (Avoid damaging the threads on the fittings!) If any unremovable polymer remains in the gauge, consult the AI; this will impair proper functioning of the gauge. Once all polymer is removed, use the grease gun to completely fill the brass fitting with grease. Clean the threads on the gauge of old Teflon tape, and wrap the threads with new Teflon tape (twice around the threads is sufficient; make sure the tape is wrapped such that the act of screwing in the gauge does not *unwrap* the tape). Screw the gauge back into the brass fitting; it should be turned so that it is visible from the front of the Killion. Use a paper towel to wipe any excess grease away from the area; excess grease will smoke when the die adapter is heated. *This procedure MUST be repeated EACH DAY the extruder is used.*
- 3) Fill the quench bucket (two-gallon pail) two-thirds full of cold tap water and place it directly beneath the die.
- 4) Make sure the screw speed control is set to zero (fully counterclockwise) and that the STOP button (red) in the Motor Control panel (red/green buttons on left side of extruder front panel) is depressed.
- 5) Make sure the setpoint on each controller is set to zero (potentiometer at bottom of module turned fully counterclockwise).
- 6) Make sure everyone working around the extruder is wearing safety goggles.
- 7) Turn on the cooling water supply to the feed throat (turn knife valve next to distillation column counterclockwise to horizontal). If the cooling water supply is inadequate, the bottom of the hopper can heat up and melt the polymer, which will clog the hopper (“bridging”) and prevent material from entering the barrel.
- 8) Flip the Master Power switch to ON. All eight digital indicators in the control bin should come on and properly indicate the room temperature in °C (though their readings may differ by a few degrees). The red “DEMAND” light (LED, right center) should be out on each controller, and all four zone ammeters should read zero. If any of these conditions is not met, *immediately* switch the Master Power to OFF and consult the AI.
- 9) Increase the setpoint in each zone to the desired temperature, by holding down the "Operate/Setpoint" toggle switch while turning the potentiometer knob clockwise until the desired temperature (°C) is displayed. Release the toggle switch. Repeat this procedure for each zone. For this experiment, set all zones to the same temperature, which must be *at least* 150°C to ensure that the LLDPE is fully melted. You should note that the current drawn

(displayed on the ammeter for each zone) will drop to a low level once the setpoint is reached. (If the demand light on the controller is off, the zone should be drawing *zero* current.)

- 10) Though the barrel should have been largely cleared of polymer at the end of the last experiment, some material always remains. If this material is not fully melted *before* the screw drive motor is turned on, one of several things could happen: the drive motor could burn out, any of several drive gears could be stripped, the drive belt could snap, or the screw could break. Given the age of the extruder, any of these failures could be terminal. Once you are satisfied that the controller has stabilized at the setpoint *and not before* (and in any case, the temperature in all zones is at least 150°C), press the Motor Control START button (green). The Motor Amps meter should indicate about 4A demand.
- 11) Observe the rotation of the screw by looking into the hopper (again, be *sure* to wear safety glasses). If the screw does not appear to be turning, press the motor control OFF button and contact the AI.
- 12) Use a plastic beaker to scoop out a quantity of pellets from the LLDPE drum and pour these into the hopper. Adjust the screw speed and temperature controls as necessary. The screw speed control should not exceed 4, while the temperature setpoint in all zones should not exceed the 220°C required for the experiments below.

KILLION SHUTDOWN

- 1) Run the Killion until the hopper is drained, and continue until the rate at which LLDPE is emerging from the die has dropped virtually to zero.
- 2) Turn the screw speed control down to zero (fully counterclockwise).
- 3) Press the Motor Control STOP button (red).
- 4) Adjust the setpoint of each controller to zero (potentiometer fully counterclockwise).
- 5) Flip the Master Power switch to OFF.
- 6) After about five minutes, shut off the cooling water (knife valve handle vertical).
- 7) Return any unused pellets, as well as the plastic beaker you've been using to fill the hopper, to the LLDPE drum. Make sure the drum is covered to prevent infiltration by dust.

TIPS ON KILLION OPERATION

- 1) Before the first LLDPE reaches the die, some grease usually drips from the die into the quench bucket. It is advisable to skim this grease off the top of the water with a paper towel *before* it sticks to either the sides of the bucket or the extruded polymer, from which it will be difficult to remove.
- 2) When waiting for the zone temperatures to equilibrate after a change in setpoint, turn the screw speed control to zero and press the Motor STOP button to shut the screw off, so as not to waste polymer.
- 3) Make sure one member of the group is charged with keeping pellets in the hopper throughout the run. The hopper need not be completely full (indeed, it should not be filled to more than about 3/4 of its height to prevent spilling pellets), but it must not be allowed to run empty, which will cause a sudden and drastic drop in the flowrate.

ANALYSIS AND DISCUSSION

Mixing

The color intensity vs. time in the extrudate is proportional to the *differential residence time distribution* in the extruder. Because diffusive mixing is negligible for materials with such high viscosity, if the exit time is scaled by the overall flowrate (to yield a *residence volume* V [=] cm^3), then the distribution should be characteristic of the extruder-screw-die combination and independent of polymer type, temperature, and screw speed.

Sketch the differential residence volume distribution (color intensity vs. V) for the extruder, at all three temperature/screw speed combinations. While the y-axis must be somewhat qualitative, since it is based on your visual estimate of color intensity, the x-axis should be quantitative. Indicate on the diagram the point at which the first colored extrudate was observed, as well as the point of maximum color intensity (V^*). Is the distribution the same for all three combinations?

Calculate the residence time distributions for three idealized cases: a tubular reactor in plug flow (volume = V^*), a tubular reactor in laminar flow (volume = $2V^*$), and a continuous stirred tank reactor (volume = V^*). Plot these three distributions. How the extruder's residence time distribution compares with these limiting cases?

Also note on the diagram the state of *radial* mixing at various points along the distribution. Are patterns found indicating the radial mixing is nonuniform? What might be the origin of the nonuniformity? Are the radial mixing patterns identical for all three temperature/screw speed combinations?

Viscosity/Constitutive Equations

From a fluid mechanics standpoint, the most important parameter of a Newtonian fluid is its viscosity μ . Knowledge of the viscosity allows one to calculate the flow pattern in an arbitrary geometry, so long as viscous forces dominate. From a practical standpoint, it is the parameter required to answer such basic questions as, "how much pump pressure do I need to flow liquid through a certain pipe at a certain rate"? As an example, for Poiseuille flow of an incompressible Newtonian fluid (in a pipe of circular cross-section), the only nonzero component of the stress tensor is τ_{rz} , and it is equal to:

$$\tau_{rz} = -\mu \dot{\gamma}_{rz} \quad (1)$$

where the *shear rate* $\dot{\gamma}_{rz} = dv_z/dr$. Any equation relating the stress tensor to the velocity profile is called a *constitutive equation*; equation (1) is one component of the constitutive equation for an incompressible Newtonian fluid, which in its full tensorial form is:

$$\underline{\underline{\tau}} = -\mu \dot{\underline{\underline{\gamma}}} \quad (2)$$

The constitutive equation for such a fluid is thus very simple: the stress and shear rate are related by a proportionality constant we term the viscosity, μ . While μ can vary with temperature, it is independent

of shear rate, and thus isothermal flow problems can be readily solved.

The difficulty with polymers and other complex fluids is that the appropriate constitutive equations are not as simple as equations (1) or (2), and indeed, are still not known exactly. However, over the years, a number of approximate constitutive equations have been developed which can yield good results for the velocity distribution under an imposed stress field in particular cases. For simple shearing flows, a common approach is to define an analog to equation (2):

$$(3) \quad \underline{\tau} = -\eta \dot{\underline{\gamma}}$$

where η plays the role of viscosity, but is now allowed to vary with shear rate. The problem then reduces to determining the functional dependence of η on $\dot{\gamma}$, which is generally determined by imposing a velocity distribution with only one nonzero component and measuring the stress component in the same direction as suggested by equation (1). (Note that this approach is approximate at the start, since for materials like polymers which have elasticity, even in simple Couette flow more than one component of the stress tensor is nonzero.)

For the present experiment, the quantities measured are the pressure drop P (equal to the reading on the pressure gauge, which reads the value *above* atmospheric) and the flowrate Q for flow through a capillary of radius R and length L . These must be related to the desired quantities, namely τ_{rz} and $\dot{\gamma}_{rz}$. A problem immediately arises for Poiseuille flow, where these quantities are not constant, but vary with radial distance r , and since the constitutive equation is unknown, the dependence of v_z on r cannot be calculated in advance. Fortunately, some useful relations can be obtained without assuming any constitutive equation. For any incompressible fluid, the momentum equation in steady Poiseuille flow reduces to:

$$dp/dz = -(1/r)[d(r\tau_{rz})/dr] \quad (4)$$

Integrating this equation with respect to r and recognizing that, by symmetry, the velocity gradient (and thus τ_{rz}) is zero at $r=0$, we obtain:

$$\tau_{rz} = (P/2L)r = \tau_w(r/R) \quad (5)$$

where the shear stress at the wall ($r=R$), τ_w , can be found by balancing the drag force on the die surface against the pressure drop across the die:

$$\tau_w = (P/2L)R \quad (6)$$

τ_w can thus be calculated from experimentally-accessible quantities. Writing the generalized version of equation (1) at the wall ($r=R$), we obtain:

$$\tau_w = -\eta \dot{\gamma} \Big|_{r=R} \equiv \eta \dot{\gamma}_w \quad (7)$$

where the shear rate at the wall $\dot{\gamma}_w$ is thus defined to be a *positive* quantity (note the sign change),

though still unknown. We know that the total flow rate Q can be obtained by integrating over the radial direction:

$$Q = 2\pi \int_{r=0}^R r v_z(r) dr \quad (8)$$

Integrating by parts and assuming the no-slip condition at the capillary wall leads to:

$$Q = -\pi \int_{r=0}^R r^2 (dv_z/dr) dr \quad (9)$$

By using equation (5), we can change the integration variable from r to τ_{rz} :

$$-Q(\tau_w^3/\pi R^3) = \int_{\tau_{rz}=0}^{\tau_w} (dv_z/dr) \tau_{rz}^2 d\tau_{rz} \quad (10)$$

Using the Liebniz formula for the derivative of an integral, this equation may be differentiated with respect to τ_w to yield:

$$[\tau_w^3 (dQ/d\tau_w) + 3Q\tau_w^2]/\pi R^3 = -\tau_w^2 (dv_z/dr)_w = \tau_w^2 \dot{\gamma}_w \quad (11)$$

Thus, using the expression for τ_w , this becomes:

$$\dot{\gamma}_w = (1/\pi R^3)[3Q + P(dQ/dP)] \quad (12)$$

This equation is known as the Rabinowitsch equation, and is valid for any fluid whose constitutive equation can be satisfactorily described by equation (7). For this experiment, it is more convenient to slightly rewrite equation (12) as:

$$\dot{\gamma}_w = (Q/\pi R^3)[3 + d\ln Q/d\ln P] \quad (13)$$

because $\ln Q$ vs. $\ln P$ is more nearly linear than Q vs. P , making the differentiation of experimental data more accurate. For a Newtonian fluid, $d\ln Q/d\ln P = 1$; for fluids obeying the *power-law* constitutive equation (described below), $d\ln Q/d\ln P$ is also a constant, but with a value different from unity.

It is the Rabinowitsch equation which makes it possible to determine the functional dependence of η on $\dot{\gamma}$ from *capillary rheometry*, where the volume flowrate of polymer through a cylindrical capillary die is determined for a range of imposed pressures. In this experiment, the extruder is being used as the pump for a capillary rheometer (the die and pressure gauge). From your measurements and equations (6), (7), and (13), you have everything you need to determine $\eta(\dot{\gamma})$ over a range of shear rates and

temperatures.

One point which should be noted here is that the Rabinowitsch equation assumes that flow is fully developed along the entire length of the capillary. The pressure drop incurred due to the sudden contraction at the capillary entrance is typically found to equal that incurred by passing through a few diameter's worth of the capillary's length; for short capillaries, this can constitute a substantial fraction of the total pressure drop. In general, this effect can be corrected for (the *Bagley correction*) by using a range of dies of the same R but varying L, and extrapolating a plot of P vs. L back to L=0 to find the pressure drop at the capillary entrance. Due to the limited time available for the experiment, entrance effects must be neglected in our case. (Since our capillary has L/R = 56, entrance effects do not dominate, and neglecting them is not unreasonable; the net effect is to increase the apparent values of η .)

To analyze your data, proceed as follows:

- 1) Convert your experimental measurements to Q (cm³/sec) and plot lnQ vs. lnP. From this, determine dlnQ/dlnP at each point, either graphically or by fitting the data to a quadratic polynomial.
- 2) Use equations (6), (7), and (13) to determine η vs. $\dot{\gamma}_w$, and plot the data sets for all three temperatures on a single graph. The die used for capillary rheometry has a uniform cross-section (R = 1/16 in.) over its entire length (L = 3½ in.). Express η in units of Pa-sec and $\dot{\gamma}_w$ in sec⁻¹.
- 3) In Appendix B are η vs. $\dot{\gamma}_w$ data for the LLDPE you have been using, measured on a Rheometrics System IV rheometer (cost ~ 10x cost of extruder). Insofar as possible, compare your data with that attached; give your best explanations (with as much quantitative justification as possible) for the origin of any differences.

Figure 4 shows a plot of log η vs. log $\dot{\gamma}$ for a low-density polyethylene (*not* the same material you are using!) at various temperatures (and over a much wider range of shear rate than we can access on the System IV). The LLDPE you have been using exhibits the same qualitative features as shown in Figure 4, namely, a roughly constant viscosity at low shear rates (the “zero-shear” or “Newtonian” limit), which decreases at higher shear rates (“shear thinning” behavior). The linear portion of the curves (between about 10 and 10⁴ sec⁻¹ in Figure 4) is known as the “power-law” regime, since the viscosity can be described by:

$$\eta = m|\dot{\gamma}|^{n-1} \quad (14)$$

where the bars denote absolute value, and the parameter n is known as the *power law index*. Typical power-law indices for polymer melts are 0.2-0.5; a Newtonian fluid has n=1. Note that the viscosities at 10⁴ sec⁻¹ in Figure 4 are about two orders of magnitude below the Newtonian viscosity, and thus the shear-thinning behavior of polymers *must* be considered to obtain even an order-of-magnitude solution to a flow problem at typical processing shear rates.

Though equation (14) cannot be valid over the entire range of shear rate (for example, it predicts η

$\rightarrow \infty$ as $\dot{\gamma} \rightarrow 0$), it is nonetheless a very commonly-used constitutive equation for solving flow problems involving polymer melts. Its chief virtue is simplicity, as it provides an analytical expression with only one more parameter than a Newtonian fluid (m and n vs. μ). Thus, flow problems which are tractable for a Newtonian fluid are readily solved for a power-law fluid by a straightforward generalization, provided m and n are known. For example, the

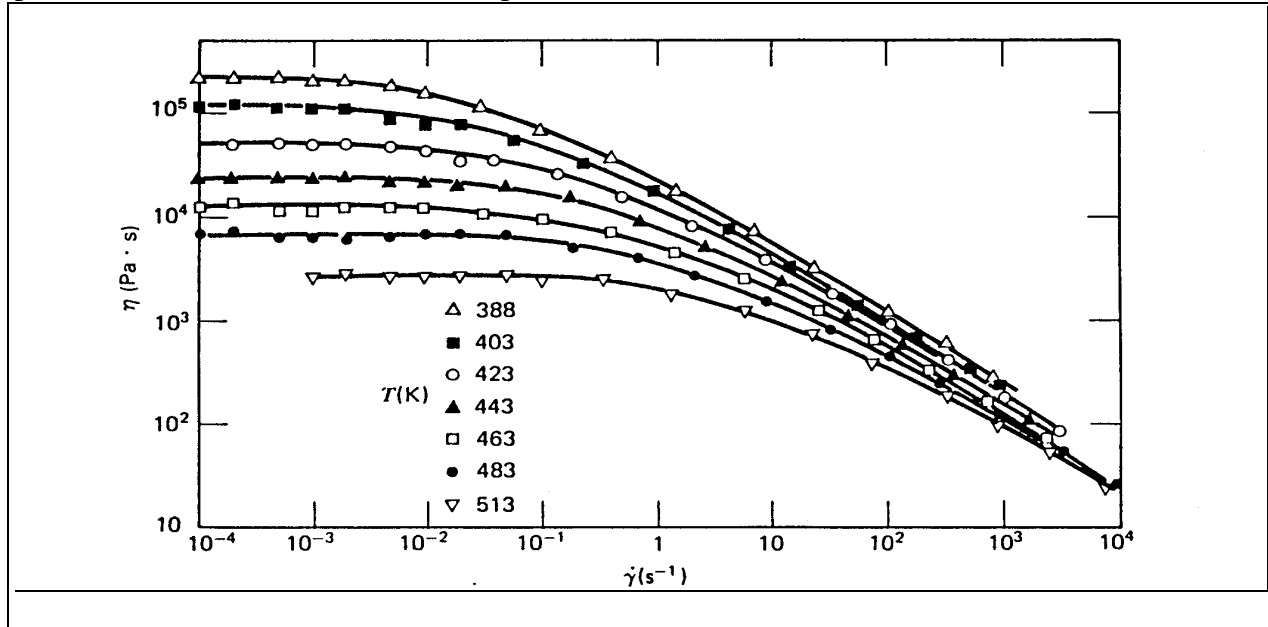


Figure 4. Viscosity η of a low-density polyethylene melt over a range of temperatures [J. Meissner, *Kunststoffe*, **61**, 576 (1971)]. Data taken below 0.05 sec^{-1} were taken in a cone-and-plate apparatus; data at higher shear rates were obtained with a capillary rheometer.

Rabinowitsch equation (13) for a power-law fluid simply reduces to:

$$\dot{\gamma}_w = (Q/\pi R^3)[3 + 1/n] \quad (15)$$

By determining values of n and m for a material in capillary rheometry, the power-law equation can be used to solve for the flow behavior in other geometries, such as film extrusion or mold filling. Moreover, nonisothermal behavior can be accommodated if necessary. It is found empirically (see Figure 4) that n is nearly independent of temperature for a given material, while m can be described by an Arrhenius relationship:

$$\ln(m) = \ln(m_0) + E_a/RT \quad (16)$$

where E_a is the flow activation energy, R is the gas constant, and T is the temperature in absolute units. From your data, it is possible to determine n , m_0 and E_a , which together form a reasonable constitutive equation for the LLDPE under consideration at the shear rates and temperatures typically encountered in processing. To analyze your data, proceed as follows:

- 1) Determine values of m and n from your η vs. $\dot{\gamma}_w$ data at each temperature via linear regression.
- 2) Take a suitable average value of n to be characteristic of the material, then fix n at this value and

redetermine m for each of the three temperatures. Comment on (and explain) any systematic variation of n with temperature.

- 3) From the m values obtained in step 2, use equation (16) to determine E_a and m_0 . Show this plot in your report.
- 4) Use the three parameters (n , m_0 , E_a) to calculate $\ln Q$ vs. $\ln P$ curves for each of the three temperatures. Plot the actual $\ln Q$ vs. $\ln P$ data points on the same graph; they should scatter around the calculated curves. Assess the accuracy of the power-law model, and the temperature dependence as given in equation (16), over the temperature and shear rate range involved, discussing any discrepancy between the data and the calculated curves.

Elastic Effects

In addition to their dramatic shear-thinning behavior, polymers differ qualitatively from Newtonian fluids in a second way: they are elastic. Functions describing the elasticity of polymers (in the same way η describes the viscosity) can be measured, though not via capillary rheometry. However, two consequences of the elastic behavior of polymers can be observed in this experiment and at least partially quantified: die swell and extrudate roughness.

Die swell describes the increase in extrudate diameter over the diameter of the capillary. This effect is a nuisance in processing, particularly in what is known as “profile extrusion”, where an object of particular cross-section is desired (such as a plastic pipe, or vinyl siding). Die swell is principally an elastic effect, and can qualitatively be explained as follows. During flow in the capillary, the polymer is subjected to stress, which causes a distortion (stretching) of the polymer coils. Once the polymer exits the die, this stretching can relax, causing a swelling of the extrudate diameter. (Think of a stretched rubber band, which on relaxation becomes shorter and thicker.) For smooth extrudates emerging from sufficiently long capillaries, where the additional deformation incurred in the entrance region to the capillary has all relaxed out by the time the polymer exits the capillary, it has been found empirically that the ratio D/D_0 is a unique function of τ_w for a particular material. Figure 5 shows such data for two polystyrenes (a distinctly different polymer from LLDPE). A slight modification

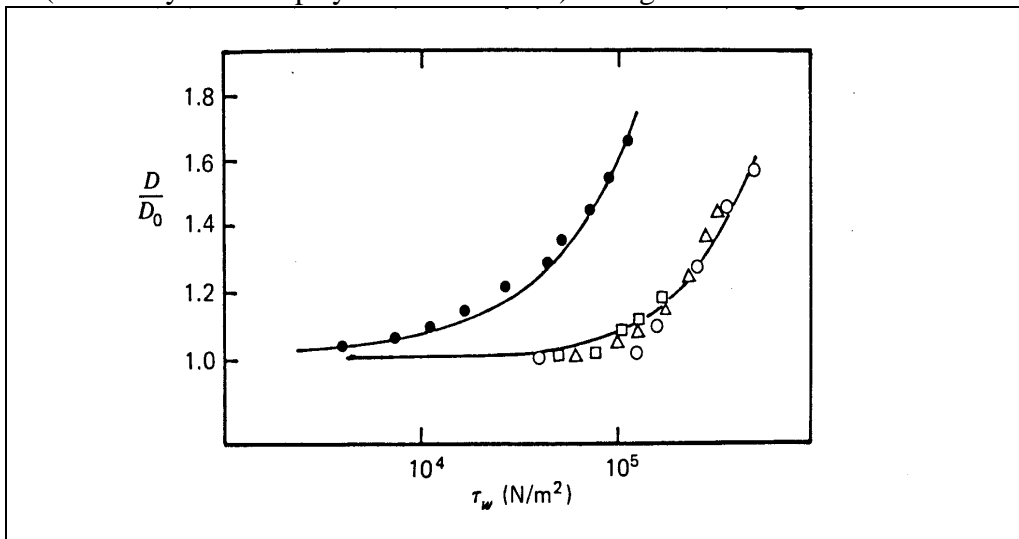


Figure 5. Die swell data for polystyrene melts, taken with long capillaries at various temperatures. Filled symbols: broad molecular weight distribution; open symbols: narrow molecular weight distribution [W.W. Graessley, S.D. Glasscock, and R.L. Crawley, *Trans. Soc. Rheol.*, **14**, 519 (1970)].

to this statement, based on rubber elasticity theory, would be that the die swell ratio should be proportional to τ_w/T , where T is the absolute temperature. (In practice, data are usually taken over a rather limited range of temperature, so it would be difficult to distinguish whether die swell scales better with τ_w or τ_w/T . The latter is chosen here to follow along with the arguments presented in the “sharkskin” section below, but in practice it would make little difference.) Though our capillary is not really “long” (residence time 1–6 sec) and our extrudate is sometimes rough, it is still worthwhile to prepare a plot like that in Figure 5. To analyze your data, proceed as follows:

- 1) Convert your measured extrudate diameters D_{meas} (at room temperature) to diameters of the melt extrudate D , using the density values given in the introduction. You may assume that upon crystallization, densification occurs isotropically, so that $D/D_{\text{meas}} = (\rho_{25C}/\rho_{\text{melt}})^{1/3}$. Calculate values of D/D_0 ; here, $D_0 = 1/8$ inch.
- 2) Use your pressure measurements to calculate τ_w . Plot all D_{act}/D_0 data vs. τ_w/T on a single log-log graph, including error bars (± 2 standard deviations. The reason for the log-log graph, which differs from Figure 5, will become apparent in the “sharkskin” discussion below.) Do the data all collapse onto a single curve?

The other significant elastic effect which you have observed in this experiment is *sharkskin*. The uncolored LLDPE melt is quite transparent, and as you have seen, where it exits the die, the extrudate is crystal clear at high temperatures and low shear rates. At higher shear rates and lower temperatures, the extrudate appears hazy on exiting the die. This is *not* due to turbidity within the extrudate, but rather to roughness at the extrudate surface. For many applications, this roughness, appropriately termed “sharkskin”, is undesirable and limits the maximum rate at which material can be extruded. As the shear rate is increased, the amplitude of the roughness increases, and the extrudate takes on a ribbed appearance. At even higher shear rates (higher than you will see in this experiment), even more gross distortions of the extrudate can occur (“melt fracture”), producing an extrudate with very large diameter variations (sometimes likened in profile to bamboo). An excellent review of the experimental and theoretical knowledge on flow instabilities of viscoelastic fluids, including those which occur on passing through a capillary die, has recently appeared (R.S. Larson, *Rheol. Acta*, **31**, 236 (1992)). Experimentally, it is found that the stress at which sharkskin begins to occur depends on the surface characteristics of the die (material, surface treatments, etc.). Therefore, sharkskin has long been thought to result from periodic stick/slip flow in the die-- that is, the no-slip condition is intermittently violated, and the material moves as a plug.

A recent paper (S.Q. Wang, P.A. Drda, and Y.W. Inn, *J. Rheol.*, **40**, 875 (1996)) treats the origin of sharkskin in some detail; the basic arguments are outlined here. Sharkskin is attributed to intermittent stick-slip (as in previous work), but this stick-slip is postulated to occur only at the die exit (die lip), whereas smooth (stick) flow occurs within the capillary itself. The die exit is unique because there is a discontinuity in the boundary condition there: inside the die, the stress at the wall is high, while outside the die, the stress at the extrudate surface is essentially zero. The same authors have shown that by coating just the die lip with materials which promote slip, sharkskin can be eliminated, because then the

oscillatory stick-slip behavior at the die exit is replaced by smooth slip. Thus, there is experimental evidence in support of the model presented below. Note that the Rabinowitsch equation, around which the viscosity analysis was based, assumed that the flow is steady and the no-slip condition applies; these assumptions are questionable if the extrusion conditions produce sharkskin. However, if slip occurs over only a very short length of the die near the exit, then the flow in the great majority of the die conforms to these assumptions. It is observed experimentally (and should be visible in your data) that no sharp discontinuity occurs in plots of Q vs. P when the extrudate turns hazy. Thus, the quantitative error in using the Rabinowitsch equation for shear rates slightly beyond the transition to haziness is not large, and this further suggests that the slip involved in the creation of sharkskin is occurring over only a small portion of the die.

Molecularly, the slip is thought to occur because a thin layer of polymer chains becomes strongly adsorbed at the die surface. (This layer is on the order of the polymer coil size, *i.e.*, 10 nm.) When the stress is sufficiently high, these chains can disentangle from the bulk, and slip of the bulk flow can occur on top of this thin layer. Thus, the slip plane is not precisely at the die surface, but is at the edge of the thin adsorbed layer. (Coating the die with materials onto which the polymer will not adsorb promotes slip, which then occurs at the coating-polymer interface.) The intermittency of the stick-slip motion occurs because once the polymer chains at the surface have disentangled and slip occurs, the stress within this thin layer becomes small, so the disentangled chains then relax and re-entangle with the bulk. This stretching and relaxation of the adsorbed chains should thus be periodic, and the observed period of the sharkskin, t_{ss} , should be comparable to the intrinsic relaxation time of the polymer, t_p .

Empirically, it is often observed that for a given polymer and die, sharkskin sets in at a critical stress (τ_w), independent of temperature, typically of the order of 100 kPa. In the context of the model described above, this stress would correspond to that required for the adsorbed chains to become disentangled from the bulk. If sharkskin sets in at a particular τ_w for a given polymer, then if $\eta(\dot{\gamma}_w)$ is known as a function of temperature (such as by knowing m_0 , n , and E_a), then the maximum rate at which a smooth extrudate can be produced at any temperature can be calculated in advance. Using your observations of the extrudate (clear vs. hazy) at various shear rates during the “capillary rheometry” experiments, determine whether or not your observations are consistent with the idea of a critical τ_w independent of temperature, and if so, estimate its value for your experimental system.

During the die swell measurements, you should also have measured the wavelength of the “ribbed” surface, λ , for those cases where it was discernable. Ignoring the change of dimensions on crystallization, λ is related to t_{ss} by:

$$\lambda = V t_{ss} \tag{17}$$

where V is the average linear velocity of the extrudate. V is necessarily related to Q by:

$$Q = (\pi D^2/4)V \tag{18}$$

where D is the extrudate diameter. Combining (17) and (18) allows for the determination of t_{ss} from the measured quantities, Q and λ :

$$t_{ss} = \pi D^2 \lambda / 4Q \tag{19}$$

However, it would be more interesting to see how λ (or t_{ss}) is predicted to scale with process conditions, namely stress or shear rate, so that we can test the model. We can further relate V to the shear rate at the wall $\dot{\gamma}_w$ with equation (15), assuming Newtonian behavior for simplicity:

$$Q = (\pi D_0^3/32)\dot{\gamma}_w \quad (20)$$

where D_0 is the die diameter. (Assuming power-law behavior would only change the value of the constant (=32) in equation (20), and thus would not affect the arguments below.) From (17), (18), and (20), we obtain:

$$\lambda = (D_0/8)(D_0/D)^2(\dot{\gamma}_{wt_{ss}}) \quad (21)$$

The ratio D_0/D in equation (21) is simply the reciprocal of the die swell; as discussed previously, this is thought to be a unique function of the wall shear stress, τ_w , or of the ratio τ_w/T . For a polymer in steady flow, the degree of chain deformation is given by $\dot{\gamma}_w t_p$. According to rubber elasticity theory, the stress is directly related to the $\dot{\gamma}_w t_p$ product and the temperature as:

$$\tau = Tf(\dot{\gamma}_w t_p) \quad (22)$$

where density changes with temperature have been neglected. The function f in equation (22) would be given explicitly by rubber elasticity theory, but the only important thing here is that f is independent of T for a given polymer. If the die swell is uniquely related to τ_w/T , then equation (22) says that die swell is also uniquely related to $\dot{\gamma}_w t_p$.

If t_{ss} is proportional to t_p (as the mechanism described above requires), then the product $\dot{\gamma}_{wt_{ss}}$ in equation (21) is also uniquely related to τ_w/T . Thus, equation (21) makes the following simple, important, and testable prediction: the sharkskin period λ should be proportional to the die diameter, and should be a unique function of the ratio τ_w/T . Since all your data are taken with a single die, this means that all your data should collapse onto a single curve if you plot λ vs. τ_w/T . Such a plot, taken from Wang *et al.*, is shown below in Figure 6 (polymer is a different LLDPE).

Examine whether or not your data are consistent with this recent model of Wang *et al.* Construct a plot analogous to Figure 6 and determine whether or not the data collapse onto a single curve. Since t_{ss} should be similar in magnitude to t_p , it is worthwhile to estimate t_p from rheological data. t_p is roughly equal to the reciprocal of the shear rate $\dot{\gamma}_c$ at which the material shifts from Newtonian to power-law behavior. From the figure provided in Appendix B (System IV data for the HS-7028), $\dot{\gamma}_c$ can be approximated by drawing lines tangent to the low- $\dot{\gamma}$ and high- $\dot{\gamma}$ portions of the curves. The results are given below:

Temperature ($^{\circ}\text{C}$)	$\dot{\gamma}_c$ (sec^{-1})	t_p (msec)
150	4.8	210
160	6.0	170
190	9.0	110
220	15.5	65

Wang *et al.* found that t_{ss} depended somewhat on τ_w , so that t_{ss} and t_p were not exactly equal; however, you should find that the two quantities are of similar magnitude. Even if you do not have sufficient data to make a firm statement about the dependence of λ on τ_w/T , as long as you have at least one “ribbing” data point, you can compare t_{ss} and t_p .

While the model just described states that λ should be a unique function of τ_w/T , we have said nothing about what the form of that function should be. In Figure 6, it is empirically found that λ is simply proportional to τ_w/T . Putting this simple proportionality into equation (21), and further assuming that t_{ss} is proportional to t_p (as the model requires) leads to:

$$(D_0/D)^2(\dot{\gamma}_w t_p) = K_1 \tau_w/T \quad (23)$$

where K_1 is a proportionality constant (which includes the factor of $1/D_0$ from equation (21)). In the power-law region, from equations (7) and (14), we know that:

$$\dot{\gamma}_w = (\tau_w/m)^{1/n} \quad (24)$$

Since the die swell is thought to be a unique function of τ_w/T (something which you should critically evaluate with your own data), this implies that equation (23) can be rewritten as:

$$(D_0/D)^2(\tau_w/T)^{1/n} = K_2 \tau_w/T \quad (25)$$

where K_2 is another proportionality constant. Rearranging equation (25) gives:

$$(D/D_0) = K_3(\tau_w/T)^{(1-n)/2n} \quad (26)$$

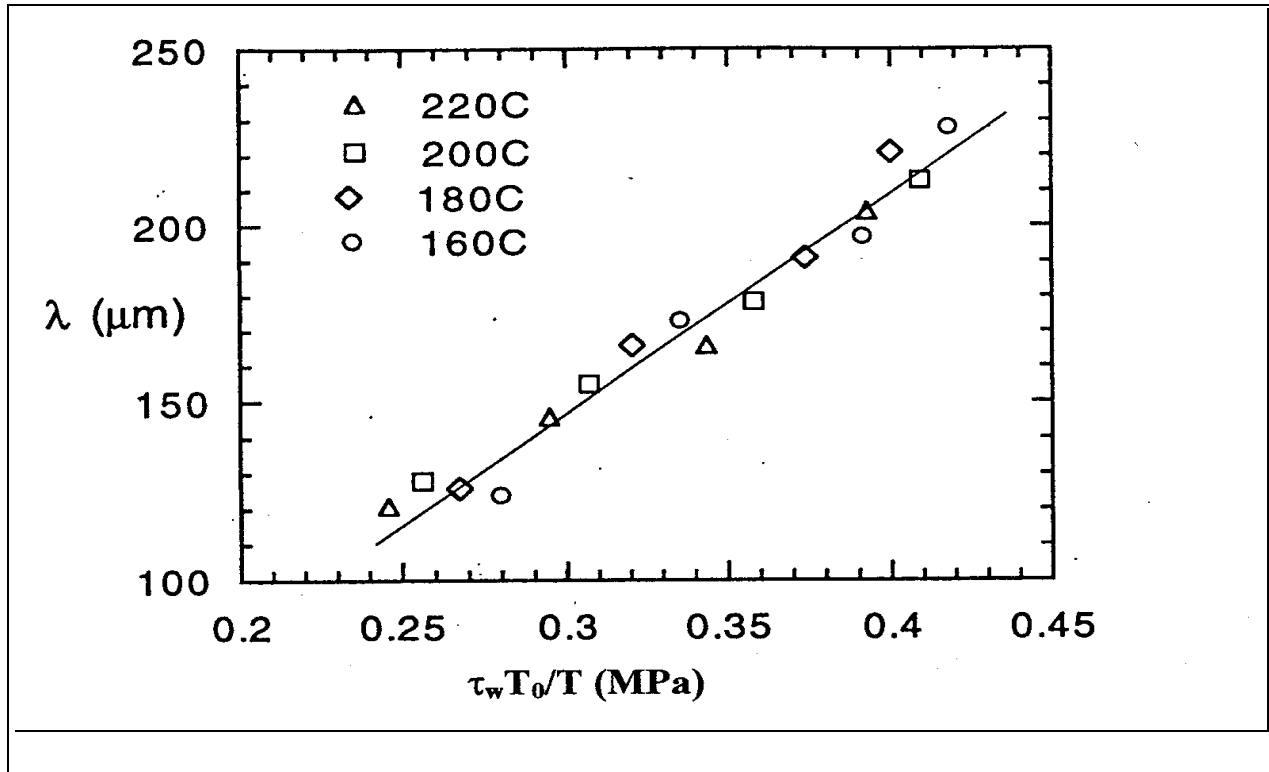


Figure 6. Master curve for sharkskin characteristics of Dowlex 2045 (LLDPE): sharkskin wavelength vs. scaled stress $\tau_w(T_0/T)$ at four temperatures, as indicated. Reference temperature $T_0 = 473\text{K}$ (200°C). From S.Q. Wang, P.A. Drda, and Y.W. Inn, *J. Rheol.*, **40**, 875 (1996).

where K_3 is another proportionality constant. If equation (26) is correct, then a log-log plot of die swell ratio vs. τ_w/T should be a straight line, with a slope of $(1-n)/2n$. Since you have determined the value of n from your capillary rheometry data, assess whether your die swell data are consistent with this simple relationship. (Recall that this simple relationship also assumes a linear relationship between λ and τ_w/T .)

APPENDIX A: PRODUCT SHEET AND MOLECULAR WEIGHT DISTRIBUTION FOR TUFLIN™ HS-7028



EXTRUSION
FILM

PRODUCT SHEET AND MOLECULAR WEIGHT DISTRIBUTION
APPENDIX A: PRODUCT



MATERIALS

FOR TUFLIN™ HS-7028
WEIGHT DISTRIBUTION

HS-7028 MATERIAL

EXCELLENT TENSILE AND PHYSICAL PROPERTIES

HS-7028 MATERIAL is an amorphous polyethylene resin with excellent toughness. It is produced using Union Carbide's UIC-80 Process and is supplied in low-density polyethylene resin with excellent toughness. It is produced using Union Carbide's UIC-80 Process and is supplied in low-density polyethylene resin with excellent toughness. It is produced using Union Carbide's UIC-80 Process and is supplied in low-density polyethylene resin with excellent toughness.

This outstanding resin can be employed as a blending resin to upgrade the film properties of other polyethylene resins that have much less toughness. However, when films with maximum performance are required, HS-7028 MATERIAL should not be diluted by the addition of less suitable resins. The use of additives such as slip and block and other polyethylene resins that have much less toughness. However, when films with maximum performance are required, HS-7028 MATERIAL should not be diluted by the addition of less suitable resins. The use of additives such as slip and block and other polyethylene resins that have much less toughness.

APPLICATIONS HS-7028 MATERIAL resin is recommended for the manufacture of films in a wide range of gauges.

PHYSICAL PROPERTIES

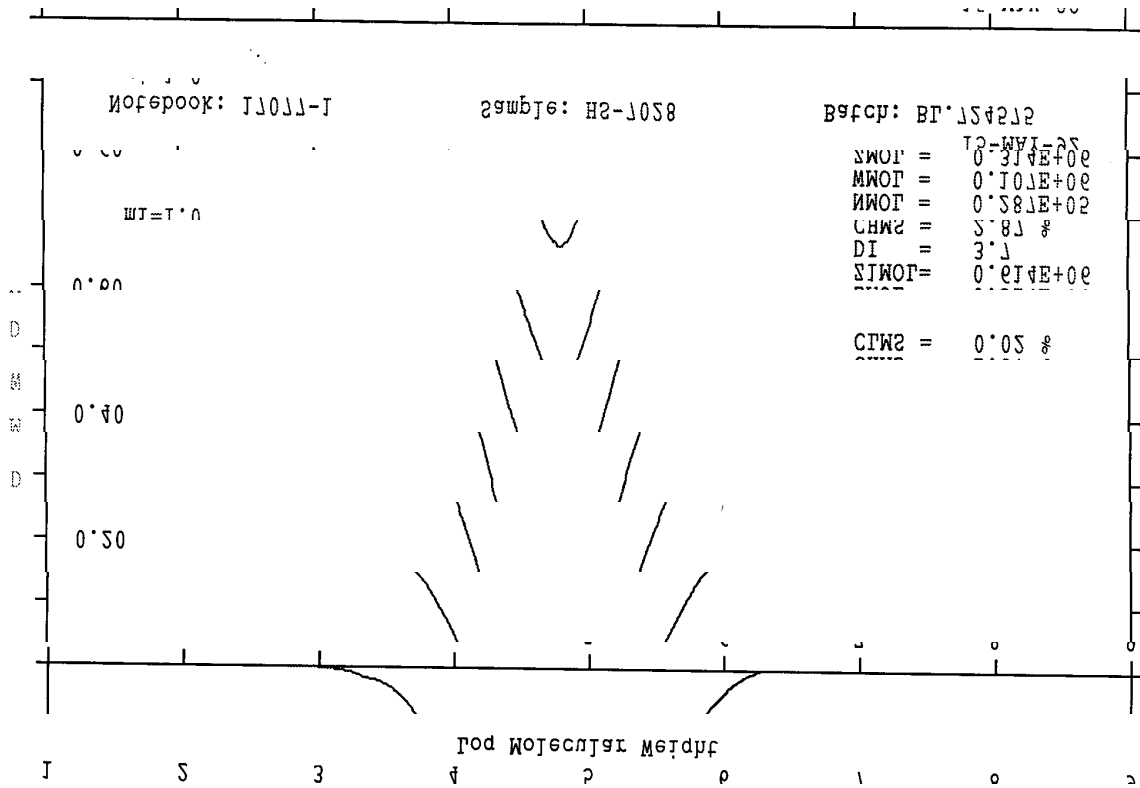
Coloring agents should be considered for some applications. HS-7028 MATERIAL should not be diluted by the addition of less suitable resins. The use of additives such as slip and block and other polyethylene resins that have much less toughness. However, when films with maximum performance are required, HS-7028 MATERIAL should not be diluted by the addition of less suitable resins. The use of additives such as slip and block and other polyethylene resins that have much less toughness.

PROPERTY	TEST METHOD	UNIT VALUE
Melt Index, g/10 minutes	D 1538	1.0
Density, g/cm ³	D 1505	0.918
Resin Properties		
Intrinsic Viscosity, dl/g		
Film Properties (a)		
Anti-block Additive Level (b)	—	0
Slip Additive Level (b)	—	0
Tensile, psi (MPa)	D 882	38,000 (2.6)
Elongation, % (MD/CD)	D 882	640 (3.4)
Tensile Strength, psi (MPa) MD/CD	D 882	2,400 (16.7) 8,000 (55.8)
Structure, in. x mil (mm)	*	18 (0.7)
Def. Prop. Impact, ft ² /lb	D 1709	540
Other Properties		
Gloss, Gardner Glossmeter 42° Dull	D 253	38
Haze, %	D 1003	18
Chemical Resistance	D 1541	Good



Union Carbide Corp. 06817-0001
38 Old Bridgeport Road
PO BOX 6817 DIVISION
UNION CARBIDE CORPORATION

* Union Carbide Test Procedure available upon request. All dependent upon operating conditions and additive package. (b) Film thicknesses are 0.001 to 0.010 in. (0.025 to 0.254 mm).



Gel-Permeation Chromatograph showing molecular weight distribution of Tufin™

APPENDIX B: RHEOMETRIC DATA FOR TUFLIN™ HS-7028 NATURAL7

Data Collected on Rheometrics System

IV

(courtesy of J.L. Adams)

Shear	Viscosity (Pa-sec) at:			
Rate (sec ⁻¹)	150°C	160°C	190°C	220°C
0.02511	23850	19830		
0.03981	23310	19400	13140	8040
0.06309	22190	18560	12450	7870
0.1	21360	17770	12090	7730
0.1584	19880	16930	11350	7510
0.2511	19280	16120	10890	7100
0.3981	17600	15140	10180	6801
0.6309	16850	14160	9373	6475
1	15360	12950	8763	6097
1.584	13850	11709	8035	5668
2.511	12170	10570	7307	5165
3.981	10580	9276	6524	4688
6.309	9004	7996	5753	4160
10	7575	6747	4980	3686
15.84	6151	5551	4189	3168
25.11	4905	4454	3469	2659
39.81	3770	3477	2795	2193
63.09	2895	2692	2236	1804
100	2145	2021	1719	1432

APPENDIX C: EXTRUDER MAINTENANCE (FOR AI)

EXTRUDER LUBRICATION

Dodge Torque-Arm Speed Reducer (top of extruder)

Grease: Remove the grease outlet plug (side of the coupling housing, front of extruder). Use the grease gun to fill the coupling through the inlet nipple (top of housing) until grease begins to squeeze through the outlet port. Replace the grease outlet plug.

Oil: Remove the magnetic drain plug (bottom of reducer housing, inside extruder) and allow the oil to drain out into a suitable container. Dispose of the oil as appropriate for organic waste. Replace the magnetic drain plug and remove the fill level plug (side of reducer housing, back of extruder). Remove the inlet plug (top of reducer housing). Add SAE 40 weight oil through the inlet port until it just begins to drain out the fill level port. Replace the fill level and inlet plugs. Capacity of the housing is nearly one quart. Recommended change interval is six months.

US Varidrive Motor (inside extruder)

Grease: As constructed, the motor has three grease nipples; two are on the top of the motor. First remove the grease outlet plug (center of motor, underside). Add grease via the nipple directly above the outlet port until grease begins to emerge from the outlet port. (For this, you will need a grease gun with a flexible neck, which you can obtain from the EQuad machine shop.) Replace the grease outlet plug. The second nipple is at the top right of the motor as viewed from the rear of the extruder. There is no outlet plug corresponding to this nipple; the grease is open to the air on the right of the motor. Add grease until you can feel its presence through the opening at the bottom of the shroud covering the right end of the motor. The third grease nipple is directly in the center of the motor; remove the grease outlet plug at the very bottom center of the motor. Add grease via the nipple directly above the outlet port until grease begins to emerge from the outlet port. Replace the grease outlet plug.

The fourth grease nipple was a necessary “in-house” modification. The gear box at the left end of the motor (as viewed from the rear of the extruder) should ideally be lubricated with SAE 30 weight oil.

However, the gasket between this box and the motor's drive shaft is shot, and cannot easily be replaced because the gear driving the belt (at the extreme end of the motor) has rusted onto the shaft. 30 weight oil drains out of the gear box rather quickly, so the gear box was packed with grease instead, which does not leak out unless the extruder is being run.

In view of this situation, the following procedures should be performed ***AT LEAST MONTHLY*** when the extruder is in use. Remove the grease outlet plug (top of gear box housing). Add grease via the nipple on the front of the gear box until it begins to emerge from the outlet port. Replace the grease outlet plug. Use a paper towel to remove the grease which has leaked through the defective gasket into the motor housing.

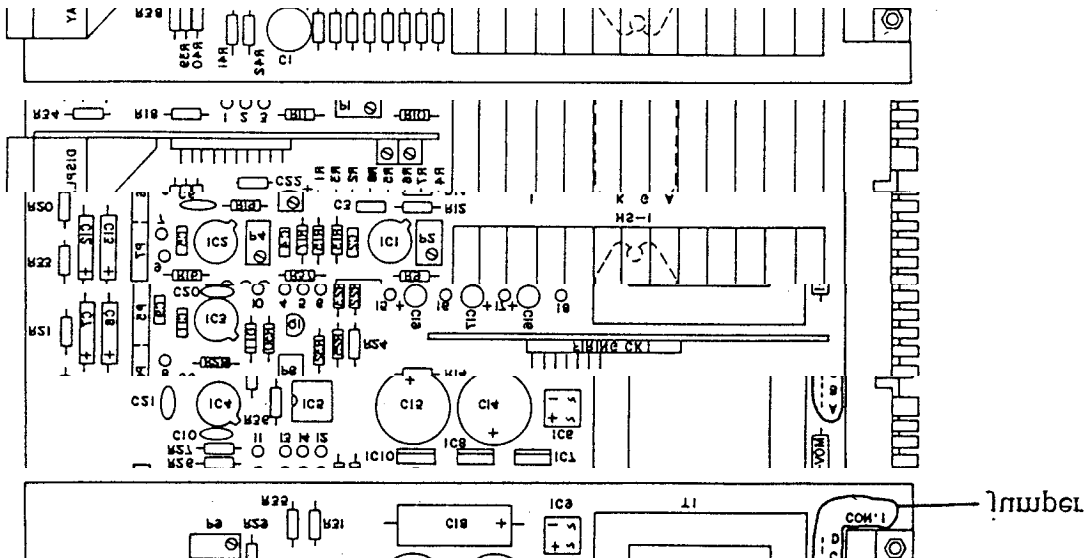
CHANGING THE DIE

- 1) Ensure that Master Power to the extruder is OFF and that the die and die adapter are cool enough to handle comfortably. When die changes are necessary, they should be performed at the *start* of the day, before the extruder has been run.
- 2) Unscrew the Swagelok cap holding the die thermocouple (Zone 4) in place and pull out the thermocouple.
- 3) Unplug the die heater from the long cable which connects to the extruder body. (This connection is about one foot from the die heater itself; if you are unsure which plugs to separate, ask the AI.)
- 4) Unscrew the die from the die adapter; this should be possible *without* the use of wrenches. If not, consult the AI. The die heater and thermocouple adapter should remain attached to the die.
- 5) Locate the second die (only two are to be used throughout this experiment, though many are lying around). This die should already have a thermocouple adapter and a die heater attached; if not, consult the AI for their whereabouts.
- 6) Clean the die threads old Teflon tape, and wrap the threads with new Teflon tape (twice around the threads is sufficient; make sure the tape is wrapped such that the act of screwing in the die does not *unwrap* the tape). Screw the die back into the die adapter, using your hands only (*no wrenches*).
- 7) Insert the thermocouple and tighten the Swagelok cap (finger-tight only).
- 8) Reconnect the die heater to the appropriate power cable.

**REPLACING/SWAPPING CONTROLLERS (ECS 6414)
AND ALARM MODULES (ECS 6460)**

The various controllers and alarm modules installed in the extruder are of slightly different vintages, and the different controllers use more than one type of fire board, but they are all essentially interchangeable. (An electronic schematic of the controller should be stored in the drawer labelled “Extruder” in A116; if not, see RAR to obtain a copy.) There should also be a spare ECS 6414 controller in the “Extruder” drawer. The interchangeability of the controllers allows this one spare to serve for any of the four zones—a useful feature, as the cost of a new controller was \$800 in 1992.

However, there is one **important** feature to note before swapping any of the controllers or alarm modules: extruder zones 1 and 2 are wired for 240VAC, while zones 3 and 4 are wired for 120VAC. The controllers will work with either power supply, but there is a jumper wire on the board which **must** be set to match the supply voltage. On the ECS 6414, this is a movable jumper wire on the lower right corner of the board, just to the right of the large transformer. Its location on the board schematic is indicated below, though the markings on the schematic (A-B-C-D-CON. I) do not correspond to the markings on the actual boards; on the boards, the jumper should connect the pin marked “H” at the extreme right of the board with the pins marked either “120V” or “240V”, as appropriate. On the ECS 6460, the jumper is actually a soldered wire in a similar position: to the right of the transformer (for 120V), or just below it (for 240V). Examine the other 6460 modules before rewiring to make sure you have the connections correct; we do not have a schematic for the 6460.



For service on the controllers or alarm modules, contact Chromalox to obtain a return authorization number. The actual service location (to which you will ship) is:

Chromalox Instruments and Controls
2716G Interstate St.
Charlotte, NC 28208
(704) 391-7887

Chromalox Instruments and Controls
1382 Heil-Quaker Blvd.
La Vergne, TN 37086-3536
phone (615) 793-3900
FAX (615) 793-5363

The standard price for repair of an ECS 6414 was \$275 in 1996.