

Chapter 2

HDPE & Propylene Materials

2.1 History and Manufacturing of Plastics and Plastic Pipes

Plastic materials used for pipes are typically manufactured with either thermosetting or thermoplastic resins. Corrugated plastic pipes used for highway and storm drainage applications belong to the thermoplastic group. The two primary types of corrugated plastic pipes used in these applications are corrugated High Density Polyethylene (HDPE) pipes and corrugated Polypropylene (PP) pipes, collectively referred to as Corrugated Plastic Pipes (CPP). Corrugated Polyvinyl Chloride (PVC) pipes are less common for these applications and will not be addressed in this handbook. HDPE and PP pipes exhibit attributes of toughness, flexibility, chemical resistance and non-conducting electrical properties. Corrugated HDPE pipes were first manufactured in the 1960s and used for agricultural drainage applications. As larger diameters were manufactured, the product soon became prevalent in highway drainage applications. Today, both HDPE and PP pipes are commonly used for storm sewers, storm drains, side drains, culverts, stormwater detention and retention systems, and other drainage applications.

2.1.1 History and Manufacturing of HDPE and HDPE Pipe

At the end of the 19th century, the German chemist Hans von Pechmann noted a precipitate while working with a form of methane in ether. In 1900, two German chemists, Eugen Bamberger and Friedrich Tschirner, identified this compound as polymethylene, a very close cousin to polyethylene. Thirty years later, a high-density residue was created by subjecting ethylene to a large amount of pressure by an American chemist at E.I. du Pont de Nemours & Company, Inc. Carl Shipp Marvel.

While experimenting with ethylene at high pressures, two British chemists, Eric Fawcett and Reginald Gibson, created a solid form of polyethylene in 1935. Its first commercial application came during World War II, when the British used it to insulate radar cables. In 1953, Karl Ziegler of the Kaiser Wilhelm Institute (later renamed the Max Planck Institute) and Erhard Holzkamp invented high-density polyethylene, or HDPE. The process included the use of catalysts and low pressure, which is the basis for the formulation of many varieties of polyethylene compounds. Two years later, in 1955, HDPE was produced as pipe. For his successful invention of HDPE, Ziegler was awarded the 1963 Nobel Prize for Chemistry.

HDPE has a density range between 0.941 and 0.965 and is a thermoplastic material composed of carbon and hydrogen atoms joined together to form high molecular weight products (Figure 2.1). Methane gas is converted into ethylene, and with the application of heat and pressure, is converted into polyethylene as shown in Figure 2.1. The polymer chain may be 500,000 to 1,000,000 carbon units long. Short and/or long side chain molecules exist with the polymer's long main chain molecules. The longer the main chain, the greater the number of atoms, and consequently, the greater the molecular weight. The molecular weight, the molecular weight distribution, and the amount of branching all can determine many of the mechanical and chemical properties of the end product.

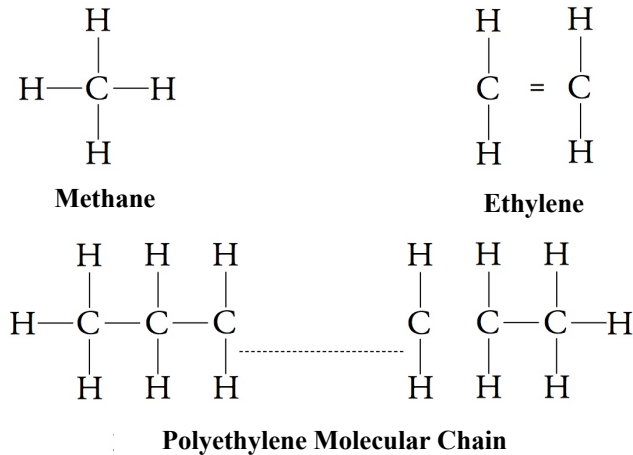


Figure 2.1: Molecular characteristics of HDPE (1)

Other common polyethylene (PE) materials are medium-density polyethylene (MDPE; $0.926 \leq \text{density} < 0.940$) commonly used for low-pressure natural gas pipelines; low-density polyethylene (LDPE; $0.910 \leq \text{density} < 0.925$), typical for small-diameter water-distribution pipes; and linear low-density polyethylene (LLDPE; $0.910 \leq \text{density} < 0.925$), which retains much of the strength of HDPE and the flexibility of LDPE, and has application for drainage pipes. Less common PE materials are ultra-high molecular weight polyethylene (UHMWPE; $0.930 < \text{density} < 0.940$) and very low density polyethylene (VLDPE; $\text{density} < 0.910$).

2.1.2 History and Manufacturing of PP and PP Pipe

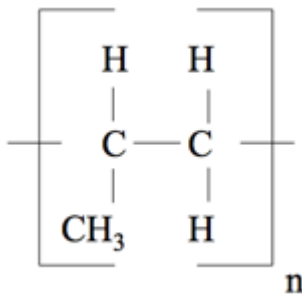


Figure 2.2: Propylene monomer

Polypropylene was invented in 1955 by the scientist F.J. Natta through an addition reaction of propylene gas with a stereospecific catalyst, titanium trichloride. The manufacturing methods and mechanical properties of PP are similar to HDPE, but there are some key differences. The chemical model of a propylene monomer is shown in Figure 2.2. Polypropylene homopolymers consist of long chains of repeating propylene monomers. It is also possible to polymerize polypropylene with other monomers, such as ethylene, which results in a polypropylene copolymer. The arrangement of the ethylene and propylene monomers in the chain affects the properties of the polypropylene copolymer. They can be arranged in alternating order, by blocks, or ordered randomly. Polypropylene random copolymers that contain more than one additional monomer are called terpolymers. Generally speaking, the PP homopolymers are stiffer than PP copolymers, but the PP copolymers will have greater toughness.

One important characteristic of PP is its tacticity, which refers to the stereochemistry of the polymer. When the propylene monomers are polymerized such that the methyl group (CH₃) remains in the same location (isolated) on the repeating chain (as depicted in Figure 2.3), the PP is classified as isotactic. Most commercial PP is 90 to 95% isotactic. When the CH₃ molecule is in a random location on the chain, the PP is termed as atactic (without order), as shown in Figure 2.4. This results in an amorphous material with rubbery characteristics. If the order of the CH₃ molecule alternates in a repeating fashion, a syndiotactic PP results (Figure 2.5).

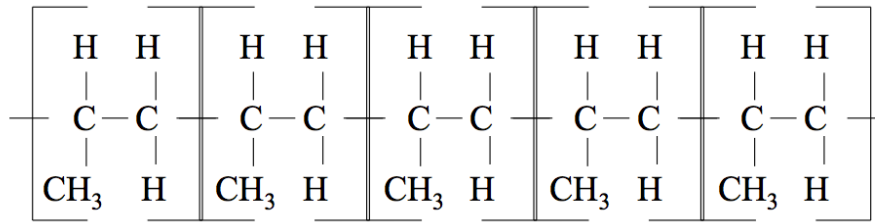


Figure 2.3: Isotactic polypropylene

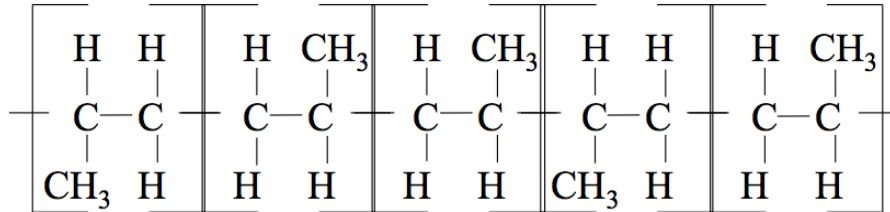


Figure 2.4: Atactic polypropylene

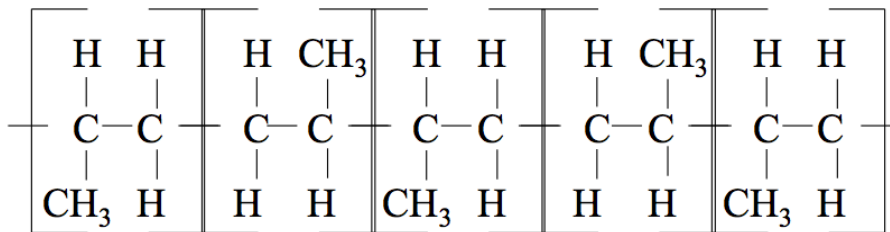


Figure 2.5: Syndiotactic polypropylene

While PP has many similarities to HDPE, there are some key differences. First, the density of PP is around 0.900 g/cc, while HDPE is more than 0.941 g/cc. This is in part due to PP being generally a more amorphous (less crystalline) material than HDPE. PP is a stiffer material than HDPE, which makes it desirable from a pipe handling standpoint. Also, it has a higher softening point than HDPE and it is extremely resistant to stress cracking, which is advantageous in warm temperature applications. One disadvantage of PP over HDPE for pipe applications is that it has a relatively high glass transition temperature of around 0 deg. F (-20 deg. C). The glass transition temperature is the temperature at which the material transitions from hard and glassy to soft and flexible. This makes the PP product more brittle than HDPE in cold temperature applications. To offset this, impact modifiers such as ethylene propylene rubbers can be added to the material; however these modifiers will reduce the stiffness. Table 2.1 summarizes some of the different properties for PP and HDPE.

Table 2.1: Comparison of key properties between PP and HDPE for corrugated pipes

| Property | Polypropylene (PP) | High Density Polyethylene (HDPE) |
|--|-----------------------------------|----------------------------------|
| Melting Point | 338 deg. F (170 deg. C) | 266 deg. F (130 deg. C) |
| Glass Transition Temp., T _g | -4 to 32 deg. F (-20 to 0 deg. C) | 0 deg. F (- 100 deg. C) |
| Density | 0.900 g/cc | 0.941 – 0.965 g/cc |
| Melt Index | 0.15 – 0.40 g/10 min | 0.20 – 0.40 g/10 min |
| Crystallinity | 40 – 70% | 70 – 90% |
| Tensile strength | 3500 – 4500 psi (24-31 MPa) | 3500 – 4000 psi (24-28 MPa) |
| Flexural modulus | 200 – 250 ksi (1379-1724 MPa) | 110 - 160 ksi (158-1103 MPa) |
| Hardness, Shore | R80 – 102 | D66 – 73 |

2.2 Viscoelastic Nature of Plastics

HDPE and PP are viscoelastic materials with time-dependent properties. This viscoelastic behavior must to be properly understood in order to determine the performance of the pipe under soil and traffic loads. There are two primary engineering principles to understand related to the viscoelastic nature of plastics: creep and stress relaxation. Creep is experienced when viscoelastic materials are placed under a constant stress (load), while stress relaxation is experienced when these materials are held at a constant strain. This is demonstrated in Figure 2.6 (2). Another important time-dependent material property to understand is the modulus of elasticity. Predicting the performance of an in-service plastic pipe (e.g. the stress, strain, deflection, and wall stability) requires knowledge of the viscoelastic nature of plastics, as well as the properties of other mechanical properties for the material resin and the geometry of the profile wall.

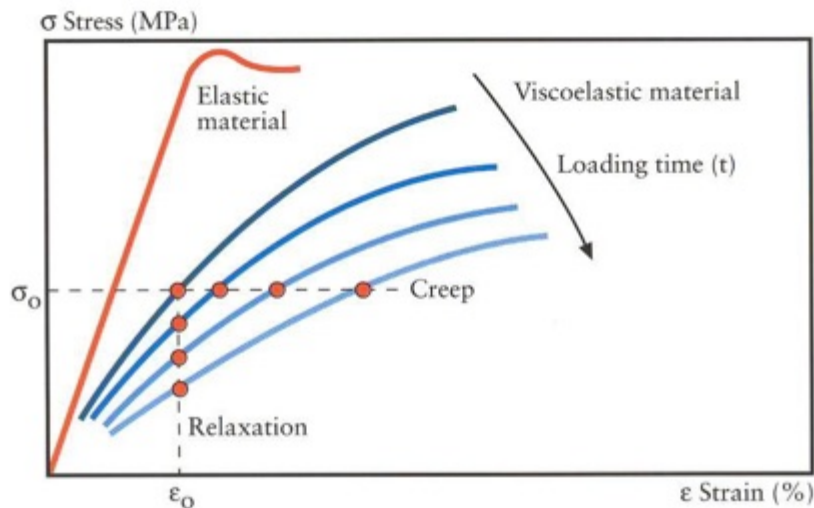


Figure 2.6: Demonstration of time-dependent nature of viscoelastic materials such as PP and HDPE (2)

2.2.1 Creep

Creep is observed when viscoelastic materials are held at a constant static load-induced stress with no restriction on the ability of those materials to deform. This is illustrated by the horizontal dashed line shown in Figure 2.6. As the loading time increases, the viscoelastic material creeps (or the strain accumulation increases). The mechanism that governs this creep is the rearrangement of the molecules in the polymer over time under a constant load. From a pipe design standpoint, creep is not typically a concern because as the pipe deflects, the loads causing the deflection are distributed into the surrounding stiffer soils, causing the creep of the polymer to eventually stabilize.

2.2.2 Stress Relaxation

Stress relaxation is illustrated by the vertical dashed line in Figure 2.6. When a viscoelastic material such as PP or HDPE is held at a constant strain, the load-induced stress relaxes over time. This is an important property that is essential to understanding buried plastic pipe performance. In buried plastic pipes, the pipe interacts with the surrounding soils to function as a system. As the pipe deflects under load, the stiffer surrounding soils restrain the pipe. The pipe deflection stabilizes and the stress on the pipe continues to decrease due to this constant strain condition. An example of this phenomenon is shown in Figure 2.7, in which a pipe was held at constant deflection (i.e. strain) between two parallel plates and the load on the pipe was measured over time. It is interesting to note how quickly the load on the pipe drops off and eventually stabilizes.

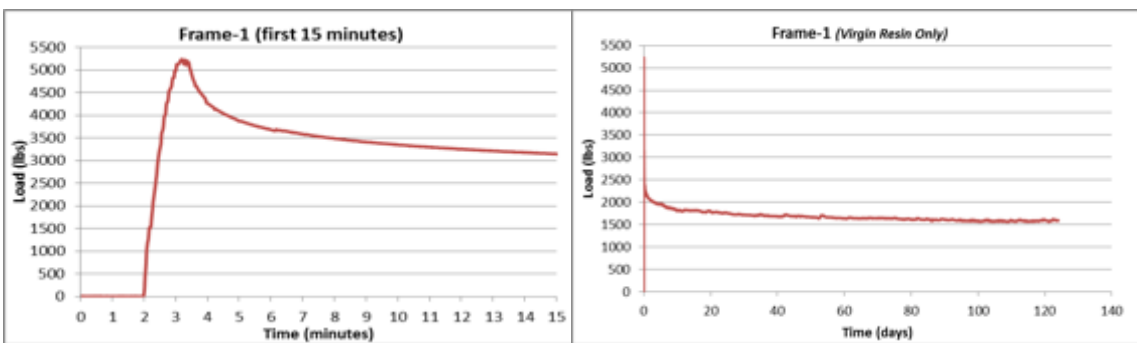


Figure 2.7: Demonstration of stress relaxation on corrugated HDPE pipe held at constant 20% deflection over time (3)

2.2.3 Modulus of Elasticity

The third key property for viscoelastic materials is the modulus of elasticity. For elastic materials, the modulus of elasticity is a constant value throughout the elastic region of the stress-strain curve and is independent of the loading rate. For example, the stress-strain curve in Figure 2.6 shows the slope of the curve for an elastic material is constant approximately to the yield point of the material. This is true regardless of the loading rate. The modulus of elasticity for an elastic material can be found by the application of Hooke’s Law, as shown in Eqn. 2.1.

$$\sigma = \varepsilon * E \rightarrow E = \sigma / \varepsilon \quad (\text{Eqn. 2.1})$$

where:

- σ = Stress, psi (MPa);
- ε = Strain, in./in. (cm/cm); and,
- E = Modulus of elasticity, psi (MPa).

For a viscoelastic material, the modulus of elasticity is time-dependent and longer duration loadings will result in a lower modulus of elasticity than shorter duration loadings. This material behavior is seen in Figure 2.6, where the slope of the stress-strain curve decreases with extended loading times.

Proper understanding of the time-dependent properties of viscoelastic materials such as HDPE and PP is critical for design purposes. The equations that govern design rely on the application of either long-term or short-term properties of the material. For example, when short-term loads are applied to the pipe (e.g., live loads from vehicles), it is appropriate to use a short-term modulus and tensile strength in design. On the other hand, for long-term loading conditions such as soil loads, it is more appropriate to use the material's long-term properties. In order to determine the long-term (defined as more than 50 years) stress in the pipe wall, one can determine the long-term strain based on the deflected condition of the pipe and can calculate the stress based on the long-term modulus. It should be noted that while the stresses in a buried pipe are greatly reduced over time, they do not scale down to zero unless the strain in the pipe wall amounts to zero strain.

2.2.4 Determining the Long-Term Properties of HDPE and PP

There are a few different test methods that can be used to determine the long-term properties of viscoelastic materials. All of them utilize some type of data extrapolation in order to project the long-term properties. Long-term property testing can be completed in a relatively short period of time by applying the concept of Time-Temperature Superposition in order to shift elevated-temperature creep data to ambient conditions. This is possible because HDPE and PP exhibit a temperature dependency based on the principles of Arrhenius, as described in Eqn. 2.2.

$$k = Ae^{-E_a/RT} \quad (\text{Eqn. 2.2})$$

where:

- k = Rate constant;
- A = Pre-exponential term;
- E_a = Activation energy (J / mol);
- R = Ideal gas constant (J / mol-K); and,
- T = Temperature (K).

The conventional method for determining the long-term creep and tensile properties of plastic materials is described in ASTM D2990, *Standard Test Methods for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics* (4). This test method measures the creep of test specimens held at various stresses. The testing can be accelerated by extending to elevated temperatures. A new test specimen is used for each stress and/or temperature condition. The time to failure which is typically the time necessary to achieve some predetermined strain or creep limit,

is plotted for each condition and a master curve is developed and extrapolated to the desired long-term service conditions related to temperature and stress.

An alternative method for determining the long-term creep, creep-rupture, and modulus properties of viscoelastic plastics such as PP and HDPE is the Stepped Isothermal Method (SIM). This method is described in ASTM D6992, *Standard Test Method for Accelerated Tensile Creep and Creep-Rupture of Geosynthetic Materials based on Time-Temperature Superposition Using the Stepped Isothermal Method* (5). It differs from ASTM D2990 in that a single test specimen is held at a constant stress and then subjected to a number of stepped temperature increases. The specimen creep at each temperature step is measured and a master curve is developed from the data. The advantage of the SIM test is that the testing can be completed within a very short timeframe (typically in less than two days) while standard creep-rupture testing can take up to a year to complete. Another advantage of the SIM test is that all of the testing is completed on the same specimen, thereby eliminating many of the variables associated with sample preparation. Furthermore, the test conditions are much easier to control for a test that lasts a few days rather than lasting an entire year as in the case of conventional testing. An example of a typical creep modulus curve for HDPE that can be obtained from either of these test methods is shown in Figure 2.8.

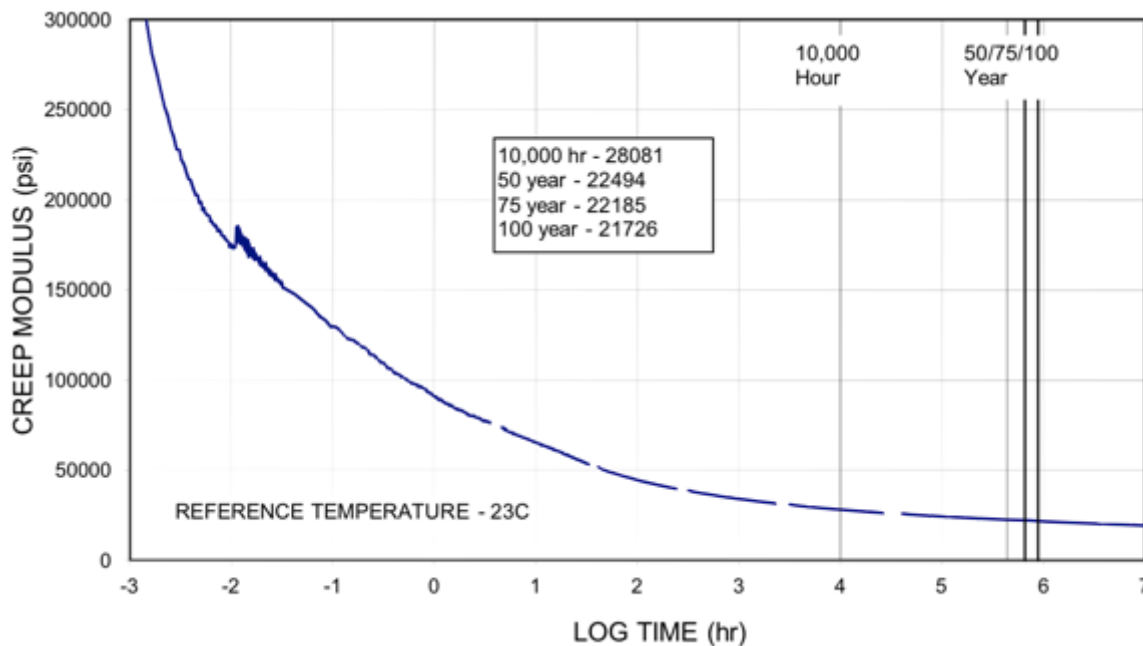


Figure 2.8: Typical creep modulus curve obtained from SIM test on HDPE pipe material (courtesy of TRI)

2.3 Properties of HDPE and PP

The characteristics of HDPE and PP materials depend upon the arrangement of the molecular chains. The molecular chains for HDPE, shown schematically in Figure 2.9, are three-dimensional and are oriented in wavy planes. Side chains can also be added to the molecular structure. The number, size, and type of these side chains determine the physical properties and characteristics of

HDPE and PP pipe materials, such as density, stiffness, tensile strength, flexibility, hardness, brittleness, elongation, creep characteristics, melt viscosity, and stress crack resistance.

2.3.1 Crystallinity

HDPE and PP are characterized as semi-crystalline polymers and are composed of both crystalline regions and amorphous regions. As shown in Table 2.1, HDPE has a higher percentage of crystalline regions than amorphous, while PP has a higher percentage of amorphous regions. Generally, the greater the proportion of crystalline regions, the higher the density of the material. As a result HDPE has a greater density than PP. Crystalline regions are those which are highly-ordered, neatly-folded, in parallel layers, and in densely-packed molecular chains. These regions occur only when the chains which branch off of the sides of the primary chains are few in number. Within crystalline regions, molecules have properties that are locally (i.e., within each crystal) and directionally dependent. In the cases where tangled molecular chains that branch off of the molecular trunk chains interfere with or inhibit the close and layered packing of the trunks, the random resulting arrangement is less dense and is termed to be amorphous. An abundance of closely-packed polymer chains results in a tough material of moderate stiffness. For clarification, the toughness of plastics refers to their resistance to impact, while stiffness refers to the rigidity of the plastic material.

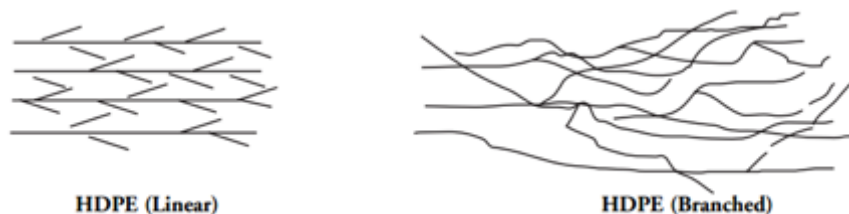


Figure 2.9: Illustrations of linear and branched HDPE

2.3.2 Molecular Weight and Molecular Weight Distribution (MWD)

The molecular weight of a polymer refers to the size of the molecules. The molecular weights for PP and HDPE can range from 200,000 to more than 1 million g/mol. The greater the molecular weight of the polymer, the greater its strength, toughness, and viscosity values. Unlike most materials, polymers do not have a uniform molecular weight and the polymerization process results in a distribution of molecular weights, or Molecular Weight Distribution (MWD). This distribution has a great effect on the properties of the material.

The distribution of differently sized molecules in PP and HDPE polymers typically follows the bell shaped normal distribution curve described by the Gaussian probability theory. As with other populations, the bell shaped curve can reflect distributions ranging from narrow to broad. A polymer containing a broad range of chain lengths is described as having a broad MWD. Resins with this type of distribution exhibit good Environmental Stress Crack Resistance (ESCR), good impact resistance, and good process ability. A polymer with a narrow MWD contains molecules that are nearly the same in molecular weight, and it will crystallize at a faster, more uniform rate. This results in a product that will hold its shape. Many HDPE resins used today are bi-modal and offer very high stress crack resistance, excellent processing characteristics, and good crystallization rates. Figure 2.10 shows the differences between the various molecular weight

distributions of HDPE.

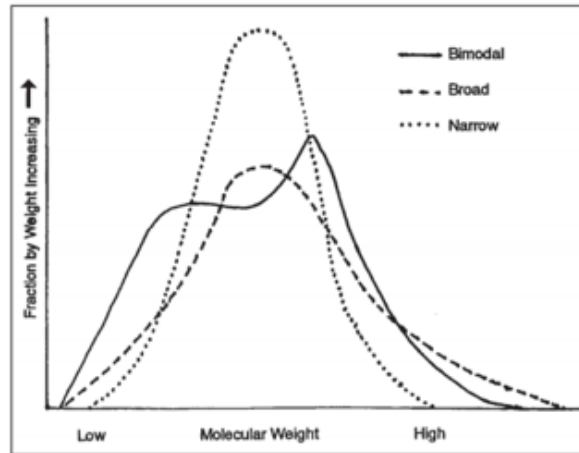


Figure 2.10: Examples of narrow, wide and bimodal molecular weight distributions

There are different ways to quantify the breadth of the distribution of the polymer. One measure is known as the polydispersity index (PI). The PI is defined as the ratio of the number-average molecular weight ($M_{n,avg}$) to the weight-average molecular weight ($M_{w,avg}$). Figure 2.11 provides a depiction of the difference between the various measures of molecular weight, as adopted from the *Handbook of Polyethylene* (6).

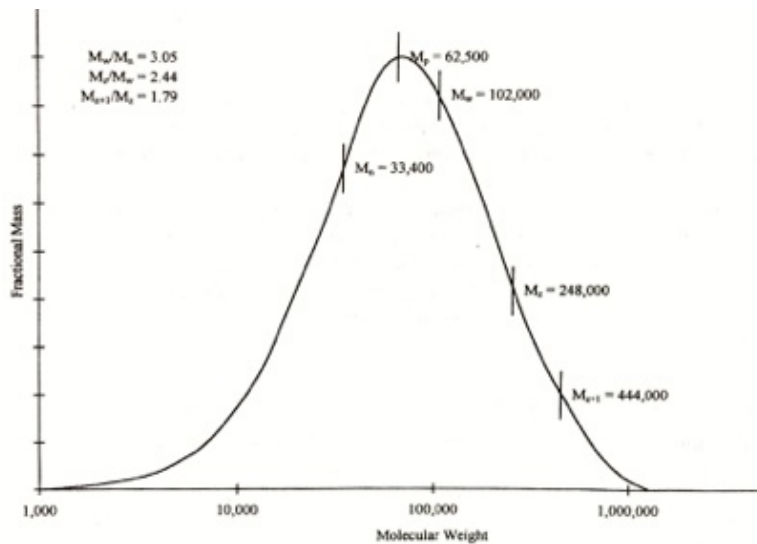


Figure 2.11: Molecular weight distribution of typical HDPE material (6).

The number-average molecular weight is calculated as the total weight of the molecules in a sample divided by the total number of molecules, as expressed in Eqn. 2.3. The weight-average

molecular weight places a greater emphasis on the higher-weight molecules in the sample and is determined by Eqn. 2.4.

$$M_{n,avg} = \frac{\sum M_i N_i}{\sum N_i} = \frac{\sum W_i}{\sum N_i} \quad (\text{Eqn. 2.3})$$

$$M_{w,avg} = \frac{\sum M_i^2 N_i}{\sum M_i N_i} = \frac{\sum M_i W_i}{\sum W_i} \quad (\text{Eqn. 2.4})$$

where:

- $M_{n,avg}$ = Number-average molecular weight;
- $M_{w,avg}$ = Weight-average molecular weight;
- M_i = Molecular weight of molecules in fraction i of the MWD;
- N_i = Number of molecules in fraction i of the MWD; and,
- W_i = Weight of molecules in fraction i of the MWD.

The molecular weight distribution of the polymer can be determined through size exclusion chromatography (SEC). One type of SEC often used for HDPE materials is gel permeation chromatography (GPC). This process consists of dissolving the polymer in a solvent and passing it through a series of columns containing a porous gel media that separates the molecules by size. Larger molecules pass through the columns faster than smaller molecules. An illustration of GPC is shown in Figure 2.12. Another means of measuring MWD, though not as common, is through rheology techniques.

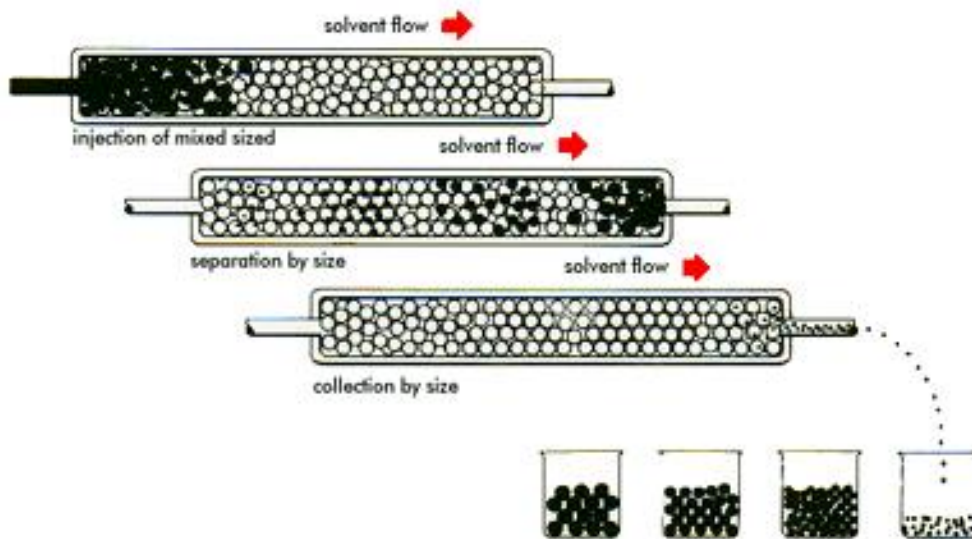


Figure 2.12: Illustration of GPC used to determine MWD of polymers

2.3.3 Density

The density of HDPE and PP is a measure of the proportion of crystals within its mass and HDPE has a more crystalline structure than PP and thus a greater density. Crystals result from the layering and close packing of the molecules and are denser than the tangled, disordered arrangement of

molecules in the amorphous regions. The crystallization is affected by cooling rate and a slow cooling rate will result in greater crystallization and greater density.

Copolymers are often used to create and control the formation of side branches in the molecular structure. The side branches reduce the density of the polymer. Homopolymers are produced without comonomers and experience very little branching; therefore, they have a very high density. In order to reduce the density of HDPE, various comonomers such as butene, hexene or octene are added. In PP, ethylene and other comonomers can be added. The greater the length of the branched carbon chains, the lower the final density.

Figure 2.13 is adopted from Peacock (6) and shows the relationship between density (also expressed as a degree of crystallinity) and branch content for various grades of HDPE, MDPE, and LDPE materials. It is also evident from the chart in Figure 2.13 that quick-cooled (or quenched) materials exhibit a lesser degree of crystallinity and therefore a lower density than their slow-cooled counterparts that allow crystallization to take place at a slower rate.

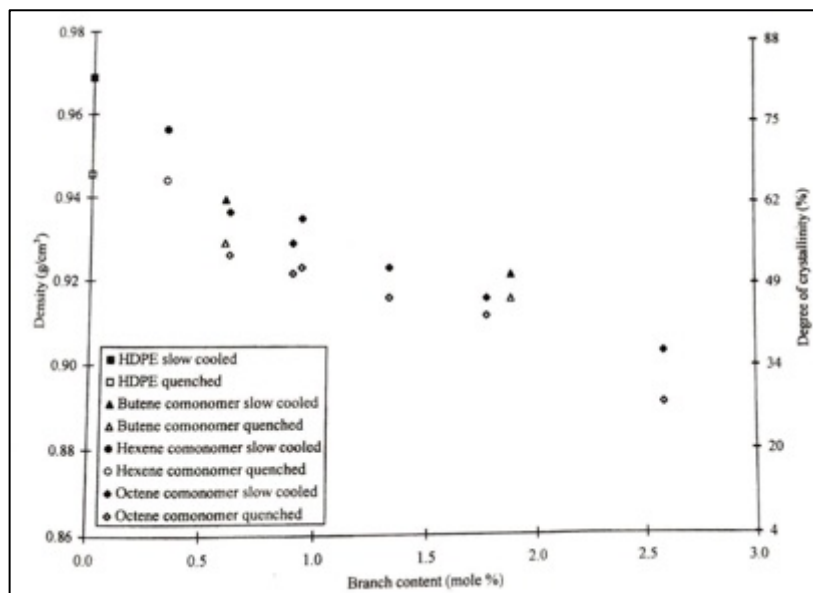


Figure 2.13: Density (crystallinity) as a function of branch content for slow cooled and quenched polyethylene materials (6)

The standard in ASTM D3350 classifies polyethylene by density as follows:

HDPE: $0.941 \leq \text{density} < 0.965 \text{ g/cc}$

LDPE: $0.910 \leq \text{density} < 0.925 \text{ g/cc}$

MDPE: $0.926 \leq \text{density} < 0.947 \text{ g/cc}$.

There are less commonly employed PE materials that are homopolymers (density $\geq 0.965 \text{ g/cc}$)

and VLDPE (density < 0.910 g/cc). Flexural stiffness and tensile strength increase with density, and results in an increase in brittleness and a decrease in toughness and stress crack resistance.

The density of HDPE is typically measured by the methods outlined in ASTM D1505, *Test Method for the Density of Plastics by the Density-Gradient Technique* (7). This method involves placement of the material in a calibrated density column and determination of the density based on its position in the column. Other acceptable methods for measuring density of HDPE include ASTM D792 *Test Methods for Density and Specific Gravity of Plastics by Displacement* (8), and ASTM D4883, *Test Method for Density of Polyethylene by the Ultrasound Technique* (9). The density of PP can be measured either by the density column technique or the displacement technique.

Most corrugated HDPE pipes and some PP pipes are produced with carbon black or other colorants that protect the product from ultraviolet (UV) degradation. These colorants will add to the density of the material. The standard ASTM D3350 (10) provides the equation in Eqn. 2.5 to determine the base resin density of HDPE if the carbon black content is known:

$$D_r = D_p - 0.0044C \quad (\text{Eqn. 2.5})$$

where:

- D_r = Base resin density, g/cc;
- D_p = Compound density, including carbon black, g/cc; and,
- C = Percent carbon black in compound.

Most of the pipe specifications for corrugated HDPE pipe (e.g., AASHTO M 294 and ASTM F2306) specify the density of the base resin and not of the compound. However, many of the specifications for PP pipe (e.g., AASTHO M 330 and ASTM F2736) specify the density of the compound (i.e., base resin plus all of the compounding ingredients, including colorant). It is important to read the specification carefully to determine whether the density measurement is to be made with or without the colorant included.

Density is a very important parameter for pipe stiffness. For a given pipe profile and geometry, a greater resin density will result in a stiffer pipe. However, denser materials are more prone to stress cracking and brittle failures and manufacturers are careful to select a material with an appropriate balance of properties.

2.3.4 Melt Index

The melt flow rate measures the viscosity of HDPE or PP in its molten state. It is a parameter related to the average molecular weight of the resin chains in a polymer and is observed in Figure 2.10. For HDPE and PP materials, the test is conducted in accordance with ASTM D1238, *Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer* (11). In this test, the polymer is heated to a molten state and is extruded through a standard size orifice under specified conditions of pressure and temperature. The mass of material extruded over a 10-minute period is called the Melt Flow Rate (MFR) and its units are typically given in g/10 min. Figure 2.14 illustrates a schematic of a typical extrusion plastometer.

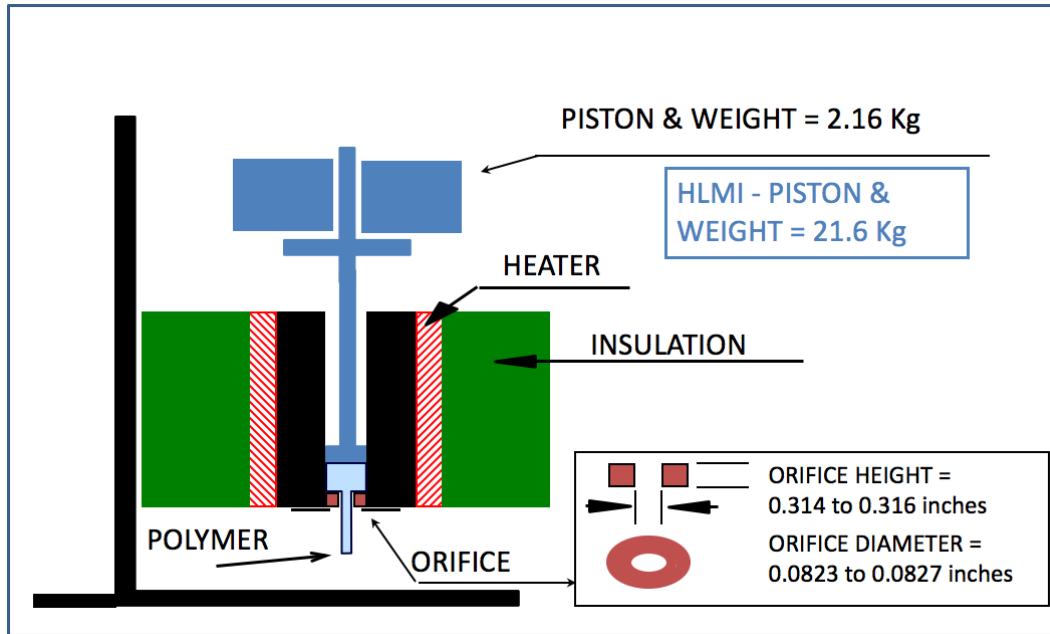


Figure 2.14: Schematic of typical device to measure melt index

Larger molecules (i.e., greater molecular weight) will have a greater viscosity and more difficulty flowing through the standard orifice. As such, the MFR of a material is inversely proportional to its molecular weight. For HDPE materials, the test is typically conducted by applying a constant pressure through the application of a 4.76-lb (2.16 kg) weight at a temperature of 374 deg. F (190 deg. C) and is termed the melt index (MI). For PP materials, the temperature of the heater is set to 446 deg. F (230 deg. C). For high molecular weight materials, a 47.6-lb (21.6 kg) weight can be used instead of the 2.16 kg weight. This is termed the high load melt index (HLMI). The ratio of the HLMI to the MI is known as the flow rate ratio (FRR) and can sometimes provide a crude indication of the MWD of the polymer, although the results should be interpreted with caution.

A lower MI which yields a higher average molecular weight is predictive of greater tensile strength, toughness, and stress crack resistance. However, a lower MI also requires greater energy and at any extrusion temperature to extrude polyethylene resin.

The MI and HLMI only give an indication of the average MW of a material. They are not indicative of the MWD of the material, which has a significant effect on the material properties. Also, it should be noted that even if two materials have identical MI readings that does not mean that they will process the same during extrusion. This is because the shear rates in the extrusion process are significantly greater than these which are applied in the melt flow rate tester. Polymers exhibit a shear thinning behavior which means that their viscosity decreases with increasing shear rates. Some polymers exhibit more shear thinning than others and an understanding of the MWD of the material can provide more insight into how the materials will process.

2.3.5 Flexural Modulus

The flexural modulus is a measure of the stiffness of material under flexural loading. It is one of the key parameters that affects the overall pipe stiffness, along with density and the geometric

properties of the profile wall (e.g., wall thickness, height and shape of the corrugation, etc.). Generally speaking, for a given polymer, the flexural modulus is directly proportional to the density. However, it should be noted that PP pipe materials, have a greater flexural modulus although their density is lower than that of HDPE pipes.

The flexural modulus of HDPE and PP materials can be determined by ASTM D790, *Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials* (12). The test is conducted by subjecting the specimens to a three-point bending load, as shown in Figure 2.15.

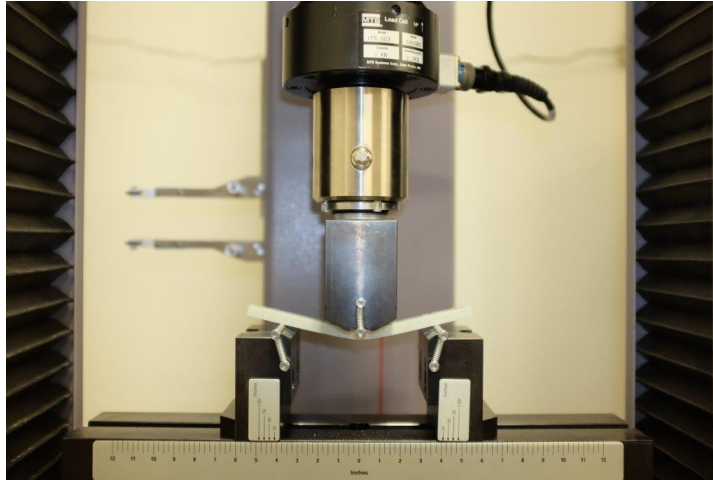


Figure 2.15: Typical flexural modulus test (photo courtesy of PSILab, Inc.)

The test is conducted in accordance with ASTM D790 Procedure B as prescribed and modified within ASTM D3350, using a 2-in (5 cm) test span. Five specimens with a cross section of 1/8 in. x 0.5 in. (0.32 cm x 1.27 cm) are inserted into the test fixture and tested at a constant crosshead speed of 0.5 in./min (1.27 cm/min). Since the slope of the stress-strain curve is not constant for most plastic materials, a secant modulus is typically reported to provide consistency when interpreting results and comparing materials. The secant modulus is obtained by taking the slope of a straight line drawn from the origin (zero strain) to some pre-defined point of strain, usually at 1% or 2% strain. For HDPE pipes made in accordance to AASHTO M 294 and ASTM F2306, a 2% secant modulus is required. The PP standards such as AASHTO M 330 and ASTM F2736 require a 1% secant modulus for PP materials. It is important to follow the standards and select the appropriate modulus to use.

For the rectangular cross section of the test specimens used in this method, the deflection required to achieve 1% or 2% strain in the outer fiber of the wall can be determined from Eqn. 2.6.

$$D = rL^2/6d \quad (\text{Eqn. 2.6})$$

where:

- D = Deflection of the center of the test specimen at the desired strain, in. (mm);
- r = Strain in the outer fiber (either 0.01 or 0.02 for 1% or 2%, respectively);
- L = Test span = 2.0 in. (5 cm); and,

d = Test specimen depth = 1/8 in.

The stress in the outer fiber of the specimen can be calculated by Eqn. 2.7.

$$\sigma = 3PL/2bd^2 \quad (\text{Eqn. 2.7})$$

where:

- s = Stress in the outer fiber of the specimen, psi (MPa);
- P = The load corresponding to the set strain level, lbf (N);
- L = Test span = 2.0 in. (5 cm);
- b = Test specimen width = 1/2 in. (1.27 cm); and,
- d = Test specimen depth = 1/8 in. (0.32 cm).

Most of the tensile machines on which these tests are conducted programmed automatically calculate the 1% or 2% secant modulus, based on the inputs provided to the software (i.e., test span, specimen dimensions). Figure 2.16 shows an illustration of the 1% and 2% secant moduli per ASTM D790.

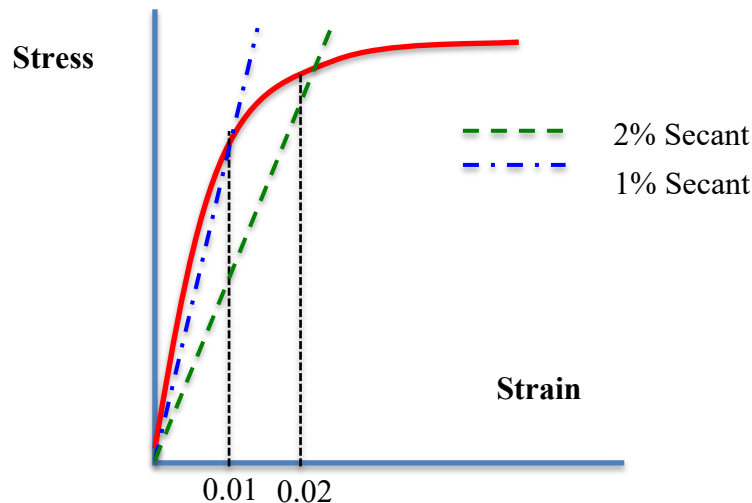


Figure 2.16: Illustration of secant modulus

For HDPE and PP pipes, the minimum pipe stiffness requirements set by the specification influences the amount of material required to produce the pipe, the cost of which dominates the cost of the finished pipe delivered to the job site. Using a material with a high flexural modulus can allow a manufacturer to potentially reduce the wall thickness of the pipe profile while maintaining the same stiffness, making for a more efficient design. However, the higher modulus materials are also more difficult to form, and manufacturers are aware of this tradeoff and will typically choose a material that optimizes their equipment capabilities.

2.3.6 Tensile Strength

The tensile strength of a plastic material is the maximum tensile stress sustained by the specimen during a tension test. The tensile test for HDPE and PP materials is conducted in accordance to ASTM D638, *Standard Test Method for Tensile Properties of Plastics* (13). Test specimens are typically fashioned in a dog-bone style, but can also be rod-shaped or tubular for certain applications. During the tensile test, the specimen is held by two opposing grips and slowly pulled apart until failure is observed. An illustration of the test is shown in Figure 2.17.

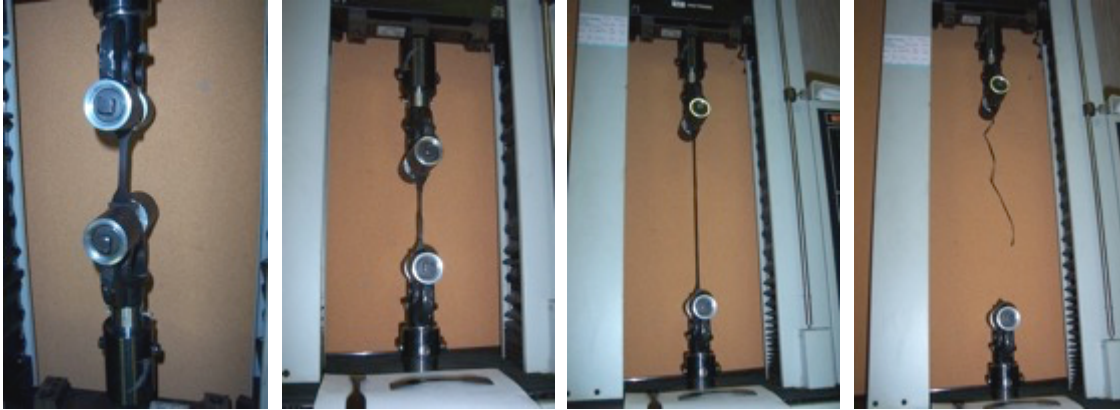


Figure 2.17: Tensile test according to ASTM D638 (courtesy of TRI/Environmental)

The yield point of the material is the first point on the stress-strain curve at which an increase in strain occurs without an increase in stress. The yield strength or yield stress is typically defined as the stress at the yield point, as seen in Figure 2.18. HDPE and PP are ductile materials that exhibit a cold drawing phenomenon once the yield strength is exceeded. The test sample develops a neck down region where the molecules begin to align themselves in the direction of the applied load. This strain-induced orientation causes the material to become stiffer in the axial direction, while in the transverse direction (i.e., 90° to the axial direction) the stiffness is lower. The stretching or elongation for materials such as PP and HDPE can be ten times the original gauge length of the sample (i.e., 1000% elongation). Failure occurs when the molecules reach their breaking strain or when defects such as edge nicks begin to grow and cause failure in the test sample. The elongation at break is typically recorded for the material. The force required to break the test sample is called the ultimate strength or the tensile strength at break. The various strengths are calculated by dividing the force (at yield or break) by the original cross-sectional area. These terms are shown in the schematic in Figure 2.18 (14), along with an image of the test equipment used to generate the curve.

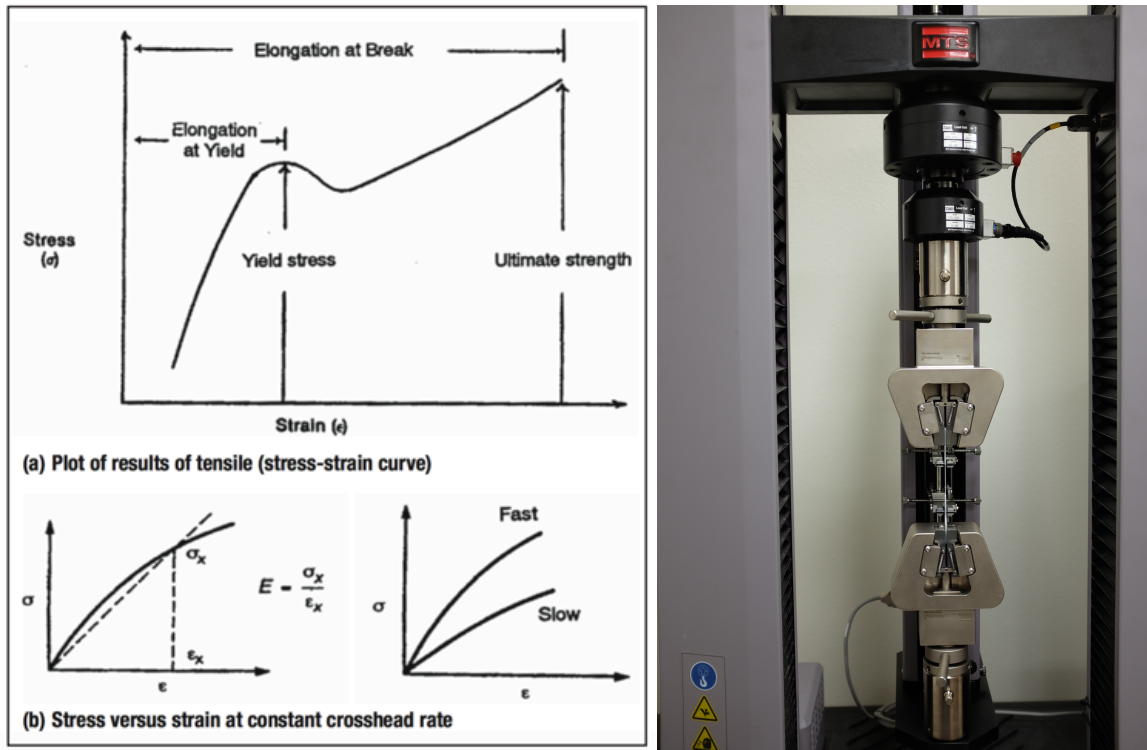


Figure 2.18: Stress-strain curve for typical polymer (14), (photo courtesy of PSILab, Inc.)

A formulation of greater density with higher fraction of crystals and a lower melt index is predictive of greater tensile strength and increased brittleness.

2.3.7 Impact Strength

The resistance to impact loads is an important factor to consider for plastic pipe materials, as the pipe may be subjected to impact loading during both handling and installation. After the pipe is installed resistance to impact loads is of considerably less importance. The impact strength of a PP or HDPE material can be determined through the Izod Impact test, described in ASTM D256, *Standard Test Methods for Determining Izod Impact Resistance of Plastics* (15). In this test, a notched material specimen is held in a vice and struck by a swinging pendulum. The amount of energy absorbed by the test specimen is determined by measuring the distance that the pendulum swings after striking the specimen. The pipe standards detail the testing conditions for the test and the minimum amount of impact resistance. Figure 2.19 shows a schematic of the Izod impact test machine set up.

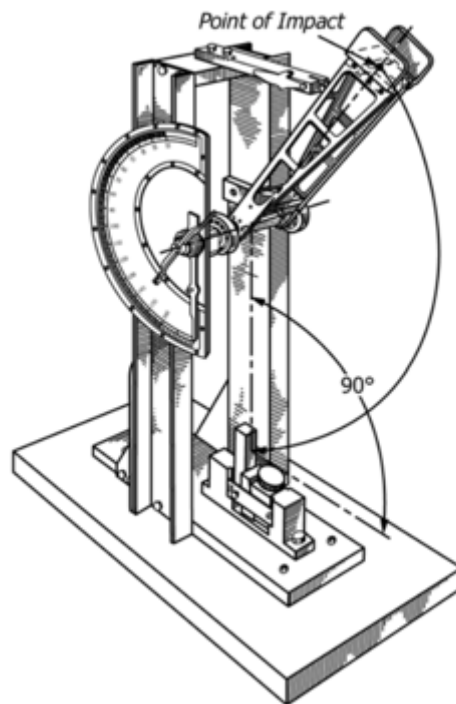


Figure 2.19: Izod Impact test machine (15)

The impact resistance of a finished pipe product can also be assessed. This is typically done with a falling weight test, in which a specified weight (tup) is dropped from a given height onto a conditioned pipe. Typically, several drops are required to be conducted within a set amount of time (usually equates to six strikes per minute). The individual pipe standard dictates the conditions and failure criteria for the test, because they vary from HDPE to PP products. The standard impact test on finished products is detailed in ASTM D2444, *Standard Test Method for Determination of the Impact Resistance of Thermoplastic Pipe and Fittings by Means of a Tup (Falling Weight)* (16).

2.3.8 Poisson's ratio

Poisson's ratio can be computed as the ratio of the decrease in lateral strain to the increase in longitudinal strain. For example, when an axial tensile force is applied to a test specimen, the specimen will increase in length (due to longitudinal strain) and decrease in thickness (due to lateral strain). The value of Poisson's ratio ranges between 0.40 and 0.45 for HDPE and PP. The Poisson's ratio is dependent on temperature and the working stress range of the material. A typical value used for design purposes is 0.45.

2.3.9 Environmental Stress Crack Resistance (ESCR)

Under certain conditions of temperature and stress in the presence of certain chemicals, HDPE may begin to crack sooner than it would at the same temperature and stress in the absence of those chemicals. This phenomenon is called environmental stress cracking (ESC).

Stress cracking agents for polyethylene tend to be polar materials such as alcohols, detergents (wetting agents), halogens, and aromatics. The property of a material to resist ESC is called

environmental stress crack resistance (ESCR). Failures from ESC are found typically to be due to the development of cracks in areas of tensile stress that slowly grow and propagate over time. Figure 2.20 provides an illustration of this phenomenon. Research has shown that ESC appears to originate at the boundary of or interphase region between the crystalline and amorphous regions of a semi-crystalline polymer such as HDPE (17). In general, the number of boundaries or interphase regions increases with the level of crystallinity of the polymer. Since stress cracking originates at these boundaries, semi-crystalline polymers such as HDPE are more prone to ESC than more amorphous polymers (such as MDPE) that have fewer of these boundaries or interphase regions. The ESCR of HDPE can be greatly improved by the presence of tie molecules that extend from one crystalline region to another. These long molecules help to hold the crystalline regions together in order to prevent stress cracking. Many of HDPE materials in use today can be formulated to exhibit extremely high SCR due to the presence of these tie molecules. Stress cracking is not a concern for PP materials.

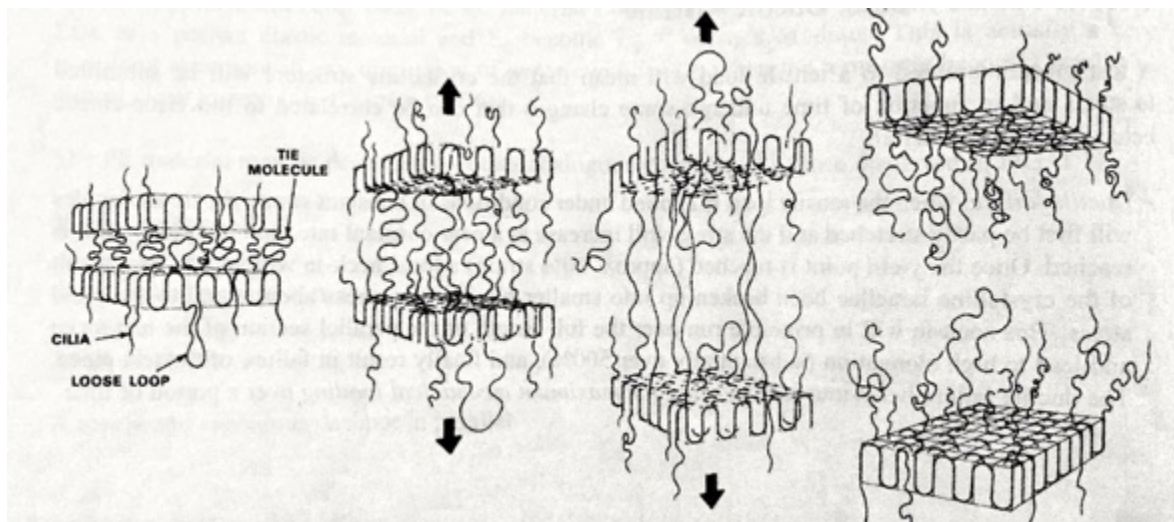


Figure 2.20: Depiction of stress cracking in HDPE at low stress levels (6)

There are a variety of ESCR test method which can be used to determine the chemical resistance of pipe materials. The standard test currently used in the polyethylene industry is the bent-strip test. It is also called the Bell Test, since it was developed during the 1950s for wire and cable coatings for the telephone industry. ASTM D1693, *Standard Test Method for Environmental Stress Cracking of Ethylene Plastics* (18), describes the test method used to determine the ESCR value for polyethylene. Ten small compression-molded specimens are notched and bent, and then placed into a holder. The holder is immersed into a tube of a surfactant, typically one such as Igepal CO-630 at 212 deg. F (100 deg. C) and at a 100% concentration, and the time to failure is documented. The results are reported using the notation F_{xx} , where xx is the percentage of samples that have failed. For example, the statement $F_{20}=500$ hours means that 20% of the samples have failed within 0 to 500 hours. Figure 2.21 shows an illustration of a typical ESCR test.

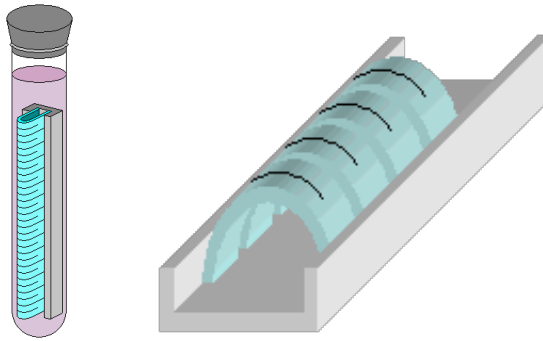


Figure 2.21: Illustration of ESCR test. Polyethylene strips are notched (indicated by the black lines) and placed in a fixture to hold them at a constant strain, then immersed in an Igepal solution (courtesy of Prospector).

It is important to note that ASTM D1693 is a constant strain test, since test specimens are held at a constant deformed shape within the holder. Due to the time-dependent nature of viscoelastic materials, it is expected that the polymer will undergo stress relaxation in the configuration (seen in Figure 2.6). While this test may be effective for materials with extremely poor SCR, it has become relatively ineffective in the current market and failures are rarely observed. This improved performance is due to both the better SCR of modern pipe materials and the nature of the test, which is a constant strain test that results in stress relaxation over time. Because of this, a test was developed to hold specimens at a constant stress and was implemented into corrugated HDPE pipe standards in AASHTO M 294 (19).

Stress cracking may be avoided by using appropriate resin formulations of SCR materials, utilizing appropriate geometric designs and manufacturing controls that prevent the occurrence of severe stress risers, and by limiting stresses and strains during pipe installation. For most buried pipe applications, the stresses are typically compressive in nature, and stress cracking does not occur in compression. Since the implementation of new SCR requirements into AASHTO M 294 in 2002, stress cracking in the field has been rarely observed.

2.3.10 Notched Constant Ligament Stress (NCLS)

The Notched Constant Ligament Stress (NCLS) test is a constant stress test that was developed primarily for corrugated HDPE pipe resins. It was adopted from the Notched Constant Tensile Load (NCTL) test, ASTM D5397 (20), which was developed for the geosynthetics industry. While the NCTL test applies a constant tensile load to the test specimen based on a percentage of the yield strength of the material, the NCLS test applies a constant tensile stress. The NCLS test method is detailed in ASTM F2136, *Standard Test Method for Notched Constant Ligament Stress (NCLS) Test to Determine Slow-Crack Growth Resistance of HDPE Resins or HDPE Corrugated Pipe* (21). The test is conducted on 2.50-in (6.4 cm) dogbone specimens that have either been machined or die-cut. The test specimens can be cut from a compression-molded plaque or prepared directly from the liner or wall of a Type S corrugated HDPE or PP pipe, provided there is a sufficient flat section from which to take the specimen. The test specimens are notched to a depth of 20% of the thickness, and then placed in an elevated temperature bath containing a surfactant (e.g., 90% distilled water and 10% Igepal). The sample is then subjected to a constant ligament stress (typically specified as 600 psi (4.1 MPa) for corrugated HDPE materials) until a brittle

failure occurs from slow crack growth as shown in Figure 2.20. Figure 2.22 for images of the NCLS test specimens and testing.

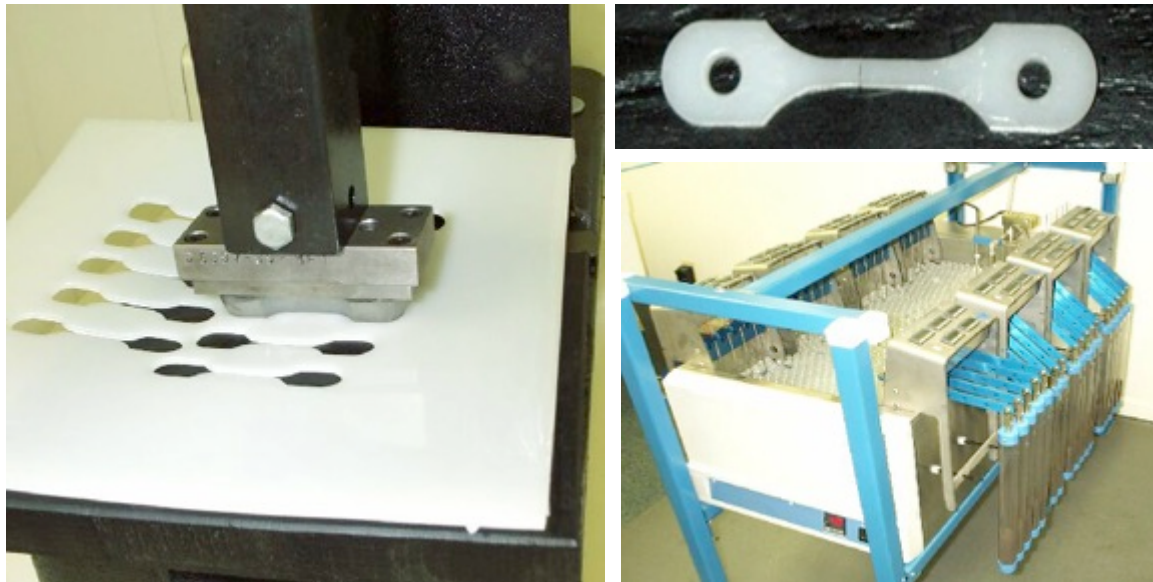


Figure 2.22: Sampling and testing NCLS specimens

2.4 Summary of effects of MW, MWD, and Density on properties

Most of the material properties of HDPE and PP are governed by one of three primary characteristics of the material: 1. Density (i.e., crystallinity); 2. Molecular Weight (typically measured from the MI); and, 3. Molecular Weight Distribution. Table 2.2 summarizes the effects of changes to these characteristics on the various pipe material properties. This table is a useful reference for evaluating new materials for pipe applications. Table 2.2 is intended to serve as a guide and it should be noted that there are always exceptions to rules as new processes and materials are developed.

2.5 Material Cell Classifications

The ASTM D3350 resin cell classifications provide the means for identification, characterization, and specification of material properties for HDPE, while ASTM D 4101 provides the classification for PP materials. The material requirements for HDPE and PP pipe are specified in their respective product standards (e.g., AASHTO M 252, AASHTO M 294 and AASHTO M 330 specify the material requirements for HDPE and PP pipe for highway applications, respectively, and these material requirements refer back to the cell classifications in ASTM D3350 and D4101). With the exception of density, manufacturers may choose higher cell classifications than the minimums required by these specifications, in order to optimize competing economic and performance constraints of production, handling, and service. Tables 2.3 and 2.4 show the cell classifications for HDPE and PP pipe materials, respectively.

Table 2.2: Effect of changes in density, MI, and MWD on HDPE and PP properties

| Property | As Density Increases, Property: | As MI Increases, Property: | As MWD Broadens, Property: |
|-----------------------------|---------------------------------|----------------------------|----------------------------|
| Tensile Strength (at Yield) | Increases | Decreases | |
| Stiffness | Increases | Decreases | Decreases |
| Impact Strength | Decreases | Decreases | Decreases |
| Low Temperature Brittleness | Increases | Increases | Decreases |
| Abrasion Resistance | Increases | Decreases | |
| Hardness | Increases | Decreases | |
| Softening Point | Increases | | Increases |
| Stress Crack Resistance | Decreases | Decreases | Increases |
| Permeability | Decreases | Increases | |
| Chemical Resistance | Increases | Decreases | |
| Melt Strength | | Decreases | Increases |
| Gloss | Increases | Increases | Decreases |
| Haze | Decreases | Decreases | |
| Shrinkage | Decreases | Decreases | Increases |

Table 2.3: AASHTO M 294 Cell Classification for HDPE Materials per ASTM D3350

| Property | Test Method(s) | Cell Classification | Requirement |
|---|---|---------------------|--|
| Density | ASTM D1505; ASTM D792; ASTM D4883 | 4 | 0.948-0.955 gm/cm ³ |
| Melt index | ASTM D1238 | 3 | 0.4 > MI ≥ 0.15 g/10 min |
| Flexural modulus (2% Secant) | ASTM D790 | 5 | 758 ≤ E _f < 1103 MPa 110,000 ≤ E _f < 160,000 psi |
| Tensile strength | ASTM D638 | 4 | 3000 ≤ φ _t < 3500 psi |
| Slow crack growth resistance - Environmental stress crack resistance (ESCR) | ASTM D1693 | 0 | Replaced by NCLS test on pipe liner or plaques; ≥ 18 hours liner ≥ 24 hours plaque |
| Hydrostatic Strength Classification Hydrostatic Design Basis (HDB) | ASTM D2837 | 0 | Not pressure rated |
| Color (C) | ASTM D4218 | C | 2% ≤ C ≤ 4% |

Table 2.4: AASHTO M 330 / ASTM F2736 cell classification requirements for PP pipe

| Property | Test Method (5) | Requirement |
|--------------------------------|-------------------|--|
| Density | ASTM D1505; D 792 | 0.900 – 0.950 gm/cm ³ |
| Melt index | ASTM D1238 | 1.50 > MI ≥ 0.15 g/10 min. |
| Flexural modulus (1% Secant) | ASTM D790 | 175,000 ≤ E _f ≤ 275,000 psi |
| Tensile strength | ASTM D638 | 3500 ≤ f _t ≤ 4500 psi |
| Elongation at yield | ASTM D638 | 5 ≤ Elongation ≤ 25 percent |
| Slow crack growth resistance | ASTM F2136 | ≥ 100 hours |
| Izod impact strength | ASTM D256 | ≥ 23.8 ft-lb/in ² |
| Oxidative-Induction Time (OIT) | ASTM D3895 | 25 ≤ OIT ≤ 200 minutes |

2.6 Recycled Materials

One advantage of thermoplastic materials is that they can be recycled and reprocessed. Incorporating these recycled materials into corrugated HDPE pipe offers both economic and environmental benefits. From an economic standpoint, recycled materials can cost less than virgin materials. While the lower cost can be partially offset by reduced manufacturing efficiencies, there can still be an economic benefit to using these materials. There are also environmental advantages in the production of the pipe because the production of the virgin raw materials is one of the largest contributors to the overall environmental impact of the pipe system when performing a cradle to grave analysis (22). Substituting recycled PE for virgin materials in the raw material stream used to manufacture the pipe will reduce the environmental impact associated with production, due to minimization of the raw materials consumed.

Several material standards allow the incorporation of recycled materials into the product formulations. Recycled materials should not be confused with reworked materials. In the AASHTO and ASTM specifications, reworked materials typically refer to those materials that originate from a manufacturer’s standard production pipe that has been scrapped. For example, during the startup of a manufacturing line, the manufacturer will typically produce some pipe that does not meet specifications while they are making adjustments to the line (e.g., die gap changes, line speed modifications, die centering, etc.). The AASHTO and ASTM standards allow manufacturers to grind up and reuse this pipe, and these ground-up materials are referred to as “reworked” materials.

Recycled materials, on the other hand, are those materials that come from products that have previously served another purpose. They can be broadly categorized as post-consumer recycled (PCR) materials and post-industrial recycled (PIR) materials. PCR materials are derived from various consumer products that have served a previous purpose such as laundry detergent containers, milk and soft drink bottles, and other consumer goods. PIR materials have been diverted from the waste stream during a manufacturing process and have never reached the end user. Technically speaking, the reworked materials are a type of PIR material, although the pipe industry generally classifies materials as PIR only if they come from an outside manufacturing process.

2.6.1 Post-Consumer Recycled Materials

PCR materials can be classified as either natural or colored. Natural materials are typically PE homopolymers from milk and water jugs. These materials possess a high strength but poor SCR. Colored PCR materials are derived from liquid detergent and fabric softener containers, shampoo and conditioner bottles, colored soft drink bottles, and other consumer products. They are typically made from HDPE co-polymers that offer improved SCR.

Contamination can be a concern when manufacturing pipe with recycled materials. Contaminants may include dirt, paper and foil from labels, rubbers from gaskets or seals, and other non-HDPE thermoplastic materials such as PP from caps, ethylene vinyl alcohol (EVOH), nylon, polyvinyl chloride (PVC) and polyester (PET). Many of these contaminants are removed in a washing and screening process by the recycled material supplier. Figure 2.23 shows a picture of PCR materials both before and after the washing process. The materials can be further filtered in a reprocessing operation. In this case, the PCR flake materials are input into an extruder where they are melted, filtered by screen packs, extruded, and pelletized. These materials are referred to as reprocessed materials, or repro for short.

The corrugated HDPE industry consumes millions of pounds of PCR materials each year. Most of these materials are utilized in agricultural drainage pipe. However, it is becoming more common practice to incorporate these materials into larger diameter pipes for commercial applications as well. For these applications, the pipes are typically manufactured in accordance with ASTM F2648, *Standard Specification for 2 to 60 inch Annular Corrugated Profile Wall Polyethylene Pipe and Fittings for Land Drainage Applications* (23), which includes a provision for manufacturing pipe with recycled content. Product specifications place limits on the amount and type of contaminants that can be present in the materials when producing pipe made from recycled content.

2.6.2 Post-Industrial Recycled Materials

PIR materials are sourced from scrap materials from industrial processes. These materials have never reached an end user and may include regrind from pipe, sheets, blown film, tubing, and large plastic drums. They typically include a variety of PE materials including LDPE, LLDPE, MDPE, and HDPE. They also vary in molecular weights from injection molding grade materials to thermoforming materials. As with PCR materials, PIR materials should be properly washed and screened prior to incorporating them into a plastic pipe manufacturing process. PIR materials can often be more variable than PCR materials, and proper blending of the materials is essential.

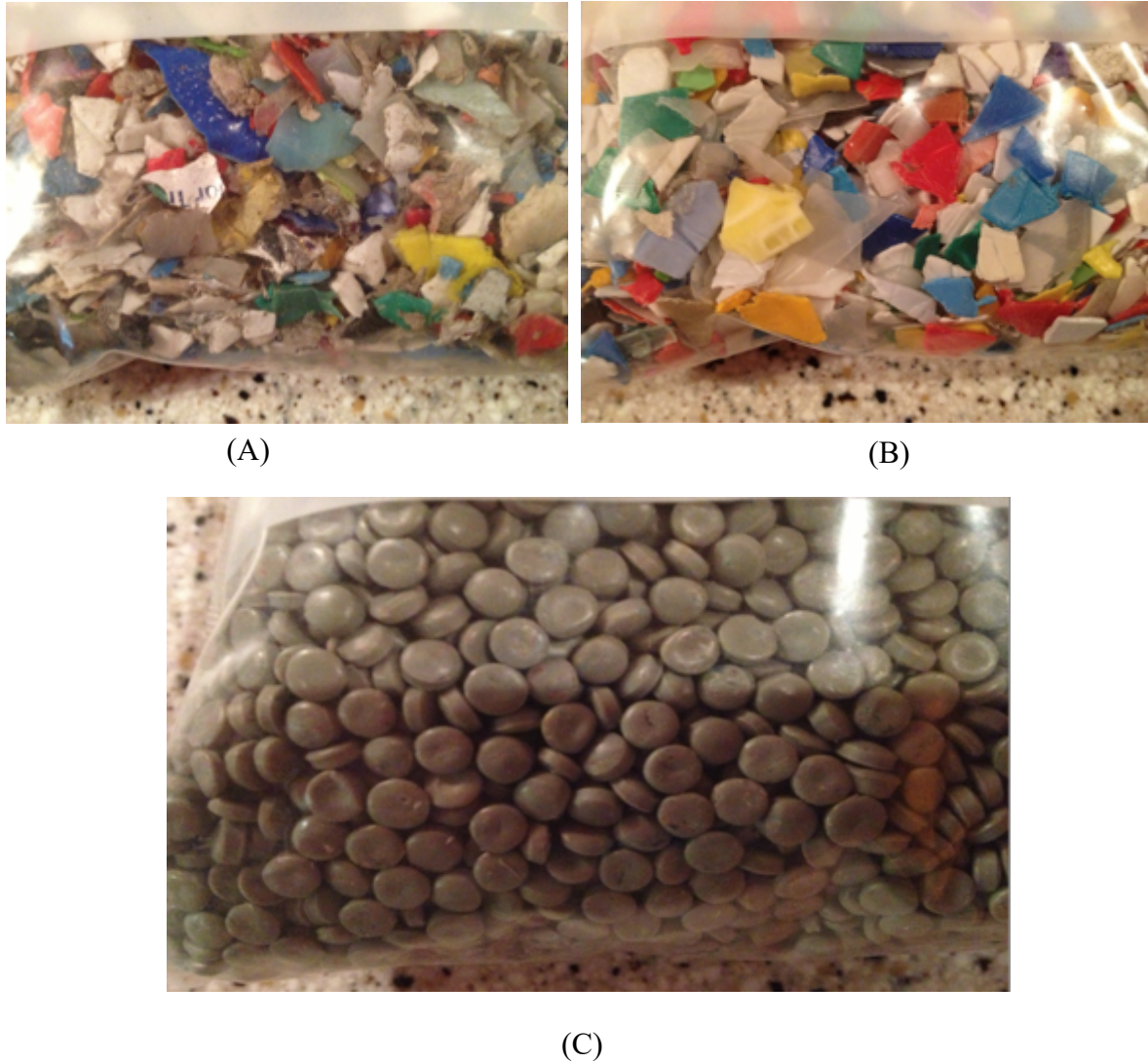


Figure 2.23: PCR materials in unwashed flake (A), washed flake (B) and reprocessed (C)

2.6.3 Resin Blending

It is often necessary to blend PCR and PIR materials with virgin resins in order to meet the end-product material specifications. For example, PCR or PIR materials with lower SCR will need to be blended with a virgin material with higher SCR in order to ensure that the overall blend performs in accordance with the specifications. Likewise, a PCR material may have a MI that is too high of a MI for processing purposes. In this case, the PCR must be blended with a material possessing a lower MI so that the final blend property meets the manufacturer’s specifications and processing requirements. Many pipe manufacturers have sophisticated blending algorithms which allow them to input the various constituent component characteristics and to predict the final finished blend properties such as SCR, impact resistance, density, MI, tensile strength, and flexural modulus.

2.6.4 Research Projects on Recycled Materials

The interest in incorporating recycled materials into corrugated HDPE pipes continues to grow as end users are becoming more cost-conscious and environmentally-conscientious. A number of agencies have funded research projects to evaluate the responsible incorporation of recycled materials into plastic pipe for long-term applications. The National Cooperative Highway Research Program (NCHRP) funded a project to study the performance of corrugated pipe manufactured with recycled content. This research project was completed in 2011 and published in NCHRP Report 696, *Performance of Corrugated Pipe Manufactured with Recycled Polyethylene Content* (24), and concluded that recycled materials could be incorporated into corrugated HDPE pipe for highway drainage applications along with guidance for the material specifications. The research was validated in a follow-up field study, NCHRP Report 870, *Field Study of the Performance of Corrugated HDPE Pipes Manufactured with Recycled Content*, which was completed in 2017 (3). This project included the development of a new test method for pipes manufactured with recycled materials, the Unnotched Constant Ligament Stress (UCLS) test, published as ASTM F3181 (25). It also included the development of a service life prediction model for pipes containing recycled materials, later published as AASHTO R 93 (26), and proposed revisions to incorporate recycled materials into AASHTO M 294 that were incorporated in 2018.

Additionally, a research project at Villanova University evaluated the service life of corrugated HDPE pipes manufactured with recycled materials, relative to fatigue and stress cracking in commuter railroad applications (27). The project involved the installation of the first large-diameter corrugated HDPE pipe manufactured with recycled materials in North America and concluded that there was no statistical difference in fatigue performance between pipes manufactured with virgin resin and pipes containing recycled materials. The research included the development of a service life model for corrugated HDPE pipes manufactured with recycled materials in railroad applications and the establishment of minimum performance criteria that ensure a 100-year service life.

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