ELECTRIC POWER
RESEARCH INSTITUTE

Advanced Nuclear Technology: Material Properties Affecting the Butt Fusion of HDPE Pipe

2014 TECHNICAL REPORT

Advanced Nuclear Technology: Material Properties Affecting the Butt Fusion of HDPE Pipe

3002003133

Final Report, September 2014

EPRI Project Managers
A. Summe
D. Munson

All or a portion of the requirements of the EPRI Nuclear Quality Assurance Program apply to this product.

YES [✓ NO]

ELECTRIC POWER RESEARCH INSTITUTE
3420 Hillview Avenue, Palo Alto, California 94304-1338 ▪ PO Box 10412, Palo Alto, California 94303-0813 ▪ USA
800.313.3774 ▪ 650.855.2121 ▪ askepri@epri.com ▪ www.epri.com

DISCLAIMER OF WARRANTIES AND LIMITATION OF LIABILITIES

THIS DOCUMENT WAS PREPARED BY THE ORGANIZATION(S) NAMED BELOW AS AN ACCOUNT OF WORK SPONSORED OR COSPONSORED BY THE ELECTRIC POWER RESEARCH INSTITUTE, INC. (EPRI). NEITHER EPRI, ANY MEMBER OF EPRI, ANY COSPONSOR, THE ORGANIZATION(S) BELOW, NOR ANY PERSON ACTING ON BEHALF OF ANY OF THEM:

(A) MAKES ANY WARRANTY OR REPRESENTATION WHATSOEVER, EXPRESS OR IMPLIED, (I) WITH RESPECT TO THE USE OF ANY INFORMATION, APPARATUS, METHOD, PROCESS, OR SIMILAR ITEM DISCLOSED IN THIS DOCUMENT, INCLUDING MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE, OR (II) THAT SUCH USE DOES NOT INFRINGE ON OR INTERFERE WITH PRIVATELY OWNED RIGHTS, INCLUDING ANY PARTY'S INTELLECTUAL PROPERTY, OR (III) THAT THIS DOCUMENT IS SUITABLE TO ANY PARTICULAR USER'S CIRCUMSTANCE; OR

(B) ASSUMES RESPONSIBILITY FOR ANY DAMAGES OR OTHER LIABILITY WHATSOEVER (INCLUDING ANY CONSEQUENTIAL DAMAGES, EVEN IF EPRI OR ANY EPRI REPRESENTATIVE HAS BEEN ADVISED OF THE POSSIBILITY OF SUCH DAMAGES) RESULTING FROM YOUR SELECTION OR USE OF THIS DOCUMENT OR ANY INFORMATION, APPARATUS, METHOD, PROCESS, OR SIMILAR ITEM DISCLOSED IN THIS DOCUMENT.

REFERENCE HEREIN TO ANY SPECIFIC COMMERCIAL PRODUCT, PROCESS, OR SERVICE BY ITS TRADE NAME, TRADEMARK, MANUFACTURER, OR OTHERWISE, DOES NOT NECESSARILY CONSTITUTE OR IMPLY ITS ENDORSEMENT, RECOMMENDATION, OR FAVORING BY EPRI.

THE FOLLOWING ORGANIZATION, UNDER CONTRACT TO EPRI, PREPARED THIS REPORT:

Bryan Hauger Consulting, Inc.

THE TECHNICAL CONTENTS OF THIS PRODUCT WERE **NOT** PREPARED IN ACCORDANCE WITH THE EPRI QUALITY PROGRAM MANUAL THAT FULFILLS THE REQUIREMENTS OF 10 CFR 50, APPENDIX B. THIS PRODUCT IS **NOT** SUBJECT TO THE REQUIREMENTS OF 10 CFR PART 21.

NOTE

For further information about EPRI, call the EPRI Customer Assistance Center at 800.313.3774 or e-mail askepri@epri.com.

Electric Power Research Institute, EPRI, and TOGETHER…SHAPING THE FUTURE OF ELECTRICITY are registered service marks of the Electric Power Research Institute, Inc.

Copyright © 2014 Electric Power Research Institute, Inc. All rights reserved.

ACKNOWLEDGMENTS

The following organization, under contract to the Electric Power Research Institute (EPRI), prepared this report:

Bryan Hauger Consulting, Inc.
4129 San Marco Drive
Longmont, Colorado 80503

Principal Investigator
B. Hauger

This report describes research sponsored by EPRI.

This publication is a corporate document that should be cited in the literature in the following manner:

Advanced Nuclear Technology: Material Properties Affecting the Butt Fusion of HDPE Pipe. EPRI, Palo Alto, CA: 2014. 3002003133.

PRODUCT DESCRIPTION

Significant efforts have been expended on several fronts to develop an understanding of butt fusion, which is the most commonly used method to assemble high-density polyethylene (HDPE) pipe. Variables that can affect the quality of the fusion joint are called essential variables. A fusion procedure specification currently needs to be developed and qualified by each organization, then re-qualified whenever an essential variable changes. This poses a very high burden on plant owners and may not be necessary for limited changes to at least some of the essential variables. Several essential variables for butt fusion must be controlled within acceptable ranges to provide reasonable assurance of strong and durable joints. This report provides detailed consideration of HDPE material properties in order to create a deeper understanding of the scientific principles that explain the fusion process.

Background

ASTM F2620 provides detailed procedures for butt fusion. ASME Section IX has issued Articles XXI – XXIV that contain detailed rules for fusing HDPE piping in boiler and pressure vessel applications and qualification of the fusing procedure. In so doing, Section IX has defined essential variables for plastic fusing in Part QF-252 for general applications. Three additional essential variables for nuclear safety-related applications are discussed here.

Objectives

- To explore the interrelationship between physical properties of HDPE with the potential to affect butt fusion (Section 2).
- To identify and discuss the physical properties of HDPE with the potential to affect butt fusion (Sections 2, 3).
- To determine the role of thermal and oxidative stabilizers (antioxidants) and other polymer additives (Section 4).
- To provide an overview of the manufacturing processes for HDPE pipe (Sections 4, 5).
- To explain the industry's use of interfacial pressure as a controlling variable in relationship to cross-sectional area and fusion force (Section 6).
- To review and evaluate the essential variables detailed in nuclear safety-related requirements from a material science perspective (Section 6).

Approach

The objectives of the project were addressed through three broader topics. The first and most significant topic centers on "Material Properties of HDPE With the Potential to Affect Butt Fusion of HDPE Pipes." Principles of chemistry and polymer science were applied to examine potential differences between HDPE polymers for the manner and extent to which these differences may affect butt fusion.

The next topic—"Manufacturing and Butt Fusion of HDPE Pipes"—addresses the industry history of using identified melt index or high load melt index (HLMI) ranges to control melt viscosity in relation to generic butt fusion procedures. Allowable ranges for HLMI and density for nuclear safety-related applications are compared to anticipated batch variability of polyethylene compound.

The final topic—"Engineering Considerations Related to Butt Fusion of HDPE Pipes"—relates previous polymer science considerations to those polyethylene compounds that meet the requirements for nuclear safety-related applications. The polyethylene compounds that currently meet nuclear safety-related applications are quite similar in HLMI and density, which acts to restrict important melting, crystallization, and viscosity properties within limited ranges. Several of the essential variables are then considered from a polymer science perspective, with many instances of scientific support being found for the essential variable ranges and trends.

Results and Findings

- The HDPE pipe manufacturing process recombines various melt strands in a strong parallel to butt fusion; thus, the same material properties control both processes (Section 4).
- The level of proprietary antioxidants included in HDPE compounds is sufficiently low that it should not affect butt fusion (Section 4).
- Review of publically available product literature indicates eight HDPE compounds have highly similar properties well within allowable limits and have the potential to comply with nuclear safety-related requirements (Section 6).
- Mathematical analysis of the HDPE pipe cross-sectional area suggests that variability in cross-sectional area is similar to the variation allowed by the essential variable pipe surface alignment (Section 6).
- Overall, the essential variables combined with the high similarity of the eight HDPE compounds provide a technical basis for limiting the number of pipe sizes, lots, manufacturing facilities, and resin suppliers used in qualification of the Fusing Procedure Specification (Section 6).

Applications, Value and Use

This report examines the testing and properties of HDPE pipes complying with nuclear safety-related application requirements for their relevance to nuclear safety-related essential variables of butt fusion. That detailed examination then forms the technical basis for conclusions and recommendations that address nuclear safety-related essential variables and their relationship to fusing procedure specifications (FPS).

Keywords

High-density polyethylene (HDPE) pipe
Butt fusion
Polyethylene fusing procedure qualification

ABSTRACT

In an effort to control the quality of butt fusion, variables which can affect the quality of the fusion joint have been identified and are called essential variables. It is acknowledged that there are several essential variables for butt fusion which must be controlled within acceptable ranges to have reasonable assurance of strong and durable joints. This report provides a detailed consideration of material properties of HDPE in order to create a deeper understanding of the scientific principles that inform butt fusion to benefit all nuclear safety-related application stakeholders.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

1

INTRODUCTION

1.1 Background

There are several essential variables for butt fusion which must be controlled in order to make strong and durable high-density polyethylene (HDPE) pipe joints through the process of butt fusion. ASME Section IX has issued Articles XXI – XXIV that provide for a standard fusion procedure specification (SFPS) for use in general application. While the currently balloted Section III Appendix permits the use of an SPFS for Class 3 Buried Polyethylene Piping, it currently places significant additional requirements on the use of the SPFS. These requirements include additional ambient temperature controls and testing to validate the fusion process using the actual fusing machine models and the actual material combinations to be fused in production. ASME Section IX for butt fusion of HDPE has defined ten essential variables for general applications. Three additional essential variables have been identified for Class 3 applications. These essential variables have grown out of the need to ensure that all proper procedures have been followed during fusion joint manufacturing. An increased focus on the material properties of the polyethylene compound and HDPE pipe may improve understanding through a closer link to the scientific principles upon which the joining process is founded. By more fully incorporating concepts from polymer science, it is anticipated that a deeper understanding will provide a technical basis for allowing greater latitude in fusion joining process changes without requalification and appropriate reductions in requalification testing where necessary.

Throughout this document, the term “Polyethylene Material – Pipe” used throughout ASME Code Case N-755-2 [1] has been called “HDPE pipe”.

1.2 Overall Project Description

This report considers the joining of HDPE pipe by introducing related concepts from the point of view of polymer and material science with the intent of creating a new and deeper understanding. Three related topics are addressed separately and include substantial references to public domain data and reports where possible in order to ensure that each topic is fully examined and well-supported by open literature.

1.2.1 Material Properties of Polyethylene Compound with the Potential to Affect Butt Fusion of HDPE Pipes

This topic starts in Section 1 by paying careful attention to the details of testing polyethylene compound properties. Section 2 focuses on polyethylene compound property requirements for nuclear safety-related applications with an emphasis placed on Certification Requirements for Polyethylene Compound. Key HDPE pipe requirements for nuclear safety-related applications are briefly considered in Section 3.

Section 4 begins by considering polyethylene as a molecule including concepts such as degree of polymerization and the incorporation of limited amounts of other olefins into polyethylene as short-chain branches. The relationship between Resin Density and the amount of other olefins incorporated is noted. Gel permeation chromatography is noted for its contribution to understanding the distribution of polyethylene compound molecular weight and the concept of poly-dispersity is introduced.

The innovation of bi-modal polyethylene compound highlights the importance of selective incorporation of an abundance of short-chain branches into the higher molecular weight portion of the total molecular weight distribution. Polyethylene molecules which bridge adjacent crystal lamella within a solid polyethylene matrix, so-called tie molecules, are noted for their importance in slowing the progression of slow crack growth. Factors with the potential to impact slow crack growth resistance such as molecular orientation and thermal history are noted.

Carbon black is then differentiated from “foreign particles” through thoughtful carbon black technology choices and skillful extrusion practices. Carbon black technologies include the industrial manufacturing process, primary particle sizes, and other properties of carbon black. The polyethylene resin that comprises the balance of the pigment concentrate compound, the carrier resin, is noted as an inconsequential contributor to bulk mechanical properties of the polyethylene compound.

Prior to considering the melt processing of polyethylene compound, long-chain branching of polyethylene is differentiated from highly cross-linked or “gelled” polymer. The importance of additive chemistry for both avoiding damage during melt processing and maintaining properties over the long-term is noted. The general trend of increasing temperature resulting in reduced viscosity applies to molten polyethylene compound as to other non-Newtonian fluids. The process of crystallization of polyethylene is considered along with the thermal properties of polyethylene including melting point, thermal conductivity, heat of fusion and the heat capacity. Key polymer properties relating to micro-mechanical theories of the fusion process are considered.

Similarities between HDPE pipe manufacturing and the butt fusion joining of HDPE pipes is touched upon. Section 4 concludes by considering the importance of extrusion screw design to obtaining adequate distribution of carbon black concentrates into a homogeneous HDPE pipe.

1.2.2 Manufacturing and Butt Fusion of HDPE Pipes

Section 5 considers the extent to which industry controls either melt index or HLMI to alter the viscosity of molten polyethylene compound in order to fit the requirements of widely varying manufacturing processes. The ranges for HLMI and density currently required for use in nuclear safety-related applications are noted as significantly larger than anticipated batch variability of polyethylene compound. Industry history is cited regarding the practice of using identified melt index (or HLMI) ranges to control melt viscosity in relation to generic butt fusion procedures.

1.2.3 Engineering Considerations related to Butt Fusion of HDPE Pipes

Section 6 focusses polymer science considerations onto those polyethylene compounds that meet nuclear safety-related requirements. Review of publically available product literature indicates a handful of potentially compliant HDPE compounds are available. Resin Density and HLMI reported for these potentially compliant HDPE compounds fall within a narrow range. Several of the essential variables are then considered from a polymer science perspective with many instances of scientific support being found for the essential variable ranges and trends. Section 6 concludes with a more detailed consideration of the essential variables for ambient temperature and cross-sectional area. Finally, conclusions and recommendations are summarized.

2

POLYETHYLENE COMPOUND PROPERTIES

2.1 Relevant Polyethylene Compound Properties

Polyethylene compound properties are referred to throughout the document. In order to ensure clarity, material properties of polyethylene compound encompassed by this work are detailed here. The polyethylene compounds under consideration are those identified as meeting the requirements for nuclear safety-related applications. The required polyethylene compound properties are included in Table 2-1 (provided below) for reference purposes.

Although a more recent version of ASTM D3350 is available, nuclear safety-related requirements are currently written specific to the 2010 year of issue and, unless noted, references in this document will be specific to that version of ASTM D3350 [2]. Similarly, other ASTM standards referenced in this document are specific to the year of issue recognized in ASME Code Case N-755-2 unless noted.

2.2 Density

Three essentially equivalent test methods are allowed for the measurement of density in relation to nuclear safety-related applications, although all are accepted industry-wide. ASTM D3350 directs that the test specimens used for these density measurements be molded in accordance with Procedure C of Annex A1 of ASTM D4703 [3] unless otherwise instructed. An explanation of the three density test methods allowed by D3350 follows.

ASTM D1505 [4] uses a column of fluid prepared by combining two liquids in a varying ratio such that the concentration of the more dense fluid is greatest at the bottom of the column and least at the top of the column. A continuous gradient of density versus depth in the column is thereby established. Floats of known and calibrated density are then used to establish this relationship in the column.

ASTM D792 [5] directs that a specimen of plastic is first weighed in air and then immersed in a liquid, commonly water. Then its apparent mass in water is determined and its specific gravity (relative density) is calculated.

ASTM D4883 [6] utilizes a known relationship between the velocity of sound through polyethylene compound and the density of the polyethylene compound. The density of a set of calibration materials is first determined by either ASTM D792 or ASTM D1505 and used to establish a calibration curve between density and sonic velocity. This calibration is then used for materials of unknown density by measuring their sonic velocity and subsequently calculating their density. ASTM D4883 has the added advantage that “inorganic materials increase density as measured by Test Methods ASTM D792 and ASTM D1505, but they have little or no effect on ultrasonic density. The ultrasonic measurement is basically a base resin density”.

Table 2-1 contains two properties that can be referred to as density. The first term will be referred to throughout this document as Resin Density which is the term used in ASTM D3350. As related in the ASTM D3350 standard, the Resin Density can be measured directly through a variety of test methods or it can be calculated. The calculation uses the formulae in Equation 2-1, (from section 6.5.1 of ASTM D3350) relating the Resin Density D_r to the density of the black polyethylene compound D_p by reducing the compound density in proportion to the concentration of carbon black in weight per cent, C.

$$D_r = D_p - (0.0044 \times C) \qquad \textbf{Eq. 2-1}$$

The black polyethylene compound density also has a required range. Although this is not a commonly specified property in ASTM standards, it can be easily understood. One can utilize Equation 2-1 to calculate the resulting product density from the minimum Resin Density required (0.9475 g/cm^3 rounded to 0.948 g/cm^3 by ASTM D3350) and the minimum carbon black content of 2.0 weight per cent. The value calculated for the minimum polyethylene compound density using these values is 0.9563 g/cm^3 and rounded to 0.956 g/cm^3 as the minimum in Appendix IV.

Similarly, the maximum Resin Density of 0.955 g/cm^3 coupled with a maximum of 2.95 weight per cent carbon black to obtain a the maximum product density of 0.968 g/cm^3. This polyethylene compound density requirement anticipates a change that was made to ASTM D3350 in April 2012 to add a section 6.2.2 which states “For PE compounds with a hydrostatic strength classification other than cell class 0, the carbon black content shall be in the range of 2.0% to 3.0%”.

2.3 Melt Index (MI)

Table 2-1 contains two properties that are both measured using test method ASTM D1238. ASTM D1238 [7] describes two options for the measurement of the amount of molten polyethylene compound passing through the orifice of a die at 190°C within a period of time which are nearly identical with the exception of the mass placed on top of the piston. The applied mass supplies the force which drives the molten polymer through the orifice by overcoming the resistance to flow of the viscous molten polymer. As the mass increases, the amount of polymer extruded through the die in a given period of time increases in a non-linear fashion as a function of the complex viscosity of the molten polymer. Throughout this report, the use of the 2.16 Kg mass at 190°C will be referred to as Melt Index (MI) while the use of the 21.6 Kg mass at 190°C will be referred to High Load Melt Index (HLMI).

When Melt Index is measured, the required material property test result value is less than 0.15 g/10 minutes. For some polyethylene compounds, the Melt Index is less than 0.05 g/10 minutes which presents difficulties in ensuring a statistically valid measurement. For this reason, some resin manufacturers do not find the use of Melt Index useful as a quality control method, but are nonetheless able to indicate that the value obtained upon testing is less than 0.15 g/10 minutes. Therefore, either the Melt Index requirement can be met with a value less than 0.15 g/10 minutes or the HLMI requirement can be met by a value between 4.0 and 20.0 g/10 minutes.

This HLMI requirement anticipates a change that was made to ASTM D3350 in April 2012 to add a section 10.1.4.1 which states “For materials having a melt index less than 0.15 (Cell 4), the manufacturer shall report a flow rate not greater than 20 g/10 minutes and not less than 4.0 g/10 minutes when tested in accordance with ASTM D1238-10, Condition 190/21.6”. Including both MI and HLMI requirements in the ASME code provides direct measurement capability to ensure compliance with the standard over the full range of PE 4710 materials in the marketplace today.

2.4 Flexural Modulus

ASTM D3350 directs that the test specimens used for flexural modulus measurements shall be molded in accordance with Procedure C of Annex A1 of ASTM D4703. ASTM D790 [8] directs the measurement of flexural modulus using a three-point loading system applied to a supported thin beam of polyethylene compound. Using Method 1, Procedure B, and a 50-mm (2-in.) test span, five replicate specimens, each 3.2 by 12.7 mm (1⁄8 by 1⁄2 in.) are tested at a crosshead travel speed of 12.7 mm/min (0.5 in./min) and the average value of the secant modulus calculated at 2% strain in the outer fibers.

Semi-crystalline polyethylene contains both high modulus crystalline regions and low modulus amorphous regions. The relationship between Resin Density and flexural modulus has been recognized for some time. Materials with a higher Resin Density are anticipated to be stiffer materials with a higher flexural modulus than lower Resin Density materials. In fact, the quantitative relationship between modulus and Resin Density has been documented [9] over the density range from approximately 0.925 and 0.970 g/cm^3. Using the allowed Resin Density range of 0.947 to 0.955 g/cm^3 and inspecting Figure 2 of Janzen and Register 1996 provides an estimate of the range of the flexural modulus of approximately 1.1 to 1.4 GPa or approximately 160,000 to 200,000 psi.

2.5 Tensile Strength at Yield

ASTM D3350 directs that the test specimens used for tensile strength at yield measurements shall be molded in accordance with Procedure C of Annex A1 of ASTM D4703 at a thickness of 1.9 ± 0.2 mm (0.075 ± 0.008 inches). Self-aligning grips are attached to tabs at the ends of a Type IV tensile bar with dimensions defined in ASTM D638 [10]. A constant rate of cross-head movement is then applied while the load imparted to one of the grips is continuously recorded. Simultaneously, an extensometer measures the strain between two locations in a reduced section, called the gage, which localizes the highest tensile stresses in the specimen away from the points of attachment. Replicate measurements are made and averaged as directed in Test Method D638 for the tensile strength at yield and elongation at break. The speed of testing is 50 mm/minute (2 inches /minute) for polyethylene compound.

Similar to flexural modulus, polyethylene compounds with a higher Resin Density are anticipated to be stronger materials with a higher tensile strength at yield than lower Resin Density materials. In fact, the quantitative relationship between modulus and Resin Density has been documented over the density range from approximately 0.925 and 0.970 g/cm^3. Using the allowed Resin Density range of 0.947 to 0.955 g/cm^3 and inspecting Figure 5 of Janzen and Register 1996 provides an estimate of the range of the tensile strength at yield of approximately 23 to 27 MPa or approximately 3,300 to 3,900 psi.

2.6 PENT

As indicated in ASTM F1473 [11], the PENT or the **Pe**nnsylvania **N**otch **T**est, captures the capability of a razor-notched sample of polyethylene compound to resist slow-crack growth.

Specimens are prepared from compression molded plaques using modified ASTM D4703 procedures, notched using precise notching techniques, and then exposed to a constant tensile stress at elevated temperatures in air. Time on test is recorded, either to rupture or to demonstrate a minimum time on test without rupture. ASTM D3350 indicates that the temperature used is 80°C and the stress on the remaining ligament is 2.4 MPa.

The minimum required value for a polyethylene compound to qualify for the cell classification of 7 for PENT testing at 80°C and 2.4 MPa is 500 hours. Nuclear safety-related requirements establish a minimum of 2000 hours and clarifies that this testing must be conducted in a lot specific test.

2.7 Hydrostatic Design Basis (HDB)

In order to understand HDB, it is appropriate to first discuss long-term hydrostatic strength (LTHS) since the two concepts are so closely related. The LTHS is obtained through analysis of initial circumferential (hoop) stress versus time-to-rupture test data (that is, stress-rupture data) over a testing duration that exceeds 10,000 hours. The initial circumferential stress is obtained through calculation while the time-to-rupture is directly measured via sustained hydrostatic pressure testing of pipe. ASTM D1598 [12] codifies the procedure for conducting time dependent hydrostatic sustained pressure testing. The data thus obtained are converted to log initial circumferential stress and log time-to-failure and then analyzed by linear regression to yield a best-fit log-stress versus log time-to-fail linear equation. This equation is then extrapolated to 100,000 hours and the corresponding hoop stress value is defined as the LTHS. ASTM D2837 [13] establishes the procedure for obtaining the LTHS. The resultant LTHS is categorized per the defined ranges in D2837 to obtain the HDB for the material. There are several premises associated with ASTM D2837 but it is of primary importance to understand the result as the HDB of the polyethylene compound, i.e. the long term stress capacity of the material, rather than as a pressure rating of HDPE pipe. Fundamentally, industry applies the HDB to a range of HDPE pipe sizes through calculations of the initial circumferential stress.

Table 2-1 contains two HDB values of interest. The required HDB at 73°F is 1600 psi which is simultaneously an ASTM D3350 requirement of all PE4710 materials. Nuclear safety-related requirements also establish the minimum HDB at 140°F to be 1000 psi which is not an ASTM D3350 PE4710 material requirement but is often obtained for HDPE materials complying with all of the other requirements in Table 2-1. Nuclear safety-related requirements go further and require that the HDB at both 73°F and 140°F be listed in PPI TR-4 [14] in accordance with PPI TR-3 [15] policies. This TR-4 listing requirement ensures several other requirements from TR-3 have been met, notably that the HDB has been measured multiple times.

2.8 Hydrostatic Design Stress (HDS) at 73°F

It should first be noted that HDS is not a material property of polyethylene compound. It is simply calculated from HDB data and a Design Factor. PPI TR-3 states that “the HDS is determined by reducing the HDB by a design factor (DF), a multiplier less than 1.0”. PPI TR-9 [16] indicates that a design factor encompasses two groups of variables. The first group is fairly well defined to include both manufacturing and testing variation (such as variations in the material, manufacture, dimensions, and evaluation procedures). The second group considers a broad variety of potential variables such as installation, temperature, transported fluid, life expectancy desired, hazard involved, and the degree of reliability selected.

Traditionally, a design factor of 0.5 was widely applied to HDB resulting in HDS. More recently, a 1000 psi HDS recommendation at 73°F was made for some polyethylene materials listed with a 1600 psi HDB at 73°F that meet additional higher requirements than the minimum necessary for PPI TR-4 listing. This PPI recommended HDS for such PE4710 materials has been utilized and adopted into several consensus industry HDPE pipe standards such as ASTM D3035 [17], ASTM F714 [18], and AWWA C901[19]. Nuclear safety-related applications also require a HDS minimum of 1000 psi ensuring that only PE4710 polyethylene compounds are accepted.

2.9 Carbon Black

Two test methods are recognized as nuclear safety-related requirements for the measurement of carbon black; ASTM D4218 [20] and ASTM D1603 [21]. In ASTM D4218, a small quantity (typically around 1 gram) of black polyethylene compound is rapidly pyrolyzed in a muffle furnace under temperature control and oxygen-deficient conditions to prevent the combustion of the carbon black. ASTM D1603 is written more broadly for all olefinic materials but is specifically applicable to black polyethylene compound. In ASTM D1603, initial high temperature exposure of the polyethylene compound sample is conducted under nitrogen. ASTM D4218 and ASTM D1603 are recognized as equivalent by ASTM D3350.

These test methods utilize pyrolysis to provide a residual weight and record that value as carbon black content. However, other incombustible materials present in the polyethylene will also be recorded as carbon black. This could include minor contributions from catalyst residues and incombustible components of stabilizers or other additives, commonly referred to as ash. Potential contributions to the calculated carbon black content from ash are not directly addressed in current standards but are commonly assumed to be less than 0.1 weight per cent.

Industry's use of carbon black to protect HDPE pipes is supported by studies conducted by Gilroy [22,23] of Bell Labs. The studies at Bell Labs indicated that permanent outdoor installations of polyethylene compound will be adequately protected against the potentially damaging effects of UV light so long as the carbon black primary particle size is sufficiently small, the dispersion of the carbon black into its primary particles is accomplished, and the amount used is at least 2 weight per cent.

2.10 Elongation at Break

Although no cell classification value is attributed to the elongation at break for polyethylene compound, ASTM D3350 includes the requirement that a determination of the extent of elongation prior to break be obtained through ASTM D638 tensile properties testing. The minimum value of 400 per cent should not be encountered in normal operations since design methods keep the extent of elongation of HDPE pipe well below the yield elongation (typically around 10 per cent). The minimum value of 400% provides adequate assurance that conditions which might limit the ductility of polyethylene compound (e.g. low degree of polymerization, contamination with macroscopic particles and material degradation) are not present to a significant extent.

2.11 Brittleness Temperature

A number of replicate specimens are placed in a specimen holder and then cooled to the desired temperature. The specimens are struck at a specified linear speed and then examined. The brittleness temperature is defined in ASTM D746 [24] as the temperature at which 50 per cent of these specimens fail. Testing is normally conducted to demonstrate conformance rather than to measure the actual brittleness temperature by taking polyethylene compound to a temperature below -60°C and then determining if less than 50 per cent of the samples failed.

Although no cell classification value is attributed to the brittleness temperature for polyethylene compound, ASTM D3350 includes this requirement to determine the brittleness temperature through impact testing. The requirement that polyethylene compound has a brittleness temperature below -60°C provides reasonable assurance that HDPE pipe will not fail in a brittle manner due to impact in typical water supply applications.

2.12 Thermal Stability

Polyethylene compound is formulated with a variety of additives to ensure the melt stabilization and long-term durability of the polymer (see Section 4.8). The testing conducted to determine the Thermal Stability Temperature is detailed in section 10.1.9 of ASTM D3350. Thin films of polyethylene compound are placed into a differential scanning calorimeter (DSC) and heated starting from 150°C (above the melting point) at a rate of 10°C / minute while exposed to static air. Under these conditions, a steep rise in the slope of the line described by a plot of heat absorption rate (Joules per second) versus temperature of the sample indicates a reaction between the polyethylene compound and the oxygen in the air. Individual values of Thermal Stability Temperature are obtained through analysis of this plot. A minimum of three individual values are averaged to provide the final Thermal Stability Temperature.

Thermal Stability Temperature is not directly applicable to calculate antioxidant content or make lifetime durability predictions for the material. However, the Thermal Stability Temperature is a required property because it is indicative of the extent or degree of stabilization of the polyethylene compound. The standard requires polyethylene compound to demonstrate a Thermal Stability Temperature in excess of 220°C. Thermal Stability Temperature has relevance to the capability of the material to undergo successful butt fusion of HDPE pipes since the polyethylene compound must be sufficiently stable, once molten, to form a strong and durable heat fusion bond. Requirements for Thermal Stability Temperatures are also found in some industry consensus standards (e.g. AWWA C906 [25], ASTM D2513 [26]) for this reason.

A different test method that is also commonly employed to confirm the presence of adequate antioxidant stabilization is ASTM D3895 [27]. In parallel to Thermal Stability Temperature, thin films of polyethylene compound are placed into a differential scanning calorimeter (DSC) and heated to above the melting point at a constant rate. In contrast to Thermal Stability Temperature, heating is initially conducted in a flowing nitrogen atmosphere rather than in static air. At a specified temperature, the gas flow is changed to pure oxygen while the sample is held at a constant temperature. A steep rise in the evolved heat of the sample indicates a reaction between the material and the oxygen. The amount of elapsed time between the start of oxygen flow and the point at which the sample begins to evolve heat is called the induction time and is reported. Duplicate measurement is required.

Table 2-1
Relevant Polyethylene Compound Properties

Properties	Required Value	ASTM D3350 Cell Class	Location of Requirement
Resin Density	0.947 to 0.955 cm^3	4	ASTM D3350 and Code Case N-755-2 Table IV-121
Density (w/carbon black)	0.956 to 0.968 cm^3	N/A	Code Case N-755-2 Table IV-121
Melt Index (2.16 Kg/190C)	Less than 0.15g / 10min	4	ASTM D3350
HLMI (21.6 Kg/190C)	4 to 20 g/10min	N/A	Code Case N-755-2 Table IV-121
Flexural Modulus	110,000 to < 160,000 psi	5	ASTM D3350
Tensile Strength at Yield	3500 psi to < 4000 psi	5	Code Case N-755-2 Table IV-121
Slow Crack Growth Resistance by PENT	> 2000 hours	7	Code Case N-755-2 Table IV-121
HDB for water @73°F (23°C)	1600 psi	4	Code Case N-755-2 Table IV-121
HDB for water @140°F (60°C)	1000 psi	N/A	Code Case N-755-2 Table IV-121
HDS for water @73°F (23°C)	1000 psi	N/A	Code Case N-755-2 Table IV-121
Carbon Black (Color and UV Stabilizer)	Minimum of 2.0 wt% Carbon Black	C	Code Case N-755-2 Table IV-121
Elongation at Break	> 400 %	N/A	ASTM D3350, Section 6.7
Brittleness Temperature	Less than -60°C	N/A	ASTM D3350, Section 6.4
Thermal Stability	> 220°C	N/A	Code Case N-755-2 Table IV-121

3

KEY HDPE PIPE REQUIREMENTS

3.1 Key HDPE Pipe Requirements

HDPE pipe properties are referred to throughout the document with a focus on those HDPE pipe properties summarized in Table 3-1. Such HDPE pipes have been previously established as constructed from materials listed in PPI TR-4 through compliance with policies and procedures in PPI TR-3. Unless stated otherwise, the HDPE pipe product properties in this document will be limited to the explicit requirements in ASTM F714-08 because this version of ASTM F714 is referenced standard in ASME Code Case N-755-2. The measurement of carbon black as a pipe material property is the only property which has direct overlap with polyethylene compound properties. Minimum testing frequencies in Table 3-1 are provided for clarity as they are established for nuclear safety-related applications.

Table 3-1
Key HDPE Pipe Requirements

Property	Manufacturing Requirement	Test Method	Test Frequency
Workmanship	ASTM F714	N/A	Hourly during ongoing production or once per length, whichever is less frequent
Outside Diameter	ASTM F714	D2122	Hourly during ongoing production or once per length, whichever is less frequent
Wall thickness	ASTM F714	D2122	Hourly during ongoing production or once per length, whichever is less frequent
Short-term Strength	ASTM F714	D1599 or D2290	At the start of production and weekly thereafter during ongoing production
Carbon black content	ASTM F714	D1603 or D4218	At the start of production and weekly thereafter during ongoing production

3.2 Workmanship

The workmanship requirement listed in Section 5.1 of ASTM F714 is, in essence, a visual inspection requirement. Although manufacturers are free to use other techniques to establish acceptable workmanship, they are not mandated by the standard. The standard requires that the pipe shall be determined to be homogeneous throughout and uniform in “color, opacity, density and other properties”.

3.3 Outside Diameter (OD)

Pipe dimensions are obviously important in standards for pipe manufacturing. ASTM F714 provides for an abundance of potential sizes. The standard contains three explicit dimensioning systems applied to OD. Additionally, ASTM F714 allows for other OD sizes through section 5.2.5 which states, in part, “other sizes or dimension ratios, or both, shall be acceptable for engineered applications when mutually agreed upon by the customer and the manufacturer”. For the sake of simplicity, the IPS sizes will be referred to throughout the document.

For HDPE pipes, ASTM F714 mandates equipment, procedures and conditions for OD dimensioning. Through sub-reference to ASTM D2122 [28], ASTM F714 mandates that OD is determined using either circumferential or vernier wrap tape placed flat against the pipe surface, around the pipe perpendicular to the pipe axis. ASTM F714 further clarifies that this measurement should be made at standard temperature without regard for humidity and at a location on the pipe no closer to the cut end of the pipe “than 1.5 pipe diameters or 11.8 in. (300 mm), whichever distance is less” to avoid any distortions in the OD measurement caused by toe-in. The relationship between OD, wall thickness, cross-sectional area and interfacial pressure is discussed in detail in section 6.6.

3.4 Wall thickness

For HDPE pipes, ASTM F714 mandates equipment, procedures and conditions for wall thickness dimensioning. Through sub-reference to ASTM D2122, ASTM F714 mandates that wall thickness is determined using either a cylindrical or ball anvil micrometer accurate to ± 0.001 inches at a minimum of eight locations around the pipe circumference. ASTM D2122 cautions against both excessive closure pressure (which may give low measurements) and misalignment of the micrometer to include wall curvature (which may give high measurements). ASTM F714 further clarifies that this measurement should be made at standard temperature without regard for humidity. The relationship between OD, wall thickness, cross-sectional area and interfacial pressure is discussed in detail in section 6.6.

3.5 Short-term Strength

3.5.1 Short-term Strength by Quick Burst

Performing a quick burst test on HDPE pipe to the point of rupture is a destructive test and therefore can not be mandated as a direct quality control measure on each length of pipe. ASTM F714 clarifies this point in non-mandatory appendix section X4.3 which indicates that pipe manufacturers are to ensure that the pipe product requirements, including short-term strength by quick burst, will be met when tested. ASTM F714 instructs that testing to rupture is applicable to nominal OD sizes up to 12 inch.

For HDPE pipes, ASTM F714 mandates equipment, procedures and temperature for short-term strength by quick burst determination through sub-reference to ASTM D1599 [29]. Equipment for ASTM D1599 testing includes a pressurization system, an appropriately located pressure gage of mandated precision with sufficient pressure range, a timing device and specimen end closures. Constant temperature is required with either air or water accepted as the fluid for provision of constant temperature. Prior to pressurization, average OD and minimum wall thickness are measured following the requirements of ASTM D2122.

Two procedures are allowed by ASTM D1599. Procedure A requires sample rupture while Procedure B does not. ASTM F714 anticipates the pressurization of the specimen to rupture between 60 and 70 seconds for HDPE pipes with nominal OD less than 12 inches. The mode of failure is required to be ductile. The pressure of the sample at the point of rupture is determined. Calculation is conducted using Equation 3-1 in which S is the hoop stress, D indicates the average OD, and t is the wall thickness. For HDPE pipes, ASTM F714 requires a minimum calculated hoop stress of 2900 psi.

$$S = P(D-t)/2t \qquad \text{Eq. 3-1}$$

3.5.2 Short-term Strength by Apparent Tensile Strength

Performing an apparent tensile strength test on an HDPE pipe to the point of yield or rupture is a destructive test. However, the length of pipe utilized is relatively small. Nuclear safety-related requirements establish the minimum frequency of testing as equivalent to quick burst testing for quality control purposes. ASTM F714 instructs that apparent tensile testing is applicable to all nominal OD sizes 3 inches and larger, which encompasses all sizes in ASTM F714. For HDPE pipes, ASTM F714 mandates equipment, specimen details, procedures and conditioning for short-term strength by apparent tensile strength determination through sub-reference to ASTM D2290. Equipment for ASTM D2290 testing includes a testing frame capable of supporting the constant rate of cross-head movement, a drive mechanism for the cross-head movement and a load cell.

Three procedures are applicable to HDPE pipes in ASTM D2290. Procedures B and C vary in some minor details regarding the sample dimensions but are otherwise very similar. In both procedures B and C, test specimens are full-diameter, full thickness rings cut from the pipe that are then reduced in length in two locations separated by 180° about the circumference of the specimen. The area of the reduced section is dimensioned for later use in calculations. The equipment for procedures B and C are two half-circumference split-disk rings sized with the disk OD closely matching the inside diameter of the ring cut from the HDPE pipe. The reduced sections of HDPE pipe are located across the split location between the two half-circumference rings and are placed in tension by the crosshead movement. It is required to provide 50 ± 10% relative humidity conditioning for at least 24 hours. A minimum of 24 hours of conditioning at 73.4 ± 3.6°F (23 ± 2°C) is required for all procedures. Cross-head movement applies equal tensile force to the two HDPE pipe ring segments at 73.4 ± 3.6°F (23 ± 2°C) and at a rate of cross-head movement of 0.5 inches per minute. Recorded values from testing include the yield point, maximum test load and crosshead separation for rupture.

The equipment used for procedure D is different than that used for procedures B and C. In procedure D, two identical rectangular, metallic (half) fixtures are supported on self-aligning central pulls to create one larger assembly. Two HDPE pipe ring segments are mounted across the two halves of the test fixture and are attached to them by pins which secure the HDPE ring segments to the test fixtures through holes machined through the ring segments. Procedure D also uses a different specimen than procedures B and C and is often used for testing HDPE pipes with OD greater than 12 inches. Test specimens start as full rings but are divided into ring segments prior to testing. Reduced areas are cut into the ring segments and dimensioned for later use in calculations. Cross-head movement applies equal tensile force to the two HDPE pipe ring segments. Procedure D has no humidity conditioning requirement. A minimum of 24 hours of conditioning at 73.4 ± 3.6°F (23 ± 2°C) is required for all procedures. Cross-head movement applies equal tensile force to the two HDPE pipe ring segments at 73.4 ± 3.6°F (23 ± 2°C) and at a rate of cross-head movement of 0.5 inches per minute. Recorded values from testing include the yield point, maximum test load and crosshead separation for rupture.

Calculation of the apparent tensile strength at yield, σ_a, is obtained using equation 3-2 in which P_b is the maximum load in pound force, d_1 and d_2 are the thicknesses measured at the two reduced sections in inches and b_1 and b_2 are the widths measured at the two reduced sections in inches. The apparent tensile strength at yield is reported to three significant figures.

$$\sigma_a = \frac{P_b}{(d_1 b_1 + d_2 b_2)} \qquad \textbf{Eq. 3-2}$$

When replicate testing is conducted, the individual replicates measurements are averaged. The minimum strength required is 2900 psi for HDPE pipes.

4

MATERIAL PROPERTIES OF POLYETHYLENE COMPOUND WITH THE POTENTIAL TO AFFECT BUTT FUSION OF HDPE PIPES

4.1 Introduction

Considering polyethylene as a molecule leads quickly to the concepts of Resin Density and melt index for a number of reasons. Significantly, many properties of solid, semi-crystalline polyethylene compound can be understood through the Resin Density while molten polyethylene compound can be understood through melt index or HLMI. The relationship between melt index and molecular weight is followed by introduction of the topic of long-chain branching. At this point, highly cross-linked or so-called "gelled" polyethylene is considered because of concerns regarding heat fusion of gelled polyethylene compound. The discovery of bimodal polyethylene compound with a dramatic improvement in ESC at a given density will be discussed and differentiated from unimodal polyethylene compound. Thermal and oxidative stabilizers (anti-oxidants) are discussed in the context of melt processing, viscosity and melt temperature. Like many other viscous liquids, polyethylene compound viscosity is a function of temperature with viscosity decreasing as temperature rises. The nature of both the carbon black particle as well as the resin used in the manufacture of the pigment concentrate compound – often called the carrier resin – is examined. Finally, the crystallization of polyethylene and its role in butt fusion will be considered from the polymer science point of view. Commonly measured polymer properties used in industry standards will be referenced throughout with a special emphasis on polyethylene compound properties contained in Table 2-1.

A more detailed understanding of polyethylene compound as a material starts with considering polyethylene compound as a molecule. As the name polyethylene implies, the manufacturing of the material starts with the gas ethylene, an unsaturated olefin containing a double bond between its two carbons. Through the use of various catalysts, the activation energy to polymerization is overcome in a spontaneous and exothermic (or heat-releasing) reaction. In the process of polymerization, the relatively reactive carbon to carbon double-bond is replaced by relatively un-reactive CH_2 groups (carbons connected to two hydrogen atoms) connected to other CH_2 groups through carbon to carbon single bonds forming long chains. If the process conditions of this chemical reaction are manipulated to intentionally limit the extent of polymerization, then the length of such chains can be limited to form oligomers of ethylene. Such oligomers can be limited to the extent that they remain liquid at room temperature, forming molecules of containing perhaps only six to sixteen carbons (constructed from three to eight ethylene units). A slightly higher degree of polymerization leads to materials that are waxy solids at room temperature often referred to as paraffin waxes constructed from perhaps ten to twenty ethylene units. The ethylene polymers of interest as structural materials for HDPE pipes are higher melting temperature polymers constructed from many thousands, often hundreds of thousands of ethylene units, often referred to as “high polymers”.

Polyethylene plastics constructed almost entirely (i.e. >99 weight per cent) of linear chains of ethylene can be properly referred to as a homopolymers of ethylene, or homopolymer polyethylene. Such homopolymer materials are high density but are of limited utility in the manufacture of HDPE pipes because of critical limitations in their mechanical properties, most importantly, low resistance to environmental stress cracking (ESC) and low resistance to slow crack growth (SCG). The incorporation of limited amounts (arbitrarily consider less than five weight per cent) of other olefins into the polyethylene chain was discovered to provide a dramatic increase in resistance to ESC and SCG while simultaneously reducing the density of the material only slightly.

The co-polymerization of ethylene with other co-monomer olefins is widely utilized by industry although very few co-monomers are commonly employed. The most commonly utilized olefins for the purpose of lowering the density of polyethylene plastics are butene (4 carbons), hexene (6 carbons) and octene (8 carbons). Only the most reactive isomer of these other olefins is commonly employed. This reactive isomer is often referred to as the alpha (α) isomer to indicate that the double bond includes a carbon at the end of the molecule (e.g. alpha-butene or α-butene). Other terminology systems refer to α-butene as 1-butene and α-octene as 1-octene. While the relative merits of the use of one co-monomer over another is a topic that is sometimes debated, the utility of their inclusion into polyethylene plastics is universally recognized.

4.2 Crystallinity and Density

The spatial consequences of the addition of a single α-hexene molecule into a chain of polyethylene molecules is important to understand the density lowering effect. It is useful to consider the chain of polyethylene molecules as the trunk of a tree. The terminal carbon and the adjacent carbon of the α-olefin are inserted into the straight chain which continues forward while the additional carbons of the α-olefin are left pendant, forming a branch on the tree. In that the weight percentage of α-olefins incorporated is typically quite low compared to the overall degree of polymerization, it should be visualized that these branches are essentially isolated from other branches and are short compared to the overall molecular length of the polymer chain. For this reason, these branch points are often referred to as short-chain branches (SCB). When polyethylene molecules crystallize into a solid micro-structure, a regular folding pattern is a preferred configuration. These folded highly dense regions also participate in larger lamellar structures in the three-dimensional network of the solid material. However, the degree of regularity in chain-folding is interrupted at the location of a SCB which results in a reduction in the overall extent of crystallinity and an increase in the amount of amorphous material in the solid. In this manner, the incorporation of varying amounts of co-monomer is a useful method for adjusting the density of the resulting ethylene co-polymer.

As discussed above, Resin Density of HDPE is largely driven by the amount of SCB present. SCB acts to disrupt regular stacking of HDPE molecules into crystalline regimes in the solid material resulting in a change to the proportion of crystalline and amorphous regions. ASTM D4883 states “Polyethylene shall be viewed as a composite structure where high-density crystalline regions are connected by lower-density amorphous material. The ratio of crystalline to amorphous material determines the final density of the material”. Polyethylene compound is sometimes referred to as a semi-crystalline material for this reason. The crystalline regions are relatively resistant to small scale distortions under applied force while the amorphous regions have a significantly lower resistance to such distortions. Also, the density of a crystalline region is much higher than the density of an amorphous region. As discussed in Sections 2.4 and 2.5 flexural modulus and tensile strength at yield are strongly related to Resin Density. Other properties like abrasion resistance and impact resistance are also related to Resin Density but include a significant contribution from the extent of polymerization of the polymer (or weight averaged molecular weight as discussed in Section 4.3) such that Resin Density has predictive value so long as the extent of polymerization remains essentially constant.

4.3 Molecular Weight, Molecular Weight Distribution and Melt Index

As discussed above, the polyethylene compound used for the manufacturing of HDPE pipes is a high polymer and is constructed of many thousands of ethylene molecules assembled into chains and occasionally interrupted by co-monomers. Some polymerization reactions can be controlled to produce polymer chains that are all essentially the same length. Such polymers are often referred to as mono-disperse to indicate this special quality. Under most polymerization conditions, polymerization reactions give rise to polymer chains that vary significantly in length resulting in poly-disperse polymers. Often, the population of polymer chains at each chain length varies in a manner that is predictable. A useful technique for understanding this population of polymer chains at each chain length is called Gel Permeation Chromatography (GPC).

4.3.1 Gel Permeation Chromatography (GPC)

Before going into the scientific principles of Gel Permeation Chromatography (GPC) in detail, it is appropriate to clarify that GPC testing is not quality control testing and should not be considered as a potential means of further specification in codes and standards for a number of reasons. Chiefly, this testing requires highly trained personnel, expensive equipment and complex analysis. The technique often uses hazardous solvents and often requires frequent calibration as the column is degraded by the extremely high pressures used.

GPC is a type of chromatographic separation that relies on size-exclusion principles. A long column is packed with an immobile matrix material containing various micropore sizes in the matrix. Polyethylene is dissolved in a solvent at elevated temperatures in a fairly dilute solution. While in solution, the dissolved molecules of polyethylene can adopt a variety of three-dimensional configurations or shapes with longer chains necessarily forming larger shapes. When the size of a micropore in the matrix is sufficiently large so that the shape the polyethylene molecule has adopted can fit inside it, the transit of the polyethylene molecule down the column is delayed by its residence time within that cavity. However, very large shapes adopted by very long polyethylene molecules may not fit into the micropores. These longest polyethylene molecules are therefore excluded on the basis of their size from retention by the immobile matrix and wash through the column relatively rapidly. Somewhat shorter chains of polyethylene find more holes into which they can fit and spend relatively more time on the column before washing off. The related use of high performance size-exclusion chromatography in the characterization of polystyrene is detailed in ASTM D5296 [30].

Detection of the polymer chains at the end of the column can be used to construct a chromatogram [31] such as shown in Figure 4-1. A sense of the distribution of molecular sizes is visualized in this figure. The distribution of molecular weight is sufficiently large that it is convenient to express the log of molecular weight as the x-axis, shown in this figure spanning more than four decades of molecular weight. The y-axis expresses a factor that is proportional to the mass at a given molecular weight. Several commonly referred to terms are illustrated on Figure 4-1 such as the number averaged molecular weight, M_n, and the weight averaged molecular weight, M_w. The ratio of M_w / M_n is commonly referred to as the molecular weight distribution, heterogeneity index (HI) or poly-dispersity index (PDI). As a PDI value approaches unity, the molecular weight distribution can be thought of as narrowing as the assembly of molecules becomes more similar in chain length.

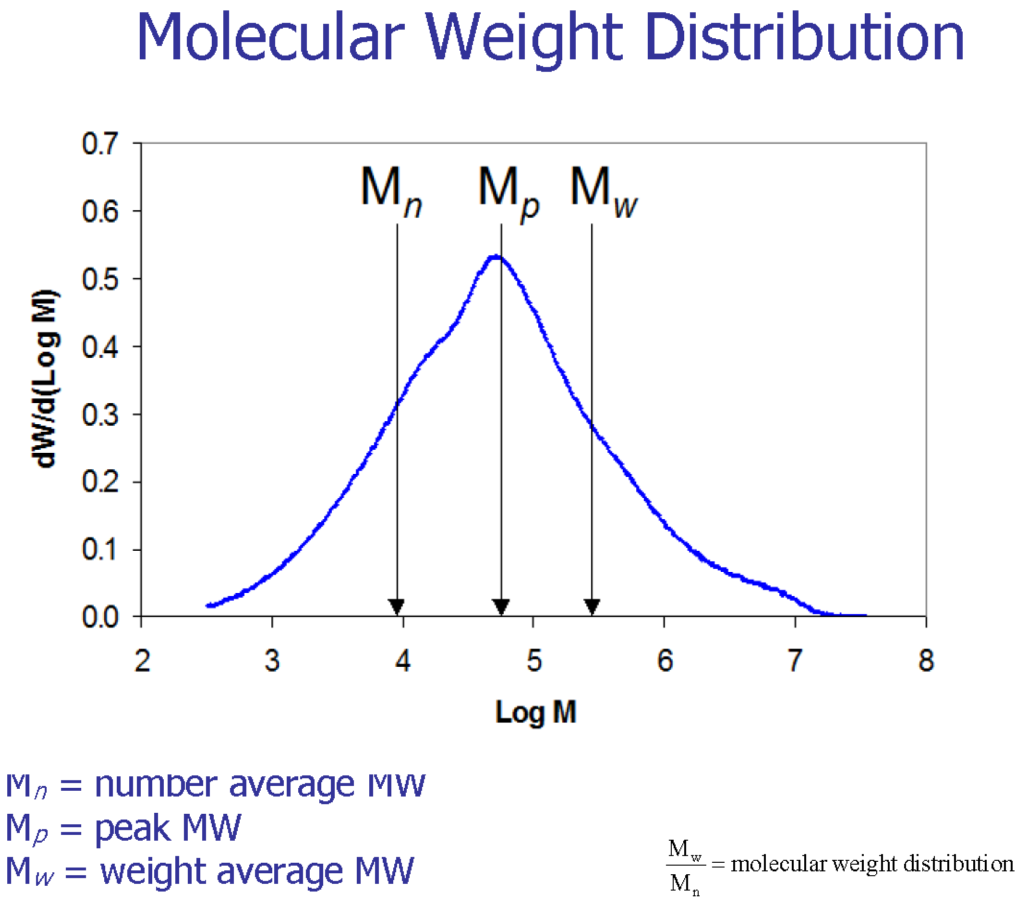

Figure 4-1
An example of a GPC chromatogram

4.3.2 Catalyst Compositions and Their Relationship to PDI

It is common to discuss the type of catalyst used to manufacture the polyethylene compound since the catalyst type can have a significant effect on PDI. A variety of metals deposited onto inorganic matrices, such as the Phillips catalyst comprised of chromium-oxide on alumina-silicates, give rise to relatively large PDI polyethylene compound. Values of PDI for such polymers may range from approximately 15 to 40. Ziegler-Natta (ZN) catalysts, typically using titanium chloride, give rise to polyethylene compounds with PDI ranging from perhaps as high as 20 down to as low as 4 when used in a single reactor configuration. More recently, single-site and metallocene catalysts, typically containing an anionic five-carbon attachment to a metal atom, may be used to generate commercial polyethylene materials with PDI approaching values as low as 2.0 when used in a single reactor configuration.

The use of melt index and HLMI as an indicator of M_w ranking is not applicable if poly-dispersity varies significantly or if the polyethylene is not essentially linear (discussed further in Section 4.5). Conversely, if poly-dispersity is maintained as essentially constant, then essentially linear polyethylene compound of different M_w can be correctly rank ordered by their melt index or HLMI. Within the context of well controlled polymerization reaction conditions using well controlled catalyst compositions, melt index or HLMI measurements are widely employed as quality control tests which act to restrict polyethylene compound within a range of M_w values.

4.4 Unimodal and Bimodal Polyethylene Compound

Considering the available polymers in the 1980s, those attempting to manufacture polyethylene compound with a designed level of environmental stress cracking (ESC) resistance had a limited set of tools. Slightly decreasing density often gave rise to significant increases in ESC resistance but both stiffness and strength would be reduced. Slightly decreasing the HLMI could also give rise to measureable increases in ESC resistance, but part manufacturing details like melt temperature and melt pressure would increase. An escape from this paradigm was strongly desired.

Full details are contained in the US patent [32] but can be conceptualized as the mechanical blending of just two ZN component resins. At high melt index, prepare a high-density polymer containing small amounts of short chain branches. At low melt index, prepare a low density polymer containing a substantial amount of short chain branches. By combining these two polymers so that they completely and uniformly mix, one can prepare a resulting polymer composition with different properties. The resulting polymer composition had remarkably high ESC performance when compared to traditional medium density polymers of similar melt index. Simply put, ESC and other properties at a given density are dramatically improved if SCB is selectively placed in the higher molecular weight portion of the total molecular weight distribution.

The micro-structural explanation for this innovation that has achieved wide acceptance [33] is represented in Figure 4-2 below. When SCB is selectively incorporated into high molecular weight components of the polymer, the statistical probability that portions of that polymer chain extend between two adjoining crystalline regimes or lamella is markedly increased. In essence, it is believed that a polymer chain that bridges two crystalline regimes mechanically reinforces the structure in such a manner that the stress crack performance is improved. In other words, these so-called tie molecules that interpenetrate neighboring crystallites are of paramount importance in obtaining improvements in slow-crack growth resistance. The incorporation of SCB into a lower molecular weight polymer chain is likely just as effective at forcing portions of that polymer chain outside of its crystal but the length of that chain has a reduced probability of extending far enough to be incorporated into a neighboring crystalline regime. It is this intentional act of placing an abundance of SCB into the higher molecular weight end of the molecular weight distribution (while depleting the lower molecular weight end of the molecular weight distribution) that establishes the elevated performance of recent bimodal polyethylene compounds.

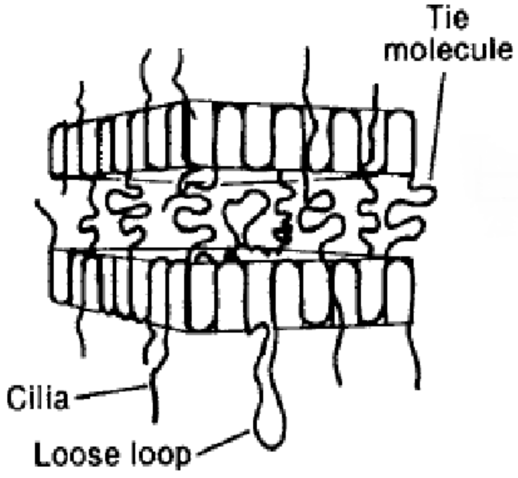

Figure 4-2
Illustration of tie molecules connecting two crystalline lamellae

Other topics are occasionally confused with bimodal polymers. One such confusion regards the presence of two local peak maxima in the GPC as rendering a polyethylene bimodal. This may or may not represent a bimodal polymer depending on the distribution of SCB. Similarly, the utilization of two polymerization reactors, in series or in parallel, is not necessary or sufficient to convey bimodal functionality. More recently, two catalysts in a single reactor have been used to generate bimodal polyethylene compound.

More sophisticated GPC techniques have been recently utilized to clearly establish the microstructure of bimodal polyethylene. By coupling GPC detection with techniques that are capable of measuring the level of SCB, a clear picture of bimodal polymers can be visualized. Shown in Figure 4-3, is an overlay [34] of two plots. In blue, a typical GPC chromatogram is illustrated for a polyethylene which shows two local maxima. However, it is not appropriate to call this a bimodal polymer yet. Data was simultaneously gathered for the amount of SCB at many separate intervals as the polymer was separated by size exclusion on the GPC column. The values for SCB concentration are shown as red dots with error bars included in red. This SCB content data is overlaid upon the molecular weight interval analyzed. As can be clearly seen from the red plot, essentially no SCB is present at the lowest molecular weights and the level of SCB increases substantially at higher molecular weights. The red dots show upward left-to-right slant. The data portrayed in red allows us to conclude that this polyethylene is truly bimodal showing excellent segregation of SCB into the high molecular weight portion of the molecular weight distribution.

Using these same techniques, the stark contrast with unimodal polymers can be visualized. Two sets of polyethylene overlays are shown [35] in Figure 4-4. Focusing our attention on the GPC for chromium on silica catalyzed polyethylene (illustrated with X), the SCB (open circles) for this polymer shows an obvious left-to-right downward slant illustrating significantly higher SCB in the low molecular weight end than in the high molecular weight end.

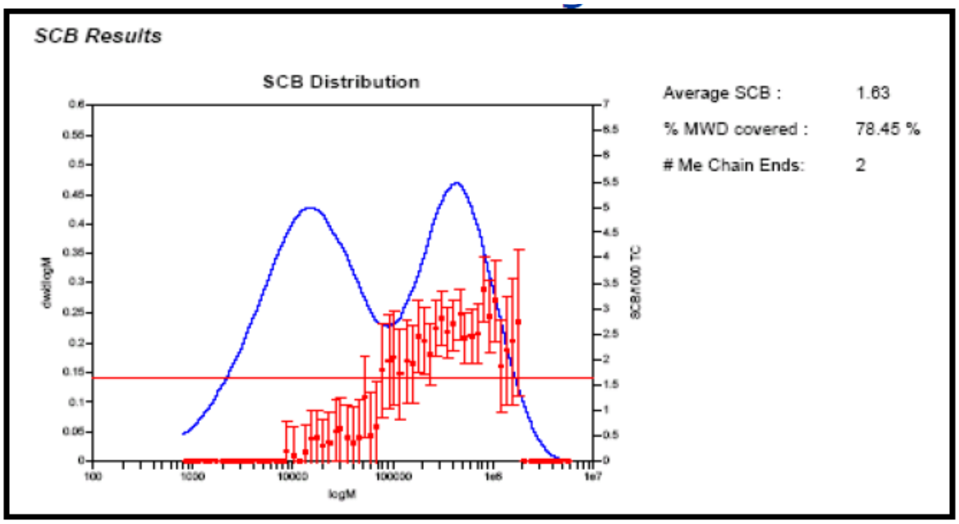

Figure 4-3
Distribution of SCB across molecular weight for bimodal polyethylene

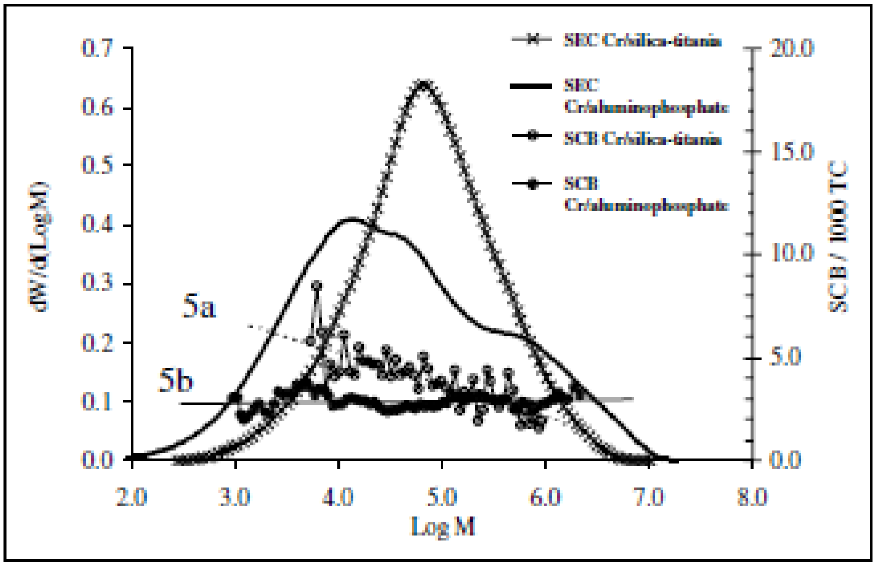

Figure 4-4
Distribution of SCB across molecular weight of two unimodal polyethylenes

Once again, it is appropriate to clarify that this specialized type of GPC testing is not quality control testing and should not be considered as a potential means of further specification in codes and standards for a number of reasons.

4.5 Tie Molecules and Slow Crack Growth Resistance

In their seminal work, Brown and Huang inferred [36] that in order for a polyethylene molecule to span the distance between two crystalline lamella, it must be longer than the thickness of the two lamella. This led them to propose that a polyethylene molecule must have a molecular weight greater than 18,000 g/mol in order to participate as a tie molecule. Above this critical molecular weight, there is a statistical probability that a polyethylene molecule will bridge adjacent crystal lamella upon crystallization and thereby serve as a tie molecule. The statistical probability of a single polyethylene molecule serving this function is increased when it incorporates short-chain branches. There is also growing literature support for a second structure of notable benefit to slow crack growth resistance of polyethylene. This inter-lamellar structure is often referred to as an "entanglement" and is composed of interactions of amorphous loops and other structures believed to be held together by van der Waals forces.

Formation of tie molecules occurs during the process of crystallization. Molten polyethylene which includes a larger number of the desirable short-chain branched high molecular weight molecules will form a greater number of tie molecule structures than a similar molten polyethylene which contains less of the desirable short-chain branched high molecular weight molecules. In essence, this can be considered a concentration effect. The linked lamella are a critical component influencing the stress crack resistance of larger spherulitic structures of the solid polyethylene.

Consensus has developed regarding the microscopic nature of slow crack growth of polyethylene in the presence of a pre-existing sharp crack. A craze zone is created ahead of the crack tip from localized yielding and drawing of the polymer creating bridging fibrils crossing the craze zone [37] (see Figure 4-5). Smaller voids are formed and combine to form larger voids. The bridging fibrils oppose the opening of the crack through their resistance to continued elongation. The net effect is commonly referred to as "crack tip blunting" and can temporarily stop the progression of the crack through the material. Eventually the fibrils thin and rupture with the crack advancing further through the material. This progression can occur in a step-wise fashion for highly SCG resistant polyethylene.

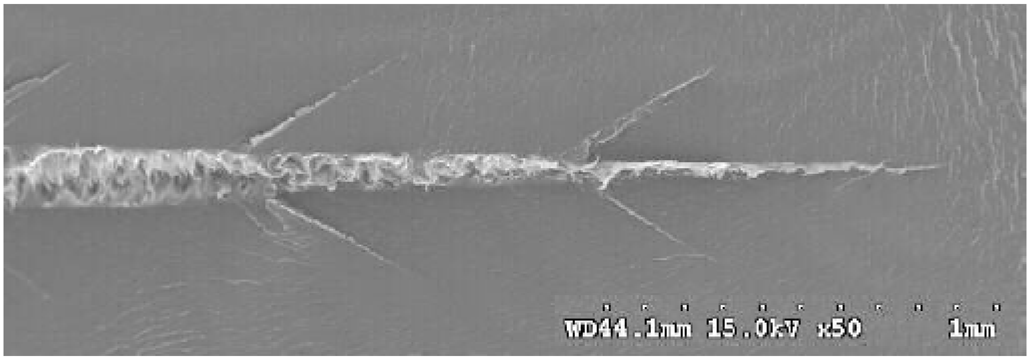

Figure 4-5
Digital microscopic image of a craze zone in polyethylene

4.6 Other Potential Impacts on Slow Crack Growth Resistance

Several factors are known to have a potential impact on slow crack growth resistance of polyethylene. Molecular orientation can play a role as noted in ASTM F1473 Section X1.1.2 which states "Notching perpendicular to the extrusion direction . . . generally gives higher results than notching parallel to the extrusion direction”. The thermal history – such as the cooling rate or annealing temperature – has also been noted [38] as changing the lamellar thicknesses and impacting slow crack growth resistance. Crack initiation has also been associated [39] with “foreign particulates or other types of heterogeneities resulting from resin formulation”. The presence of carbon black is sometimes cited [40] as having a negative impact on slow crack growth resistance depending on the technical details of the compound such as carbon black size, dispersion of the carbon black, polyethylene density, crack size, etc. The critical beneficial contributions of carbon black to the UV stability of polyethylene are realized through skillful extrusion practices and informed by thoughtful choices regarding carbon black technology.

Thus far, this Section has focused on either unpigmented polyethylene or the black polyethylene compound that is converted into HDPE pipes. Although some resin manufacturers provide polyethylene compound with carbon black already added, commonly called pre-compounded polyethylene compound, others provide natural resin and a resin that contains carbon black at levels significantly higher than 2.0 to 3.0 weight per cent. The resin manufacturer provides the pipe manufacturer with instructions for combining the two materials, the natural resin and the black pigment concentrate compound, often referred to as “salt and pepper” compounding. The instructions provided will likely include important manufacturing details such as the blending ratios of the two components, instructions for proper drying techniques for the pigment concentrate compound and information regarding the details of mixing and extrusion used to create desirable properties in finished HDPE pipes.

The carbon black used industrially is manufactured through incomplete combustion of hydrocarbons under low oxygen conditions. The carbon atoms are contained in graphitic layered structures which make up larger spherical particles, commonly called primary particles, with diameter from 10 to 100 nm. During the production process it is desirable that the primary particles assemble into larger agglomerate structures in order to facilitate bulk handling of carbon black. While there are relatively few code or standard requirements on the carbon black used in pipe formulations in the U. S., the indirect requirements introduced through various performance criteria on pipe materials have resulted in commonly selected properties for the carbon black. In general, carbon blacks are selected based on several factors but a heavy emphasis is placed on ease of dispersion of the carbon black into its primary particles during melt processing. A property that has received significant industry interest is the average or mean primary particle size measurement [41] based on transmission electron microscopy. Other properties of carbon black that are normally referred to by suppliers include specific surface area [42] and oil absorption number [43].

Speaking very generally, pigment concentrate compounds for use in pressure pipe formulations are typically manufactured at greater than 25 weight per cent carbon black. Carbon black has a fairly high surface affinity for polyethylene and dramatic viscosity increases can be anticipated during the preparation of pigment concentrate compounds. Because of this tendency for a dramatic viscosity increase during the preparation of pigment concentrate compounds, it is generally not practical to produce pigment concentrate compounds using low melt index materials. Using a natural grade of resin with a melt flow significantly less than 0.15 gram / 10 minutes in the preparation of a pigment concentrate compounds containing greater than 25 weight per cent carbon black would likely result in a pigment concentrate compound of such high viscosity that it would not distribute effectively when combined with natural resin in the extrusion of HDPE Pipe products. The polyethylene resin that comprises the balance of the pigment concentrate compound will be referred to throughout this document as “carrier resin”. Generally polyethylene materials with a melt flow greater than 0.15 gram / 10 minutes are used and are referred to as carrier resins.

It is natural to consider the implications to the mechanical properties of the black compound from the incorporation of the carrier resin. Undoubtedly, some effect from the incorporation of this carrier resin should be anticipated. As an illustrative example, consider that the combination of a pigment concentrate compound that is 33.3 weight per cent carbon black to deliver the minimum amount of 2.0 weight per cent carbon black would only incorporate 4 weight per cent carrier resin. In practice, the incorporated amount of this carrier resin is fairly small and its actual contribution to bulk mechanical properties are difficult to directly measure due to statistical variation in the testing. It is important to note that the industry approach to HDB generation is to require that this testing be done for a fully formulated compound. If HDPE pipe manufacturing will include a pigment concentrate compound, then industry has created requirements through PPI TR-3 that establish a formulation specific testing protocol.

4.7 Long-chain Branches and Cross-linking of Polyethylene Compound

In the proceeding Sections, the terminology has carefully referred to “essentially linear polyethylene”. This term has been chosen with great care because the impact to the properties of molten polyethylene depends heavily on the presence or absence of even a small number of long-chain branches (LCB). Considering polyethylene molecules as straight-lines is useful in the explanation of LCB. Taking the terminus of one straight-chain polyethylene molecule and attaching it directly to the center of another straight-chain polyethylene molecule can be considered to result in a molecule that is roughly shaped like a capital T. Similarly, the base of the T-shaped molecule thus formed can intersect another straight-chain polyethylene molecule to result in a molecule that is roughly shaped like a capital H. These points of attachment are termed LCB understanding that the number of carbons between these branching points are considerably more (and therefore longer) than the four carbons resulting from incorporation of a molecule of 1-hexene.

It is now useful to consider the consequences of taking cross-linking to the other extreme. In the limit that an assembly of polyethylene molecules are all intersected at their terminus as discussed above, the entire assembly becomes a single molecule. At this point, polyethylene retains similar physical characteristics as a solid material but the characteristics in the molten state are completely different. In fact, this highly cross-linked polyethylene molecule will no longer flow in any predictable manner when in the molten state and is often referred to as “gelled”. In the limit of essentially complete cross-linking, this polyethylene material is no longer properly referred to as a thermoplastic material but is rather a thermoset material. It is often stated inaccurately that “gelled polyethylene does not melt” when in fact it melts but does not flow. Similarly, molecular diffusion becomes ineffective because of the extremely high molecular weight of gelled polyethylene. In a simplistic sense, there is an approximate doubling of molecular weight for each instance of cross-linking. Highly cross-linked materials are therefore much higher in molecular weight than the original polyethylene materials that they arise from. Strong efforts are therefore taken throughout the HDPE pipe manufacturing process to avoid cross-linking polyethylene such as process monitoring and controls during HDPE pipe manufacturing, frequent QC testing of HDPE pipe, heating elements which fail safe rather than resulting in run-away heating, and streamlined die and screw design.

Catalyst composition can also affect the amount of LCB present in polyethylene. Although low density polymers are not discussed in detail in this report, they are useful to illustrate this point. Low density polyethylene (LDPE) contains an abundance of LCB based on its use of peroxide initiators at high temperatures and ethylene pressures. The use of ZN catalysts to produce linear low density polyethylene (LLDPE) essentially free from LCB provides similar materials with differentiated properties. When ZN catalysts are used to make high-density polymers, once again, these polymers are essentially free from LCB. It has long been understood that Phillips-type catalysts provide HDPE with a small but important amount of LCB in many of their polymers. However, the flow properties of these polymers have more in common with the linear polymers than they do with LDPE.

In summary, although small amounts of LCB may be present in the polyethylene compound the effect is generally minimal. The molecular similarity between LCB and "gelled" polyethylene compound is offered for the purposes of illustration and clarification. The presence of adequate antioxidant stabilization, discussed further in Section 4.8, addresses the potential for polyethylene compound to degrade to the point of extensive cross-linking.

4.8 Melt Processing of Polyethylene Compound and Additive Chemistry

Aside from the intentional process of cross-linking, it is important to discuss the potential for unintentional cross-linking of polyethylene and additives used to protect against this chemical reaction. Polyethylene, similar to all other hydrocarbons, is subject to oxidation as both a solid and as a molten liquid. The rate of oxidation is significantly enhanced for the molten liquid for a number of reasons including the significantly elevated temperature required to obtain melting. Degradation of polyethylene through oxidation is widely understood to be a one-electron process involving high reactivity radicals. Unfortunately, in the absence of chemical intervention, this reaction is also self-catalyzing (autocatalytic) and, if left unchecked, has significant deleterious results. Figure 4-6 provides an illustration [44] of several routes of chemical intervention to defeat catalytic degradation of polymers.

Two distinct degradation pathways for polyethylene are commonly discussed. The degradation pathway most commonly noted as problematic for solid polyethylene are reactions that lead to chain-breaking, or scission. This reaction acts to reduce the overall molecular weight of the polymer accompanied by both physical changes (such as cracking, discoloration, and part distortion) as well as mechanical changes (such as reduction in impact resistance, and reduced ductility).

The degradation pathway most commonly noted as problematic for molten polyethylene are reactions that lead to cross-linking, or chain growth. Uncontrolled cross-linking of molten polyethylene leads first to increases in the viscosity of the resin, followed by uneven flow regimes and finally by the creation of gelled polyethylene that will not flow when molten. Since this gel material is very dissimilar to the surrounding polyethylene, it remains localized in the finished article where its dissimilar properties give rise to a variety of problems. In HDPE pipes, the most serious consequence of gelled material in the pipe wall is that this is a site of localized stresses in excess of the adjacent material such that SCG can initiate.

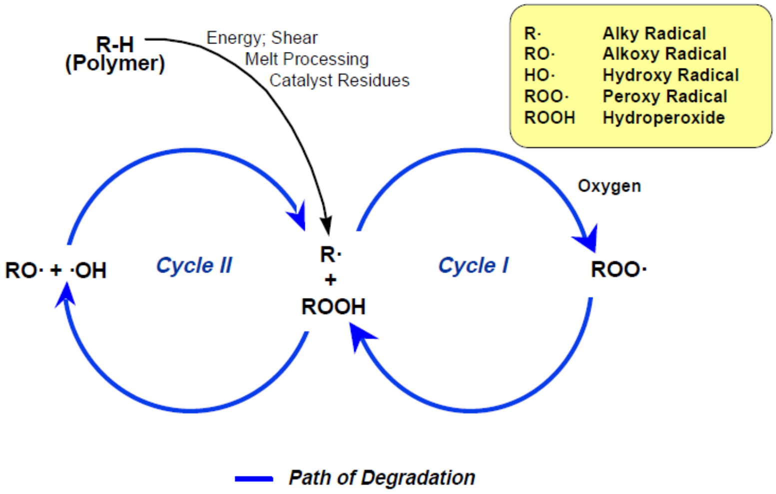

Figure 4-6
The autocatalytic radical processes leading to polymer degradation

It is a useful over-simplification to consider that there are two types of additives for polyethylene. Often additives that are most effective at protecting molten polyethylene are called “processing stabilizers”. In contrast, other additives are referred to as “long-term stabilizers” or “thermal stabilizers” since they are more effective at protecting solid HDPE compound. Synergistic roles for each type of additive in contributing to the full beneficial use of the other is sometimes noted.

Another useful over-simplification is to consider that there are two types of chemical compositions adapted to either process stabilization or long-term stabilization. In it important to note that all of these chemical additives are present in low amounts, tend to concentrate in the amorphous phase of HDPE compound and therefore do not interfere with butt fusion or the development of the key tie molecule structures so important for slow crack growth resistance.

4.8.1 Hindered Phenols

A critical role in long-term stabilization chemistry of polyethylene compound is played by a class of chemicals commonly called hindered phenols. These chemicals are designed to donate a hydrogen atom to highly reactive peroxy compounds rapidly so that neither cross-linking reactions nor chain scission reactions damage the mechanical properties of the polymer over time. In donating this hydrogen atom, the hindered phenol becomes activated to further reactions. However, the phenolic structure is generally protected from further reactions by large groups located on the adjacent carbons. In so doing, the phenolic region of the hindered phenol is rendered ineffective for further stabilization reactions and is, in essence, consumed by the reaction although it remains present in the polyethylene plastic. It is relatively common for hindered phenols in commerce today to include more than one phenolic region. Figure 4-7 illustrates [45] the chemical structure of some hindered phenols.

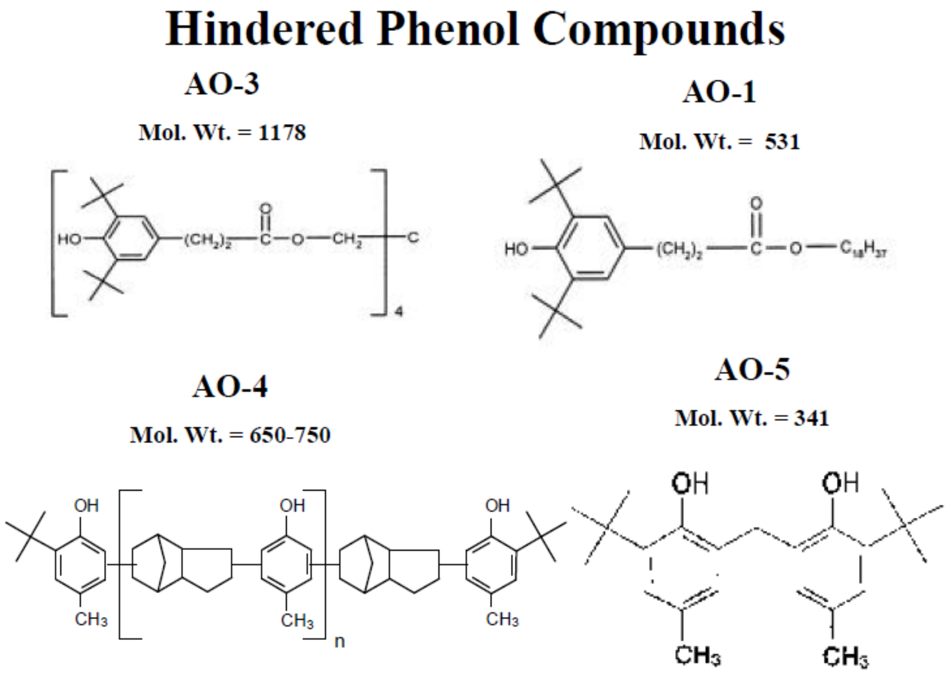

Figure 4-7
Illustration of hindered phenols

4.8.2 Phosphites

A critical role in melt stabilization chemistry of polyethylene compound is played by a class of chemicals commonly called processing stabilizers or phosphites. These chemicals have an important role to play that can’t be managed by hindered phenols. Phosphites are able to react with oxidized polyethylene molecules that can arise from multiple chemical pathways including incomplete degassing of the molten polymer. This reaction results in oxidation of the phosphite to a phosphate. This reaction results in stabilization of the oxidized polyethylene molecule such that it does not participate in either cross-linking reactions or chain scission reactions. In so doing, the phosphorus atom is unavailable for further oxidation reactions and is, in essence, consumed by the reaction although it remains present in the polyethylene plastic. It is relatively common for phosphites in commerce today to contain elements of hindered phenols which can be liberated to participate in other stabilization reactions, thereby acting as multi-site stabilizer. Figure 4-8 illustrates [46] the chemical structure of some phosphites that also contain hindered phenols.

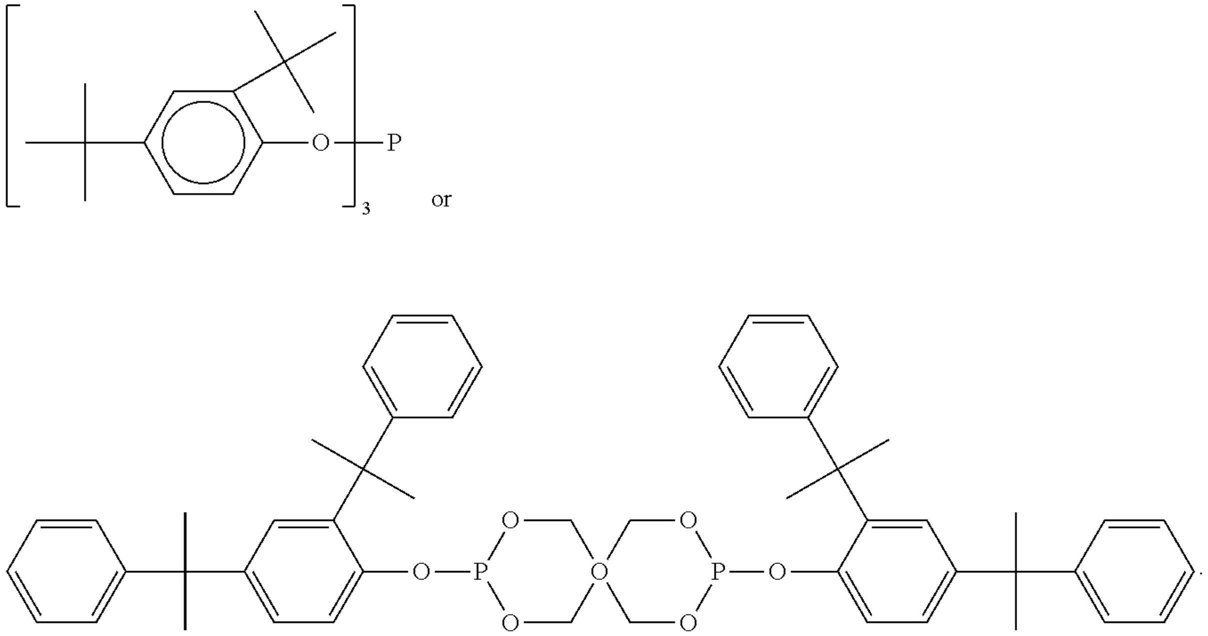

Figure 4-8
Illustration of Phosphites

Typically the level of hindered phenol antioxidants or phosphites used will not exceed 0.25 weight per cent for a number of reasons. As Figure 4-7 and 4-8 illustrate, modern antioxidants are chemicals designed for a high degree of effectiveness resulting in high stabilization at low use levels by weight. Another reason for low use levels of antioxidants is the relatively high cost of the additives which often exceeds the value of the polyethylene by factors of two to ten-fold. Low use levels of antioxidants also relates to utility. Increased levels of additives do not always lead to enhancement in stabilization. Eventually, additives exceed their limit of solubility in the polymer matrix and can result in effects such as chalking and dusting. Finally, it is important to note that many pressure rated polyethylene compounds that meet nuclear safety-related requirements are also used in the transport of potable water. In potable water application, the chemicals used and the levels at which they are used are regulated by NSF/ANSI 61 [47] which places limits on the chemicals that may be present in drinking water and – by extension – on the formulary of the polyethylene compounds used to manufacture the HDPE pipe. Several addition factors limit the concentration of antioxidants used in polyethylene compounds including the relatively high cost of antioxidants as well as the potential for appearance difficulties generally referred to as “chalking”. It is generally assumed that the total level of antioxidants comprise less than 1 weight per cent of the total polyethylene compound composition. This level of antioxidants is sufficiently low that it should not affect butt fusion. While there are certainly other additives available to be utilized in formulation, this brief over-view has focused on the topics of greatest interest for our later consideration of butt fusion of HDPE pipes.

4.9 The Viscosity of Polyethylene Compound and Melt Temperature

There is a well-known relationship between the viscosity of non-Newtonian fluids, such as lubricating oils and molten polyethylene, and temperature with the general trend of increasing temperature resulting in reduced viscosity. An application of particular interest to the extrusion of polyethylene compound into HDPE pipes is shown [48] in Figure 4-9 below which shows the increase in shear stress with increasing shear rate as well as the decrease in shear stress with increasing temperature. Please note that this set of curves starts at 150°C, slightly above the melting temperature for HDPE of approximately 130°C, and increases to 230°C. A melt temperature around 230°C is within the capability of modern extrusion equipment and polyethylene compounds. This information provides an insight into the challenges of polyethylene compound stabilization. We will return to various processing considerations later.

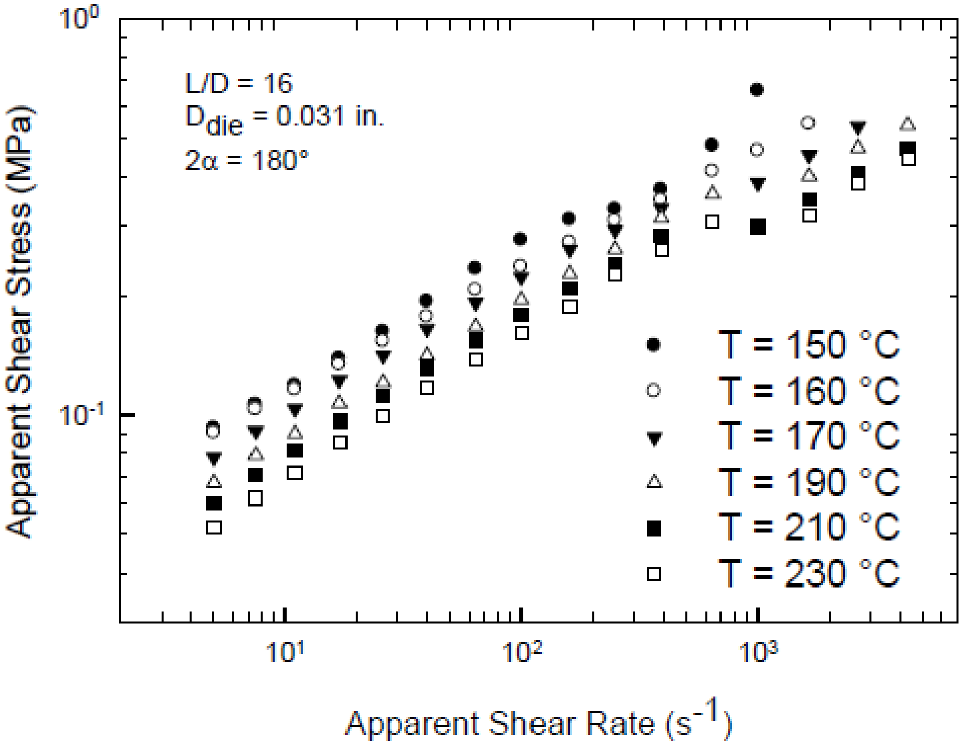

Figure 4-9
Apparent flow curve for ZN catalyzed HDPE at various temperatures

4.10 Thermal Properties of Polyethylene Compound and Butt Fusion

It is important to identify and discuss polymer crystallization, melting and material properties of the molten polymer in order to consider the potential effects of butt fusion. Similar to the process of crystallization of salts from water, as molten polyethylene compound cools to a critical temperature, the process of crystallization begins. When polyethylene solidifies from the melt, initially formed crystalline sites create nucleation points for further crystal growth. The crystals grow through the addition of polymer molecules until they intersect an adjacent growing crystal. The highly ordered crystalline phases are substantially higher density than the melt and therefore, the process of crystallization is a densification process resulting in a volumetric reduction normally referred to as melt shrinkage. The rate of cooling is also an important variable in that greater rates of cooling through the critical crystallization temperature result in a lower density solid than is produced if the rate of cooling is slower. For this reason, the measurement of polyethylene compound properties in ASTM D3350 includes a requirement that test specimens be molded under specified conditions in accordance with ASTM D4703 as noted in Section 2.

The thermal properties of polyethylene compound, both in the molten state and in the solid state are of central importance to understanding the crystallization behavior of polyethylene. As described in ASTM D3418 [49], Differential Scanning Calorimetry (DSC) is a convenient test method for measurement of the heat flows into polyethylene. A DSC measurement of heat flow into polyethylene is shown [50] in Figure 4-10. As the temperature of the solid is slowly increased, the heat flow into the solid slowly increases until the temperature of the solid approaches the melting point. Near the melting point, the heat flow rapidly rises and melting occurs. Polyethylene compounds with a density of approximately 0.950 g/cm^3 material have melting point range of 130-133°C when measured using test method ISO 3146 [51]. This range of melting point is fairly reflective of the melting point of polyethylene compound generally although slight increases in melting point can be anticipated as Resin Density increases in the allowed range [52]. Following melting, the liquid polyethylene compound is still capable of absorbing additional heat resulting in a reduction in the viscosity of the molten phase.

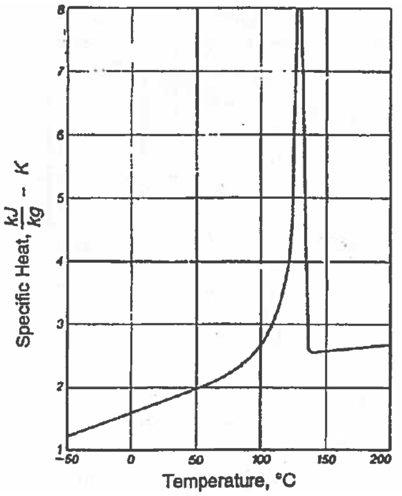

Figure 4-10
Specific heat as a function of temperature for Polyethylene Compound

The low rate of thermal conductivity of solid polyethylene compound compared to metals is widely appreciated with sources indicating metals that have rates of thermal conductivity hundreds or even thousands times higher than solid polyethylene compound. Sources [53] typically cite thermal conductivity in units of W/m/°C and indicate solid polyethylene compound with a value between 0.2 and 0.5 W/m/°C. Due to this low rate of thermal conductivity (perhaps it is more direct to say the insulating capability of solid polyethylene compound), solid polyethylene compound can increase in temperature at an interface with metallic materials more quickly than the heat moves through the part thickness. Also, the rate of thermal conductivity is sufficiently slow for polyethylene compound that heat losses to the air through convective cooling can be an important part of total cooling.

Two additional values of interest are the heat capacity and the heat of fusion of polyethylene compound. However, it should first be noted that the heat capacity of polyethylene compound will be dependent on temperature, crystallinity and density [54]. Heat capacity of polyethylene compound will also be different in the solid phase than as a molten liquid. The heat capacity (also called Specific Heat) is defined as the amount of heat required to raise the temperature one degree Celsius and is reported [55] as 2300 Joules / kg °C while the heat of fusion is reported as significantly larger (250,000 Joules / kg) and represents the amount of heat required to melt polyethylene compound without increasing its temperature. This same amount of heat is given off as molten polyethylene compound solidifies.

The complexity of using butt fusion to join two pieces of HDPE pipe through crystallization to form a new strong and ductile joined material requires additional explanation. It is important to consider the joining process in relation to key polymer properties. Theories that emphasize the key importance of molecular diffusion are routinely highlighted [56]. At the point that molten polyethylene compound interfaces are brought into contact with each other under pressure and during the time period that the interface remains molten, polyethylene molecules from each of the flow boundaries diffuse into each other. This diffusion process facilitates the formation of crystalline structures and entangled amorphous regions containing molecules from each of the original molten boundaries creating an interfacial bond. The blending of the materials on the micro-structural level allows for the transmission of forces acting at the interfacial boundary to be transmitted into the bulk of the material. Because this theory focuses on molecular diffusion, it is emphasized that the time that the interface remains molten, the temperature of the molten interface and the diffusion coefficient of the polyethylene molecules are important considerations. It is important to emphasize that the diffusion coefficient of a polyethylene molecule is related in turn to the weight-averaged molecular weight, M_w, which is related inversely to the melt index (or HLMI) as discussed above. It should be noted that the process of molecular diffusion which develops a strong bonding interface between two polyethylene compound melt fronts operates on a similar time-scale as the development of the key tie molecule structures so important for slow crack growth resistance.

4.11 Broad Overview of HDPE Pipe Manufacturing

At a high level, a single screw extruder is a simple machine composed of a few parts. Pellets of polyethylene compound are fed onto a screw which is driven by a motor. The screw is composed of a helical metal section that closely fits inside the barrel and a channel that carries forward the polyethylene compound. The channel depth of the screw is variable down the length of the screw to accomplish various functions [57] including solids conveyance, melting, mixing, and melt conveying. A significant portion of the polyethylene compound heating is supplied by conversion of energy used to turn the screw into heat once extrusion starts, but supplemental barrel heat must always be available to melt residual polymer especially during start up. In large extruders, specific energy may be closely approximated through a calculation of the kilowatts of energy supplied by the extruder motor divided by the polyethylene output in kilograms / hour.

Prior to the extruded melt exiting from the extruder, it is necessary to change the molten polyethylene compound into the cylinder shape of a HDPE pipe. The creation of this cylinder is accomplished by a metallic form held motionless in the melt stream and supported against the outer barrel. Conventionally, the melt stream was formed into a cylinder by action of a spider-die [58], so called because the multiple points of attachment to the barrel can be compared to the eight legs of a spider (see Figure 4-11). At times a “torpedo” section is attached to the leading edge of the spider-die to streamline the splitting of the melt into a cylinder. Of course, these points of attachment obstruct the flow of the molten polyethylene compound causing the melt to separate. At the point that the polyethylene compound streams recombine, a line (“spiderline”) is created [59]. As embodied currently in most extrusion facilities, separating the melt into hundreds of small strands using a very different die design has proven more effective for maintaining properties than separating the melt into a few large strands. Regardless of the specifics of the die design, it is emphasized that all HDPE pipe extrusion will necessarily require various separate melt strands to re-combine in the molten state, under the combined conditions of heat, pressure and time prior to the polyethylene material exiting the die. This joining together of melt strands into a high strength and integrity HDPE pipe during extrusion is an inherent demonstration of the capability of the polyethylene compound that comprises the HDPE pipe to undergo butt fusion under comparable conditions.

Figure 4-11
A traditional spider die equipped with a "torpedo" section

In normal commercial production, the polyethylene melt is typically at a temperature 100 – 200°F above the melting point and is typically held at greater than 1500 psi of pressure. The pressure drop at the die exit is therefore substantial and the elastic properties of the melt act to cause the molten polyethylene compound to swell as it exits the die into air. After rapidly crossing this air gap, the molten polyethylene compound then enters a sizing sleeve or series of rings which acts to conductively cool the exterior of the melt thus rapidly forming the a solid skin of polyethylene compound. The inside dimension of the sizing sleeve is carefully dimensioned to deliver a properly sized outside diameter of the HDPE pipe. The sizing device is mechanically attached to a quench cooling chamber. The chamber is most commonly supplied with both vacuum and cold water mist spray because of enhanced cooling efficiency and commonly referred to as a "vacuum tank". However, a "flooded tank" is sometimes used in which the quench cooling chamber is run substantially or completely full of water with the HDPE pipe submerged. In either type of quench tank, it is not the intent to fully cool the molten polyethylene compound because it is not practical. Because of its previously noted low rate of thermal conductivity, solid polyethylene compound is slow to cool. Several additional water spray tanks are needed to provide a residence time for successively cooling the solidifying HDPE pipe cylinder as it advances down the extrusion line. Thicker walled pipes can develop residual stresses across the pipe wall due to the differential cooling.

A puller is located after the cooling spray tanks and provides the required force to move the entire length of HDPE pipe through all operations. The ratio of puller rate to extrusion production rate allows control of the wall thickness of the finished HDPE pipe. Slowing the puller down without changing extrusion rate will result in the wall becoming slightly thicker. Increases of puller rate in concert with increases in extrusion rate, accelerates the rate of linear production without changing the wall thickness.

4.12 Barrier Screw Design, Mixing and HDPE Pipe Manufacturing

The mixing of natural polyethylene compound with pigment concentrate compounds using a single screw is a sufficiently important part of HDPE pipe manufacturing that it is worthy of additional consideration. Polymer sticks to the barrel and slides on the polished surface of the screw. Tight tolerances of the screw flights to the inside of the barrel ensure forward motion of the polymer through the channels. It is useful to discuss the ratio of the length of the flighted section of the screw, L, divided by the screw diameter, D with a common value of L/D for HDPE pipe manufacturing in North America of 24:1.

It is common that extrusion of a pigment concentrate compounds in combination with natural resin utilizes a modified screw design called a barrier screw. Following a traditional conveyance section of the screw which accepts polymer feed, the design of a barrier screw [60] uses an additional flight to separate the screw channel into two sections. The dimensions of the flight are such that molten material may pass over the barrier but solids may not. Progressively down the length of the screw, the relative volume of these two channels change with the molten volume starting small and growing down the length of the screw. Prior to the end of the screw, the channel for solids is eliminated which largely prohibits the movement of solids further forward. The major advantage of the barrier design is this added restriction against conveyed solids. However, high production rates can sometimes overcome even a barrier screw design. In order to accomplish an even higher level of distributive mixing, high shear mixing tips near the end of the screw may be used. Static mixing sections are not attached to the screw but can also be used to improve distributive mixing. The net result is to present a homogeneous melt to the die for shaping into the pipe product.

The critical importance of adequate distributive mixing should be clear. Even and uniform carbon-black distribution is desirable. The pigment concentrate compound has different mechanical and thermal properties than the natural polyethylene resin. These pigment concentrate compounds typically are less ductile than the natural polyethylene resin. Incomplete distribution of pigment concentrate compound throughout the melt could therefore give rise to reduced ductility regions of the HDPE pipe. By contrast, areas of low relative carbon black content can, if present and when observed in thin sections [61], appear as “windows” (see Figure 4-12) due to their relatively higher translucency. Slides prepared in accordance with procedures in ISO 18553 [62], examined at low magnification, and compared to the reproduction photomicrographs in Figure 4 of Canadian Standards Association B137.0 [63], may be useful in evaluating the overall distribution of pigment concentrate compound to eliminate agglomerates, smears and streaks.

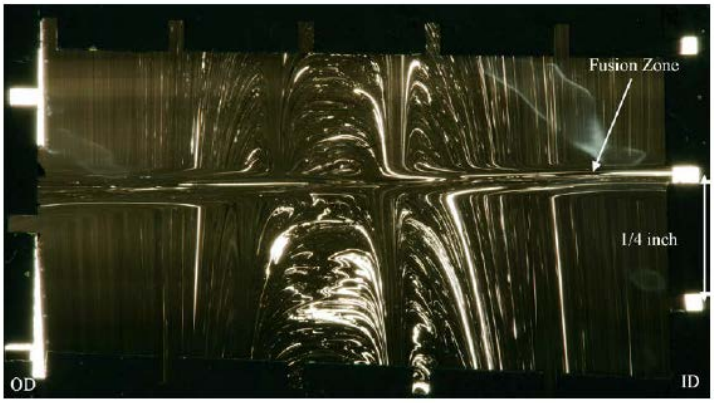

Figure 4-12
Photograph of a 25 – 35 micron slice from the fusion zone of unevenly mixed PE3408 HDPE pipe

Several factors are commonly noted as having an impact on distributive mixing in single screw HDPE pipe extrusion. As mentioned above, screw design is of primary importance to obtaining acceptable mixing but can also include maintenance considerations such as screw and barrel wear [64]. Improper alignment of the screw within the barrel can lead to accelerated wear and should be avoided. Insufficient or inconsistent melt temperature [65] can increase the viscosity of both the pigment concentrate compound and the natural polyethylene compound resulting in decreased mixing efficiency. Reduction in residence time can lead to a reduction in distributive mixing. This is commonly thought of as related to increased production rate but can also be influenced by equipment choices (e.g. screw design, die design, breaker plate, screen pack).

5

MANUFACTURING AND BUTT FUSION OF HDPE PIPES

5.1 Introduction

Understanding the basics of the extrusion manufacturing of HDPE pipes provides important insights related to the butt fusion of these same materials. In several important ways, the extrusion process and butt fusion share key features in common. In both processes, molten polymeric materials are brought together rapidly to effect finished articles that are both strong and durable.

5.2 Polyethylene Compound Properties and HDPE Pipe Manufacturing

Undoubtedly, the most important characteristic for the conversion of polyethylene compound into a manufactured article is the viscosity of the molten phase as measured by either melt index or HLMI. In order to allow polyethylene compound to be fashioned into finished articles in a variety of shapes and sizes, the viscosity of the melt is often dramatically altered in order to fit the requirements of a specific manufacturing process. It is desirable for injection molding to utilize a material with a low melt viscosity (melt index typically ranging from 10 to 60 g/10 minutes) to facilitate mold filling and rapid cycle times. It is desirable for rotational molding to use materials with a somewhat higher melt viscosity (melt index typically ranging from 1 to 10 g/10 minutes) so that the melt coats the interior of a mold evenly but quickly. Blow molding utilizes materials that are somewhat higher still in melt viscosity (melt index typically ranging from 0.1 to 1.0 g/10 minutes) so that the molten parison may hang momentarily while a mold block closes around it prior to the blow pin inflating the part. Extrusion processes, such as HDPE pipe manufacturing, often utilize quite low melt viscosity (HLMI typically ranging from 6 to 10 g/10 minutes) materials in comparison to other manufacturing processes.

HLMI has provided HDPE pipe processors a valuable tool for many years. For several decades, the relationship between several processing variables for smooth-bore extrusion of polyethylene compound and the HLMI was of paramount practical importance for HDPE pipe manufacturers. Production rate, extrusion temperature, extrusion pressure, and melt temperature were all well understood to relate to HLMI. Assuming the extruder rpm was kept constant, higher HLMI materials commonly exhibited lower extrusion pressures and lower melt temperatures. In those instances where either extrusion pressure or melt temperature was a limitation to production rate, the extruder speed could be increased resulting in increases in pounds of production per hour. In this regard, the design of the extrusion plastometer commonly used to measure melt index and HLMI allowed it to function, to a large degree, as an extrusion simulator.

It should be noted that the range of HLMI allowed (for the purposes of specification of all potentially useful PE4710 materials) is significantly larger than anticipated product variation for a single PE4710 material. For a given pressure rated HDPE compound, a typical manufacturing control range is likely to be constrained within approximately some few g/10 minutes and should not be anticipated to cover the 16 g/10 minutes range allowed by nuclear safety-related requirements. Secondary to HLMI in terms of practical impact to HDPE pipe processors, the thermal stability value is potentially worthy of mention. Although the specification formalized for nuclear safety-related applications allows natural resins with a thermal stability value of 220°C minimum, typical values are substantially greater, perhaps 10 to 30°C greater. This arises from the industry understanding that the HDPE pipe manufacturing conditions will consume some additives and reduce the thermal stability value.

Density is of primary importance to the mechanical properties as discussed in detail in other sections of this report. However, Resin Density has little practical implication for melt processing of HDPE. It should be noted that the range of Resin Density allowed (for the purposes of specification of all potentially useful PE4710 materials) is significantly larger than anticipated product variation for a single PE4710 material. For a given pressure rated material, a typical manufacturing control range is likely to be constrained within approximately 0.004 to 0.005 g/cm^3 and should not be anticipated to cover the 0.008 g/cm^3 range allowed in nuclear safety-related application for Resin Density or the 0.012 g/cm^3 range allowed for polyethylene compound density. Other polyethylene compound properties are not significant to HDPE pipe manufacturing and are not commented on further here.

5.3 Butt Fusion of HDPE Pipes

The joining of multiple molten strands of polyethylene compound to form a high strength and durable HDPE pipe inside the extrusion die is an autohesion joining process. Simply put, molten surfaces are bonded without the use of an adhesive such that they resist separation. The process of butt fusion joining two polyethylene compound surfaces from two HDPE pipes is a similar process that can be considered in three separate steps.

The first step relates to the point of contact between the molten polyethylene compound surfaces. This step is critical in establishing a bond and the presence of any material that presents a non-polymeric boundary can interfere. ASTM F2620 [66] indicates that HDPE pipes should be cleaned to remove any foreign matter and faced to create a smooth surface and this is widely repeated in other industry documents. The faces of the HDPE pipe segments are then squarely brought into contact with clean heater plates coated with a non-stick material in an effort to further eliminate the presence of other materials as well as to present a smooth surface for bonding. The faced ends are then allowed to heat under pressure until evidence of melt is visible around the entire circumference, at which point the net pressure is reduced to zero. At this point, the HDPE pipe surfaces maintain contact with the heater plate without the application of continuous pressure for a sufficient time to ensure complete coalescence during the fusing process.

In the second step, the heater plate is removed and the molten surfaces of the HDPE pipe are rapidly forced together at fusion pressure. Following contact, interfacial pressure establishes a flow zone of molten polyethylene compound outward from the center of the wall thickness toward both the inside diameter and outside diameter of the joined HDPE pipe. In the third step, the newly joined HDPE pipe continues to be held together under pressure while the molten polyethylene compound becomes solid and cools further.

From a mechanistic point of view, the presence of non-polymeric materials (such as carbon black) in the developing fusion interface can only be viewed as counter-productive. However, there are reasons to anticipate that the potentially negative impact of carbon black in the developing fusion interface will be small. As previously discussed, carbon black primary particles are spherical and small compared to the scale of the fusion interface. Additionally, the interfacial bond between carbon black and polyethylene is fairly strong as indicated by the viscosity changes which are observed at higher concentrations of carbon black. Moreover, the content of carbon black is required to range between 2 and 3 weight per cent. Carbon black in the fused joint is in the same concentration as the carbon black in the pipe, which was extruded in a molten state similar to the fusing process. So, the presence of carbon black in the fused joint should be no more deleterious than in the pipe itself. Each of these considerations suggests that the potential negative impacts of carbon black on butt fusion are both controlled and limited; the inherent presence of carbon black is of course mandatory. Similarly, the anticipated impact of anti-oxidants on the development fusion joining interface is negligible due to their lower relative concentration and smaller relative size compared to carbon black.

As mentioned in Section 4.7, molten polyethylene compound fronts coalescence into a single material largely through molecular diffusion. Experimental support for material coalescence through diffusion has been summarized [67]. Entanglement in the amorphous regions and co-crystallization into lamellar structures both act to form a new strong and ductile single solid material. All of these processes are general to polyethylene compound and will occur in the manufacturing of HDPE pipes in a highly similar manner as they occur in butt fusion of HDPE pipes. All of these processes are general to polyethylene compound and will also apply equally well to cross-fusion between PE4710 HDPE pipes comprised of two different polyethylene compounds meeting nuclear safety-related requirements.

Historically, industry has recognized [68] the utility in limiting the melt index or HLMI range of HDPE compounds in order to control critical properties during a standardized butt fusion process. A PHMSA website [69] acknowledges this historical precedent when it states that some polyethylene compounds “required separate qualified fusion procedures due to significant differences in melt viscosity compared to other” polyethylene compounds.

Combining the polymer science concepts from Sections 4 and 5 of this report provides greater insight into the reasons behind these melt index (or HLMI) limitations. At low HLMI (such as below the nuclear safety-related minimum of 4 g/10 minutes), polymer science suggests a high weight-averaged molecular weight leading to both an increased melt viscosity and a slower rate of molecular diffusion in the melt at a given melt temperature. Both effects can be overcome by intentional changes to fusion parameters but this runs counter to the intent of a Generic Butt Fusion procedure. Similarly, at high HLMI (such as above the nuclear safety-related maximum of 20 g/10 minutes), polymer science suggests a low weight-averaged molecular weight leading to both a reduced melt viscosity and a faster rate of molecular diffusion in the melt at a given melt temperature. An increased rate of molecular diffusion is not problematic. An adjustment to reduce interfacial pressure is anticipated to retain a proper amount of molten polymer in the fusion zone between the two pipes. Again, however, this adjustment runs counter to the intent of a Generic Butt Fusion procedure. Therefore, nuclear safety-related fusion parameters combine with the limits on HLMI of polyethylene compound to establish a field butt fusion process that is refined based on an understanding of those properties of the HDPE pipe and polyethylene compound essential for formation of a strong and durable fusion joint.

Recent studies [70] have raised a concern regarding dramatic reductions in SCG resistance at the butt fusion joint when compared to the HDPE pipe material. Later work suggests [71] differences in the molecular orientation in the butt fusion area as contributing to reduced SCG resistance. Separate testing has been conducted [72] and suggests that lower SCG resistance in the butt fusion joint "may be due to higher orientation of the crystals in the radial direction". It should be clear that the concern raised relates to the material performance in the joined interface and does not indicate the presence of a notch or flaw in the butt fusion joint. Current balloting in Section III paragraph 4451(a) states "Indications of defects in fusion joints detected by the examinations required by nn-5000, or by the tests of nn-6000, shall cause rejection of the joint. Repair of a fused joint is not permitted. All unacceptable joints shall be removed and replaced."

6
ENGINEERING CONSIDERATIONS RELATED TO BUTT FUSION OF HDPE PIPES

6.1 Introduction

ASME Section IX has approved Articles XXI-XXIV for butt fusion of HDPE pipe. Three additional essential variables have been identified for nuclear safety-related applications; HDPE pipe Cross-sectional Area, the Fusion Machine Model Number and the ambient temperature. Prior to considering the 3 additional essential variables, it is appropriate to discuss in detail the specific PE4710 materials which are both listed in PPI TR-4 and confirm in their product literature that they meet several nuclear safety-related requirements. This group of polyethylene compounds are found to range more narrowly in density and melt index than the range allowed in nuclear safety-related application. This conclusion has the potential to add clarity to a brief overview of the butt fusion process and the existing essential variables.

6.2 Specifics of Polyethylene Compounds that Meet Code Case Requirements

In the previous Section, consideration has been given to the polyethylene compound, the HDPE pipe, the manufacturing of HDPE pipe and its similarities to butt fusion joining in general terms. It is appropriate to bring our examination full circle and return to a more narrow focus on those polyethylene compounds that meet several of the requirements for nuclear safety-related applications.

First, PPI TR-4 was reviewed for those polyethylene compounds listed as PE4710 materials with a listed HDB of 1000 psi at 140°F in addition to a listed HDB of 1600 psi at 73°F as discussed in Chapter 2. This creates a finite list of polyethylene compounds. Publically available product literature for these polyethylene compounds was reviewed to ensure that the TR-4 listed compound contains between 2 and 3 weight per cent carbon black. Further review of the publically available product literature allows for the selection of those polyethylene compounds that are identified as meeting an ASTM F1473 PENT minimum requirement of > 2000 hours. These criteria lead quickly to a small group of polyethylene compounds with the potential to meet the full requirements for nuclear safety-related applications. The identity of these polyethylene compounds are codified in this document to allow for more detailed review without implying any endorsement.

Table 6-1 contains a summary of publically available product literature data for this small group of polyethylene compounds. While nuclear safety-related requirements limit the density of unpigmented polyethylene compound to a range between 0.947 to 0.955 g/ cm^3, the potentially compliant PE4710 materials range more narrowly in their listed density (0.9485 – 0.949 g/ cm^3). Similarly, the density of the polyethylene compound (including the carbon black) is allowed in nuclear safety-related requirements to range from 0.956 to 0.968 g/ cm^3. Those materials in

Table 6-1 which report this value, all report the single value of 0.959 g/ cm^3. This leads to the inference that other properties with a strong relationship to density are likely to also fall in a narrow range. This relationship was noted in Chapter 2 for mechanical properties such as flexural modulus, and tensile strength at yield while the relationship between density and thermal properties such as polymer melting temperature, polymer softening temperature, and heat capacity was discussed in Chapter 4.

A similar situation is found for HLMI values reported in Table 6-1. Nuclear safety-related requirements limit polyethylene compounds to a value between 4 and 20 g/10 min. However, the reported values range more narrowly from 5.5 to 9.0 g/10 min. Similarly, the melt index of the polyethylene compound is allowed by nuclear safety-related requirements to be any value less than 0.15 g/10 min. All of the reported values in Table 6-1 range narrowly from 0.04 to 0.08 g/10 min. This leads to the inference that other properties with a strong relationship to melt index are likely to also fall in a narrow range. A relationship between melt index and the viscosity of the molten polyethylene compound as a function of temperature was discussed in Chapter 4. Some relationship between melt index and M_w values was also noted in Chapter 4. Nuclear safety-related requirements also mandate a Thermal Stability value greater than 220°C. Several of the polyethylene compounds are indicated as complying with this requirement and one material reports a value of 250°C suggesting more than adequate stabilization for HDPE pipe fabrication. Combination of the process controls provided by the essential variables with the highly similar properties of the eight HDPE compounds that potentially comply with nuclear safety-related requirements provides a technical basis to limit the number of pipe sizes, lots, manufacturing facilities, and resin suppliers used in qualification of the Fusing Procedure Specification.

Table 6-1
Potentially Compliant Nuclear Safety-Related PE4710 Material Properties

Compound Identity	Resin Density (g/cm^3)	Density w/carbon black (g/cm^3)	HLMI (g/10 min.)	Melt Index (g/10 min.)	Thermal Stability (°C)
Material A	0.949	NR	7.0	0.04	250
Material B	0.9485	0.959	8.5	NR	>220
Material C	0.949	NR	9.0	NR	NR
Material D	0.949	NR	9.0	NR	NR
Material E	0.949	0.959	7.5	0.080	>220
Material F	0.949	0.959	5.5	0.060	>220
Material G	0.949	0.959	8.5	0.080	>220
Material H	0.949	0.959	6.8	0.080	>220

Table entries shown as NR are Not Reported in public domain literature on the compounds.

6.3 Butt Fusion Process

The butt fusion joining process is covered in detail in ASTM F2620 and the ASME code. As stated previously, butt fusion of HDPE pipes is highly similar to the PE Pipe Material extrusion process in that heated surfaces at a controlled temperature are brought together by application of a sufficient force which causes the melted materials to flow, mix, and finally fuse. In the specific instance of butt fusion joining, an appropriate machine clamps and aligns two HDPE pipes, a tool is used to make the adjacent surfaces flat and parallel, the surfaces are held flat against a metal heating plate until a melt is formed, then the two molten surfaces are rapidly brought together and held under force while the newly formed joint cools.

The joining of HDPE depends to a great extent on three variables; melt temperature, interfacial pressure applied to establish flow and the time period that the material remains molten. This is reflected in the industry history leading up to the initial establishment of a generic fusion procedure. Recommended industry practice varied considerably because many unique combinations of temperature, interfacial pressure, and cooling time all lead to strong and durable HDPE pipe joints. However, it was counter-productive for the industry to persist in using several slightly different fusion procedures. Therefore, several of the recommended fusion parameter ranges are fairly large since they define a joining process that has broad tolerance of process variations.

Interfacial pressure is defined in ASTM F2620 as a value between 60 and 90 psi that is multiplied by the HDPE pipe cross-sectional area in square inches to obtain the fusion force in pounds force. If a manual fusion device is used, a torque wrench can be utilized to control the fusion force and thereby the applied interfacial pressure. For hydraulic fusion machines, the fusion force is obtained through the same multiplication of HDPE pipe cross-sectional area by a chosen interfacial pressure. In the simplified case of insignificant drag forces, this fusion force is then divided by the total effective piston area of the movable carriage cylinders to give the butt fusion gauge pressure in psi.

6.4 Essential Variables for Butt Fusion

Consider the Essential Variables for Butt Fusion to be separated into two categories; essential variables in which polymer science may play a role and those in which it does not. Table 6-2 contains a list of those Essential Variables which provide few insights to polymer science. For example, Pipe Position and Pipe Surface Alignment have little if any relevance to polymer science although they are traditionally required welding procedure specifications and are useful for butt fusion. Proper surface alignment may be important to distributing melt flow evenly around the joined circumference in addition to even melt bead formation on the inner and outer pipe walls, but these simplistic mechanical requirements are easily met by current fusion equipment design. Only butt fusion joining is currently under consideration for nuclear safety-related applications.

The fusion machine model number again has little to do with polymer science. To be certain, the use of a properly qualified fusion machine is important for the delivery of the proper interfacial pressure. The machine must deliver both the required hydraulic pressure for interfacial pressure and to overcome any drag pressure. Inclusion of the Fusion Machine Model Number as an essential variable is useful for tracking, traceability, management of change and for confirming that the equipment is appropriate to the task of field joining.

Table 6-2
Essential Variables With Few Implications for Polymer Science

Item	Condition	Required Value or Range	Change Limitations	Location of Requirement
1	Pipe Position	Horizontal ± 45°		Code Case N-755-2 Appendix I-221(b)-1
2	Pipe Surface Alignment	Within 10% of the pipe wall thickness		Code Case N-755-2 Appendix I-221(b)-3
3	Joint Type		In Joint Type	Section IX 2013 QF-402.1
4	Position		In the fusion position beyond that qualified	Section IX 2013 QF-404.1
5	Fusing Machine Manufacturer		In the equipment manufacturer from that qualified	Section IX 2013 QF-406.1

Table 6-3 lists Essential Variables in which polymer science has some meaningful role for consideration. It is important to consider each of the essential variables to determine if our consideration of the material science of HDPE provides any insights into the appropriateness of values, ranges or trends.

In Section IX, butt fusion joining is considered for a broad range of polyethylene compounds for use in a variety of general industrial applications. Table 6-3 Item 1 seeks to limit changes in polyethylene compounds used to manufacture pipe. Significant additional requirements are placed on polyethylene compounds for nuclear safety-related applications. Sections 2 and 4 of this document have indicated that the limited range of HLMI values for nuclear safety-related applications restricts the potential range of polymer viscosities during butt fusion fabrication. It was noted in Section 5 that industry past practice has similarly attempted to limit the materials addressed by generic fusion practices to those materials with an appropriate range of HLMI values. As discussed in Section 6-1, the polyethylene compounds which have the potential to meet nuclear safety-related requirements are available in only a narrow range of HLMI and density values which should limit concerns regarding butt fusion between HDPE pipes made from “dis-similar” materials. In summary, the polyethylene compounds which currently meet nuclear safety-related applications are quite similar in HLMI and density which acts to restrict important melting, crystallization and viscosity properties within limited ranges.

Throughout this document, the necessity of pressure, time and melt temperature to form a durable bond between polyethylene compound interfaces has been emphasized. It is entirely appropriate that some value for pressure should be controlled such as Item 2, Interfacial Pressure. Interfacial pressure is multiplied by the HDPE pipe cross-sectional area to generate a value for force that is applied during joining with the viscosity of the molten polyethylene compound providing the opposing force.

Similarly, heater plate surface temperature (Item 3, Table 6-3) seems an entirely appropriate process control variable for obtaining molten HDPE. The temperature range of 400 to 450°F is substantially above the melting point of HDPE noted as approximately 130°C (or 266°F) in

Section 4. It is interesting to note the similarity of this temperature range to that used in HDPE Pipe product manufacturing noted in Section 5 (100 to 200°F above melting point or approximately 360 to 460°F). It was also noted in Section 4 that specific additives are included in the polyethylene compound in order to stabilize melt processing in this temperature range. The HDPE pipes allowed in nuclear safety-related applications were discussed in Section 2 as being required to meet a minimum value of Thermal Stability provided by the additives. The relatively high rates of thermal conductivity of metals and the relatively low rates of thermal conductivity of polyethylene compound noted in Section 4 suggests that heat will rapidly flow into the polyethylene compound at the point of contact with the metal heater plate but then will take some time to fully melt a volume of polyethylene compound. Note that "Minimum Melt Bead Width during Heat Soak" (Item 4, Table 6-3) has a range of specified values which depend on both HDPE pipe OD and wall thickness. Section 6.6 will return to the topics of HDPE Pipe OD and "Pipe Cross-Sectional Area" (Item 10, Table 6-3).

The "Maximum Heater Removal Time" (MHRT) considers two factors that relate to the rate of cooling of the fused pipe. The MHRT in the field (Item 5, Table 6-3) starts at 8 seconds and increases to 30 seconds as the size of the pipe wall increases. Similarly, the MHRT in a manufacturer facility (Item 6, Table 6-3) increases as the size of the pipe wall increases and is approximately double the MHRT for the same pipe size as in the field. The insulating capability (i.e. low thermal conductivity) of HDPE has been noted in Section 5 and thicker pipe wall should result in longer cooling times to fully remove the heat. Therefore, the amount of time allowed to remove the heater and bring the two melt fronts together can be expanded. Additionally, the approximate doubling of the MHRT in a manufacturing facility recognizes that a manufacturing facility may often be warmer than field conditions. Since the two different locations for the butt fusion operation are noted as having potentially different cooling rates, Item 7 in Table 6-3 seeks to limit changes between the two locations without further qualification work. Since the difference in melt temperature and ambient temperature (Table 6-3, Item 11) is the driving force for convective cooling, this difference in MHRT is in a direction that is supported by the heat exchange capabilities of polyethylene compound.

The "Cooling Time Under Fusion Pressure" (Item 8, Table 6-3) is indicated as a range which increases with the pipe diameter and that cooling times at the higher end of the range are needed when the Pipe Wall Thickness (Item 9, Table 6-3) exceeds two inches. The insulating capability (i.e. low thermal conductivity) of polyethylene compound has been noted in Section 4 with larger sizes of HDPE pipe requiring longer cooling times to fully remove the heat and are therefore supported by current understanding of the heat exchange capabilities of polyethylene compound. In Section 4.5, the critical role of entanglements and tie-molecule formation to SCG performance was noted. In Section 4.6, it was noted that foreign particles, molecular orientation and rate of cooling can play a role in SCG performance. The considerations of polymer science therefore suggest that a reproducible butt fusion process must allow time for tie-molecule structures to develop, be free from foreign particles, have a rate of cooling that is within a controlled range and that the rate of polymer flow is controlled. The heat fusion protocols established within the code accomplishes these key requirements. Highlighted in Section 5.3, investigations into the nature and extent of molecular orientation within the polymer fusion zone are worthy of continued investigation.

Table 6-3
Essential Variables with Meaningful Polymer Science Implications

Item	Condition	Required Value or Range	Change Limitations	Location of Requirement
1	PE Material		To other than listed in Table QF-422	Section IX 2013 QF-403.1
2	Interfacial Pressure		Beyond the range qualified	Section IX 2013 QF-405.2
3	Heater Surface Temperature		Beyond the range qualified	Section IX 2013 QF-405.2
4	Minimum Melt Bead Width during Heat Soak	Depends on pipe OD		Code Case N-755-2 Appendix I-221(b)-7 and Table I-221(b)-1 and I-222(d)
5	Maximum Heater Removal Time (Field)	Depends on pipe wall thickness		Code Case N-755-2 Appendix I-221(b)-9 and Table I-221(b)-2 and I-222(e)
6	Maximum Heater Removal Time (Manufacturer Facility)	Depends on pipe wall thickness		Code Case N-755-2 Appendix I-221(b)-9 and Table I-221(b)-2 and I-222(e)
7	Location of Fusing, Shop or Field		In the location of Fusing Operations from that qualified	Section IX 2013 QF-407.1
8	Cooling Time Under Fusion Pressure	Depends on wall thickness		Section IX QF221.1(l)
9	Pipe Wall Thickness		Beyond the range qualified	Section IX 2013 QF-403.3
10	Pipe Cross-sectional Area		Beyond the range qualified	Section IX 2013 QF-403.4
11	Ambient Temperature		Additional procedures when < 50°F or > 125°F	Code Case N-808 Section 4111 and Section III Mandatory Appendix NN Ballot

6.5 The Essential Variable for Ambient Temperature

As discussed in Section 5, providing polyethylene compound at the proper melt temperature for fusion is a crucial element in ensuring that sufficient time is available for entanglement in the amorphous regions and co-crystallization into lamellar structures to form a single solid material.

The heater plate temperature is already noted as an essential variable. It is important to note that the narrow range in the density of the polyethylene compounds with the potential to meet the requirements for nuclear safety-related applications implies a narrow range in heat capacity, rate of heat loss and similar cooling times for these compounds at practical ambient temperature.

However, if ambient temperature is 50°F or lower, then the total rate of cooling from both conduction and convection of the HDPE pipe butt fusion joint is likely to be accelerated. This may not allow sufficient time for entanglement in the amorphous regions and co-crystallization into lamellar structures to fully develop into a single solid material. It has also been stated that the process of molecular diffusion which develops a strong bonding interface between two HDPE interfaces operates on a similar time-scale as the development of the key tie molecule structures so important for slow crack growth resistance. Accelerated cooling may thus affect the strength, ductility and durability of joined HDPE pipe.

Conversely, if the ambient temperature is greater than 100°F, then the total rate of cooling from both conduction and convection of the HDPE Pipe product involved in butt fusion joining is likely to be delayed. Slower cooling should not interfere with the availability of sufficient time for entanglement in the amorphous regions and co-crystallization into lamellar structures to form a single solid material to fully develop. However, premature movement of a joint which has not cooled sufficiently may affect the strength, ductility and durability of the joined materials.

Requirements related to ambient temperature are formalized in Code Case N-808 section 4111 as "Fusing shall not be performed at ambient temperatures less than 50^{o}F (10^{o}C), unless an environmental enclosure is used to control work area temperature at or above 50^{o}F (10^{o}C)". The balloted language for Section III goes slightly further in nn-4412 "Fusing shall not be performed at ambient temperatures less than 50°F (10°C) or greater than 125°F (52°C), unless an environmental enclosure is used to control work area temperature between 50^{o}F (10^{o}C) and 125°F (52°C). For ambient fusing temperatures between 100°F (38°C) and 125°F (52°C), minimum cooling time shall be 13 minutes per inch of thickness". This seems appropriate based on an understanding of heat transfer.

6.6 Cross-sectional Area as an Essential Variable

The inclusion of an essential variable for Pipe Cross-section Area (Item 10, Table 6-3) for nuclear safety-related joining of HDPE pipe deserves further review. Pipe Wall Thickness (Item 9, Table 6-3) and Pipe Cross-sectional Area (CSA) are considered essential variables but HDPE pipe OD is not considered an essential variable. Clearly all three concepts are mathematically inter-related and several items in Table 6-3 relate to either HDPE pipe OD or wall thickness.

$$CSA = t\pi(OD - t) \quad \textbf{Eq. 6-1}$$

Industry sources agree on the use of a simple equation to obtain the same value for CSA as shown in Equation 6-1. This equation assumes a perfect circular shape although HDPE pipe in the field may vary slightly from perfectly circular.

The inclusion of CSA as an essential variable relates to its use in the calculation of fusion force which is simply the product of interfacial pressure (in psi) multiplied by the CSA (in square inches) leading directly to the calculation of fusion force as pounds-force. In actual practice, hydraulic fusion machine operators must also account for two additional important terms (e.g.

total effective piston area and drag pressure) to calculate the desired gauge pressure. Let us now consider CSA in detail.

6.6.1 Mathematical Analysis of Cross-Sectional Area for 8" IPS DR11

Calculating the CSA using outside diameter and wall thickness dimensions from ASTM F714 is instructive. Consider the example of 8" IPS DR11 at the minimum OD of 8.586 inches and at the minimum wall thickness of 0.784 inch. One can easily calculate the minimum CSA for 8" IPS DR11 HDPE pipe as 19.22 sq. inches (see Table 6-4). At the maximum OD of 8.664 inches at the minimum wall thickness, the CSA increases to 19.41 sq. inches, an increase of approximately 1 per cent. In this context, direct measurement of outside diameter with the intent of understanding variation in CSA is of minor benefit. The ASTM F714 tolerance on OD, which ranges from 0.53 per cent at small diameters to 0.45 per cent on large sizes, fixes the range of CSA values based on allowed OD variation at approximately 1 per cent for all IPS sizes encompassed by ASTM F714.

Minimum wall thickness in ASTM F714 for 8" IPS DR11 is 0.784 inches. In spite of the fact that ASTM F714 has no direct dimensional requirement for maximum wall thickness, every HDPE pipe manufacturer carefully manages their use of raw materials by limiting wall thickness within their own control limits. For the sake of illustration, we will select a maximum wall thickness of 0.878 inches (a wall thickness that is 12% larger than the minimum). At the maximum wall thickness 0.878 inches and the maximum OD of 8.664, the maximum CSA is calculated as 21.48 sq. inches (see Table 6-4). This demonstrates that for 8" IPS DR11 HDPE pipe, the per cent variation from minimum CSA to maximum CSA is essentially equivalent to the per cent wall thickness variation with little contribution from variation in the OD. This relationship is maintained regardless of OD dimension across all sizes in ASTM F714.

Table 6-4
Values Related to Cross-Sectional Area Analysis for 8" IPS DR11

Outside Diameter (inches)	Wall Thickness (inches)	Cross-sectional Area (sq. in.)	Percent Greater than Minimum Area	Comment
8.586	0.784	19.22	-	Min. wall Min. OD
8.664	0.784	19.41	1.0	Min. wall Max. OD
8.664	0.878	21.48	11.8	Max. wall Max. OD

6.7 Conclusions and Recommendations

The following conclusions result from a consideration of polymer science as applied to polyethylene compound.

- A correlation exists between flexural modulus values and Resin Density. The range of Resin Density allowed by nuclear safety-related requirements predicts a range of flexural modulus values that is consistent with Code Case requirements. (Section 2)

- A correlation exists between tensile strength at yield and Resin Density. The range of Resin Density allowed by nuclear safety-related requirements predicts a range of tensile strength at yield values that is consistent with Code Case requirements. (Section 2)

The following conclusions result from a consideration of polymer science as applied to butt fusion of HDPE pipes.

- HDPE pipe manufacturing processes re-combine various melt strands under the combined conditions of heat, pressure and time to form a high integrity HDPE pipe in strong parallel to butt fusion process. (Section 4)
- The ranges for HLMI and density allowed under current requirements for nuclear safety-related applications are significantly larger than anticipated batch variability of polyethylene compound. (Section 5)
- Industry has historically used melt index (or HLMI) ranges to control melt viscosity in relation to generic butt fusion procedures in strong parallel to nuclear safety-related requirements. (Section 5)
- Publically available product literature indicates several HDPE compounds are available that are potentially compliant with nuclear safety-related requirements. (Section 6)
- Resin Density and HLMI reported for potentially compliant HDPE compounds fall within a range substantially more narrow than that mandated by nuclear safety-related requirements. (Section 6)
- Mathematical analysis of the HDPE pipe Cross-sectional Area suggests that the likely variability in cross-sectional area is approximately the same magnitude as the variation allowed through the essential variable Pipe Surface Alignment. (Section 6)

7
REFERENCES

---

1. ASME Code Case N-755-2, "U*se of Polyethylene (PE) Plastic Pipe for Section III, Division 1, Construction*" ASME Boiler and Pressure Vessel Code, 2013.
2. ASTM D3350, "*Standard Specification for Polyethylene Plastics Pipe and Fittings Materials*" ASTM International, West Conshohocken, PA, 2010.
3. ASTM D4703, "*Standard Practice for Compression Molding Thermoplastic Materials into Test Specimens, Plaques, or Sheets*" ASTM International, West Conshohocken, PA, 2008.
4. ASTM D1505, "*Standard Test Method for Density of Plastics by the Density-Gradient Technique*" ASTM International, West Conshohocken, PA, 2010.
5. ASTM D792, "*Standard Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement*" ASTM International, West Conshohocken, PA, 2008.
6. ASTM D4883, "*Standard Test Method for Density of Polyethylene by the Ultrasound Technique*" ASTM International, West Conshohocken, PA, 2008.
7. ASTM D1238, "*Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer*" ASTM International, West Conshohocken, PA, 2010.
8. ASTM D790, "*Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials*", ASTM International, West Conshohocken, PA, 2010.
9. J. Janzen and D. F. Register, "*Crystallinity Dependence of Modulus and Yield Properties in Polyethylenes*", SPE ANTEC Proceedings, 1996.
10. ASTM D638, "*Standard Test Method for Tensile Properties of Plastics*", ASTM International, West Conshohocken, PA, 2010.
11. ASTM F1473, "*Standard Test Method for Notch Tensile Test to Measure the Resistance to Slow Crack Growth of Polyethylene Pipes and Resin*s", ASTM International, West Conshohocken, PA, 2007.
12. ASTM D1598, "*Standard Test Method for Time-to-Failure of Plastic Pipe Under Constant Internal Pressure*", ASTM International, West Conshohocken, PA, 2009.
13. ASTM D2837, "*Standard Test Method for Obtaining Hydrostatic Design Basis for Thermoplastic Pipe Materials or Pressure Design Basis for Thermoplastic Pipe Products*", ASTM International, West Conshohocken, PA, 2008.
14. PPI TR-4, "*PPI Listing of Hydrostatic Design Basis (HDB), Hydrostatic Design Stress (HDS), Strength Design Basis (SDB), Pressure Design Basis (PDB), and Minimum Required Strength (MRS) Ratings for Thermoplastic Piping Materials or Pipe*", Plastics Pipe Institute, Irving, TX, 2010.

15. PPI TR-3, "*Policies and Procedures for Developing Hydrostatic Design Basis (HDB), Pressure Design Basis (PDB), Strength Design Basis (SDB), and Minimum Required Strength (MRS) Ratings for Thermoplastic Piping Materials or Pipe*", Plastics Pipe Institute, Irving, TX, 2010.
16. PPI TR-9, "*Recommended Design Factors and Design Coefficients for Thermoplastic Pressure Pipe*", Plastics Pipe Institute, Irving, TX, 2002.
17. ASTM D3035 "*Standard Specification for Polyethylene (PE) Plastic Pipe (DR-PR) Based on Controlled Outside Diameter*", ASTM International, West Conshohocken, PA, 2010.
18. ASTM F714 "*Standard Specification for Polyethylene (PE) Plastic Pipe (SDR-PR) Based on Outside Diameter*", ASTM International, West Conshohocken, PA, 2010.
19. AWWA C901 "*Standard for Polyethylene (PE) Pressure Pipe and Tubing, 1/2 in. (13 mm) through 3 in. (76 mm), for Water Service*" Denver, CO, 2008.
20. ASTM D4218, "*Standard Test Method for Determination of Carbon Black Content in Polyethylene Compounds By the Muffle-Furnace Technique*", ASTM International, West Conshohocken, PA, 2008.
21. ASTM D1603, "*Standard Test Method for Carbon Black Content in Olefin Plastics*", ASTM International, West Conshohocken, PA, 2006.
22. J. B. Howard, H. M. Gilroy "*Natural and Artificial Weathering of Polyethylene Plastics*" Polymer Engineering & Science, Volume 9, Issue 4, pages 286–294, July 1969.
23. H. M. Gilroy "*Polyolefin Longeveity for Telephone Service*" SPE ANTEC Proceedings, 1985.
24. ASTM D746, "*Test Method for Brittleness Temperature of Plastics and Elastomers by Impact*", ASTM International, West Conshohocken, PA, 2007.
25. AWWA C906 "*Polyethylene (PE) Pressure Pipe and Fittings 4 In. (100 mm) Through 63 In. (1,600 mm) for Water Distribution and Transmission*" Denver, CO, 2007.
26. ASTM D2513, "*Standard Specification for Polyethylene (PE) Gas Pressure Pipe, Tubing, and Fittings*", ASTM International, West Conshohocken, PA, 2013.
27. ASTM D3895, "*Standard Test Method for Oxidative-Induction Time of Polyolefins by Differential Scanning Calorimetry*", ASTM International, West Conshohocken, PA, 2007.
28. ASTM D2122, "*Standard Test Method for Determining Dimensions of Thermoplastic Pipe and Fittings*", ASTM International, West Conshohocken, PA, 2010.
29. ASTM D1599, "*Standard Test Method for Resistance to Short-Time Hydraulic Pressure of Plastic Pipe, Tubing, and Fittings*", ASTM International, West Conshohocken, PA, 2005.
30. ASTM D5296, "*Standard Test Method for Molecular Weight Averages and Molecular Weight Distribution of Polystyrene by High Performance Size- Exclusion Chromatography*", ASTM International, West Conshohocken, PA, 2011.
31. Figure adapted from "*Basic Properties of HDPE and Material Classification*" presented by Dr. Bryan E. Hauger at the Use of HDPE for Power Plant Piping Systems Workshop, Charlotte, NC, June 2010.

32. F. W. Bailey and W. M. Whitte, "*Ethylene polymer blends*" U.S. Patent 4461873, July 1984.
33. Figure adapted from "*Environmental Stress Crazing*" Author, A. Lustiger page 306 in "Characterization and Failure Analysis of Plastics" ASM International, 2003.
34. Figure abstracted from "*Basic Properties of HDPE and Material Classification*" presented by Dr. Bryan E. Hauger at the Use of HDPE for Power Plant Piping Systems Workshop, Charlotte, NC, June 2010.
35. Figure 2 abstracted from P. J. DesLauriers, et. al "*Novel Phillips Loop-Slurry based Polyethylene Resins using Modified Chromium Catalysts for High Performance Pipe Applications*" SPE ANTEC Proceedings, 2005, p. 3172.
36. Y. Huang, N. Brown "*The Effect of Molecular Weight on Slow Crack Growth in Linear Polyethylene Homopolymers*", Journal of Materials Science, Vol. 23, pp. 3648-3655.
37. Figure 2 abstracted from Z. Zhou, et. al "*Temperature Effects on Slow Crack Growth in Pipe Grade PE*", SPE ANTEC Proceedings, 2010, p. 680.
38. N. Brown, X, Lu, Y, Huang, I. Harrison and N. Ishikawa "*The Fundamental Material Parameters that Govern Slow Crack Growth in Linear Polyethylenes*", Plastics Rubber and Composites Processing and Applications, 1992, pp 255-258.
39. Z. Zhou, A. Caratus, A. Chudnovsky, W. Michie "*Characterization of Slow Crack Growth in HDPE under Creep Condition*", SPE ANTEC Proceedings, 2008.
40. McGrath, T. J., Moore, I. D., Hsuan, G. Y., "*NCHRP Report 631— Updated Test and Design Methods for Thermoplastic Drainage Pipe*", Transportation Research Board, Washington, D.C., 2009.
41. ASTM D3849, "*Standard Test Method for Carbon Black—Morphological Characterization of Carbon Black Using Electron Microscop*y", ASTM International, West Conshohocken, PA, 2013.
42. ASTM D6556, "*Standard Test Method for Carbon Black-Total and External Surface Area by Nitrogen Adsorption*", ASTM International, West Conshohocken, PA, 2010.
43. ASTM D2414, "*Standard Test Method for Carbon Black—Oil Absorption Number (OAN)*", ASTM International, West Conshohocken, PA, 2013.
44. Figure 1 abstracted from R. E. King III, "*Advances in Polyolefin Stabilization*", SPE ANTEC Proceedings, 1999.
45. Figure abstracted from P. Jacoby, "*The Effect of Hindered Phenol Stabilizers on Oxygen Induction Time (OIT) Measurements,* and *The Use of OIT Measurements to Predict Long Term Thermal Stability*" obtained at http://www.pstc.org/files/public/Jacoby.pdf.
46. Figure abstracted from J. Wu, K.T. Dinh, T. L. Street, R. W. Hedrick, L. L. Ferrarese, B. P. Gilmartin, "*Phenolic Phosphite containing Photoconductors*", U.S. Patent 8507161 B2, August 2013.
47. NSF/ANSI 61, "*Drinking Water System Components - Health Effects*", NSF International, Ann Arbor, MI, 2012.

48. Figure 4a abstracted from M. Ansari, S. G. Hatzikiriakos, A. M. Sukhadia and D. C. Rohlfing, "*Melt Fracture of Linear Polyethylenes: Molecular Structure and Die Geometry Effects*" SPE ANTEC Proceedings, 2009, p. 12.
49. ASTM D3418, "*Standard Test Method for Transition Temperatures and Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry*", ASTM International, West Conshohocken, PA, 2012.
50. Figure abstracted from Handbook of Polyethylene Pipe. First Edition. Plastics Pipe Institute, 2006. Page 83.
51. ISO 3146, "*Plastics – Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers by capillary tube and polarizing-microscope methods*", International Organization of Standardization, Geneva, Switzerland, 2000.
52. M. Varma-Nair, M.G. Williams and N.R. Dharmarajan "*Structure-Property Relationships in Metallocene Based Ethylene-Octene Plastomers*" SPE ANTEC Proceedings, 2004.
53. An example is found online at http://www.engineeringtoolbox.com/thermal-conductivity-d_429.html.
54. B. Wunderlich, "*The ATHAS Database on Heat Capacities of Polymers*" Pure &App. Chem. 1995, No. 6, pp. 1019-1026.
55. J. Vlachopoulos and D. Strutt, "*Basic Heat Transfer and Some Applications in Polymer Processing*" obtained at http://www.polydynamics.com/heat_transfer_revised.pdf.
56. Ch. Bonten, "*A New Hypothesis To Describe The Acting Mechanisms In A Welded Joint of Semi-crystalline Thermoplastics*", SPE ANTEC Proceedings, 1999.
57. C. Rauwendaal, "*Mixing in Extrusion Part One, General Considerations*" SPE ANTEC Proceedings, 2002.
58. Image abstracted from website location www.seekpart.com/company/53294/products/201253095495923691906021.html
59. A.-S. Lichtlé, L. Meylheuc, D. Kucharavy, E. Caillaud, "*Design of PVC Pipes for shock resistance*", SPE ANTEC Proceedings, 2005.
60. Chris Rauwendaal, "*Recent advances in barrier screw design*", Elsevier Ltd., Plastics Additives & Compounding Magazine, September/October 2005.
61. Image abstracted from S. L, Crawford, S. R. Doctor, A. D. Cinson, M. W. Watts, S. E. Cumblidge, T. E. Hall, and M. T. Anderson, "*Assessment of NDE Methods on Inspection of HDPE Butt Fusion Piping Joints for Lack of Fusion*", Office of Nuclear Regulatory Research and Pacific Northwest National Laboratory, 2012.
62. ISO 18553, "*Method for the assessment of the degree of pigment or carbon black dispersion in polyolefin pipes, fittings and compounds*" International Organization of Standardization, Geneva, Switzerland, 2002.
63. CSA B137.0-13 "*Definitions, general requirements, and methods of testing for thermoplastic pressure piping*", Canadian Standards Association, Mississauga, Ontario, 2013.
64. C.-K. Jen, Z. Sun, and M. Kobayashi "*Ultrasonic Monitoring of Barrel Wear and Screw Status*", SPE ANTEC Proceedings, 2004.

65. A. W. Christie, “*Using Thermal Process Imaging for Extruder Analysis and Improvement*”, ANTEC Proceedings, 2005.
66. ASTM F2620 “*Standard Practice for Heat Fusion Joining of Polyethylene Pipe and Fittings*”, ASTM International, West Conshohocken, PA, 2012.
67. S. Wu, “*Polymer Interface and Adhesion*”, Dekker, New York, 1982.
68. PPI TR-33, “*Generic Butt Fusion Joining Procedure for Field Joining of Polyethylene Pipe*” Plastics Pipe Institute, Irving, TX, 2012.
69. Quotation abstracted from website location http://phmsadotc.sabanow.net/prodcontent/cninv000000000001033/tj_03.htm
70. D.-J. Shim, P. Krishnaswamy, E. Focht, “*Comparison of Parent and Butt Fusion Material Properties of High Density Polyethylene*”, Proceedings of the ASME 2009 Pressure Vessels and Piping Conference, July 26-30, 2009, Prague, Czech Republic.
71. S. Kalyanam, D.-J. Shim, P. Krishnaswamy, Y. Hioe, “*Slow Crack Growth Resistance of Parent and Joint Materials from PE4710 Piping for Safety-related Nuclear Power Plant Piping*”. Proceedings of the ASME 2011 Pressure Vessels and Piping Conference, July 17-21, 2011, Baltimore, Maryland.
72. D. D. Keller, “*Analytical Testing on 5" Thick HDPE Butt Fusion Joint*”, Presentation to ASME WG on HDPE Research & Development, August. 2011.

Program:

Nuclear Power

3002003133

Electric Power Research Institute
3420 Hillview Avenue, Palo Alto, California 94304-1338 • PO Box 10412, Palo Alto, California 94303-0813 USA
800.313.3774 • 650.855.2121 • askepri@epri.com • www.epri.com