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Property-processing relationships in linear-low-density and high-density polyethylene

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PROPERTY-PROCESSING RELATIONSHIPS IN LINEAR-LOW-DENSITY AND HIGH-DENSITY POLYETHYLENE

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ABSTRACT

PROPERTY-PROCESS RELATIONSHIPS IN LINEAR-LOW-DENSITY AND HIGH-DENSITY POLYETHYLENE

by Eric W. Leopold

The material properties of two linear-low-density and two high-density polyethylene resins were evaluated to determine the relationship of resin properties to extruded tubing functional properties. The effect of electron beam irradiation on the functional properties of extruded tubing was also investigated.

Material properties tested included molecular weight, density, and melt index. The extrusion functional properties tested were elastic modulus, yield strength, elongation, ultimate tensile strength, and flexural modulus. There was an unexpected decrease in functional properties with increased average number molecular weight and decreased molecular weight distribution. A trend of improved functional properties was observed for increasing density and decreasing melt index. Also found was an increased strength and increased brittleness as an effect of electron beam irradiation.

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TABLE OF CONTENTS

LIST OF FIGURES

Page

Page

LIST OF TABLES

Chapter 1

INTRODUCTION AND BACKGROUND

Heart disease is currently the number one cause of death. A leading cause of heart disease is atherosclerotic plaque. This plaque can cause a blockage in the coronary artery resulting in decreased blood flow to the heart muscle. The coronary artery system consists of three main arteries, the left anterior descending, the right coronary, and the circumflex arteries, and their branches which supply blood to the heart and thereby supply oxygen and nutrients. Loss of blood supply to a section of the heart can cause heart muscle atrophy, potentially leading to a heart attack.

The longstanding means of reinstating blood flow in the heart has been open heart surgery, also known as coronary artery bypass grafting, in which a peripheral vein or artery is removed and grafted into the coronary artery system to circumvent the diseased section of artery. A more recent means of reinstating blood flow is opening of the artery with percutaneous transluminal coronary angioplasty (PTCA), which was introduced in 1977 by Dr. Andreas Gruentzig. The practice of PTCA has grown in recent years and in 1993 approximately 408,000 cases were performed. The main reasons for the quick growth of PTCA include the cost effectiveness of the procedure and the low level of trauma in comparison to open heart surgery.

PTCA consists of inflating a balloon in a coronary artery to open a plaque blockage. The balloon is advanced into the coronary artery system by a catheter system consisting of a guiding catheter, a guide wire, and a coronary balloon catheter. The guiding catheter is inserted into the brachial or femoral artery and advanced to the ostium of the coronary artery. A guide wire is then passed through the guiding catheter and into the diseased coronary artery, where the guide wire is passed through the blockage. Next, the balloon catheter is advanced over the wire and across the coronary blockage into the distal anatomy. The balloon is then inflated to compress and/or crack the plaque to increase the inner and outer diameter of the coronary artery.

The basic design of a balloon catheter consists of a coaxial shaft attached on the proximal end to a luer adapter and distally to a balloon, as shown in Figure 1. The inner lumen of the balloon catheter is for passage of the guide wire. The outer lumen of the catheter is for inflation and deflation of the balloon.

The balloon is inflated using an inflation device filled with contrast (fluid) solution. This allows for visibility under fluoroscopy during the procedure and eliminates the risk of an air embolism if the balloon were to rupture. The inflation pressure required during a procedure is dependent

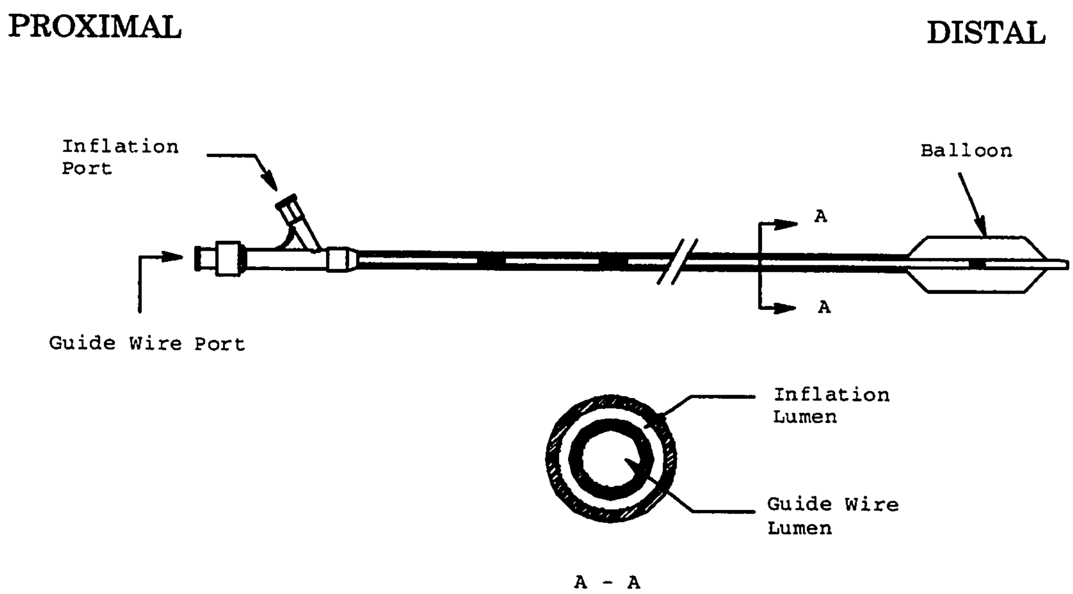


Figure 1: Balloon catheter design.

on the lesion consistency - the more calcified a lesion, the higher the pressure needed. Highly calcific lesions often require pressures as high as 12 to 20 atmospheres. Pressure needs for the majority of cases, however, range from 2 to 10 atmospheres.

The balloon's performance during PTCA procedures is the major determinant of success. The crossing profile of the balloon contributes to the ability of the deflated balloon catheter to pass through the lesion. Improved crossing profiles are achieved in part by decreasing the wall thickness of the balloon. Thin walls also improve the flexibility of the balloon, as does the softness of the material. The flexibility of the balloon aids in the ability of the balloon catheter to track down a tortuous artery to approach a lesion.

The compliance of the balloon is important in setting the inflation strategy. A balloon's compliance curve defines the diameter of the balloon at a given pressure. Samples of compliance curves for multiple balloon sizes of polyethylene balloons are given in Figure 2. Balloons with low compliance curves, such as those shown in Figure 2, have a relatively low pressure-diameter slope to allow for a wide range of pressures to be used while maintaining a narrow diameter range. A low compliance curve limits

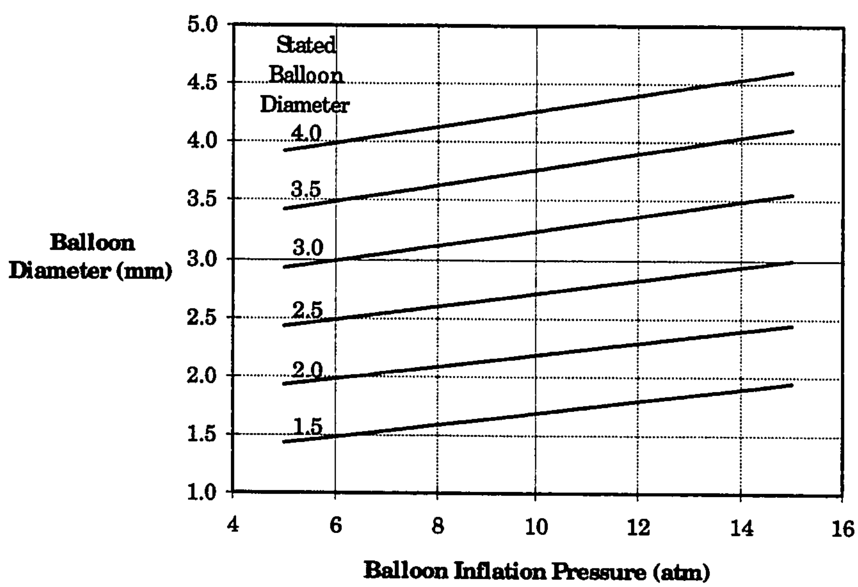


Figure 2: Polyethylene compliance curve.

overextension of the artery when using high pressures. Control of balloon compliance can be achieved by irradiation of the balloon for some materials, such as polyethylene. Irradiation creates a cross-linking of the polymer, thereby limiting the amount of growth during pressurization. A balloon with a high compliance curve, on the other hand, allows for an inflation strategy such that higher pressures can be used to further increase the arterial inner diameter as needed.

The pressure the balloon is capable of withstanding without rupturing contributes to the ability of the balloon to successfully open an artery. When a balloon ruptures, having the correct failure mode is critical if trauma occurring in the artery is to be limited. The least traumatic failure mode is a longitudinal tear. A second failure mode is radial tearing where the tear occurs circumferentially. This failure mode is often traumatic due to a higher chance of dissection (unplanned tearing) of the artery and the possibility of the distal end of the balloon tearing off the catheter during removal of the catheter system. A third failure mode is pin holing of the balloon. This can also be traumatic to patients due to high concentrations of energy on the arterial wall, often creating dissections.

Materials often used for balloons with good all around properties include polyethylene, polyolefin copolymer (POC), and nylon. These materials offer pressure capabilities of 12 to 15 atmospheres with a reasonable wall thickness and crossing profile. They have good flexibility and are essentially free from radial tear and pin hole failures. The compliance curves for these materials generally range from intermediate compliance, allowing for controlled growth during inflations, to a high compliance.

A material often used because of its high pressure capability is polyethylene terephthalate (PET). It can be used to attain pressures in the range of 20 to 30 atmospheres while maintaining a very thin wall thickness. The disadvantage of PET is its high stiffness and greater potential for pin hole failures.

Future balloon materials should have smaller crossing profiles, higher pressures, and improved flexibility. To obtain an improvement in these areas, new materials and new blends of materials need to be considered to balance properties. To assist in the process of determining acceptability of materials, balloon properties can be evaluated by testing of extruded tubing. Functional properties that are measured include elastic modulus and functional modulus, to determine the material stiffness and projected compliance. Also measured are ultimate tensile strength and yield strength, to predict the balloon rupture pressure.

In order to maximize the functional properties for a material, it is necessary to have a firm understanding of how they are affected by material properties and processing steps. The objective of this investigation is to correlate the resin properties of polyethylene to the functional properties of extruded tubing, through theory and experimentation. The effect of electron beam radiation to cause cross-linking of polyethylene will also be studied. These results will aid in the screening of future balloon resins by identifying the critical parameters to evaluate.

Chapter 2, Review of Polyethylene Studies, covers studies performed on various polyethylene materials, evaluating the effect of material properties and electron-beam irradiation on functional properties. The research hypotheses and objectives for this research are presented in Chapter 3. Sample preparation and testing methods are presented in Chapter 4, Research Approach, along with the analysis of the results. The results, discussion, and conclusions, which will present the findings of this study and the conclusions and recommendations based on the results, are contained in Chapters 5, 6, and 7, respectively.

Chapter 2

EFFECT OF RESIN PROPERTIES AND PROCESSING ON POLYETHYLENE

The mechanical properties of polyethylene as a thin film have been shown to be affected by variations in material properties and processing steps. Section 2.1 covers investigations focused on material property variations and Section 2.2 addresses the effects of gamma and electron-beam irradiation.

2.1 Material Property Studies

Thin film polyethylene materials investigated include linear-low-density (LLDPE), low-density (LDPE), and high-density (HDPE). A key difference among these polyethylene material types is the extent and type of branching. A visual illustration of the type of branching in LLDPE, LDPE, and HDPE is given in Figure 3. As illustrated, LDPE has a more highly branched nature than either LLDPE or HDPE, producing less crystalline regions. This decreased crystallinity results in a lower density which correlates to the lowering of the tensile and yield strength of LDPE(1,2).

Ulku Yilmazer(3) investigated the properties of LDPE and LLDPE by testing samples of extruded film made from various percentages of LLDPE

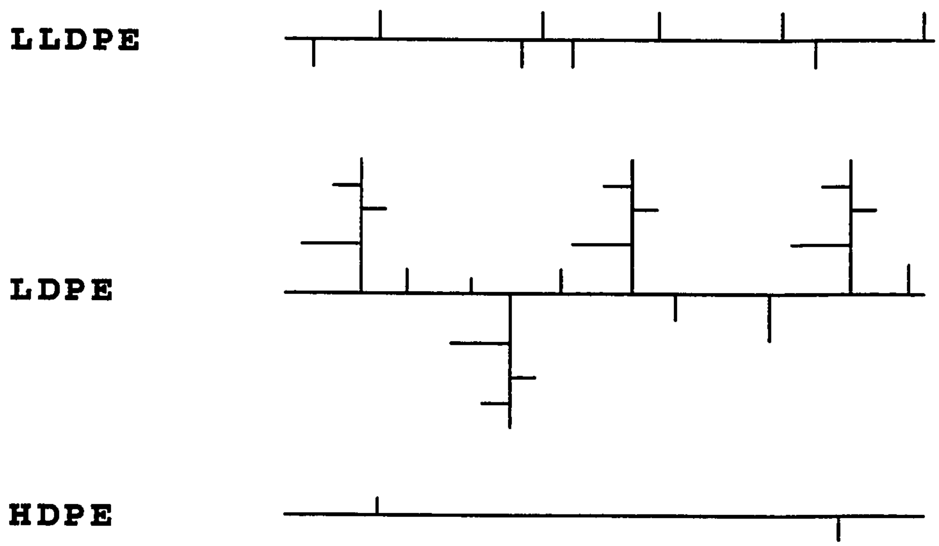


Figure 3: Branching of LLDPE, LDPE, and HDPE

blended with LDPE. This study demonstrated that a higher percent of the LLDPE created a higher tensile strength and strain at break in the machine and transverse directions. In the transverse direction a higher tear strength was also observed. The results of the tests for tensile strength, strain at break, and tear strength are shown in Figures 4 through 6, respectively.

The higher strength for LLDPE is attributed by U. Yilmazer to the lower melt index value for the LLDPE, 1 g/10 min, compared to LDPE, 1.2 g/10 min, which implies a higher molecular weight. The densities for the two materials were listed as 0.923 g/cm^3 and 0.920 g/cm^3 for the LDPE and LLDPE, respectively. It is interesting to note that the effect of additional LLDPE to LDPE seems additive in nature except at levels of twenty to forty percent. This is shown by the non-linear rise in the slopes in Figures 4 through 6 in the 20 to 40 % LLDPE range.

The molecular weight (MW) and the molecular weight distribution

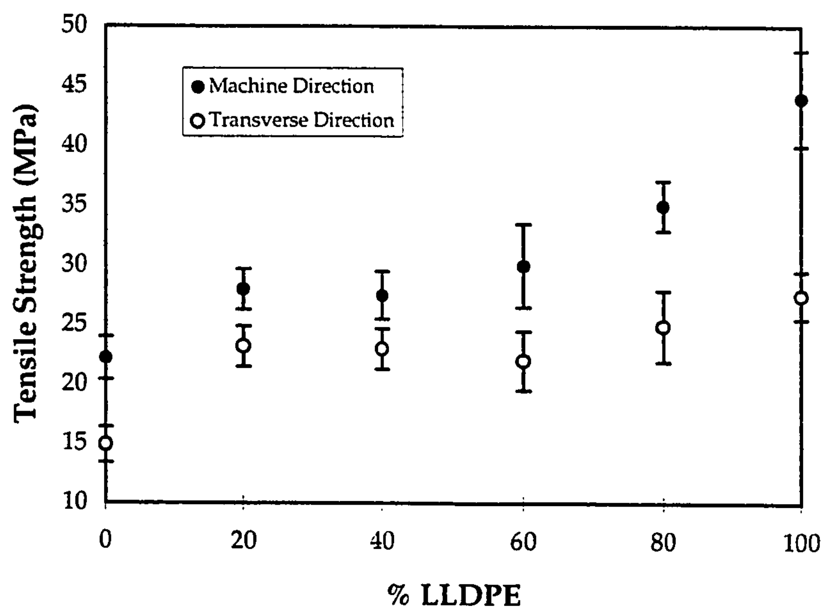


Figure 4: Tensile Strength versus
% LLDPE/LDPE [3]

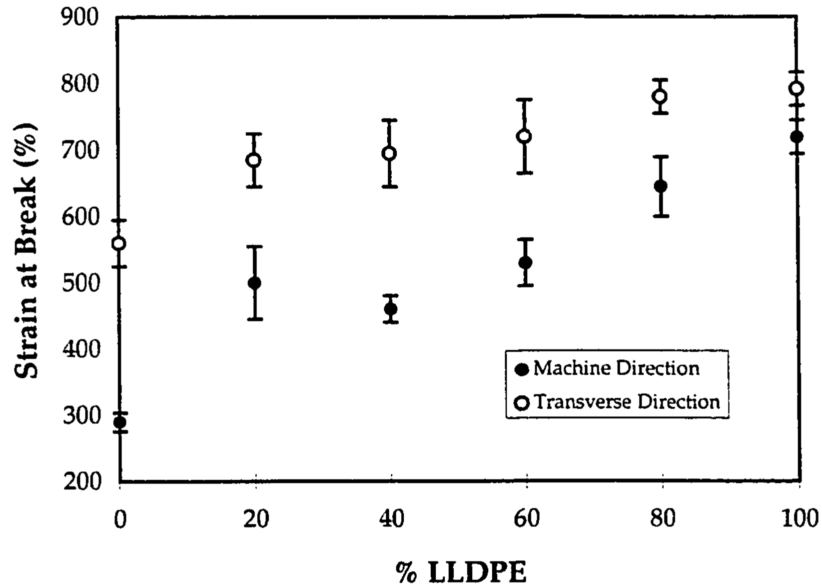


Figure 5: Strain at Break versus
% LLDPE/LDPE (3)

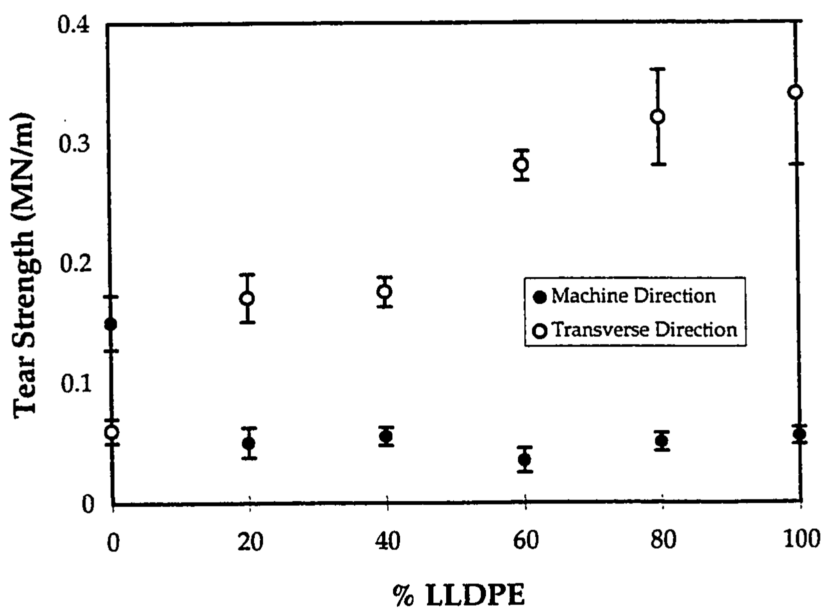


Figure 6: Tear Strength versus
% LLDPE/LDPE (3)

(MWD) are also contributing factors to the functional properties of polyethylene. Edward S. Sherman[4] characterized the effects of varying MW and MWD of LDPE and HDPE on the orientation of the polymer fibers in polymer films. Sherman found that lower MW and narrower MWD polyethylene films had a more uniaxial orientation of stacked lamellar crystals, whereas higher MW and broader MWD polyethylene films consisted of nearly orthotropically oriented crystals. The orientation of fibers in the lower MW and narrower MWD polyethylene were seen to be in the machine direction, i.e. the direction of drawing. The fiber orientation was determined with a scanning electron microscope and film samples.

D.V. Bibee and K.K. Dohrer[5] studied the effects of MW and MWD changes on the physical properties of LLDPE film. Their results showed that a decrease in the melt index, corresponding to an increase in molecular weight, results in an increase in tensile strength and puncture resistance, as shown in Figures 7 and 8, respectively. Also shown in these figures is the effect of decreasing short-chain branching distribution, corresponding to a narrowing MWD, which results in an increase in tensile strength and puncture resistance.

The effect of the MWD on ultra-high-molecular-weight polyethylene (UHMW-PE) films was investigated by Liang Bao Liu et al[6]. They tested two types of UHMW-PE - narrow MWD and broad MWD films. The narrow

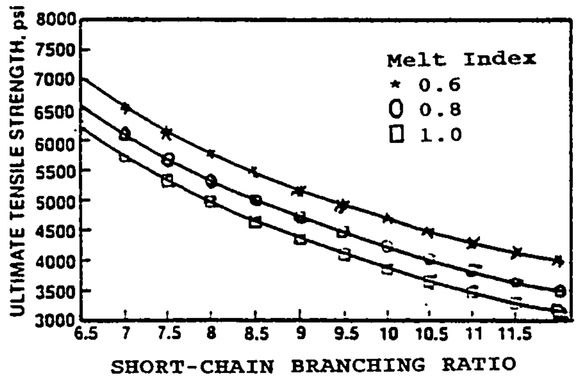


Figure 7: Ultimate Tensile Strength versus chain side branching and melt index[5]

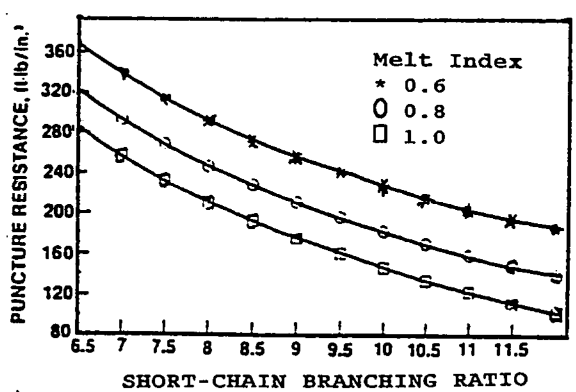


Figure 8: Puncture resistance versus short short chain side branching and melt index[5]

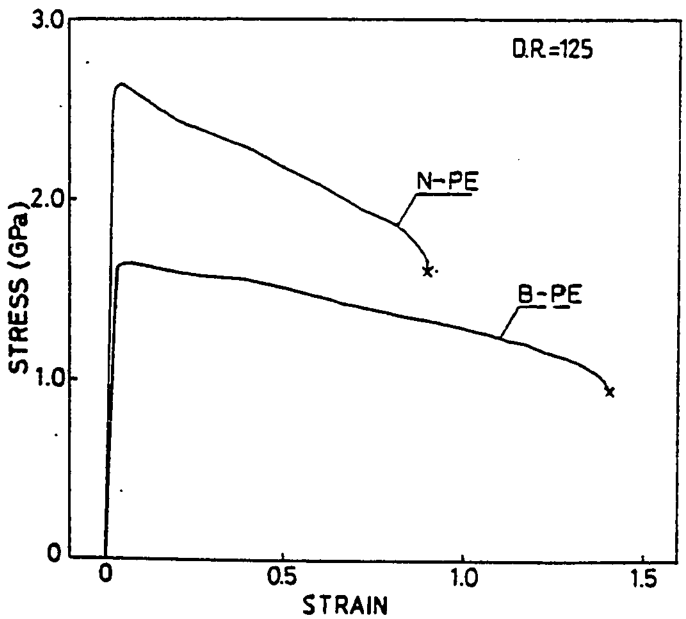


Figure 9: Stress-strain curve for narrow and broad MWD drawn films[6]

MWD film was hypothesized to have some improved mechanical properties due to its higher crystallinity. Tests did confirm that the narrow MWD film had a higher tensile strength than the broad MWD film, but that the narrow MWD film has lower ultimate elongation, as shown in Figure 9. This phenomenon is explained by the presence of long intercrystalline tie chains which are more prevalent in the narrow MWD film. These tie chains absorb larger amounts of energy upon elongation. However, the toughness of both the narrow MWD film and the broad MWD film are similar.

Increased crystallinity has been shown by several investigators to improve mechanical properties, namely tensile strength, strain at break, and tear strength. This increase in mechanical properties can further be obtained by choosing a high molecular weight polyethylene or a narrow MWD.

2.2 Irradiation Studies

Many studies have been performed to evaluate the effect of electron beam irradiation on polyethylene[7,8,9,10,11,12]. A. Valenza et al.[10] used gel fraction analysis to measure the extent of cross-linking during gamma irradiation of LLDPE. Their findings demonstrated an increase in cross-linking due to increased irradiation levels, with gel fractions of 84, 89, and 95 for irradiation levels of 50, 200, and 400 kGy, respectively. Shown in

Figure 10 is the resulting increase in the yield strength for the increasing levels of irradiation.

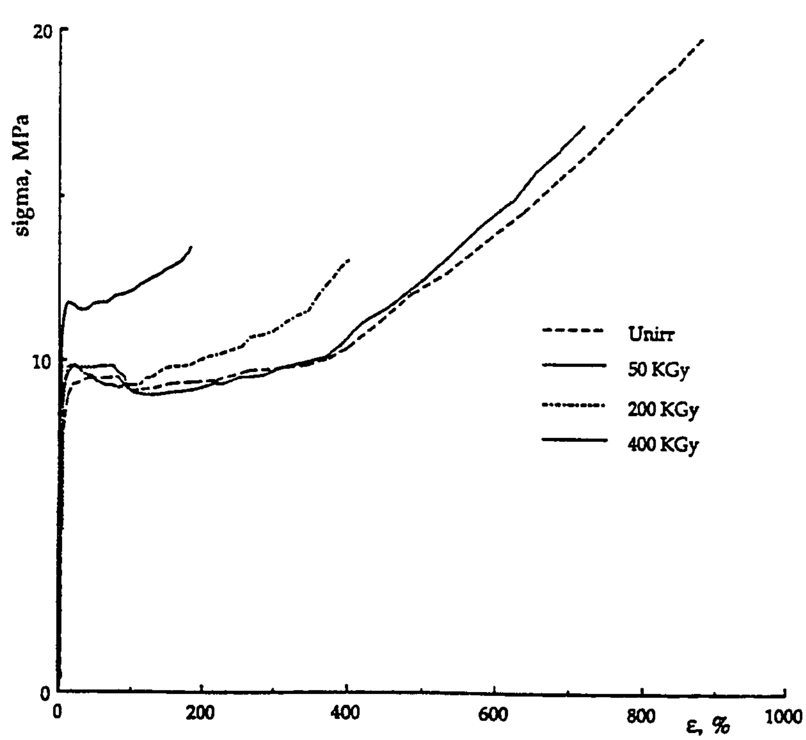


Figure 10: Stress-strain curves for unirradiated and irradiated LLDPE[10]

D.W. Woods and I.M. Ward[11] showed an increase in the gel fraction of HDPE due to gamma irradiation and electron-beam irradiation, as shown in Figures 11 and 12, respectively. The increase in gel fraction was attributed to the attachment of free radicals to polymer chains. These form cross-links by reacting with neighboring radicals. This study further showed chain scission at levels of gamma irradiation above 20 MRad, shown in Figure 11 as a decreasing slope of the gel fraction versus dose curve.

N.H. Ladizesky et al.[12] evaluated the effects of electron beam irradiation and draw ratio on LLDPE. Results of tensile testing displayed small increases in tensile strength for increasing irradiation and large increases in tensile strength for increases in the draw ratio, as shown in Figures 13 and 14, respectively. A further effect of increased brittleness was seen with increasing levels of irradiation, shown in Figure 15 by a reduction in the extension to break for a given draw ratio.

The increase in degree of polyethylene cross-linking with increasing levels of irradiation has been demonstrated by several investigators. The effect of irradiation on material properties include a rise in the tensile strength of the material and an increase in brittleness. At a high level of irradiation chain scission is expected to balance out or surpass the extent of

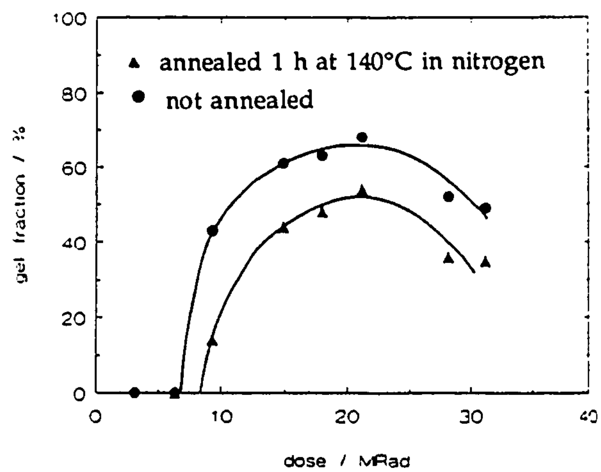


Figure 11: Gel fraction versus dose using gamma irradiation on HDPE[11]

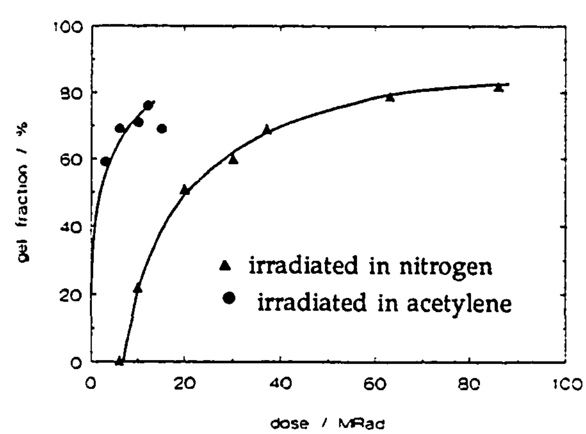


Figure 12: Gel fraction versus dose using electron-beam irradiation on HDPE[11]

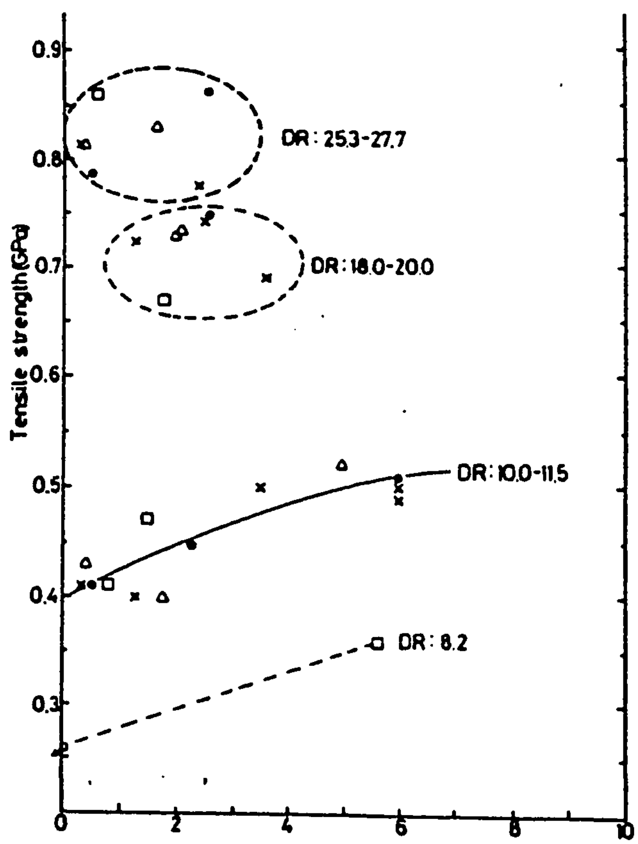


Figure 13: Tensile strength versus irradiation dose for LLDPE[12]

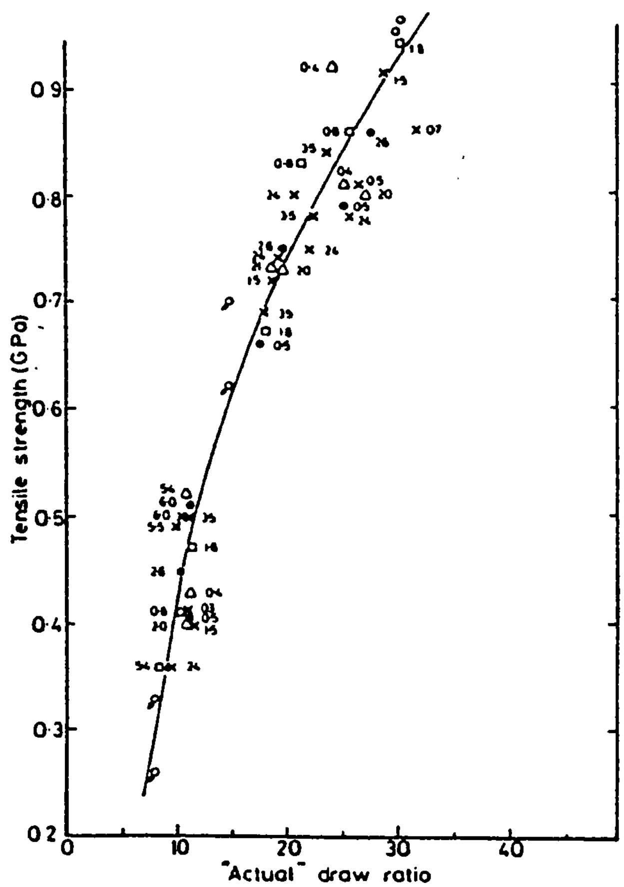


Figure 14: Tensile strength versus draw ratio for LLDPE[12]

cross-linking, potentially causing a decrease in the strength of the material.

While the trends described in this chapter are based on thin film samples, a better understanding of the relationships between processed tubing performance and resin properties is still needed. Also needed is a more thorough analysis that compares the effect of electron-beam irradiation on different material resins and different types of polyethylene, namely LLDPE and HDPE.

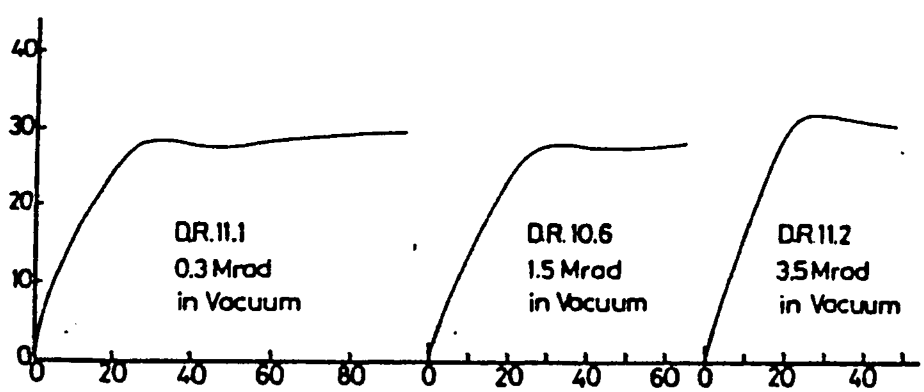


Figure 15: Stress-strain curves for increasing doses of irradiation on LLDPE[12]

Chapter 3

RESEARCH HYPOTHESES AND OBJECTIVES

The balloons used for coronary angioplasty catheters have many desired properties as described in the introduction and background sections. To best meet these needs polyethylene has historically been used as a balloon material. The objective of this investigation is to evaluate different polyethylene resins to correlate material properties to functional properties of the extruded tubing. Material properties to be examined include density, molecular weight, molecular weight distribution, and melt index. Functional properties to be determined include elastic modulus, flexural modulus (stiffness), tensile strength, yield strength, and ultimate elongation. This investigation will also evaluate the effect of increasing electron-beam irradiation on these functional properties of polyethylene.

It is expected that an increase in resin density would have a positive effect on the mechanical properties of the extrusions by increasing the elastic modulus, strength, and elongation. This is expected since ultimate failures occur in amorphous regions of the polymer, which are less abundant in higher density materials. HDPE is expected to have a higher strength and elongation than LLDPE due to its higher density. It is also expected that density variations between the two HDPE resins and the two LLDPE resins would result in strength and elongation variations, namely a higher strength and ultimate elongation for higher density resins.

A high molecular weight and narrow molecular weight distribution is hypothesized to result in a higher strength and elongation of an extrusion than an extrusion from a low molecular weight or broad molecular weight distribution material. This effect from molecular weight is anticipated due to the lower mobility associated with a higher molecular weight. The effect from molecular weight distribution is expected due to shorter chains acting as plasticizers in the polymer matrix. These phenomena are expected within all groups of polyethylene to be tested.

Irradiation of the materials is expected to further influence the mechanical properties of the extrusion. An anticipated effect of irradiation is cross-linking of the material, resulting in higher strength and decreased elongation. At high irradiation levels, however, chain scission can occur resulting in decreased strength and elongation. At the levels used for this experiment, the irradiation is hypothesized to create a sufficient rate of cross-linking to counter the chain scission effect. Therefore, increasing levels of electron-beam irradiation are expected to result in a higher strength and a more brittle material.

Chapter 4
RESEARCH APPROACH

Four different polyethylene resins were used in this study. Two of the resins were linear-low-density polyethylene, designated LLDPE #1 and LLDPE #2, and two were high-density polyethylene, designated HDPE #1 and HDPE #2. Section 4.1 describes the method of measuring the properties of resins. Sections 4.2 and 4.3 describe the techniques for analyzing the extruded tubings and irradiated tubings, respectively. Section 4.4 describes the data analysis technique to be used on the data from extruded and irradiated tubing tests.

4.1 Properties of Resins

The measured properties of the resins are listed in Table 1. This table also lists the general test procedure used for measurements.

TABLE 1: Properties of Resins

Resin Properties	Test Procedure
Molecular Weight	ASTM D3593-80 using GPC
Molecular Weight Distribution	ASTM D3593-80 using GPC
Density	ASTM D792-91 Method B
Melt Index	ASTM D1238-90b Method A, Condition "E"
Bond Characteristics	FTIR Spectrophotometry

The molecular weight and molecular weight distribution were determined using a Waters Associates 150C Gel Permeation Chromatograph (GPC) with a 1 x 10^6 Ultrasyragel HT column and two Linear Ultrasyragel columns at 135°C with trichlorobenzene (TCB) at 1.0 ml/min. A sample quantity of 0.045 g was dissolved in 30 ml of TCB at 150°C and poured into a vial to be injected into the GPC. Testing was performed according to ASTM D3593-80. The data were analyzed using a Waters Associates 845 Chromatography Data Station. Six samples were measured and averaged to obtain number average molecular weight (Mn) and weight average molecular weight (Mw). The polydispersity was then calculated as Mw/Mn.

The density of the resins was measured with a Quantachrome Multipycnometer, by Powder Technology Instrumentation and Services, according to ASTM D792-91, Method B, using helium gas as the displacement medium. Three samples were measured and averaged to obtain the density of each resin.

The melt flow index was measured using a Kayeness Melt Indexer, Model D2051. Testing was performed using ASTM D-1238, Method A, Condition "E", with a 2.16 kg load and a temperature of 190°C. A sample quantity of 6 to 7 g of pellets was used. Three samples for each resin were measured and averaged.

The bond characteristics were determined using a Bio-Rad FTS-40 Fourier Transform Infrared Spectrophotometer. Sample preparation consisted of the use of a heated Carver Press for pressing the films to be scanned. One sample of each resin was evaluated.

4.2 Extruded Tubing Evaluations

Each raw material was extruded into tubing with an outer diameter of 0.040" ± 0.001", an inner diameter of 0.025" ± 0.001", and a length of greater than 1000 feet. The extruded tubing was evaluated according to the test methods listed in Table 2.

TABLE 2: Extruded Tubing Test Methods

Tubing Properties	Test Procedure
Dimensional Analysis	Toolmaker's Microscope
Elastic Modulus	Dynamic Mechanical Analysis
Loss Tangent	Dynamic Mechanical Analysis
Yield Strength	ASTM D638-91
Ultimate Tensile	ASTM D638-91
Ultimate Elongation	ASTM D638-91
Flexibility Test	ASTM D747

The dimensional analysis was performed using a IX adapter with a Zoom 6000 II lens from Do Industries which was connected to a Sony Video Camera CCD-IRIS and Tinitron Color Video Monitor, Model PVM-13428 with cross-hairs. Samples were sectioned and placed on an x-y platform, Model 14-2902, which was connected to a digital readout, Model 12-6682, made by Semprex Corporation. Five samples of each extrusion, pre- and post-irradiation, were measured in two directions for outer diameter and inner diameter. These dimensions were then used to calculate the average cross-sectional area.

The elastic modulus and loss tangent were determined using a Dynamic Mechanical Analyzer RSA-11 by Rheometrics Incorporated. The fiber and film test fixture was used with a 22 mm span. A temperature sweep was performed using the range of -150°C to 100°C. One sample of each extrusion was cut to a length of approximately 30 mm to load in the fixture. Cross-sectional areas were measured for each sample prior to loading into the test fixture. Procedures according to the RSA-11 instruction manual were followed.

The ultimate tensile strength and elongation were determined by tensile testing according to ASTM D638-91. Testing was performed on an Instron Model 4202 manufactured by Instron Corporation. Ten samples of each extrusion were cut into three inch lengths. The test setup consisted of a one inch gauge length, twenty points/second sampling rate, and ten inches/minute crosshead speed. The data were analyzed by a Series IX Automated Materials Testing System 1.15 before conversion into Excel 5.0

for further analysis.

Flexibility testing was performed utilizing a 1 Inch-Pound Tinius Olsen Stiffness Tester, manufactured by Tinius Olsen Testing Machine Company. Testing was performed using ASTM Standard D747-86 modified to use cylindrical samples. Ten samples of each extrusion were cut into lengths of two inches and a 0.045 in. lbs. bending moment and 0.5 in. span was used. The flexural modulus (stiffness), E, was found using the scale reading at 9° angular deflection (.1571 radians). The flexural modulus was calculated, using the average readout for the ten samples tested, with the following equation:

$$E\ (\text{MPa}) = \frac{0.5\ \text{in} \times .045\ \text{in lbs} \times \text{readout}}{3\pi/64 \times (D^4 - d^4) \times 100 \times .1571} \times \frac{1\ \text{MPa}}{145\ \text{psi}} \qquad [1]$$

where D and d are the outer and inner diameters in inches, respectively.

4.3 Irradiated Tubing Evaluations

Approximately three hundred feet of each reel of tubing was irradiated at 30, 50, and 70 MRad. The measurements that were done on irradiated tubing are listed in Table 3. All testing and sample preparation was the same as the testing and sample preparation for the extruded tubing described in section 4.2.

TABLE 3: Irradiated Tubing Test Methods

Irradiated Tubing Properties	Test Procedure
Elastic Modulus	Dynamic Mechanical Analysis
Loss Tangent	Dynamic Mechanical Analysis
Yield Strength	ASTM D638-91
Ultimate Tensile	ASTM D638-91
Ultimate Elongation	ASTM D638-91
Flexibility Test	ASTM D747

4.4 Analysis Technique

The resin properties of molecular weight, polydispersity, density, and melt index from the raw material testing were plotted against the extruded tubing results of elastic modulus, yield strength, ultimate tensile strength, ultimate elongation, and flexural modulus (stiffness). Linear regression using the least squares method was used to determine correlations between the resin properties and tubing functional properties. The r-squared, square of the Pearson product moment correlation coefficient, was calculated using Excel 5.0 software. The r-squared value is interpreted as the proportion of the variance in y attributable to the variance in x. Where no correlation was determined, trends were identified.

To evaluate the effect of irradiation on the tubings, the irradiation dose was plotted against the results of yield strength, ultimate tensile strength, ultimate elongation, and flexural modulus. The elastic modulus and loss tangent of the tubings were analyzed for trends in increased cross linking and changes in glass transition temperature.

Chapter 5

RESULTS

The experimental results are separated into three sections. Section 5.1 covers the results of resin testing. Section 5.2 covers the results of extrusion testing, as well as correlations to the resin properties. Section 5.3 describes the results of testing on irradiated tubings.

5.1 Properties of Resins

Table 4 summarizes the properties of number average molecular weight (Mn), weight average molecular weight (Mw), polydispersity (Mw/Mn), density, and melt index for each of the resins tested. The results of the molecular weight determination are plotted in Figure 16, with one standard deviation error bars. The molecular weight charts are contained in Appendix A. The chart for polydispersity, Figure 16 (a), shows that the high density polyethylene resins are 1.94 to 2.51 higher in polydispersity than the linear low density polyethylene. There is only a 0.04 difference between the two HDPE materials and a 0.54 difference between the two LLDPE materials.

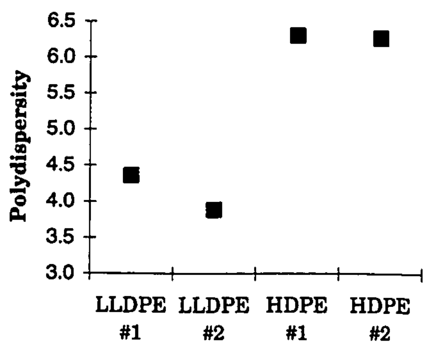
The plot comparing the number average molecular weight (Mn) of the resins is shown in Figure 16 (b). Shown in this plot are 40 to 57% (18 to 25 kg mol) higher values of Mn for the LLDPE materials compared to the

HDPE materials. Also seen in this plot is a larger variation for readings of the LLDPE materials compared to the HDPE materials. The plot comparing the weight average molecular weight (Mw), Figure 16 (c), has similar Mw values for the two HDPE materials with a difference of only 4.4% (12 kg mol). However, a large difference, 24% (57 kg mol), was observed between the LLDPE materials, with the LLDPE #2 having a lower Mw value.

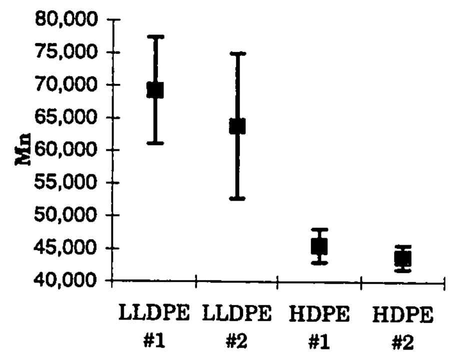
TABLE 4: Resin Material Properties

	Mn (g mol)	Mw (g mol)	Mw/Mn	Density (g/ml)	Melt Index (g/10 min)
LLDPE #1	69,260	299,080	4.319	0.911	1.17
LLDPE #2	63,840	241,600	3.784	0.919	2.31
HDPE #1	45,550	286,600	6.292	0.935	0.77
HDPE #2	43,810	274,140	6.257	0.963	0.70

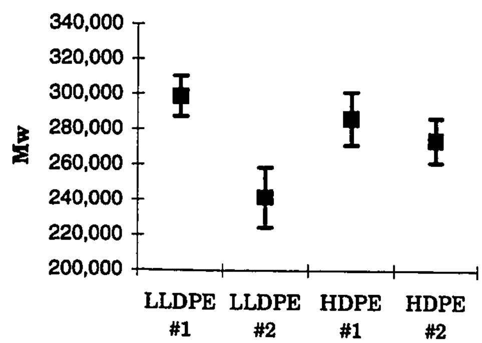
There was considerable variation in density of the resins. The LLDPE materials had densities of 0.911 and 0.919 g/ml. These values span the range of commercially available extrusion/film grade LLDPE resins which are noted have densities of 0.910 to 0.925 g/ml [13]. The HDPE resins, with density readings of 0.935 and 0.963 g/ml, were also observed to span the range of commercially available resins, which are listed as 0.941 to 0.965 g/ml for extrusion/film grade HDPE resins [13]. The HDPE materials



16 (a)



16 (b)



16 (c)

Figure 16: Molecular Weight Determination: (a) Polydispersity, (b) Mn, and (c) Mw

were observed to have densities 0.016 to 0.052 g/ml higher than the LLDPE materials.

The melt index values for the two HDPE materials were relatively close, given that commercially available extrusion/film grade HPDE materials range from 0.02 to 18 g/10 min[14]. The fact that the melt indices are relatively close suggests that the molecular weight determinations are also close. This corresponds well to the molecular weight determinations of this study. The LLDPE #2 material has a slightly higher melt index than the LLDPE #1, given that commercially available extrusion/film grade LLDPE materials range from 0.28 to 6 g/10 min. This result suggests a lower molecular weight value for LLDPE #2, due to the inverse relationship between the melt index and the molecular weight. This observation corresponds well with weight average molecular weight determinations.

The FTIR spectra for each resin are shown in Figures 17 through 20. These spectra show peaks for =C-H stretch at approximately 2800 wavenumber, =C-H bend at 1500 wavenumber, and C=C at 750 to 800 wavenumbers. Observation shows that all materials have the same major peaks.

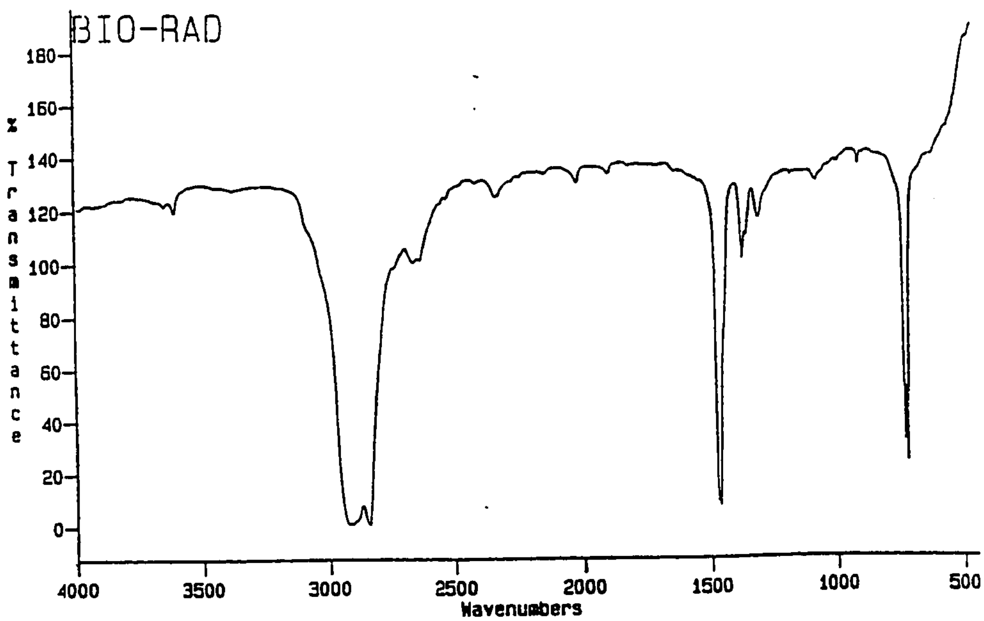


Figure 17: FTIR Spectrum for LLDPE #1

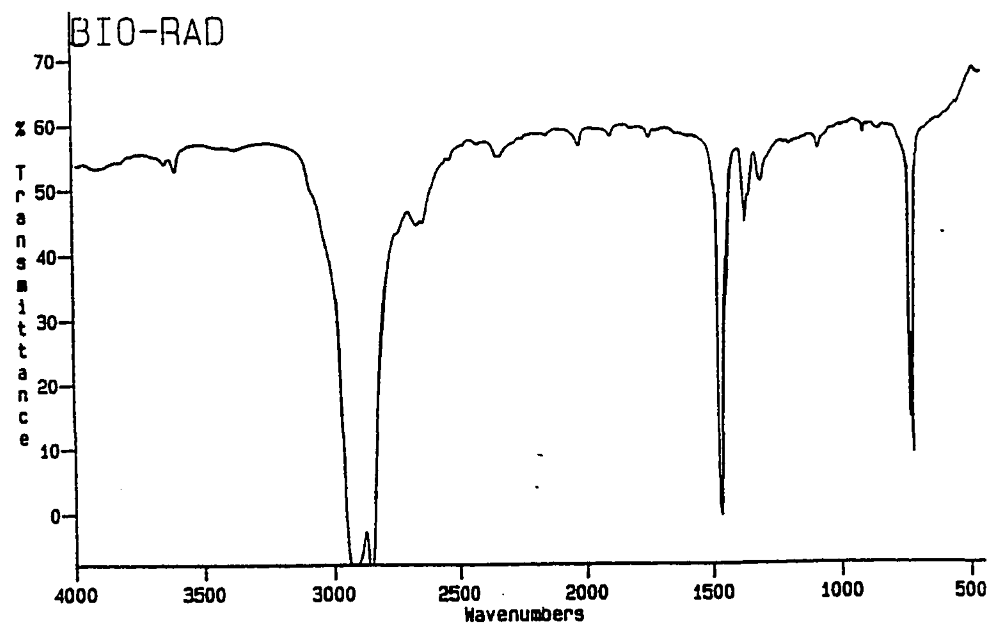


Figure 18: FTIR Spectrum for LLDPE #2

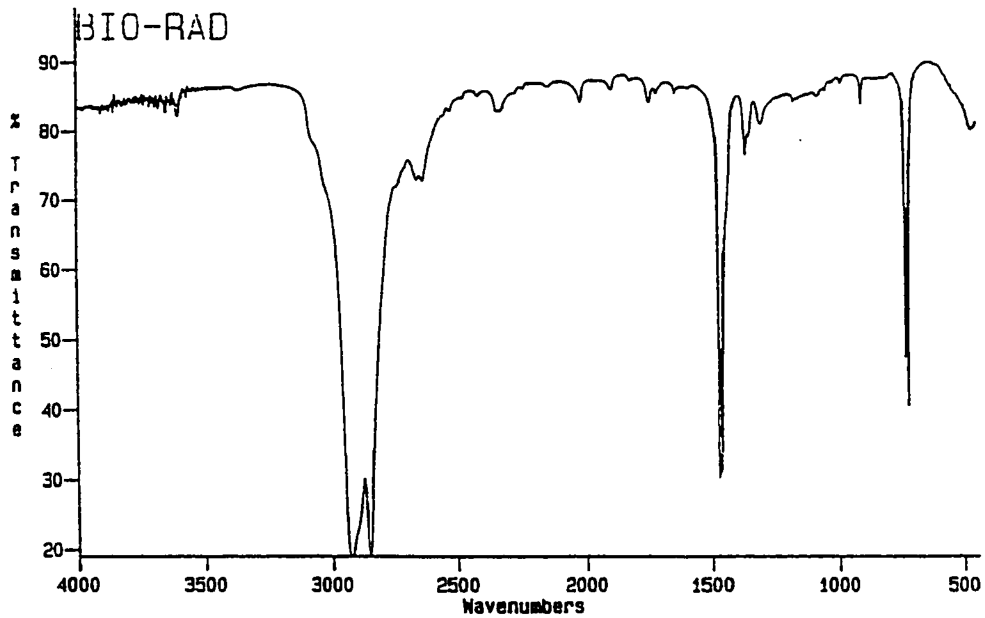


Figure 19: FTIR Spectrum for HDPE #1

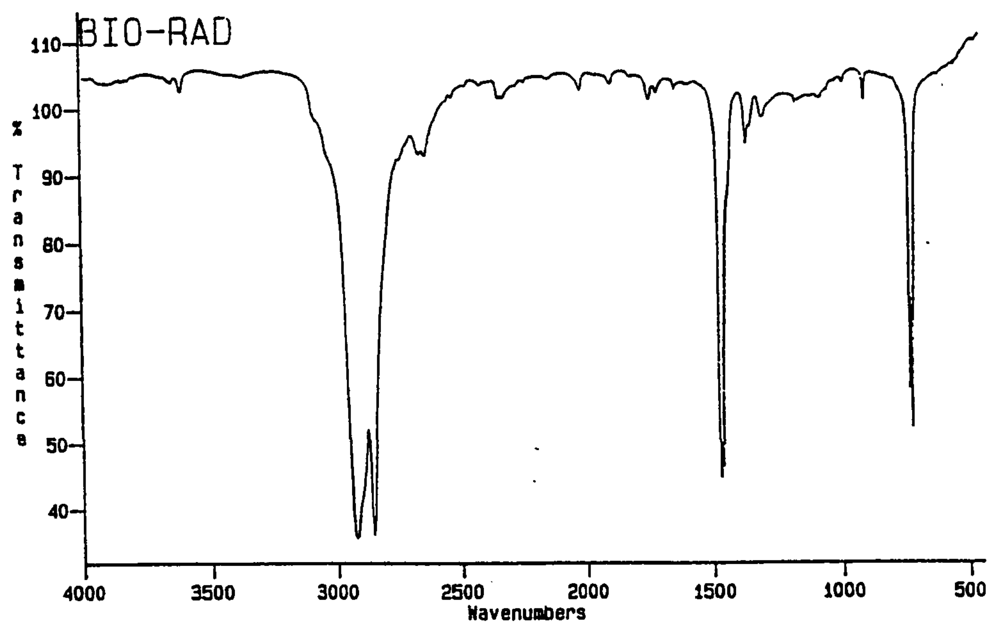


Figure 20: FTIR Spectrum for HDPE #2

5.2 Functional Properties of Extruded Tubings

A summary of the average outer diameter (OD), inner diameter (ID), and the corresponding cross-sectional area for the tubings is presented in Table 5. Appendix B provides the individual readings taken for all extruded tubings. The cross-sectional area for each extrusion is shown in Figure 21, with one standard deviation error bars. This cross-sectional area determination shows differences of only 0.4 to 3.9% between the four extrusions. The actual dimensions, however, were used for flexibility, tensile, and DMA calculations.

TABLE 5: Extruded Tubing Dimensional Analysis

	OD (mm)	ID (mm)	Cross-Sectional Area in mm^2
LLDPE #1	0.995	0.613	0.482
LLDPE #2	1.011	0.639	0.481
HDPE #1	0.989	0.620	0.464
HDPE #2	1.009	0.654	0.466

The DMA determinations of elastic modulus at 25°C, loss tangent at 25°C, and the glass transition temperature (T_g) are contained in Table 6. These determinations show an average of 172% higher elastic modulus for the HDPE samples, which is indicative of a higher degree of crystallinity,

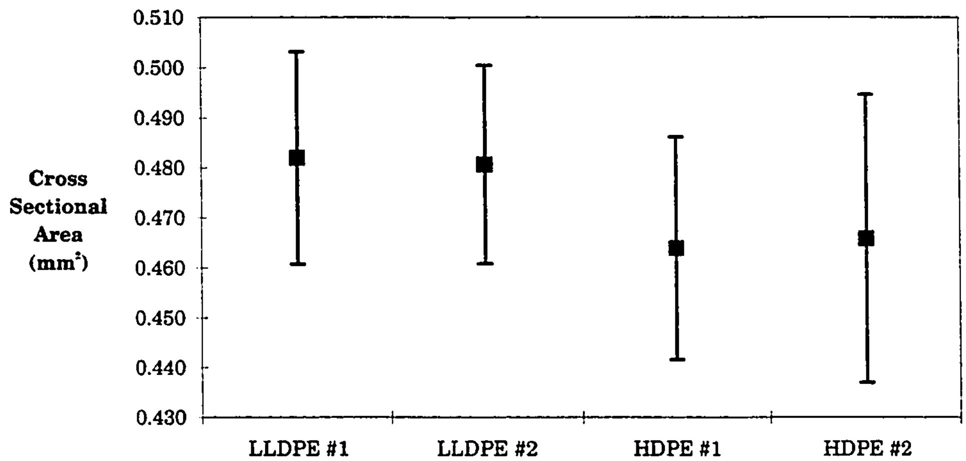


Figure 21: Cross-sectional area of extrusions

than the LLDPE samples. Figure 22 shows the elastic modulus over a range of -150°C to 100°C for all extrusions. This figure shows a consistently higher modulus of elasticity for the HDPE materials in 0°C to 100°C temperature range compared to the LLDPE materials, with HDPE #1 having the highest modulus. This figure shows that the two LLDPE materials are similar to each other in the 0°C to 100°C temperature range.

TABLE 6: Dynamic Mechanical Analysis Results of Non-Irradiated Extrusions

	Elastic Modulus at 25 C (dyn/cm^2)	Loss Tangent at 25°C	T_g (°C)
LLDPE #1	3.31 x 10^8	0.185	-114
LLDPE #2	3.12 x 10^8	0.215	-99
HDPE #1	10.16 x 10^8	0.120	-114
HDPE #2	7.32 x 10^8	0.147	-114

The correlations between the elastic modulus and each of the following are plotted in Figure 23: Mn, polydispersity, Mw, melt index, and density. A linear correlation is observed between the elastic modulus and Mn and between the elastic modulus and polydispersity, with R^2 values greater than 0.8, as shown in Figures 23 (a) and (b), respectively. The slope of the Mn versus the elastic modulus plot is -2.4 x 10^7 MPa/kg mole. The slope of the polydispersity versus the elastic modulus plot is 3 x 10^8 MPa. No trend is observed between the elastic modulus and Mw in Figure 23 (c). Trends of increased elastic modulus are observed in Figures 23 (d) and (e) for a decreasing melt index and an increasing density, respectively. The R^2 values were calculated to be 0.5 and 0.4 for the linear regression of elastic modulus versus the melt index and density, respectively.

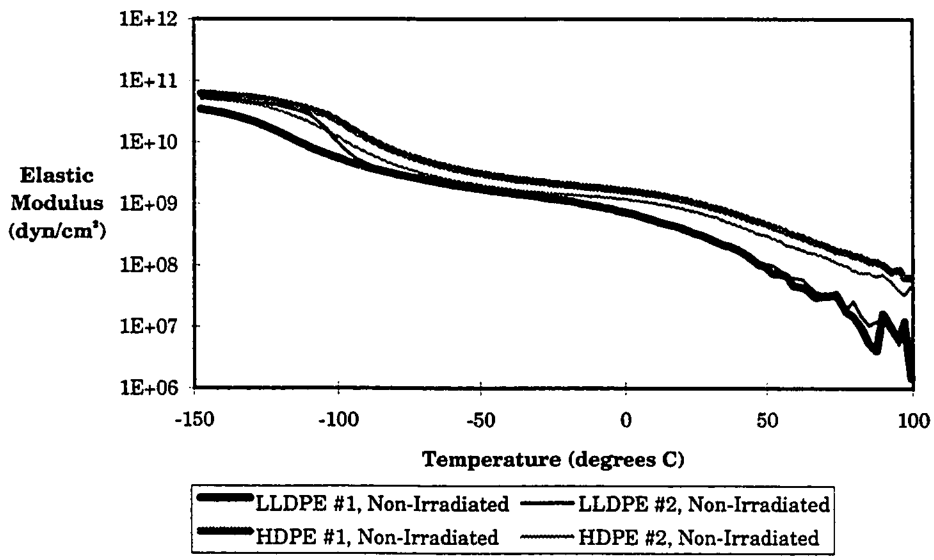


Figure 22: Elastic Modulus for non-irradiated extrusions

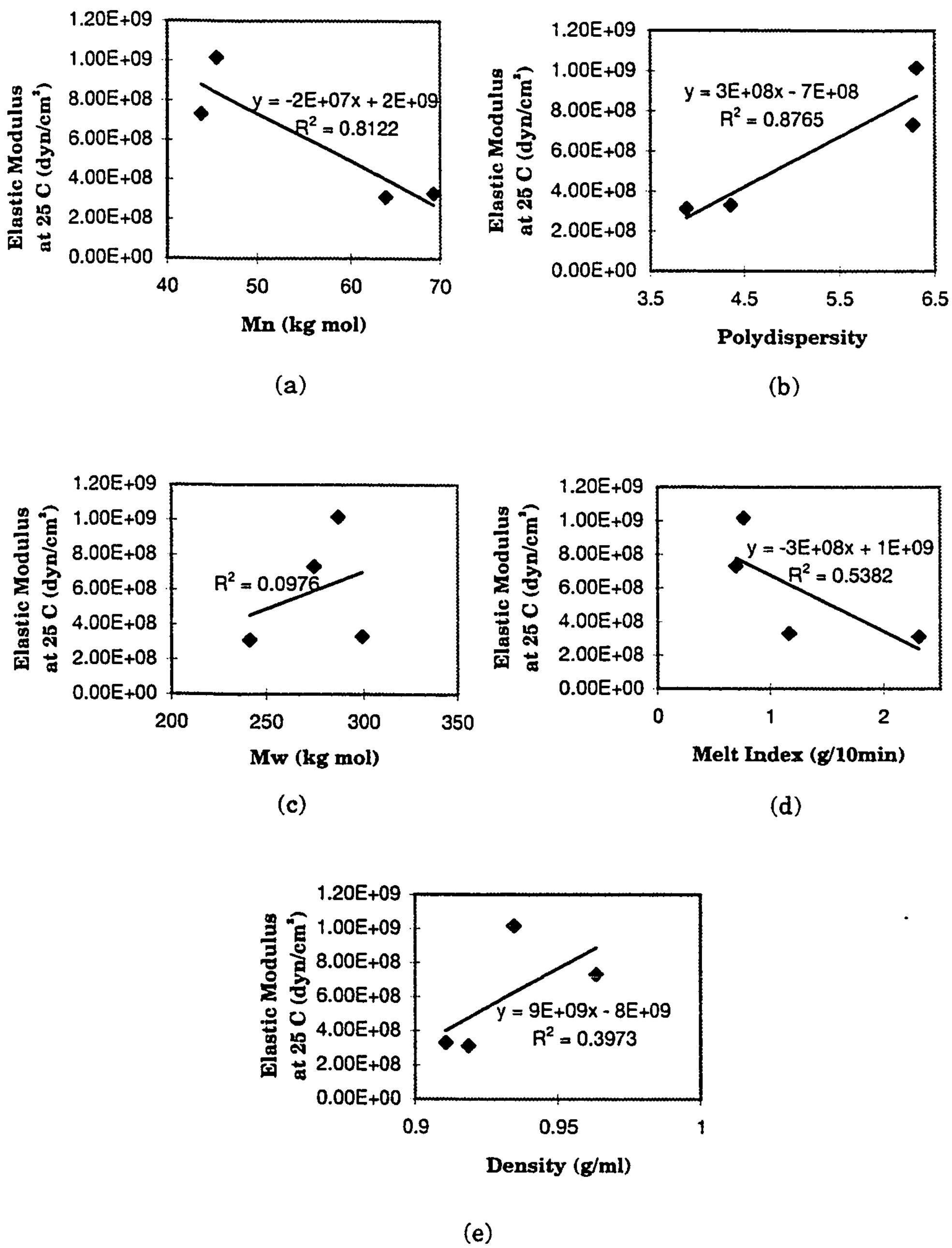


Figure 23: Correlation between elastic modulus and (a) Mn, (b) polydispersity, (c) Mw, (d) melt index, and (e) density

Figure 24 is a chart of the loss tangent determinations associated with each extrusion. This chart shows that all materials have a peak, designating the glass transition temperature, at -114°C except for LLDPE #2, which has a peak at -99°C, 5 degrees higher than the other materials. This peak, however, is thought to be an outlier after evaluation of irradiated samples, as described in section 5.3.

Table 7 contains the determinations of yield strength, ultimate tensile strength, and ultimate elongation for non-irradiated extrusions of all four materials. The stress-strain curves for the four extrusions are shown in Figure 25. Appendix C contains the stress-strain curves for individual samples. These results in Table 7 demonstrate that the HDPE extrusions have an average of 99% higher yield strengths and an average of 89% higher ultimate tensile strengths than the LLDPE extrusions. Within the HDPE material group, HDPE #1 has only a 9% higher yield strength, but a 20% higher tensile strength and a 179% higher elongation compared to HDPE #2. The LLDPE materials have only a 13% difference in tensile strength and no difference in the yield strength as can be seen from Table 7. The LLDPE #2, however, has a 176% higher ultimate elongation than LLDPE #1.

TABLE 7: Tensile and Flexibility Test Results of Non-Irradiated Extrusions

	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Ultimate Elongation (%)	Flexural Modulus (MPa)
LLDPE #1	14.8	19.7	293	227
LLDPE #2	13.0	19.7	809	201
HDPE #1	18.8	40.1	1,490	702
HDPE #2	26.3	33.8	534	565

Correlations of ultimate tensile strength (UTS) are plotted in Figure 26 versus the following material properties: Mn, polydispersity, Mw, melt index, and density. A negative linear relationship, with a slope of -0.764 MPa/kg mol and R^2 =0.87, was calculated between Mn and UTS, shown in Figure 26 (a). A positive linear correlation, based on a calculated slope of 7.94 MPa and R^2 of 0.913, was observed between polydispersity and UTS, as shown in Figure 26 (b). No trend was observed in the plot of UTS versus Mw in Figure 26 (c). Trends toward higher UTS, R^2 >0.4, were observed with a decreasing melt index and an increasing density in Figures 26 (d) and (e), respectively.

Figure 27 contains plots of the correlations between the yield strength and the following properties: Mn, polydispersity, Mw, melt index, and density. These plots show that the yield strength determinations correlate to the material properties in a similar manner as the UTS. A negative linear relationship was demonstrated between Mn and yield strength, based on a calculated slope of -0.589 MPa/kg mol and R^2 of 0.90, as shown in

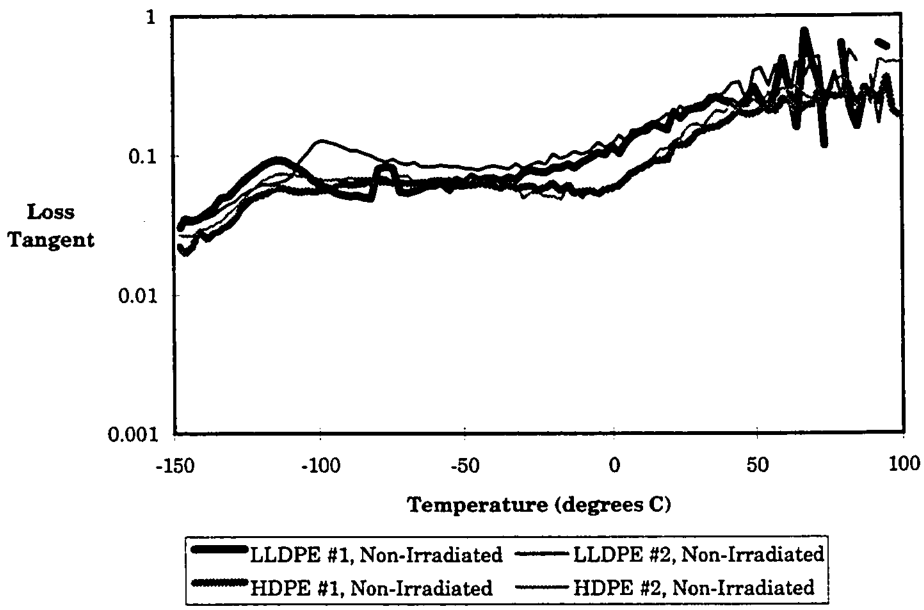


Figure 24: Loss tangent for non-irradiated extrusions

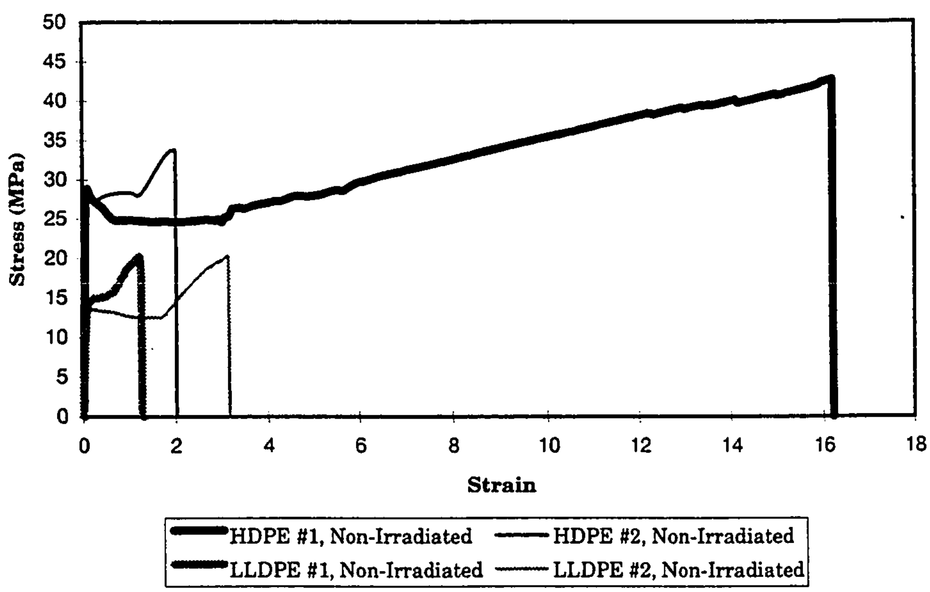
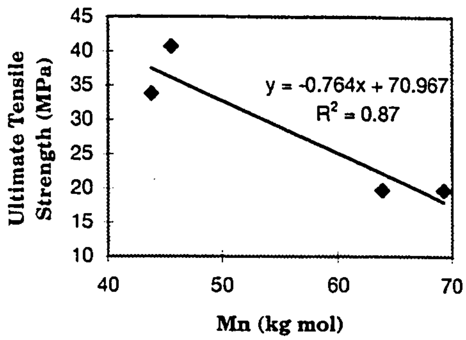
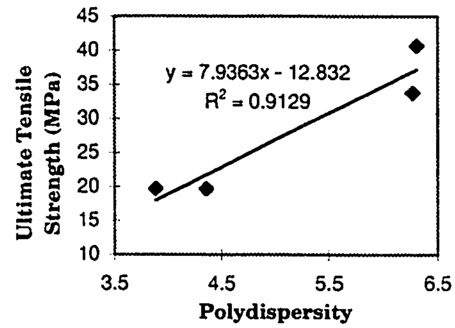


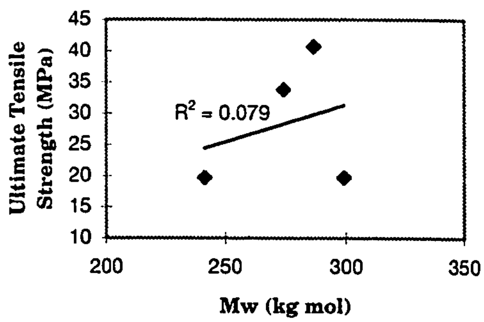
Figure 25: Stress-strain curves for non-irradiated extrusions



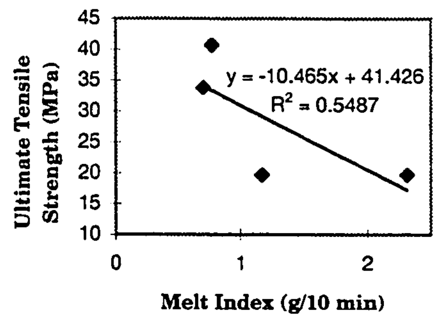
(a)



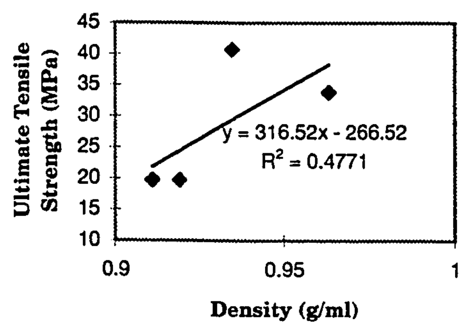
(b)



(c)

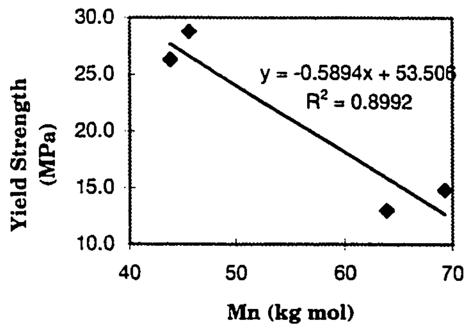


(d)

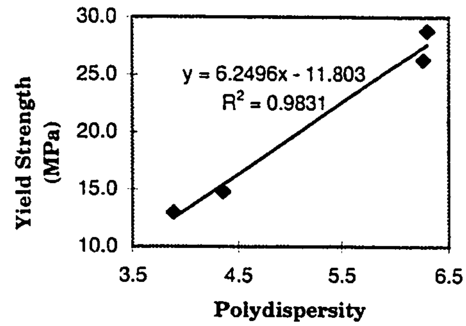


(e)

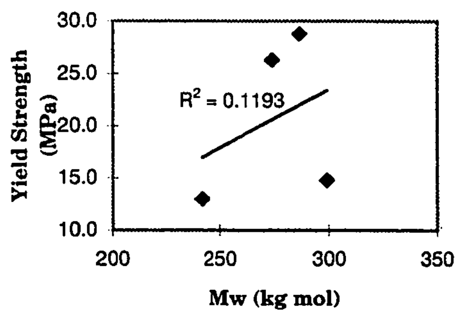
Figure 26: Correlation between ultimate tensile strength and (a) Mn, (b) polydispersity, (c) Mw, (d) melt index, and (e) density



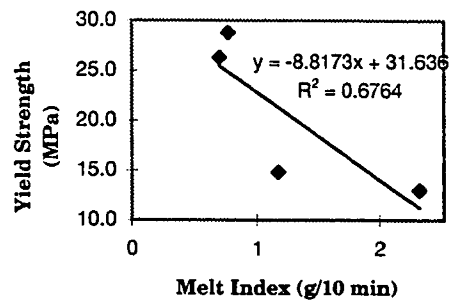
(a)



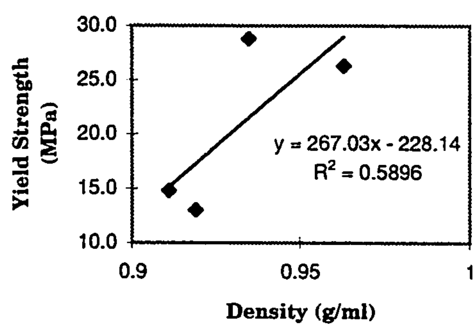
(b)



(c)



(d)



(e)

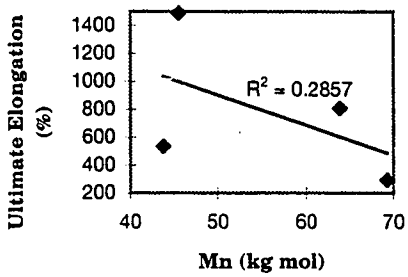
Figure 27: Correlation between yield strength and (a) Mn, (b) polydispersity, (c) Mw, (d) melt index, and (e) density

Figure 27 (a) . A positive linear correlation, with a slope of 6.25 MPa and R^2 = 0.983, was calculated between polydispersity and the yield strength, shown in Figure 27 (b). No trend was observed between the yield strength and Mw, plotted in Figure 27 (c). Trends toward higher yield strength, R^2 >0.5, were observed with a decreasing melt index and an increasing density in Figures 27 (d) and (e), respectively.

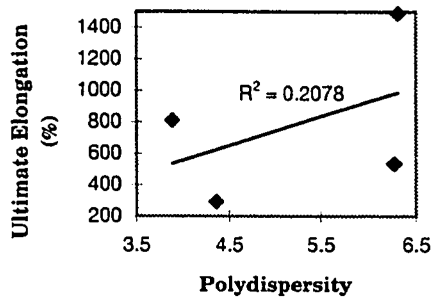
Figure 28 contains plots of ultimate elongation versus the following material properties: Mn, polydispersity, Mw, melt index, and density. Calculations of R^2 for each of these plots resulted in values consistently below 0.30. Therefore, these material properties are considered to not have linear correlations with the ultimate elongation.

The flexural modulus for all non-irradiated extrusions are shown in Table 7. The raw data from the flexibility testing is contained in Appendix D. A 66% higher flexural modulus was observed for the HDPE materials compared to the LLDPE materials. HDPE #1 was observed to be 24% higher than HDPE #2. The flexural modulus of the LLDPE materials were seen to be similar, with LLDPE #1 being only 13% higher.

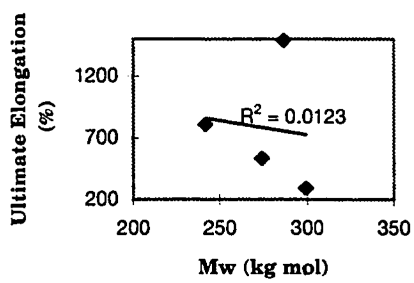
Plotted in Figure 29 are the correlations of flexural modulus versus the following material properties: Mn, polydispersity, Mw, melt index, and density. A negative linear relationship was calculated, with a slope of -18.17 MPa/kg mol and R^2 = 0.88, between Mn and the flexural modulus,



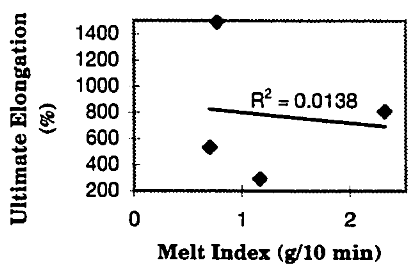
(a)



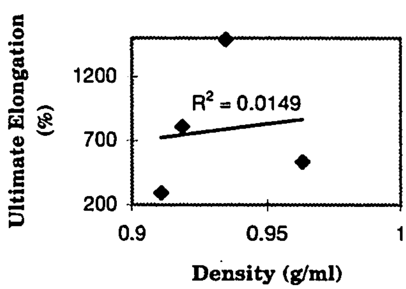
(b)



(c)



(d)



(e)

Figure 28: Correlation between ultimate elongation and (a) Mn, (b) polydispersity, (c) Mw, (d) melt index, and (e) density

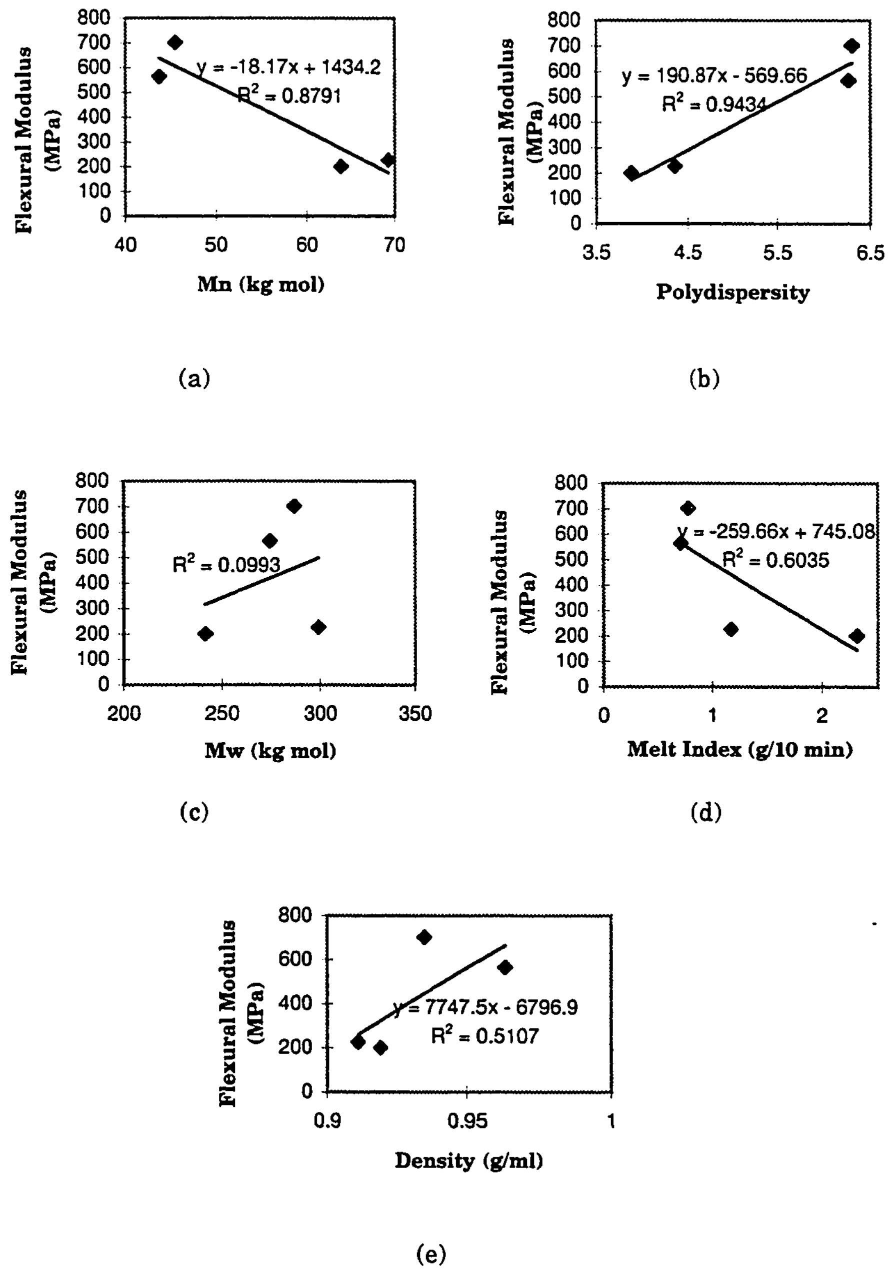


Figure 29: Correlation between flexural modulus and (a) Mn, (b) polydispersity, (c) Mw, (d) melt index, and (e) density

shown in Figure 29 (a). A positive linear correlation was seen between polydispersity and the flexural modulus, based on the calculated slope of 190 MPa and R^2 = 0.913, shown in Figure 29 (b). No trend was observed between the flexural modulus and the Mw, plotted in Figure 29 (c). Trends toward a higher flexural modulus, R^2 of 0.60 and 0.51, were observed with a decreasing melt index and an increasing density, shown in Figures 29 (d) and (e), respectively.

5.3 Functional Properties of Irradiated Tubings

The DMA results of elastic modulus at 25°C, loss tangent at 25°C, and the glass transition temperature are summarized in Table 8. These results demonstrate a higher elastic modulus for the HDPE samples, indicative of a higher extent of crystallinity, compared to the LLDPE samples. Figures 30 through 33 are charts of the elastic modulus over a -150°C to 100°C temperature range for LLDPE #1, LLDPE #2, HDPE #1, and HDPE #2, respectively. The only extrusion to exhibit an increasing elastic modulus with increasing temperature in the temperature range of 0°C to 100°C is the HDPE #2 material, as plotted in Figure 33. This result suggests that irradiation results in an increase in the extent of crystallinity for the HDPE #2 material.

Figures 34 through 37 are plots of the loss tangent plotted against temperature for each level of irradiation for LLDPE #1, LLDPE #2, HDPE #1, and HDPE #2, respectively. All materials were observed to have peaks, designating the glass transition temperature, between -116°C and -111°C, with the exception of non-irradiated LLDPE #2. All materials show a trend of decreasing glass transition temperature with increasing irradiation. This trend indicates that increased irradiation is potentially

TABLE 8: Dynamic Mechanical Analysis Results of Irradiated Extrusions

	Elastic Modulus at 25°C (dyn/cm^2)	Loss Tangent at 25°C	T_g (°C)
LLDPE #1			
0 Mrad	3.31×10^8	0.185	-114
30 MRad	3.51×10^8	0.182	-116
50 MRad	4.04×10^8	0.169	-111
70 MRad	3.48×10^8	0.206	-111
LLDPE #2			
0 MRad	3.12×10^8	0.215	-99
30 MRad	3.95×10^8	0.193	-114
50 MRad	3.88×10^8	0.178	-114
70 MRad	4.10×10^8	0.171	-112
HDPE #1			
0 MRad	10.16×10^8	0.120	-114
30 MRad	9.82×10^8	0.122	-116
50 MRad	10.02×10^8	0.122	-111
70 MRad	10.10×10^8	0.127	-113
HDPE #2			
0 MRad	7.32×10^8	0.147	-114
30 MRad	10.15×10^8	0.125	-114
50 MRad	8.05×10^8	0.137	-111
70 MRad	13.28×10^8	0.138	-111

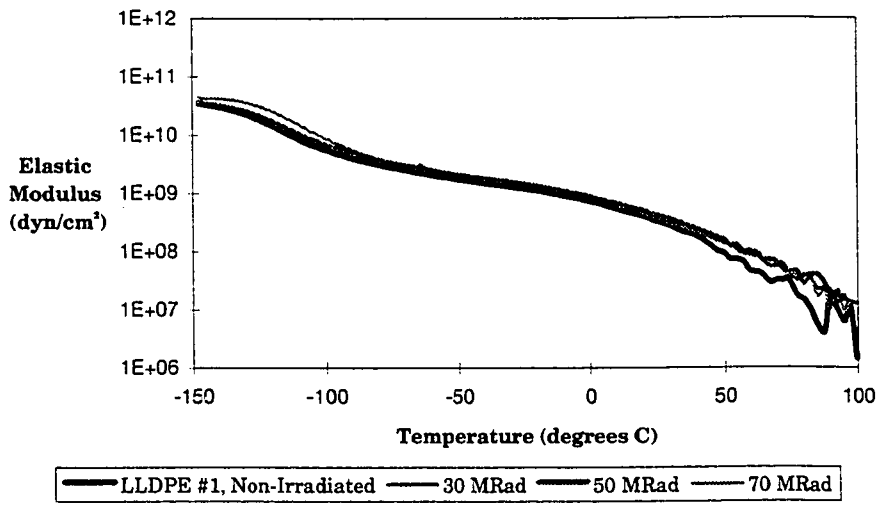


Figure 30: Elastic Modulus for LLDPE #1

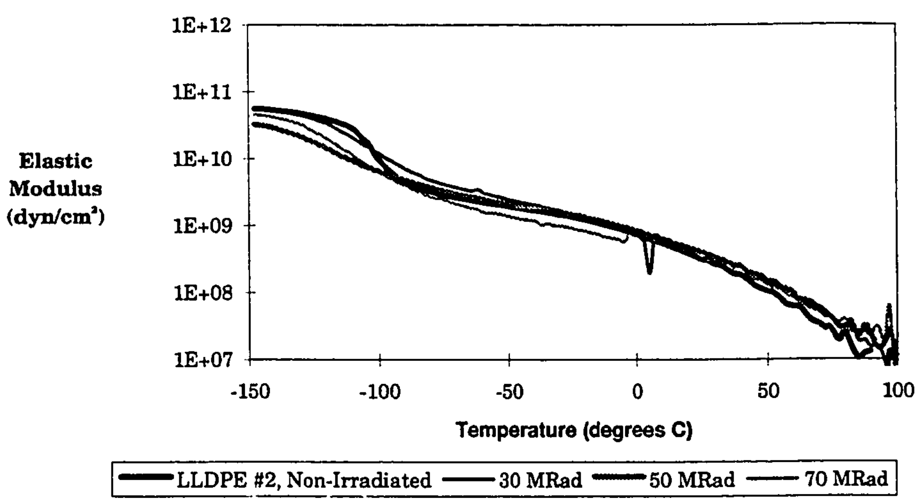


Figure 31: Elastic Modulus for LLDPE #2

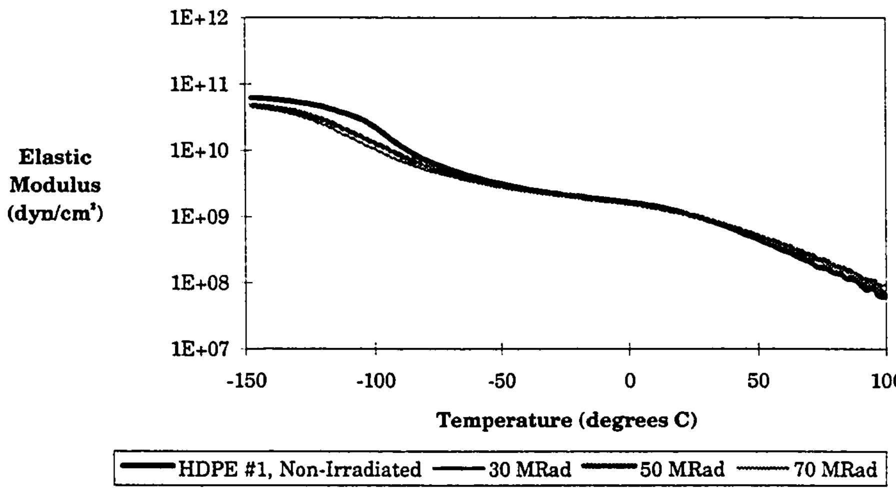


Figure 32: Elastic Modulus for HDPE #1

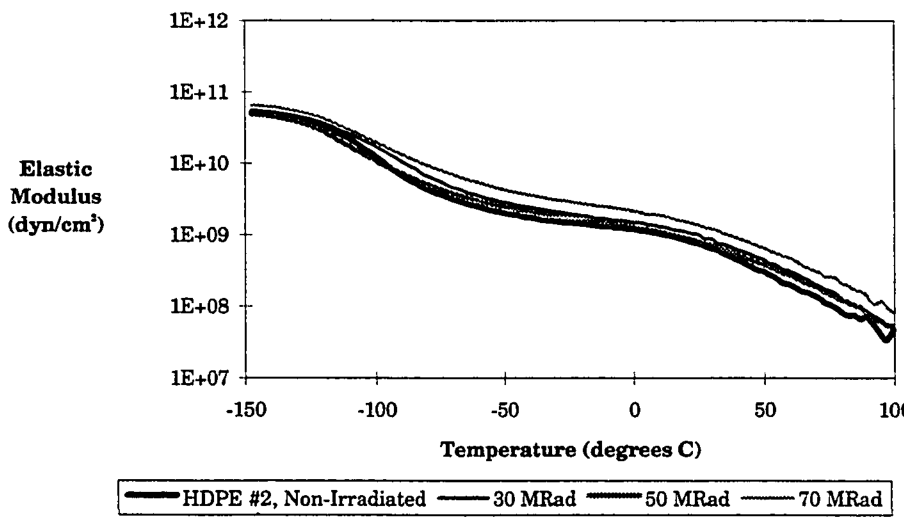


Figure 33: Elastic Modulus for HDPE #1

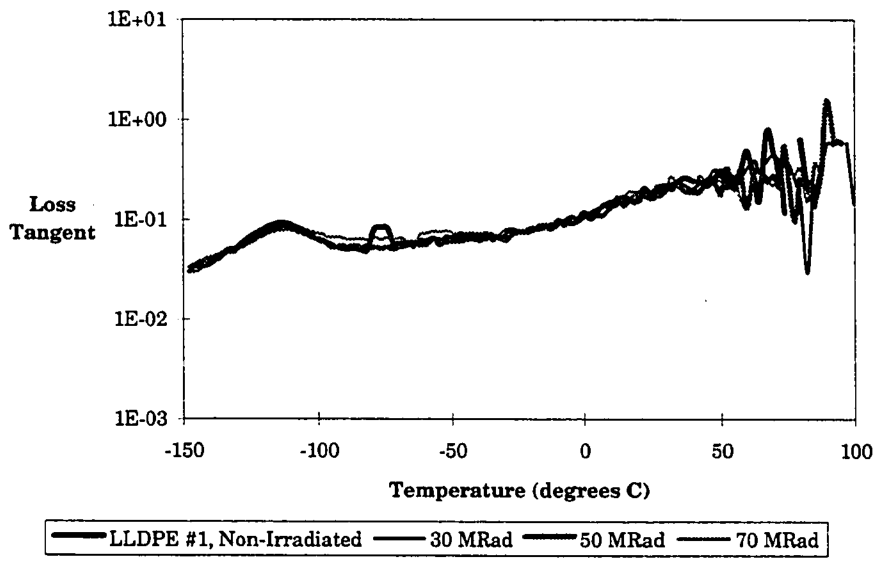


Figure 34: Loss Tangent for LLDPE #1

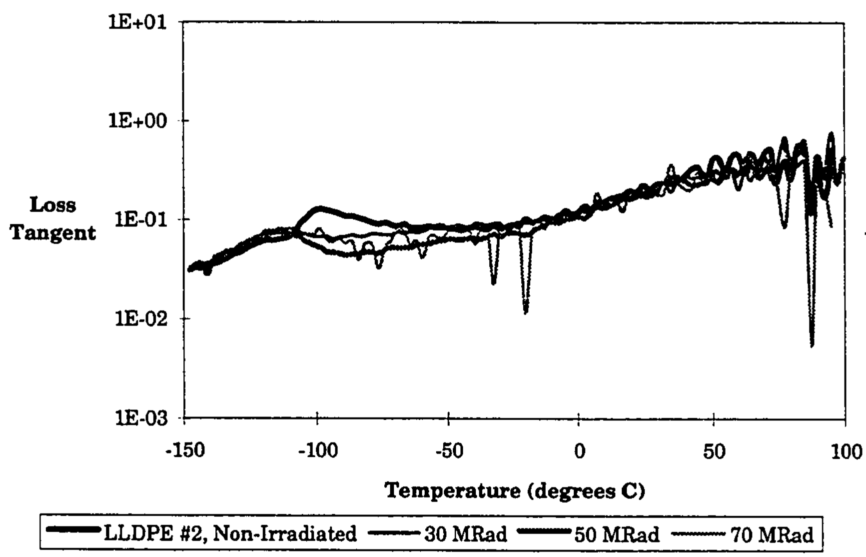


Figure 35: Loss Tangent for LLDPE #2

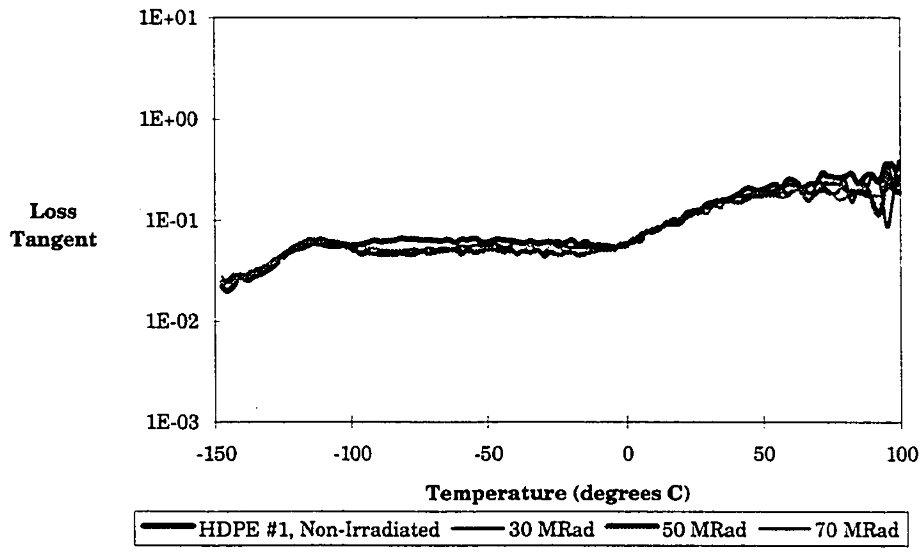


Figure 36: Loss Tangent for HDPE #1

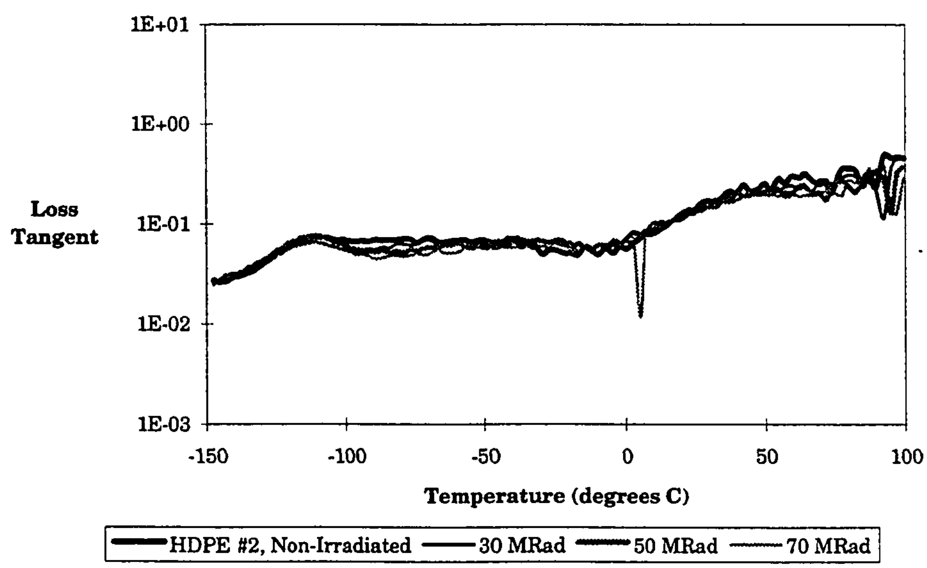


Figure 37: Loss Tangent for HDPE #2

creating a higher degree of cross-linking. The LLDPE #2 extrusion transition temperature of -99°C seems too high and is considered an outlier from the remaining data based on the significantly (15%) lower glass transition temperature obtained after irradiation.

The tensile test determinations of yield strength, ultimate tensile strength, and ultimate elongation are summarized in Table 9 for the irradiated tubings. Figure 38 is a plot of the ultimate tensile strength against the irradiation dose. The ultimate tensile strength and the irradiation dose was determined to have a linear relationship, with $R^2 > 0.7$, for the two LLDPE materials. It was determined that the HDPE have a polynomial relationship between the ultimate tensile strength and the irradiation level, with an initial decrease seen in the 0 MRad to 30 MRad dose region. The yield strength versus the irradiation dose is plotted in Figure 39. This figure depicts a linear relationship, $R^2 > 0.7$, between yield strength and irradiation level. The slope of the yield strength versus irradiation level was calculated to be low for each material, with slopes of 0.0343 to 0.0412 MPa/MRad. The ultimate elongation and the irradiation level were determined to have a linear correlation, with R^2 values greater than 0.7, shown in Figure 40. The slopes of these plots were calculated to range from -1.45 to -18.05 %/MRad.

The stress-strain curves for LLDPE #1, LLDPE #2, HDPE #1, and

HDPE #2 are shown in Figures 41 through 44, respectively. A trend toward a higher degree of brittleness with increased irradiation levels was observed for all materials, characterized by the increasing strength and decreasing elongation.

The flexural modulus determinations for irradiated tubings are contained in Table 9 and plotted in Figure 45. A linear relationship was calculated between the flexural modulus and the irradiation level, with $R^2 > 0.60$. A slope of 0.754 to 1.794 MPa/MRad was calculated, suggesting only minimal increases of flexural modulus with increasing irradiation levels.

TABLE 9: Tensile and Flexibility Test Results of Irradiated Extrusions

	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Ultimate Elongation (%)	Flexural Modulus (MPa)
LLDPE #1				
0 MRad	14.8	19.7	293	227
30 MRad	16.6	19.7	312	277
50 MRad	17.5	20.4	244	283
70 MRad	17.5	21.3	197	287
LLDPE #2				
0 MRad	13.0	19.7	809	201
30 MRad	14.8	21.2	608	233
50 MRad	15.6	20.6	443	249
70 MRad	15.9	22.1	380	252
HDPE #1				
0 MRad	28.8	40.7	1,490	702
30 MRad	29.7	36.1	564	774
50 MRad	30.7	37.7	353	778
70 MRad	31.7	39.7	228	837
HDPE #2				
0 MRad	26.3	33.8	534	565
30 MRad	28.8	31.6	355	657
50 MRad	28.7	33.7	254	689
70 MRad	28.8	36.6	212	657

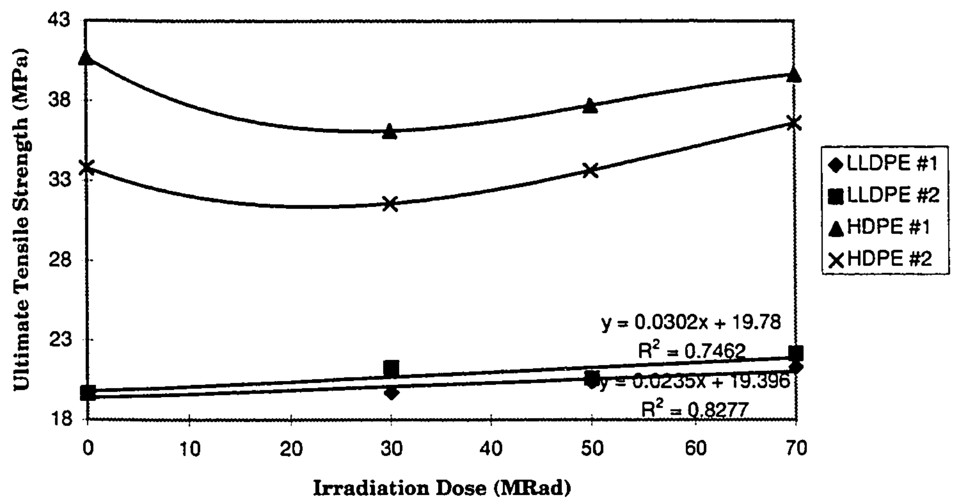


Figure 38: Ultimate tensile strength versus irradiation dose

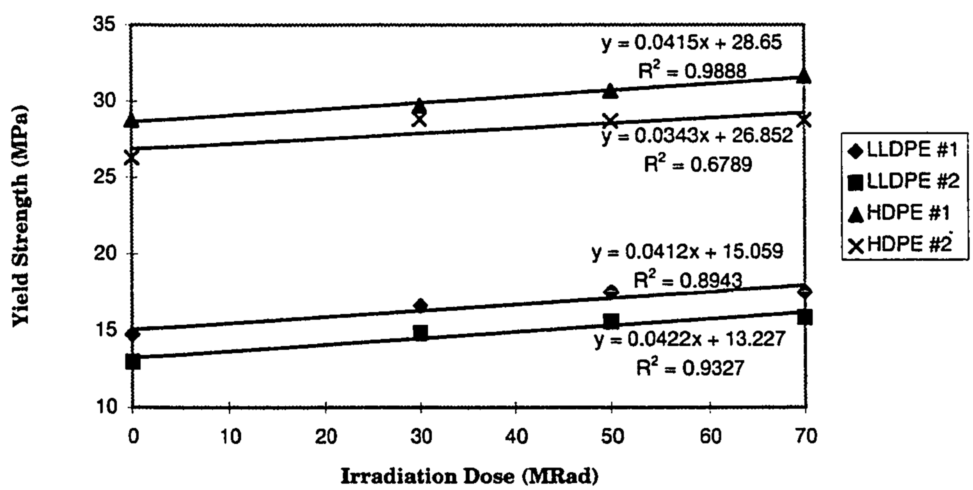


Figure 39: Yield strength versus irradiation dose

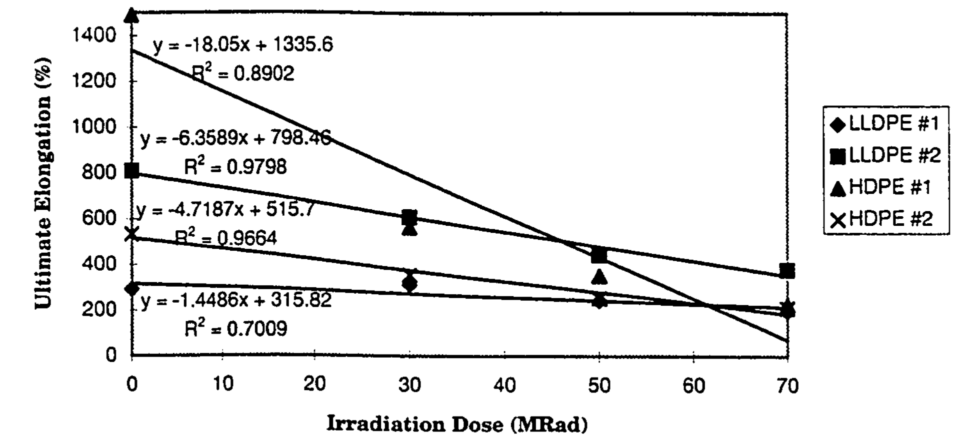


Figure 40: Ultimate elongation versus irradiation dose

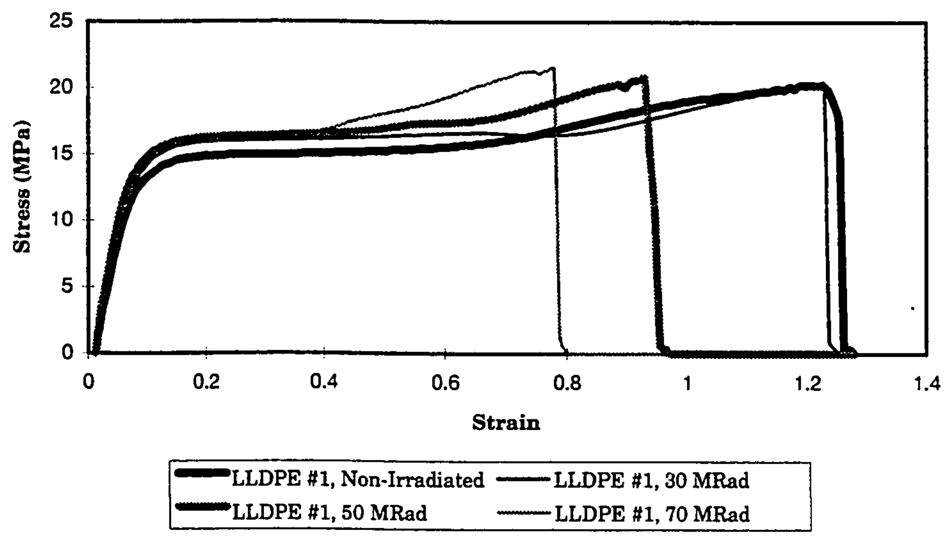


Figure 41: Stress-strain curves for LLDPE #1

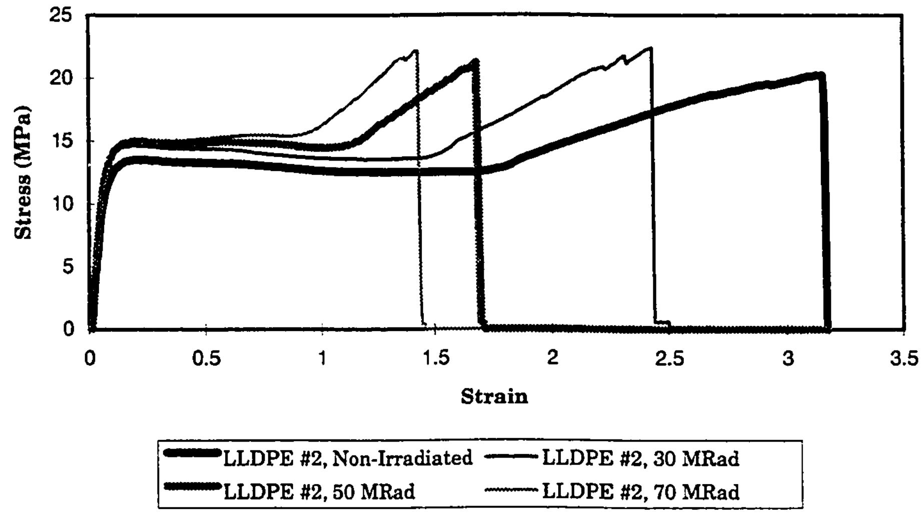


Figure 42: Stress-strain curves for LLDPE #2

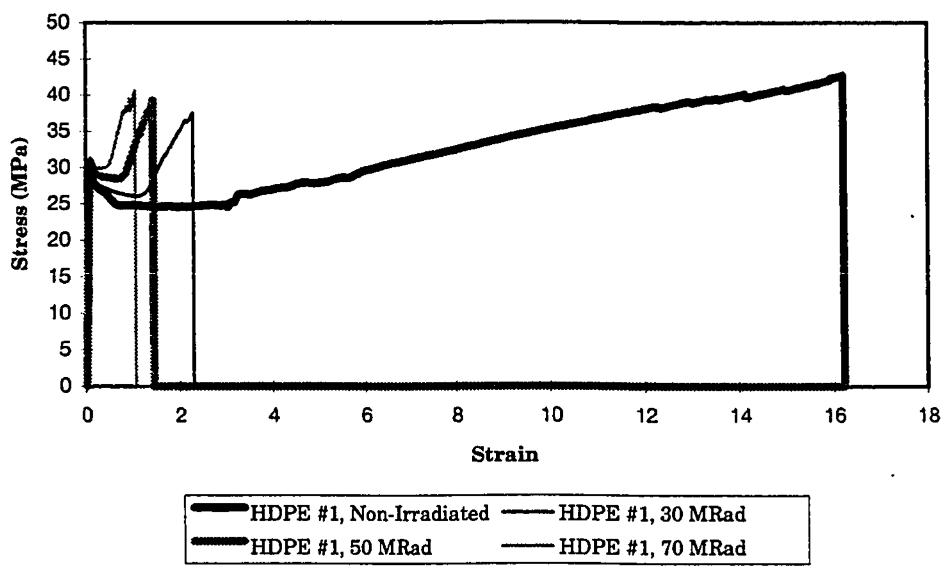


Figure 43: Stress-strain curves for HDPE #1

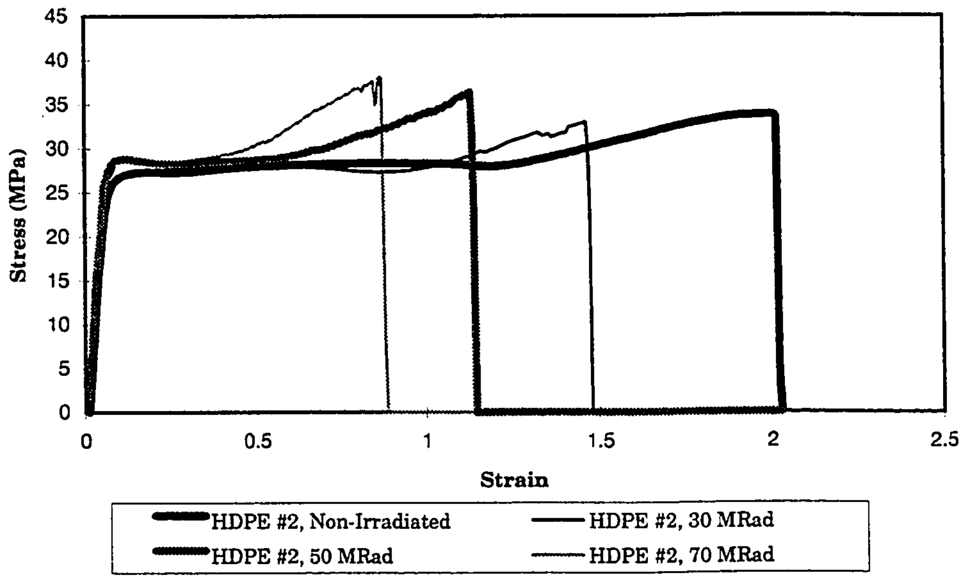


Figure 44: Stress-strain curves for HDPE #2

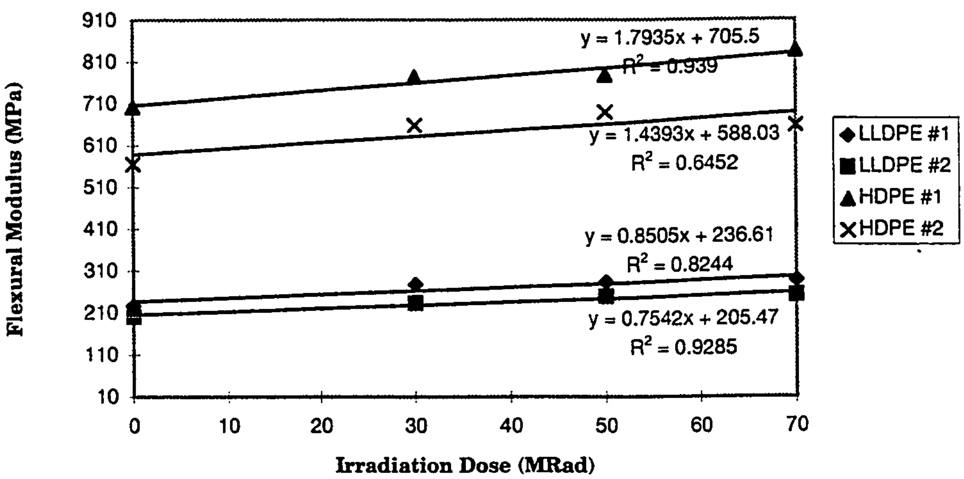


Figure 45: Flexibility test results for irradiated samples

Chapter 6

DISCUSSION

Due to the small number of resins investigated in this study only limited generalizations can be made with respect to correlations between material properties and functional properties. A further complication is the fact that many characteristics of the resins differ, such as branch level, type of sidechains, etc. Therefore, the following discussion points out the correlations seen in this study which are applicable only to the resins studied.

The negative correlations between UTS, yield strength, and flexural modulus and the number average molecular weight do not match the expected result of increased molecular weight providing improved strength. A positive correlation was expected due to increased Mn providing lower mobility, as described in Chapter 3. This result is possibly due to the overriding effect of density creating an improvement in the HDPE materials. A slight trend of increasing functional properties was seen between the two HDPE materials and also between the two LLDPE materials.

The effect of varying molecular weight distribution resulted in decreased strength with lower MWD. This result varies from the expected result of lower MWD causing improved material properties due to less chain length variability, as also described in Chapter 3. As was noted for Mn, the density possibly overrides the effect of molecular weight distribution. In this case, however, the trend in each polyethylene type matched the overall trend of improved material properties with increased molecular weight distribution. This deviation from theory is most likely due to having too little variability of MWD between the two LLDPE materials and the two HDPE materials, with 14% and .5% differences, respectively.

The trend of increased density creating increased ultimate tensile strength, yield strength, elastic modulus, and flexural modulus was found and matches the expected results. The ultimate elongation, however, was found to have no correlation to the density. An increase in ultimate elongation was expected due to the increased extent of crystallinity. The reason for the lack of increased elongation is potentially due to variation of material characteristics not controlled in this study.

The melt index was found to have trends toward improved properties with lower values of melt index, with the exception of ultimate elongation. This result was expected since a small melt index is associated with more entanglement of chains and increased cross-linking.

This study of material and extrusion properties suggests that within a type of polyethylene, i.e. LLDPE or HDPE, the number average molecular weight can be used to obtain limited information on expected extrusion properties. In general, however, testing of resins within a type of polyethylene may not provide a direct correlation to the functional properties of extruded products, as proposed in Chapter 3. It therefore seems necessary to perform testing directly on extrusions to provide information to prove desired functional properties are met.

The results of the FTIR testing, Figures 17 through 20, demonstrated that all materials have the same major peaks but did not provide a method of differentiating polyethylene materials. It is suggested that future work does not include FTIR analysis.

The dynamic mechanical analysis of the irradiated materials showed only slight increases in loss tangent peak between -116°C and -99°C, signifying T_g. This suggests an increase in cross-linking, as hypothesized in Chapter 3.

A large effect from irradiation was seen during tensile testing. Each material was found to have an improved yield strength and lower elongation as a result of irradiation, creating an increased degree of brittleness. The correspondence of stiffness to irradiation level was also determined in this study. A linear relationship was observed with higher irradiation levels providing higher flexural modulus values.

Further work is needed to understand the relationship between resin properties and extruded tubing functional properties, with better material characteristics control. Future work also needs to entail a greater range of molecular weight and molecular weight distributions to obtain a significant correlation. It is further recommended that future research entail only one type of polyethylene with better controlled material characteristics. Dependent on the characteristic control achievable and the quantity of acceptable resins, it is recommended that a statistical analysis be performed to determine two factor interactions as well as individual factor effects.

Chapter 7

CONCLUSION

High-density polyethylene was found to have consistently improved mechanical properties when compared to linear-low-density polyethylene, with the exception of elongation. The measured densities correlated positively to ultimate tensile strength, yield strength, elastic modulus, and flexural modulus, i.e. increased densities correlated to increased functional properties.

The effect of the molecular weight and molecular weight distribution were found to have opposite trends from what was expected. An increase in the number average molecular weight corresponded to a decrease in ultimate tensile strength, yield strength, elastic modulus, and flexural modulus. An increase in the molecular weight distribution, however, corresponded to an increase in ultimate tensile strength, yield strength, elastic modulus, and flexural modulus. This disagreement with the hypothesis is thought to be due, in part, to the large difference of density between the HDPE and LLDPE materials. Density also has an overriding effect on the functional properties of these materials.

In general, the material properties were found not to correlate to extrusion functional properties, as hypothesized. This was most likely due to the lack of control of material characteristics such as the branch level and the type of sidechain. This area needs further research, with a greater variety of HDPE and LLDPE resins, before any conclusions can be drawn.

This study has shown that when selecting a material for a coronary balloon catheter, it is important to consider the type of material to be used, such as LLDPE or HDPE, due to the large difference in material characteristics. When selecting individual resins, within a material type, it is necessary to perform testing on the extruded tubing. This is necessary due to the current lack of correlation between the resin properties and the extrusion function properties.

Electron beam irradiation was found to have a significant effect on the properties of the extruded tubing. Increased levels of irradiation resulted in an increase in the ultimate tensile strength, yield strength, elastic modulus, and flexural modulus on the one hand, and a decrease in elongation and a higher degree of brittleness on the other hand. These results are thought to be due to increases in cross-linking, as demonstrated by the increases in the glass transition temperature.

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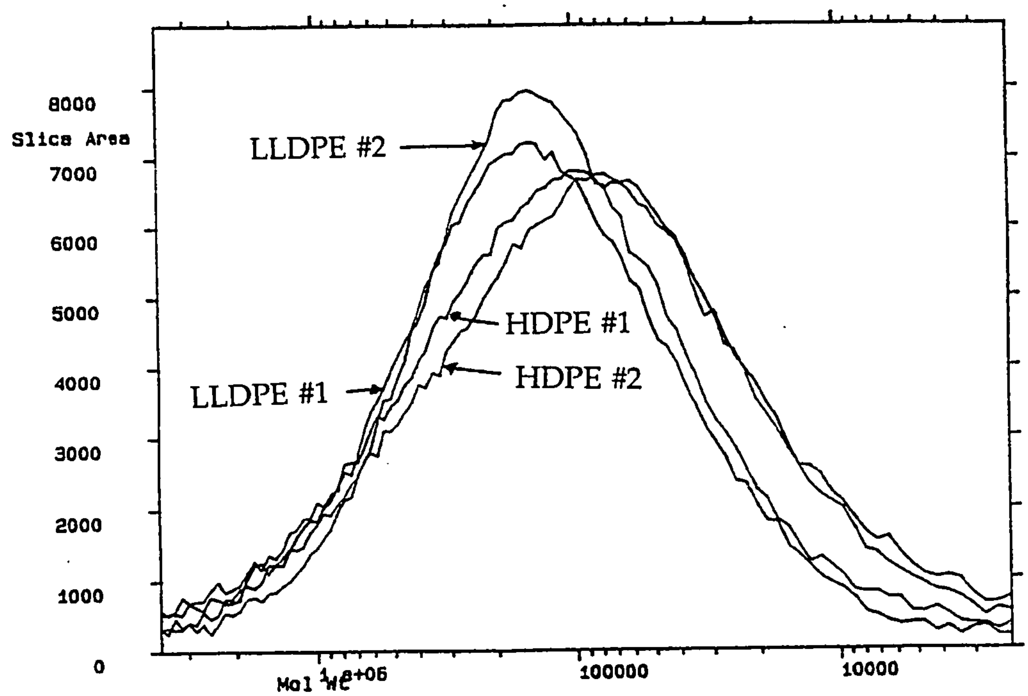
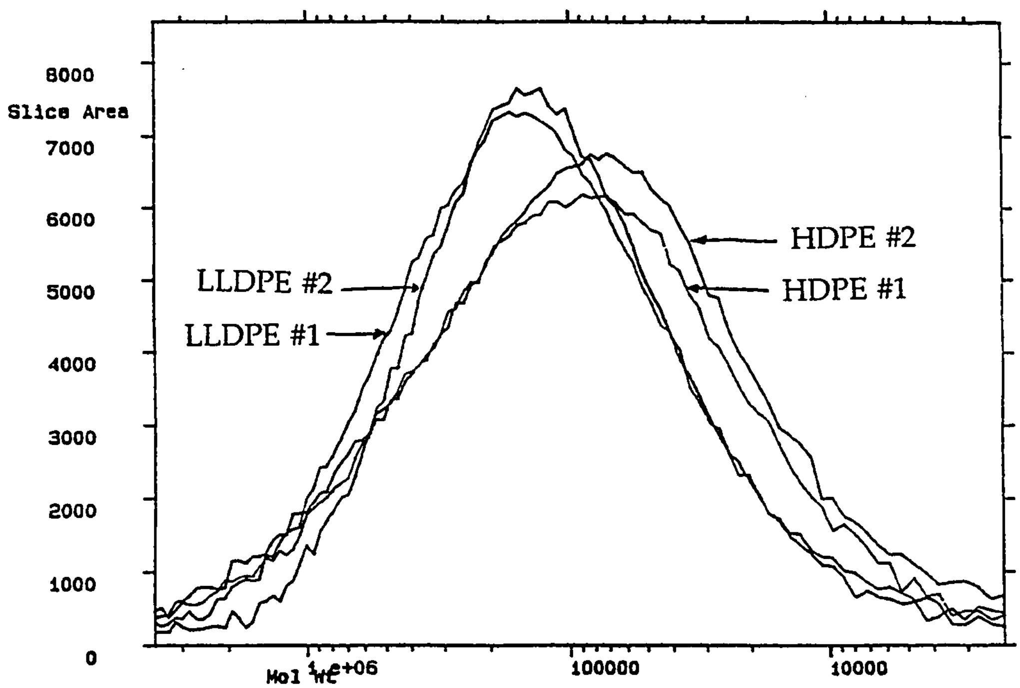
Appendix A

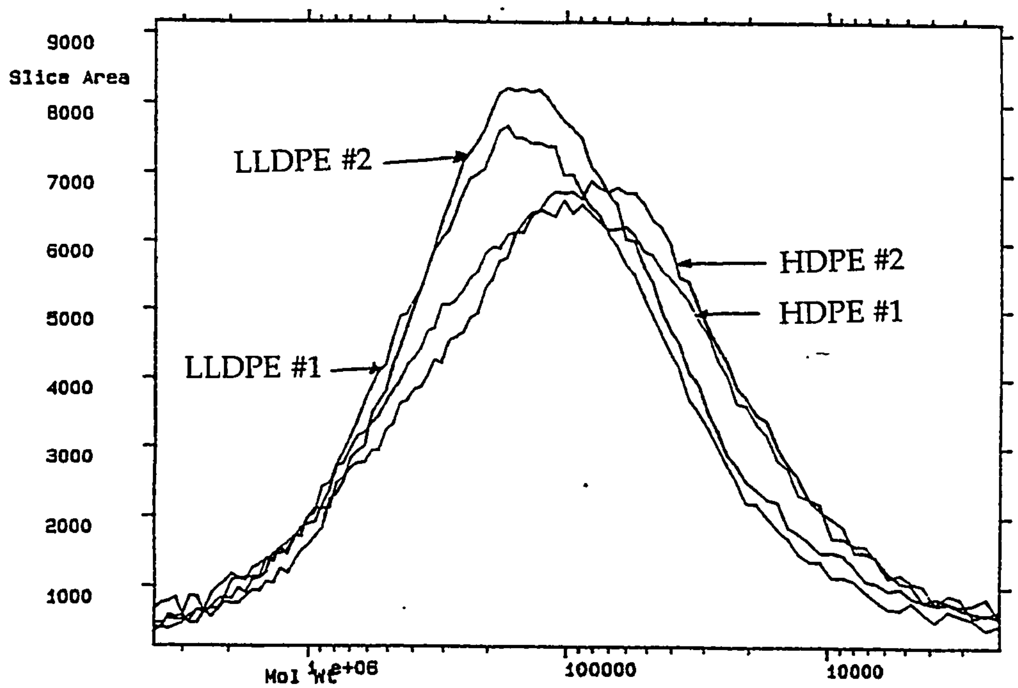
MOLECULAR WEIGHT TEST RESULTS



Molecular Weight Test Results: Average of runs number 1 and 2



Molecular Weight Test Results: Average of runs number 3 and 4



Molecular Weight Test Results: Average of runs number 5 and 6

Appendix B

DIMENSIONAL DATA

Material	OD - X	OD - Y	ID - X	ID - Y
LLDPE #1 (0 Mrad)				
Unit 1	0.998	0.950	0.637	0.571
Unit 2	1.025	0.956	0.633	0.557
Unit 3	1.050	0.953	0.656	0.572
Unit 4	1.058	0.969	0.658	0.586
LLDPE #1 (30 Mrad)				
Unit 1	1.017	0.972	0.637	0.594
Unit 2	1.000	1.005	0.640	0.612
Unit 3	1.017	0.970	0.646	0.587
Unit 4	1.050	1.016	0.653	0.600
LLDPE #1 (50 Mrad)				
Unit 1	1.043	0.929	0.646	0.541
Unit 2	1.065	0.925	0.646	0.559
Unit 3	0.996	0.960	0.636	0.583
Unit 4	1.019	0.937	0.639	0.579
LLDPE #1 (70 Mrad)				
Unit 1	1.028	0.940	0.661	0.572
Unit 2	1.029	0.961	0.660	0.570
Unit 3	1.035	0.975	0.635	0.570
Unit 4	1.044	0.939	0.649	0.632
Outer Diameter				
Average	**0.995**			
Standard Deviation	**0.042**			
Inner Diameter				
Average	**0.613**			
Standard Deviation	**0.037**			

Dimensional Data: LLDPE #1 (data in mm)

Material	OD - X	OD - Y	ID - X	ID - Y
LLDPE #2 (0 Mrad)				
Unit 1	0.989	1.028	0.644	0.634
Unit 2	0.991	1.000	0.639	0.647
Unit 3	0.992	0.988	0.608	0.642
Unit 4	1.015	0.971	0.653	0.624
LLDPE #2 (30 Mrad)				
Unit 1	1.018	1.026	0.617	0.634
Unit 2	1.014	1.014	0.636	0.633
Unit 3	1.043	1.023	0.668	0.635
Unit 4	1.002	1.033	0.668	0.624
LLDPE #2 (50 Mrad)				
Unit 1	0.989	1.012	0.638	0.632
Unit 2	1.010	1.004	0.646	0.627
Unit 3	1.014	1.041	0.644	0.644
Unit 4	1.030	1.039	0.634	0.651
LLDPE #2 (70 Mrad)				
Unit 1	0.989	1.011	0.633	0.645
Unit 2	1.010	1.002	0.634	0.633
Unit 3	1.014	1.005	0.640	0.647
Unit 4	1.030	0.992	0.642	0.665
Outer Diameter				
Average	**1.011**			
Standard Deviation	**0.018**			
Inner Diameter				
Average	**0.639**			
Standard Deviation	**0.013**			

Dimensional Data: LLDPE #2 (data in mm)

Material	OD - X	OD - Y	ID - X	ID - Y
HDPE #1 (0 MRad)				
Unit 1	1.026	0.906	0.629	0.556
Unit 2	1.022	0.927	0.642	0.588
Unit 3	1.038	0.931	0.651	0.578
Unit 4	1.029	0.941	0.651	0.593
HDPE #1 (30 MRad)				
Unit 1	1.051	0.993	0.655	0.579
Unit 2	1.041	0.994	0.666	0.588
Unit 3	1.046	0.983	0.665	0.586
Unit 4	1.037	0.985	0.678	0.603
HDPE #1 (50 MRad)				
Unit 1	1.011	0.906	0.663	0.557
Unit 2	1.017	0.903	0.661	0.558
Unit 3	1.021	0.928	0.663	0.555
Unit 4	1.002	0.922	0.667	0.570
HDPE #1 (70 MRad)				
Unit 1	1.030	0.934	0.670	0.574
Unit 2	1.038	0.956	0.674	0.595
Unit 3	1.019	0.956	0.666	0.595
Unit 4	1.087	0.950	0.668	0.587
Outer Diameter				
Average	**0.988**			
Standard Deviation	**0.051**			
Inner Diameter				
Average	**0.620**			
Standard Deviation	**0.044**			

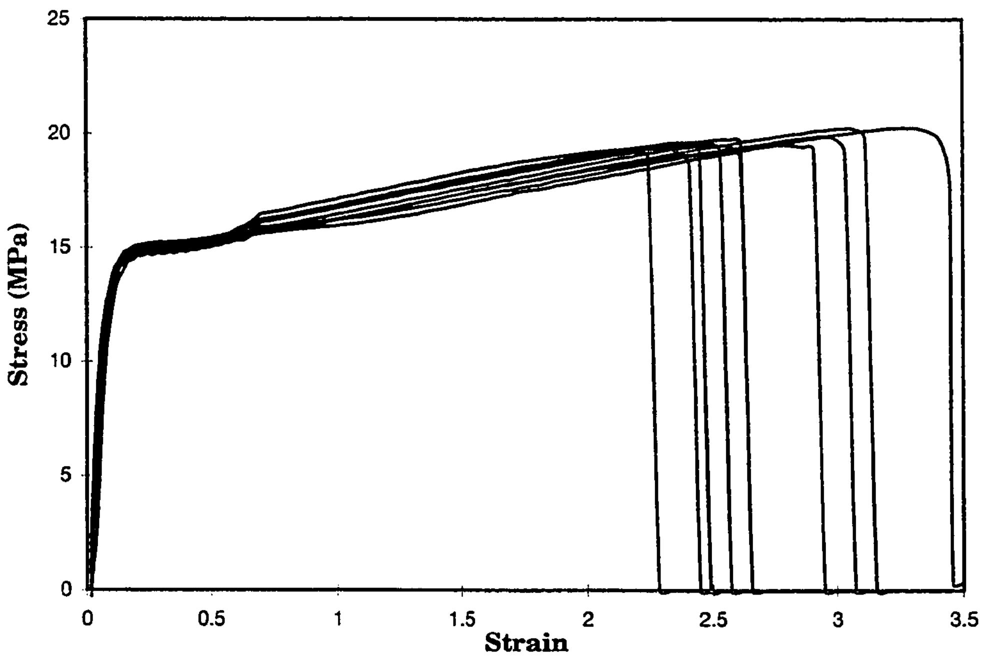
Dimensional Data: HDPE #1 (data in mm)

Material	OD - X	OD - Y	ID - X	ID - Y
HDPE #2 (0 MRad)				
Unit 1	1.063	0.970	0.711	0.597
Unit 2	1.067	0.983	0.704	0.651
Unit 3	1.102	0.940	0.694	0.619
Unit 4	1.069	0.961	0.699	0.644
HDPE #2 (30 MRad)				
Unit 1	1.033	0.998	0.675	0.613
Unit 2	1.064	0.993	0.678	0.620
Unit 3	1.006	1.014	0.663	0.657
Unit 4	1.038	1.021	0.656	0.645
HDPE #2 (50 MRad)				
Unit 1	1.005	0.964	0.657	0.606
Unit 2	1.026	0.957	0.670	0.613
Unit 3	0.996	0.986	0.673	0.650
Unit 4	1.043	0.991	0.668	0.658
HDPE #2 (70 MRad)				
Unit 1	1.036	0.973	0.678	0.635
Unit 2	1.046	0.948	0.676	0.626
Unit 3	1.016	0.954	0.686	0.617
Unit 4	1.050	0.979	0.685	0.597
Outer Diameter				
Average	**1.009**			
Standard Deviation	**0.041**			
Inner Diameter				
Average	**0.654**			
Standard Deviation	**0.032**			

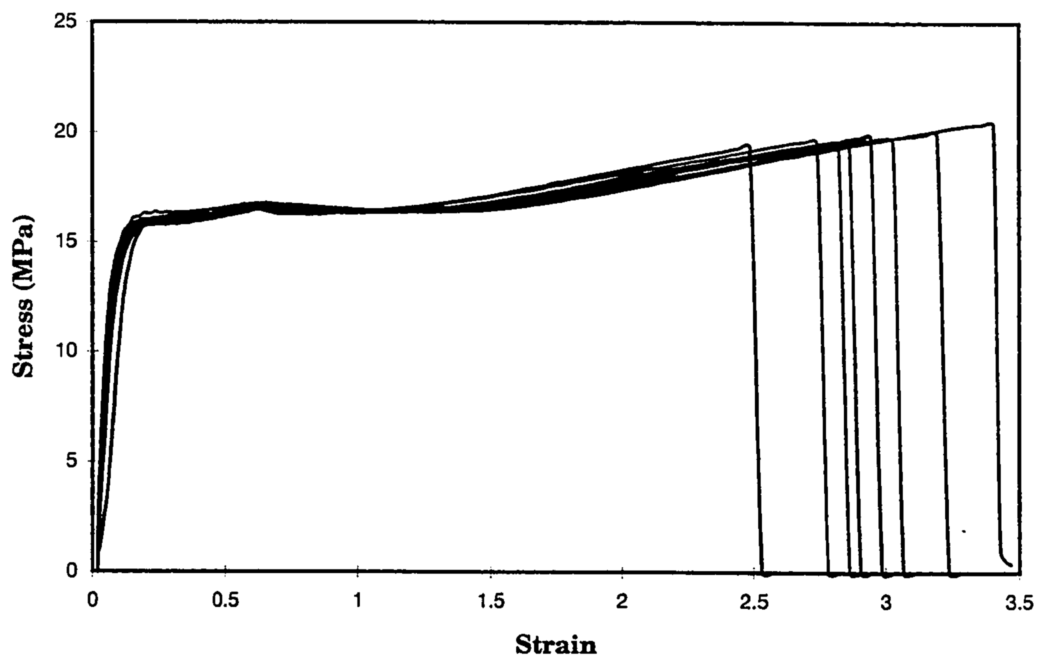
Dimensional Data: HDPE #2 (data in mm)

Appendix C

TENSILE TEST STRESS-STRAIN CURVES



Tensile Test Stress-Strain Curves: LLDPE #1 (0 MRad)



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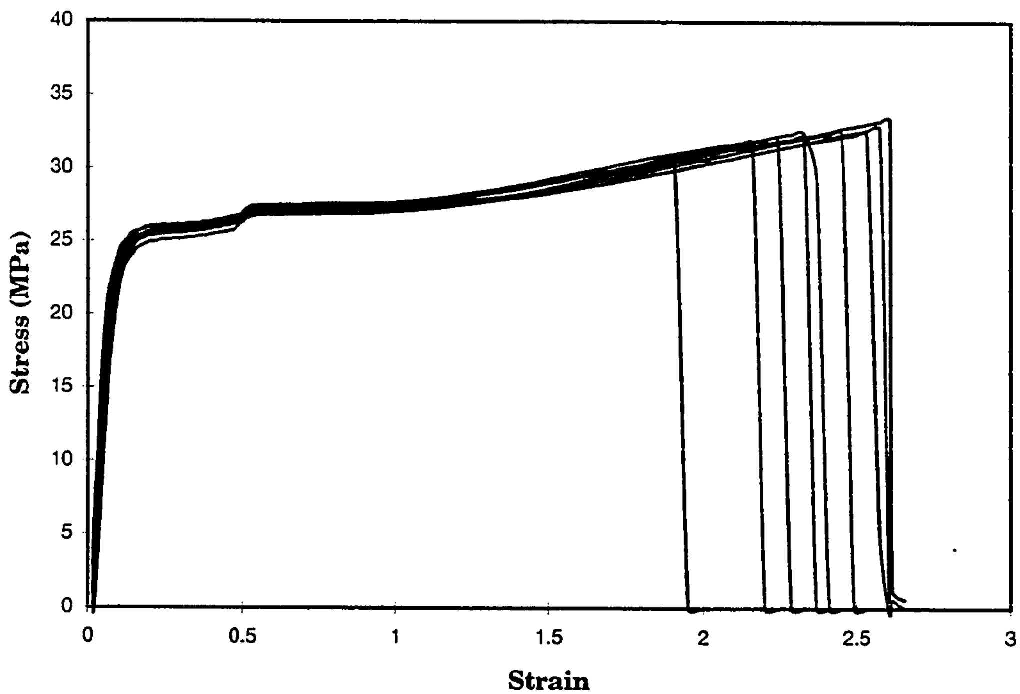

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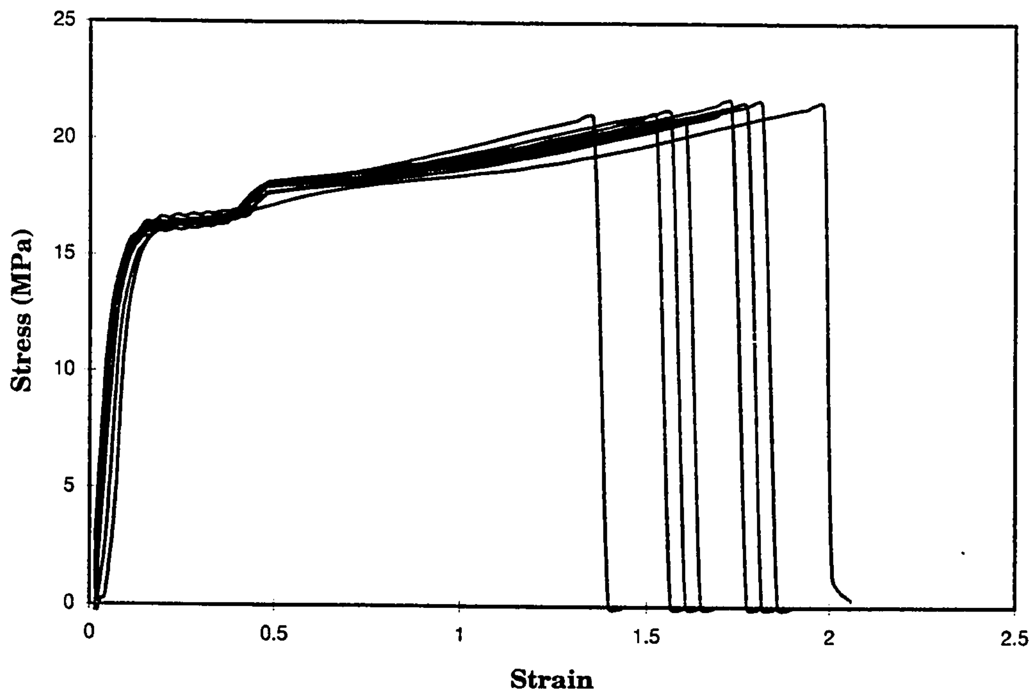

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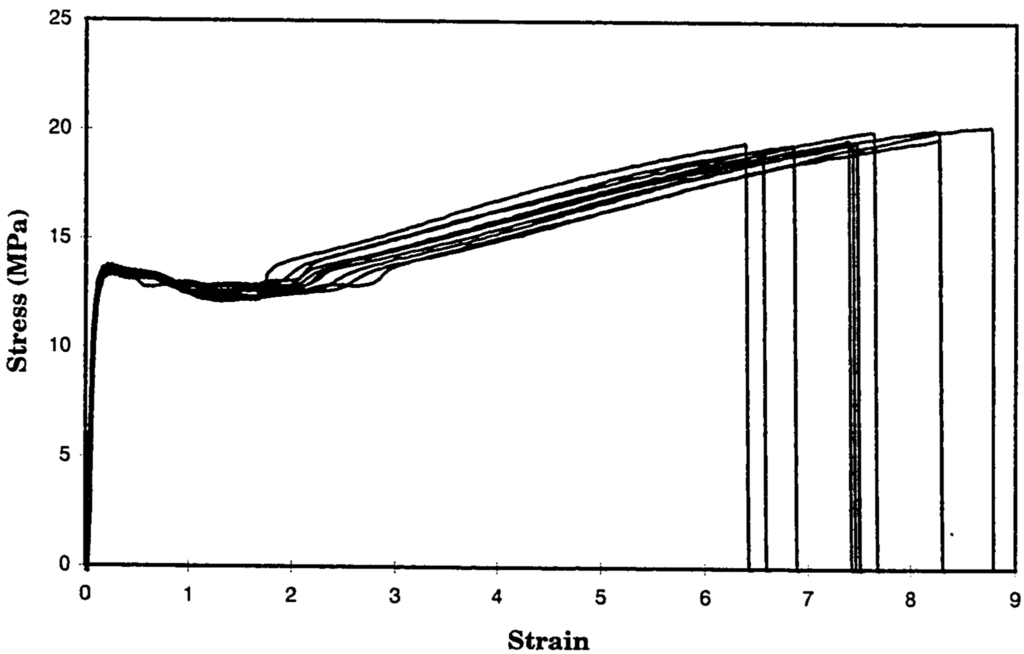
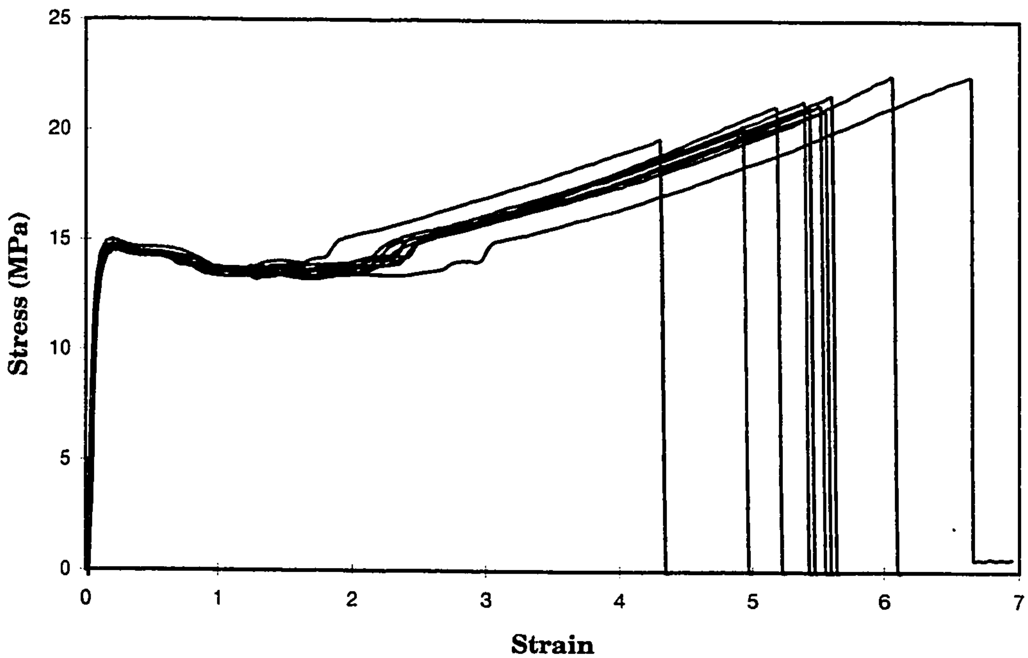


Tensile Test Stress-Strain Curves: LLDPE #2 (0 MRad)



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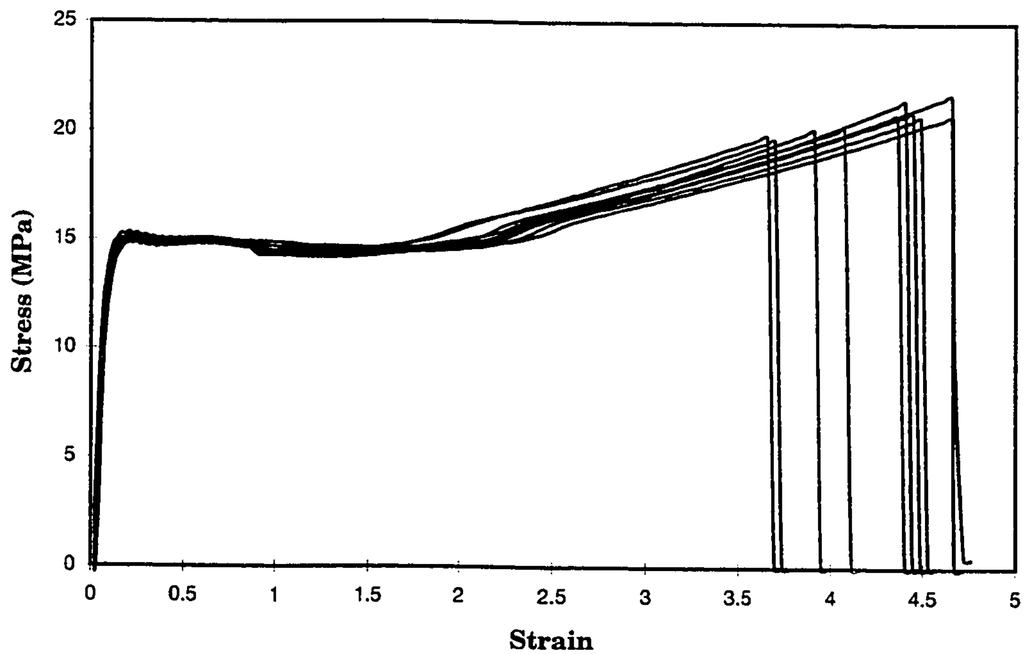

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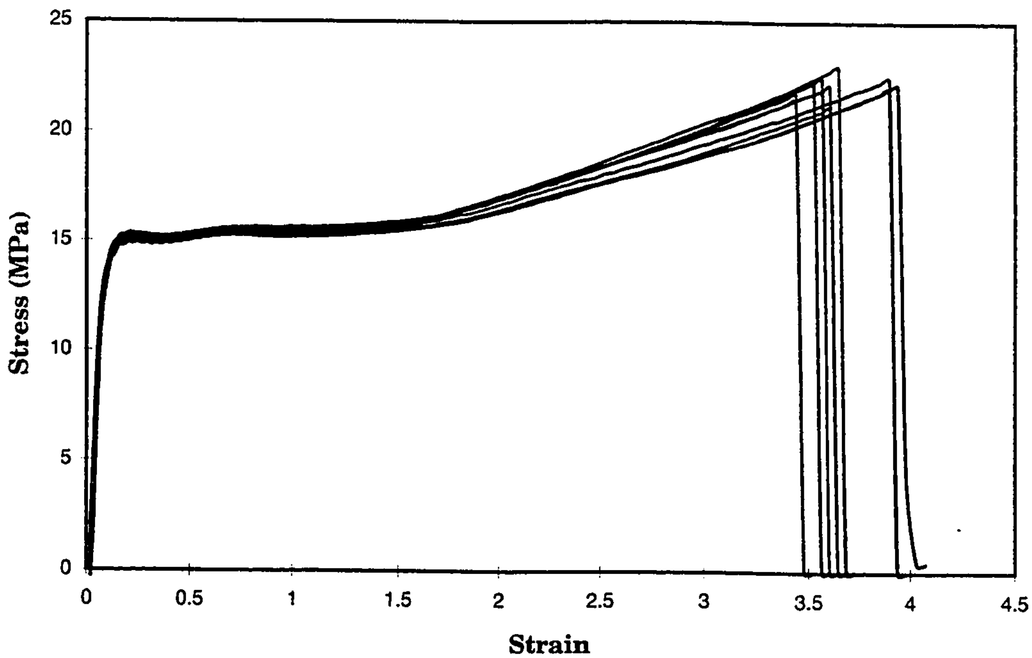
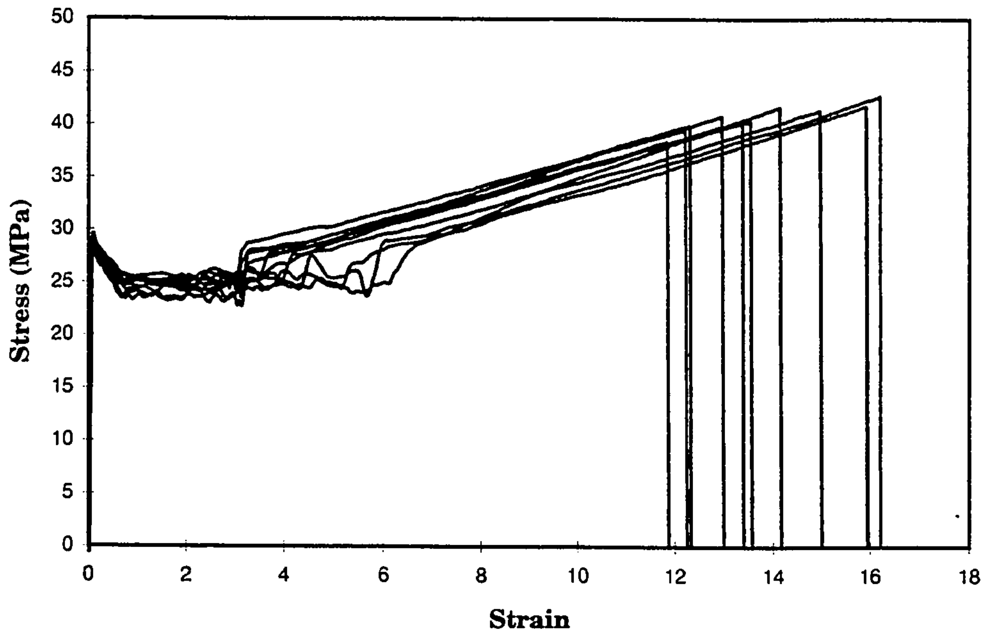
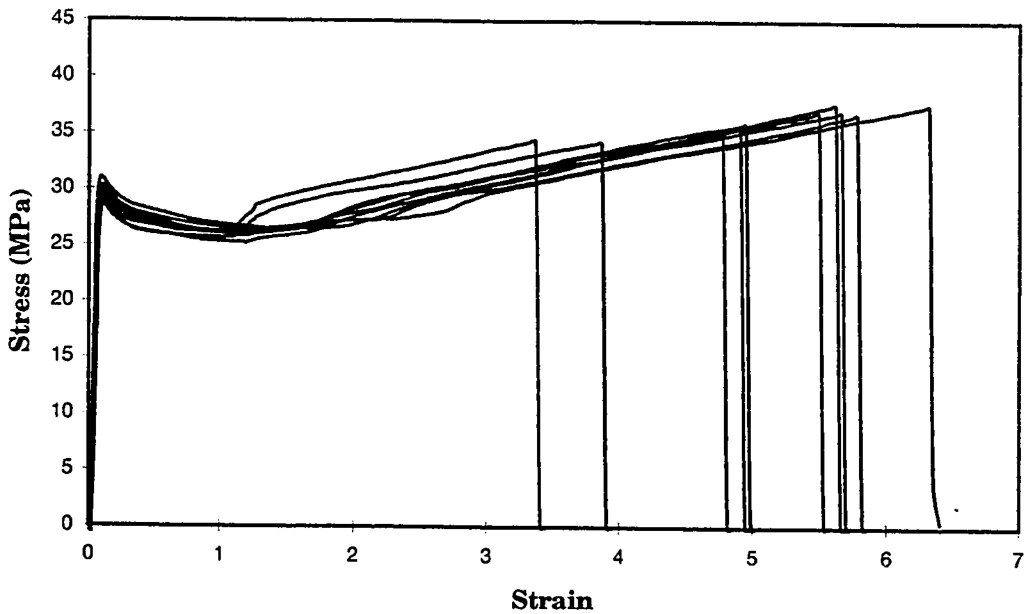


Tensile Test Stress-Strain Curves: LLDPE #2 (70 MRad)



Tensile Test Stress-Strain Curves: HDPE #1 (0 MRad)



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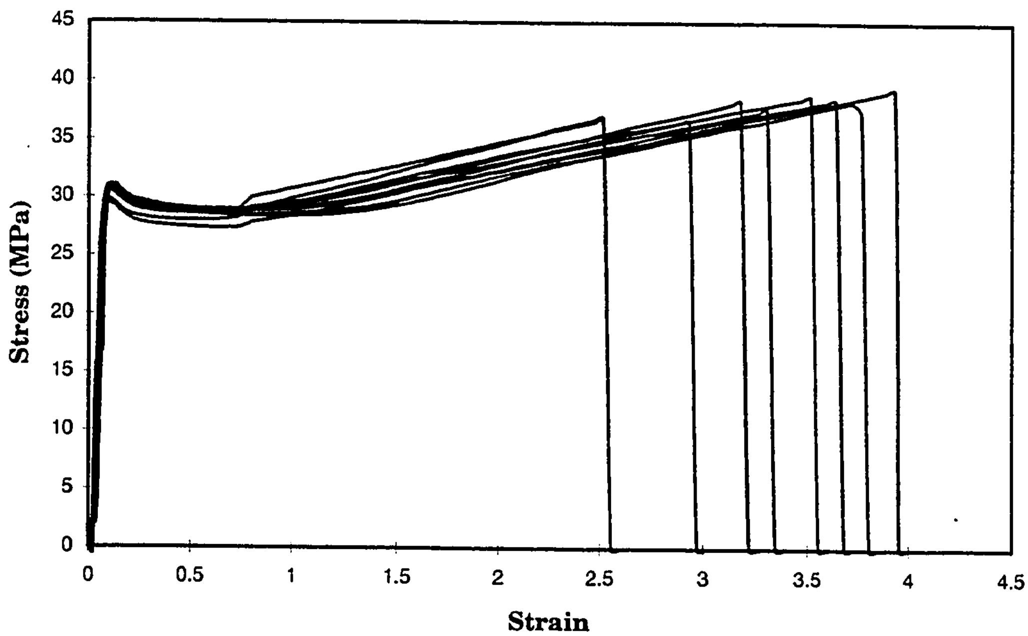
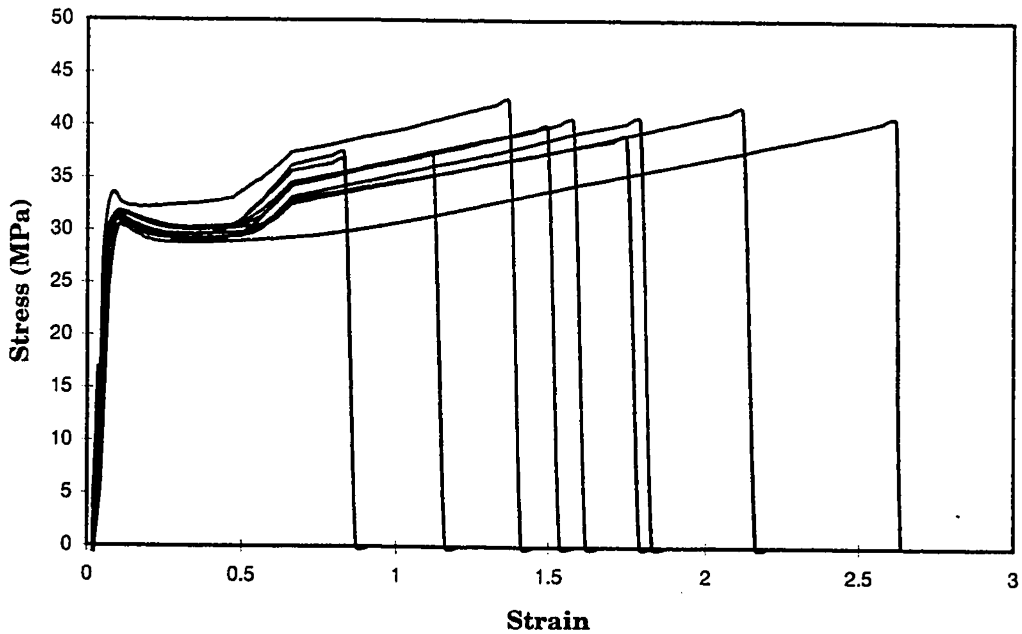
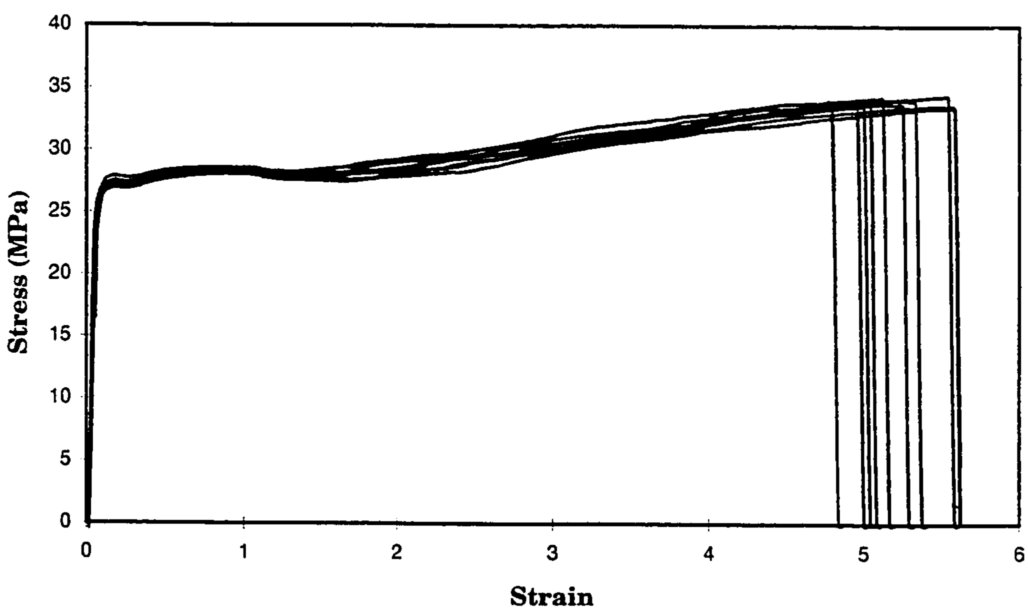


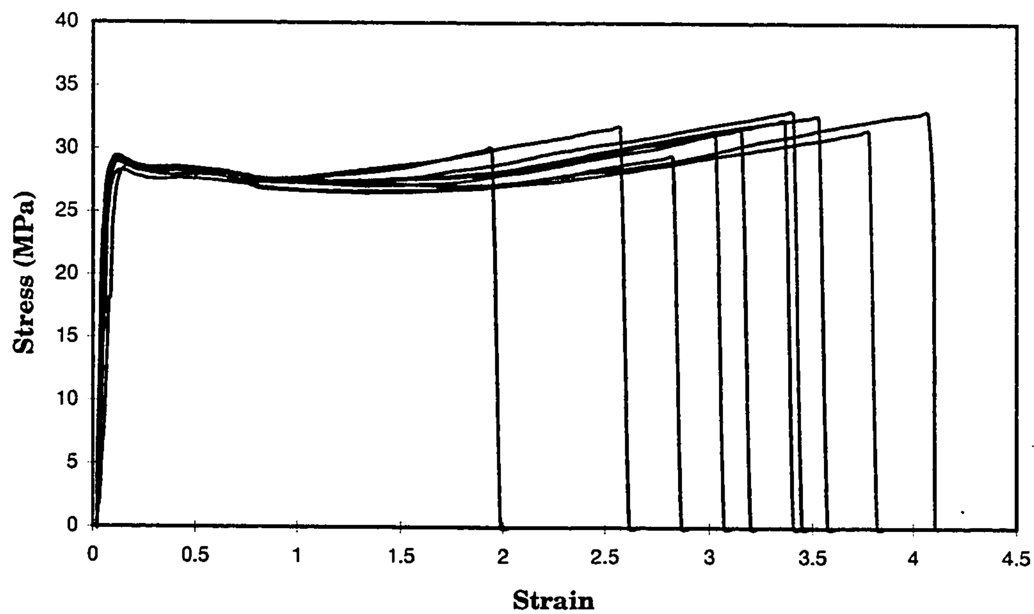
Tensile Test Stress-Strain Curves: HDPE #1 (50 MRad)



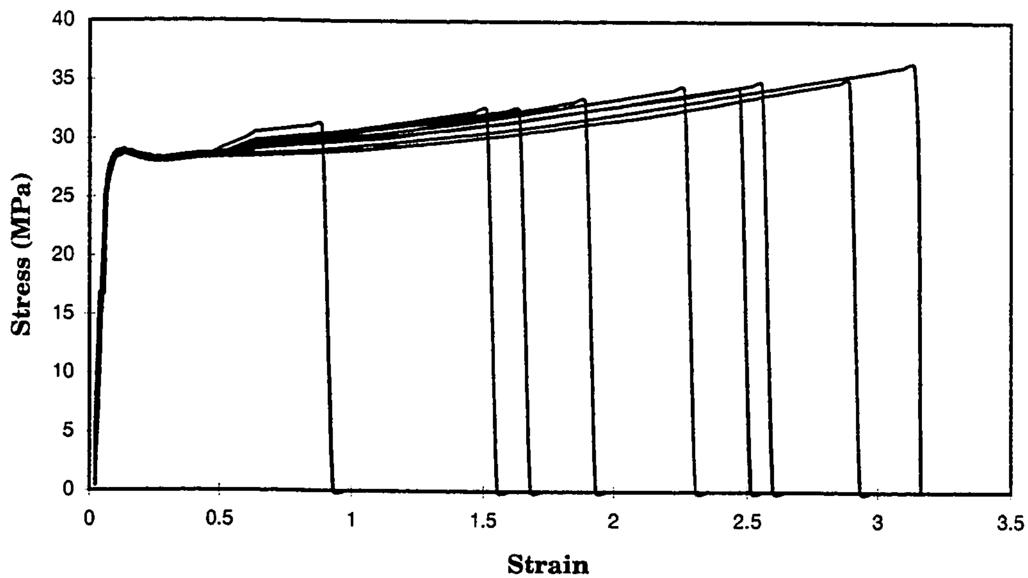
Tensile Test Stress-Strain Curves: HDPE #1 (70 MRad)



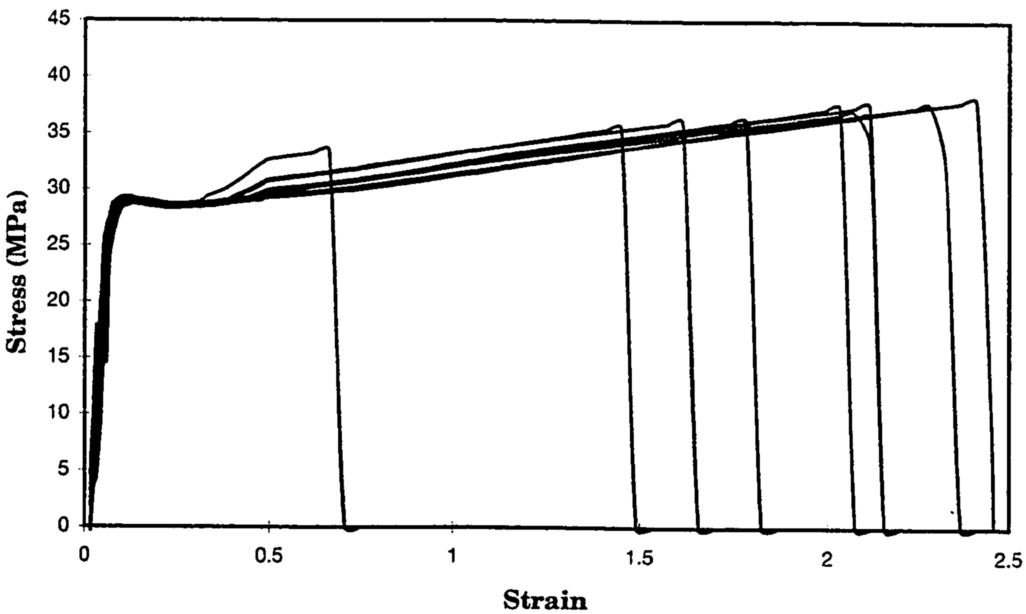
Tensile Test Stress-Strain Curves: HDPE #2 (0 MRad)



Tensile Test Stress-Strain Curves: HDPE #2 (30 MRad)



Tensile Test Stress-Strain Curves: HDPE #2 (50 MRad)



Tensile Test Stress-Strain Curves: HDPE #2 (70 MRad)

Appendix D

FLEXIBILITY TEST DATA

Deflection Angle	3	6	9	12	15	20	25	30	35	40	50	60	70	80	90
LLDPE #1 (0 MRad)															
Unit 1	3	5	7	8	10	12	14	16	17	18	21	22	23	23	23
Unit 2	3	5	7	9	10	13	15	16	18	19	21	22	23	24	24
Unit 3	3	5	7	9	10	13	14	16	18	19	21	22	23	23	24
Unit 4	3	5	7	9	11	13	15	16	18	19	21	22	23	23	24
Unit 5	2	4	6	8	10	12	14	16	17	18	19	21	22	23	23
Unit 6	3	5	7	9	11	13	15	17	18	20	22	23	23	24	24
Unit 7	2	4	6	8	9	11	13	15	16	17	19	21	21	22	22
Unit 8	3	5	7	9	10	13	15	17	18	20	22	23	23	24	24
Unit 9	2	5	7	9	11	13	15	17	18	20	22	23	24	24	24
Unit 10	3	5	7	9	10	13	15	17	19	20	22	23	24	24	24
AVERAGE	2.7	4.8	6.8	8.7	10.2	12.6	14.5	16.3	17.7	19.0	21.0	22.2	22.9	23.4	23.6
STD. DEV.	0.5	0.4	0.4	0.5	0.6	0.7	0.7	0.7	0.8	1.1	1.2	0.8	0.9	0.7	0.7
LLDPE #1 (30 MRad)															
Unit 1	3	5	8	10	11	14	16	18	19	20	22	22	24	24	25
Unit 2	4	6	8	10	12	15	17	19	21	22	24	25	26	26	26
Unit 3	4	7	9	11	13	16	18	20	22	23	25	26	27	27	27
Unit 4	3	5	8	10	12	14	17	19	21	22	24	25	26	26	27
Unit 5	3	6	9	10	13	16	18	20	22	23	25	26	27	28	28
Unit 6	3	6	8	10	12	15	17	19	21	22	24	25	26	26	26
Unit 7	3	6	8	10	12	15	17	19	21	22	24	25	26	26	27
Unit 8	3	6	8	11	13	15	18	20	22	23	25	26	27	27	28
Unit 9	3	6	8	10	12	15	18	20	22	23	26	27	28	28	29
Unit 10	4	7	9	11	13	16	18	21	22	24	26	27	28	28	28
AVERAGE	3.3	6.0	8.3	10.3	12.3	15.1	17.4	19.5	21.3	22.4	24.5	25.4	26.5	26.6	27.1
STD. DEV.	0.5	0.7	0.5	0.5	0.7	0.7	0.7	0.8	0.9	1.1	1.2	1.4	1.2	1.3	1.2
LLDPE #1 (50 MRad)															
Unit 1	3	6	8	10	12	15	17	19	21	23	24	26	27	27	27
Unit 2	4	8	10	12	14	17	20	21	23	25	26	28	28	29	29
Unit 3	3	6	8	10	12	15	17	20	22	23	25	27	28	28	28
Unit 4	4	6	9	11	13	15	18	20	22	23	25	27	28	28	28
Unit 5	4	6	8	10	12	15	17	19	21	23	25	26	27	27	27
Unit 6	4	6	8	10	12	14	17	19	20	22	24	26	26	27	27
Unit 7	3	6	8	11	12	15	18	20	22	23	25	27	28	28	28
Unit 8	4	6	9	11	13	16	18	20	22	23	25	27	28	28	28
Unit 9	4	6	8	11	13	15	18	20	22	23	25	26	27	27	28
Unit 10	4	7	9	12	13	16	19	21	23	24	26	27	28	28	28
AVERAGE	3.7	6.3	8.5	10.8	12.6	15.3	17.9	19.9	21.8	23.2	25.0	26.7	27.5	27.7	27.8
STD. DEV.	0.5	0.7	0.7	0.8	0.7	0.8	1.0	0.7	0.9	0.8	0.7	0.7	0.7	0.7	0.6
LLDPE #1 (70 MRad)															
Unit 1	4	6	9	11	13	16	18	20	22	24	26	28	29	29	30
Unit 2	4	7	9	11	13	16	19	21	23	24	26	28	29	29	29
Unit 3	4	6	8	10	12	15	17	19	21	22	24	26	27	27	27
Unit 4	4	6	9	11	13	15	18	20	22	23	25	26	27	27	27
Unit 5	3	6	9	11	13	16	18	20	21	23	25	27	28	28	28
Unit 6	4	7	9	11	13	15	18	20	22	23	25	27	27	28	28
Unit 7	3	6	8	10	12	15	17	19	21	22	24	26	26	27	27
Unit 8	4	7	9	11	12	15	17	19	21	22	24	26	26	27	27
Unit 9	4	6	8	11	13	15	18	20	22	23	25	27	28	28	28
Unit 10	3	6	8	10	12	15	17	20	22	23	25	26	27	28	28
AVERAGE	3.7	6.3	8.6	10.7	12.6	15.3	17.7	19.8	21.7	22.9	24.9	26.7	27.4	27.8	27.9
STD. DEV.	0.5	0.5	0.5	0.5	0.5	0.5	0.7	0.6	0.7	0.7	0.7	0.8	1.1	0.8	1.0

Flexibility Test Data: LLDPE #1

Angular Deflection	3	6	9	12	15	20	25	30	35	40	50	60	70	80	90
LLDPE #2 (0 MRad)															
Unit 1	3	5	7	8	10	12	13	15	16	17	19	20	21	21	21
Unit 2	2	4	6	8	9	11	13	15	17	18	20	21	21	21	22
Unit 3	3	5	6	8	9	11	13	14	16	17	19	20	20	20	20
Unit 4	2	4	5	7	8	10	12	13	15	16	17	18	19	19	19
Unit 5	3	5	7	8	9	11	13	15	16	17	19	20	21	21	21
Unit 6	3	5	7	8	10	12	14	15	16	18	19	20	21	21	21
Unit 7	2	4	6	8	9	11	13	15	16	17	19	20	21	21	22
Unit 8	2	4	5	7	8	10	12	13	15	16	17	18	19	19	20
Unit 9	4	6	8	9	10	12	14	16	17	18	20	21	21	21	22
Unit 10	3	5	6	8	9	11	13	14	16	17	18	19	20	20	21
AVERAGE	2.7	4.7	6.3	7.9	9.1	11.1	13.0	14.5	16.0	17.1	18.7	19.7	20.4	20.4	20.9
STD. DEV.	0.7	0.7	0.9	0.6	0.7	0.7	0.7	1.0	0.7	0.7	1.1	1.1	0.8	0.8	1.0
LLDPE #2 (30 MRad)															
Unit 1	4	6	8	10	11	13	16	18	19	20	23	24	24	25	25
Unit 2	3	5	7	8	10	12	14	16	17	19	21	22	22	23	23
Unit 3	3	6	7	9	11	13	15	17	18	20	22	23	23	24	24
Unit 4	3	6	7	9	11	13	15	17	18	20	21	23	23	24	24
Unit 5	3	6	7	9	11	13	15	17	18	20	21	23	23	23	24
Unit 6	4	6	8	10	11	14	16	18	19	21	23	24	25	25	25
Unit 7	3	5	7	9	10	13	15	17	18	19	21	22	23	23	24
Unit 8	4	6	8	9	11	13	16	17	19	20	22	23	24	25	25
Unit 9	4	6	7	9	10	13	15	17	18	19	21	22	23	23	23
Unit 10	3	6	7	9	11	13	15	17	18	20	21	22	23	24	24
AVERAGE	3.4	5.8	7.3	9.1	10.7	13.0	15.2	17.1	18.2	19.8	21.6	22.8	23.3	23.9	24.1
STD. DEV.	0.5	0.4	0.5	0.6	0.5	0.5	0.6	0.6	0.6	0.6	0.8	0.8	0.8	0.9	0.7
LLDPE #2 (50 MRad)															
Unit 1	4	6	8	10	11	14	16	18	20	21	23	24	25	25	25
Unit 2	3	6	8	10	12	14	16	18	20	21	23	24	24	25	25
Unit 3	4	6	8	10	12	15	17	19	20	22	24	25	26	26	26
Unit 4	4	6	8	10	11	14	16	18	20	21	23	24	25	25	25
Unit 5	4	6	7	9	11	14	16	18	19	21	23	24	25	25	25
Unit 6	4	7	8	10	12	14	16	17	19	20	22	23	24	24	24
Unit 7	4	6	7	9	11	13	15	17	18	19	21	22	23	23	24
Unit 8	4	7	8	10	12	14	17	18	20	21	23	25	25	26	26
Unit 9	4	7	8	10	12	15	17	19	21	22	24	25	25	26	26
Unit 10	3	6	8	10	11	14	17	18	20	21	23	25	26	26	26
AVERAGE	3.8	6.3	7.8	9.8	11.5	14.1	16.3	18.0	19.7	20.9	22.9	24.1	24.8	25.1	25.2
STD. DEV.	0.4	0.5	0.4	0.4	0.5	0.6	0.7	0.7	0.8	0.9	0.9	1.0	0.9	1.0	0.8
LLDPE #2 (70 MRad)															
Unit 1	4	6	8	10	12	14	17	19	20	22	24	25	26	26	26
Unit 2	4	7	9	11	12	15	17	19	20	21	23	24	25	25	26
Unit 3	3	6	7	9	10	12	15	17	18	20	22	22	23	23	24
Unit 4	4	6	8	9	11	13	15	17	18	19	21	22	23	23	23
Unit 5	4	6	8	10	12	14	17	18	20	21	23	25	25	26	26
Unit 6	4	6	8	10	11	14	16	18	19	20	23	24	24	25	25
Unit 7	3	6	8	10	12	14	17	19	21	22	24	25	26	27	27
Unit 8	4	6	8	10	12	14	17	19	20	22	24	25	26	26	27
Unit 9	4	6	7	9	11	14	16	18	19	20	23	24	25	25	25
Unit 10	3	5	8	9	11	13	15	17	18	19	21	23	23	24	24
AVERAGE	3.7	6.0	7.9	9.7	11.4	13.7	16.2	18.1	19.3	20.6	22.8	23.9	24.6	25.0	25.3
STD. DEV.	0.5	0.5	0.6	0.7	0.7	0.8	0.9	0.9	1.1	1.2	1.1	1.2	1.3	1.3	1.3

Flexibility Test Data: LLDPE #2

Angular Deflection	3	6	9	12	15	20	25	30	35	40	50	60	70	80	90
HDPE #1 (0 MRad)															
Unit 1	9	15	21	26	31	37	40	44	47	48	51	51	51	51	49
Unit 2	8	16	21	26	31	36	40	43	46	48	50	51	51	51	49
Unit 3	8	14	19	25	29	34	38	42	45	47	49	50	50	50	48
Unit 4	8	15	21	27	32	38	42	45	48	50	52	53	52	52	50
Unit 5	7	14	19	24	28	34	38	42	44	47	49	50	50	50	48
Unit 6	8	15	20	25	30	35	39	42	45	47	49	50	50	49	48
Unit 7	8	14	20	25	29	34	39	42	45	46	49	50	50	50	49
Unit 8	7	14	20	25	29	34	38	41	44	45	47	48	48	47	46
Unit 9	9	15	21	25	30	35	40	43	46	48	50	51	51	51	49
Unit 10	7	15	21	26	31	37	41	44	47	49	51	51	51	50	48
AVERAGE	7.9	14.7	20.3	25.4	30.0	35.4	39.5	42.8	45.7	47.5	49.7	50.5	50.4	50.1	48.4
STD. DEV.	0.7	0.7	0.8	0.8	1.2	1.5	1.4	1.2	1.3	1.4	1.4	1.3	1.1	1.4	1.1
HDPE #1 (30 MRad)															
Unit 1	7	14	19	25	30	37	42	47	50	53	55	56	55	53	51
Unit 2	8	16	22	28	33	40	44	49	52	55	57	58	58	55	52
Unit 3	8	16	23	29	34	41	46	50	54	56	59	60	59	57	53
Unit 4	9	17	23	29	33	40	45	49	51	54	56	57	57	54	51
Unit 5	8	15	21	27	32	38	44	48	51	53	55	56	55	53	50
Unit 6	8	16	23	28	34	41	46	50	53	56	58	59	58	56	52
Unit 7	10	17	23	30	34	40	45	49	52	53	55	55	54	52	49
Unit 8	10	18	24	29	34	41	46	49	51	53	55	55	54	52	49
Unit 9	9	17	23	30	34	41	46	50	53	55	57	58	57	55	52
Unit 10	9	16	23	28	33	39	44	48	50	53	55	55	54	53	50
AVERAGE	8.6	16.2	22.4	28.3	33.1	39.8	44.8	48.9	51.7	54.1	56.2	56.9	56.1	54.0	50.9
STD. DEV.	1.0	1.1	1.4	1.5	1.3	1.4	1.3	1.0	1.3	1.3	1.5	1.8	1.9	1.7	1.4
HDPE #1 (50 MRad)															
Unit 1	9	16	23	29	34	40	46	49	51	53	56	57	56	54	51
Unit 2	9	16	23	29	34	41	46	50	52	55	57	58	57	55	52
Unit 3	9	15	22	28	34	40	45	49	52	54	57	58	57	55	52
Unit 4	10	16	22	28	33	39	44	48	51	53	55	56	55	53	50
Unit 5	9	17	24	30	35	42	47	52	56	58	61	62	61	58	55
Unit 6	8	15	21	27	31	38	44	48	51	53	55	55	55	53	50
Unit 7	8	16	23	28	33	41	46	50	54	56	58	59	58	55	52
Unit 8	8	16	23	29	34	42	47	51	53	56	58	59	58	55	51
Unit 9	9	16	22	27	32	39	44	47	50	52	54	54	53	51	48
Unit 10	8	16	22	28	32	39	45	48	52	54	55	56	55	52	50
AVERAGE	8.7	15.9	22.5	28.3	33.2	40.1	45.4	49.2	52.2	54.4	56.6	57.4	56.5	54.1	51.1
STD. DEV.	0.7	0.6	0.8	0.9	1.2	1.4	1.2	1.5	1.8	1.8	2.1	2.3	2.2	2.0	1.9
HDPE #1 (70 MRad)															
Unit 1	10	18	25	31	37	44	50	54	58	61	63	64	63	59	56
Unit 2	10	18	26	33	38	46	52	57	61	65	68	68	68	64	60
Unit 3	10	17	25	31	37	44	49	53	57	60	61	61	60	56	53
Unit 4	9	18	25	32	37	45	50	55	58	60	62	62	61	56	54
Unit 5	10	18	25	31	36	44	49	53	58	61	63	63	62	60	56
Unit 6	9	16	22	28	33	40	45	49	52	55	57	57	57	54	50
Unit 7	9	17	23	29	34	42	47	52	56	59	61	62	61	58	55
Unit 8	10	18	25	31	37	44	49	53	57	59	62	62	62	57	54
Unit 9	8	16	23	29	34	42	47	51	54	57	59	59	58	54	51
Unit 10	7	16	23	30	35	43	49	53	57	61	63	63	63	59	56
AVERAGE	9.2	17.2	24.2	30.5	35.8	43.4	48.7	53.0	56.8	59.8	61.9	62.1	61.5	57.7	54.5
STD. DEV.	1.0	0.9	1.3	1.5	1.7	1.7	1.9	2.2	2.4	2.7	2.9	2.9	3.0	3.0	2.8

Flexibility Test Data: HDPE #1

Angular Deflection	3	6	9	12	15	20	25	30	35	40	50	60	70	80	90
HDPE #2 (0 MRad)															
Unit 1	7	12	17	21	25	30	33	36	39	41	43	45	45	45	44
Unit 2	6	11	16	20	23	28	32	34	37	39	41	42	43	43	42
Unit 3	7	12	17	21	25	30	33	36	38	40	42	44	44	44	43
Unit 4	7	13	18	22	26	31	34	37	40	42	44	45	46	46	45
Unit 5	7	13	18	22	26	30	34	37	39	41	43	44	45	45	44
Unit 6	7	13	17	21	25	30	33	36	38	40	42	43	43	43	42
Unit 7	7	13	17	21	25	29	33	35	38	40	42	43	43	43	42
Unit 8	7	13	17	21	24	28	32	34	37	39	41	42	42	42	42
Unit 9	8	14	18	23	27	31	35	37	40	41	43	44	44	44	42
Unit 10	7	13	18	22	25	29	33	35	38	40	42	43	43	43	42
AVERAGE	7.0	12.7	17.3	21.4	25.1	29.6	33.2	35.7	38.4	40.3	42.3	43.5	43.8	43.8	42.8
STD. DEV.	0.5	0.8	0.7	0.8	1.1	1.1	0.9	1.2	1.1	0.9	0.9	1.1	1.2	1.2	1.1
HDPE #2 (30 MRad)															
Unit 1	8	14	19	24	29	35	39	43	46	48	51	52	52	51	50
Unit 2	8	15	20	25	29	35	39	43	46	48	51	52	52	51	49
Unit 3	8	15	21	26	31	37	42	46	49	51	54	56	55	55	52
Unit 4	8	14	20	25	30	36	41	45	48	50	53	55	55	55	53
Unit 5	7	13	18	23	27	31	35	39	42	44	47	48	47	46	43
Unit 6	7	14	20	25	29	35	40	43	46	49	51	53	53	52	50
Unit 7	8	15	21	26	30	36	40	44	47	49	52	53	52	52	49
Unit 8	8	15	20	25	30	35	40	44	46	48	51	51	51	51	50
Unit 9	8	14	20	24	28	35	39	42	44	46	48	49	49	48	47
Unit 10	9	16	22	26	31	37	41	44	47	49	52	53	53	52	50
AVERAGE	7.9	14.5	20.1	24.9	29.4	35.2	39.6	43.3	46.1	48.2	51.0	52.2	51.9	51.3	49.3
STD. DEV.	0.6	0.8	1.1	1.0	1.3	1.7	1.9	1.9	2.0	2.0	2.1	2.4	2.5	2.8	2.8
HDPE #2 (50 MRad)															
Unit 1	8	15	21	25	30	36	40	43	46	48	50	51	51	50	47
Unit 2	8	15	20	25	29	35	40	44	46	48	51	52	52	51	46
Unit 3	9	16	22	27	31	38	43	46	49	52	54	55	55	55	52
Unit 4	8	14	20	24	28	34	39	42	45	47	49	50	50	49	47
Unit 5	9	15	22	27	31	37	42	45	48	50	53	54	54	54	51
Unit 6	9	16	22	28	32	38	43	47	50	53	56	57	57	56	53
Unit 7	8	15	21	26	31	37	42	45	48	50	53	54	54	53	49
Unit 8	9	16	22	27	32	38	43	46	49	51	54	54	55	54	51
Unit 9	8	14	20	25	29	36	40	44	47	49	51	52	52	51	48
Unit 10	9	15	21	26	31	37	41	45	48	49	52	52	52	51	49
AVERAGE	8.5	15.1	21.1	26.0	30.4	36.6	41.3	44.7	47.6	49.7	52.3	53.1	53.2	52.4	49.3
STD. DEV.	0.5	0.7	0.9	1.2	1.3	1.3	1.5	1.5	1.6	1.9	2.1	2.1	2.1	2.3	2.4
HDPE #2 (70 MRad)															
Unit 1	9	15	21	26	30	37	41	44	47	48	51	51	51	50	47
Unit 2	9	16	22	27	32	38	43	46	50	52	55	56	56	55	52
Unit 3	8	14	20	24	29	35	40	43	48	50	51	51	50	49	48
Unit 4	7	14	19	24	29	35	40	43	47	49	52	53	53	51	48
Unit 5	9	16	21	27	31	38	42	45	49	52	55	56	56	55	52
Unit 6	6	13	18	23	27	34	38	42	45	48	50	51	51	50	48
Unit 7	7	15	21	26	30	37	42	45	48	51	54	55	55	54	50
Unit 8	8	15	20	25	30	36	41	45	48	50	54	55	55	54	52
Unit 9	8	14	20	25	30	36	40	44	47	49	50	51	51	50	49
Unit 10	8	14	19	24	28	35	39	42	46	48	49	50	50	49	47
AVERAGE	7.9	14.6	20.1	25.1	29.6	36.1	40.6	43.9	47.5	49.7	52.1	52.9	52.8	51.7	49.3
STD. DEV.	1.0	1.0	1.2	1.4	1.4	1.4	1.5	1.4	1.4	1.6	2.2	2.4	2.5	2.5	2.1

Flexibility Test Data: HDPE #2