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The Effect of Liquid Hot Filling Temperature on Blow-Molded HDPE Bottle Properties

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THE EFFECT OF LIQUID HOT FILLING TEMPERATURE ON BLOW-MOLDED HDPE BOTTLE PROPERTIES

by

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GRADUATE COMMITTEE APPROVAL

of a thesis submitted by

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This thesis has been read by each member of the following graduate committee and by majority vote has been found to be satisfactory.

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BRIGHAM YOUNG UNIVERSITY

As chair of the candidate's graduate committee, I have read the thesis of Benjamin S. Hudson in its final form and have found that (1) its format, citations, and bibliographical style are consistent and acceptable and fulfill university and department style requirements; (2) its illustrative materials including figures, tables, and charts are in place; and (3) the final manuscript is satisfactory to the graduate committee and is ready for submission to the university library.

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ABSTRACT

THE EFFECT OF LIQUID HOT FILLING

TEMPERATURE ON BLOW-MOLDED

HDPE BOTTLE PROPERTIES

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Master of Science

The occurrence of deformation in plastic bottles is a common problem in the bottling industry where bottles are blow molded, hot filled at high temperatures and sealed. Plastics have unique properties that make it difficult to predict when and why such changes may occur. The root cause of such deformation is unknown by many bottle producers and recent attempts have been made to minimize the occurrence of such defects.

The purpose of this research is to determine which variables involved in the bottle production process influence bottle shape. Earlier variables that were tested included both blow molding resin and total bottle sidewall thickness. The result of changing these variables did not create a decrease in defects. The use of an Ishikawa fishbone diagram identified hot filling temperature a major variable that influences final bottle shape.

This research summarizes the results of a series of tests that were developed to observe the effect of hot filling temperature on final bottle shape. A positive correlation between sidewall deflection and liquid hot filling temperature was observed.

A series of tensile tests were also developed to analyze the strength of various regions of a blow molded bottle. An early Pareto Analysis determined that the parting line is more susceptible to defects than any other region of the bottle. This weakness was confirmed after the tensile tests proved that there is a statistically significant difference between measurements on the sidewall and parting line (pvalue $< .001$).

The results of this thesis highlight the consequences of arbitrarily choosing a filling temperature with little understanding of the bottle's strength at high temperatures. Plastic bottle producers and hot filling companies should unite to determine the appropriate hot filling temperature before bottles are molded and filled.

ACKNOWLEDGMENTS

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I am also grateful to various individuals at Brigham Young University who have shared their time and talents to assist in the research and testing. I am equally grateful to my wife, Chelsea, who has been a constant support to me throughout the creation of this thesis.

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1 Introduction

Within the bottling industry, blow molded high-density polyethylene (HDPE) bottles have replaced heavier metal glass bottles for a variety of products (Miller 2005). The use of HDPE in the bottling industry has provided numerous advantages such as versatility, low weight, safety and hygiene, cost-effectiveness, and durability (Sunderland 2000). Conversely, the application of HDPE has generated inherent weaknesses in and limitations to the bottles being used. These weaknesses are not linked to the material alone but to the processes being used to produce the bottles.

These weaknesses and limitations include inconsistent wall thickness due to variation in the blow molding process, low melting points of blow molding resins, and variations in the mechanical and thermal properties among resin grades. In some applications these weaknesses can lead to costly defects such as morphological shape changes in the bottles that emerge after the liquid hot filling phase of production.

This thesis was inspired by a need to identify an appropriate method to use in understanding and diagnosing HDPE bottle defects that have been identified after the liquid hot filling phase of production.

The results of this thesis provide a framework that bottle producers can use to identify potential sources of defects. The thesis also presents a new method of testing that determines the impact of hot filling temperature on the final shape of HDPE bottles.

1.1 Statement of the Problem

The occurrence of paneling and other forms of deformation in plastic bottles is a common problem in the plastic bottling industry where bottles are blow molded, hot filled at high temperatures for pasteurization, and sealed. For most bottling companies, discovering a solution to overcome these defects is often a trial and error process and can be very costly. Many companies are tolerating a certain percentage of defects because of the tremendous savings and flexibility associated with using plastics. As the percentage of defects increases, the cost of defects can offset these savings. The emergence of defects can also have a negative impact on the end customer's perception of product quality.

The main problem of bottle deformation is a process problem, wherein the underlying source of defects is unknown. An additional problem with deformation is root cause traceability of such defects. This issue has lead to disputations between bottle manufacturing companies and hot filling companies concerning accountability for the cost of defective bottles. Although many types of defects can occur, the study is only concerned with those defects related to visible deformation at the surface of the bottle.

1.2 Purpose of the Study

The purpose of the research is to determine which variables involved in the production of blow molded HDPE bottles influence final bottle shape. The researcher will identify these variables and perform further tests to determine their influence on final bottle shape. The research will also test the impact of hot filling temperature on final bottle shape.

1.3 Significance of the Study

There is a need for a greater understanding of the source of defects in the plastic bottling industry. Blow molded plastic bottles comprise the majority of bottles used in the bottling industry today (Harper 2006), and information related to improvements in their production process will provide benefits to both bottle producers and consumers. These improvements will also allow bottle producers to test new bottle designs without lost production time. Many companies have experienced problems with groups of defects in production batches and have worked independently to overcome such problems.

To fully understand and control defects, organizations should begin by having a standard model that is understood and used by everyone and may be improved over time (Okes 2003). The development of a systematic approach to deal with such problems will be beneficial to those individuals experiencing various types of defects.

The major focus of the research is to determine the effect of liquid hot filling temperature on blow molded HDPE bottle properties. The research will also seek to determine the root cause(s) of defects occurring in a specific HDPE bottle after the blow molding, hot filling, and sealing processes have occurred, and to provide a framework for discovering such causes.

The findings of the research will be used to help manufacturers understand the influence of hot filling temperature on the final shape of HDPE bottles. The results will also be used to resolve a dispute between two manufacturing companies, Sonic Plastics (a custom blow molding company in Lindon, UT) and Fill Co. Custom Fillers (a hot filling company in Saint George, UT). Prior to this research, both companies agreed to participate in the effort in an attempt to receive a non-biased analysis of the true nature of bottle deformation, which they have experienced on numerous occasions.

The research will be used to provide information that will be instrumental in resolving their debate dealing with accountability for high levels of defects among recent production batches involving a specific bottle design and liquid. These companies have made changes to some variables associated with their product and process. These changes are documented in Chapter 3 of the thesis and provide an overview of their prior work.

In addition, the research will provide a framework that companies can use to determine the processing capabilities of the bottles that they are hot filling. The research methodology will also provide a way for companies to diagnose future defects occurring among HDPE bottles.

In the research, a defect will be defined as any morphological shape change or deformation that occurs on the bottle's outer surface and significantly differs from the virgin bottle's shape prior to filling. This type of defect is often referred to as paneling. Through research, expert opinion and observations, various tests have been selected to determine which variables have the most significant influence on final bottle shape.

1.4 Hypothesis

Through initial observations of samples of defective bottles, and deep analysis of the blow molding and hot filling processes used to create these bottles, the researcher has formed the hypothesis that the hot filling temperature is the primary variable leading to the creation of defects. Therefore the null hypothesis of this thesis is that hot filling temperature has no effect on the final shape of the bottle. This hypothesis will be thoroughly tested through a series of temperature tests known as "hot filling simulations" which will be documented later in the research.

1.5 Assumptions

The major assumptions of this study include the following:

- The type of filling liquid used has not effect on the occurrence of defects
- Third order effects associated with liquid texture and content are negligible
- All HDPE bottles used in testing are identical (See Table 4-9)

1.6 Delimitations

The major delimitations of this study include the following:

- Only 32 oz. Boston Round HDPE bottles (manufactured by Sonic Plastics) were observed and used in the study
- Fill volume was chosen to be 950 mL based on bottle filling specifications used for a sample of 32 oz. Boston Round bottles containing defects
- Other bottle sizes were not considered in the study
- PAXON™ High Density Polyethylene AD60-007 Blow Molding Resin was the only blow molding material used for testing samples
- The liquid hot filling phase of the bottle production is the only process that was simulated. Other processes involved in the production of HDPE bottles were not simulated (See Figure 2-1 for an overview of bottle production process)

- All variables that influence the polymer microstructure of the HDPE resin used in the research will not be tested. A brief overview of the polymer microstructure is included in Chapter 2 of the thesis to provide sufficient background to understand the chemical, mechanical and thermal properties of HDPE
- The research results may not be reproducible at other elevations and in other climates. The testing has taken place in Provo, Utah and results may fluctuate in testing environments with different elevations and levels of humidity
- Ozonated water rinsing of bottles and nitrogen filling will be bundled with the liquid hot filling phase of production in processing descriptions
- The defects analyzed in the thesis only include morphological shape changes and visible deformation that occur on the surface of the bottle after the liquid hot filling operation (paneling). Other common defects such as punctured seals, spoiled liquid, and non-concentric necks will not be considered
- The testing liquid has been limited to a blend of gogi and noni juices, referred to as “Nogi” juice. Other liquids were not tested. The juice has been provided by Bruce Strong of Sonic Plastics and George Hansen of Advantage Marketing
- The research will only consider bottles formed by the extrusion blow molding process and hot filled in a separate facility. Bottles produced with

a preform or via the blow-fill-seal production method (Oshmann 1999) will not be considered

Although many variables have the potential to influence the shape of the bottle, the research will only test the influence of liquid hot filling temperature on the final shape of the bottle. This hypothesis is based on existing research, industry data, and conversations with custom hot fillers and experts in the field of plastics including Dr. Brent Strong and other members of the thesis committee.

2 Background and Review of Literature

The production process for plastic bottles will be summarized in this section, in addition to an analysis of high-density polyethylene (HDPE). A flowchart of the production process of plastic bottles can be seen in Figure 2.1 below.

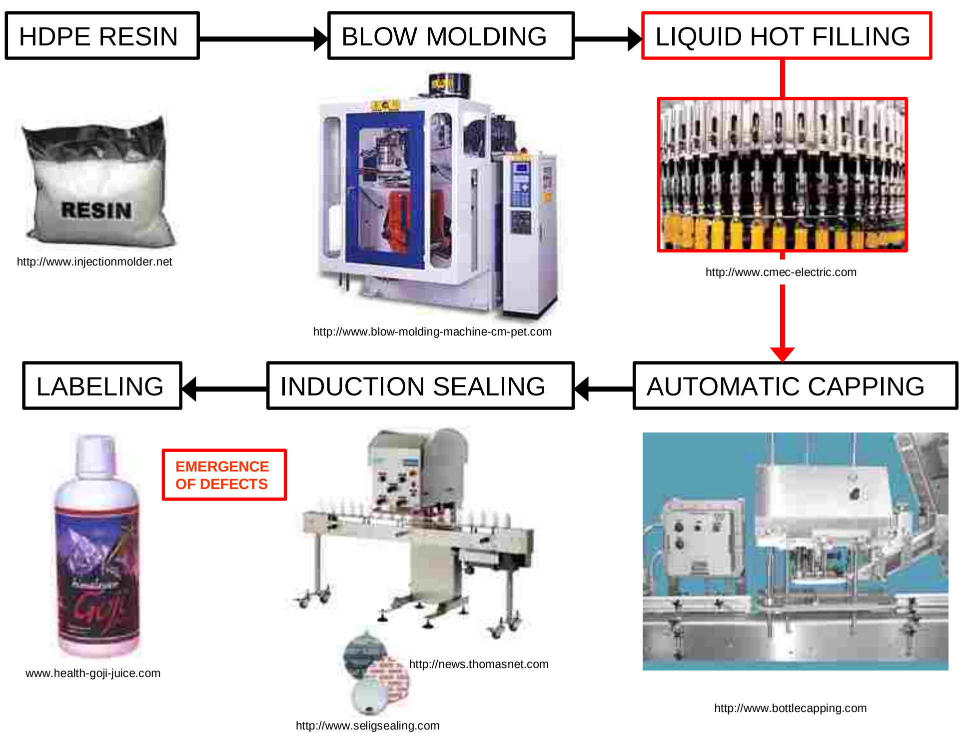

Figure 2-1 Commercial beverage production process flow chart

2.1 High Density Polyethylene (HDPE)

The initial stage of the production of bottles begins with plastic resin. The resin used in this study is known as high-density polyethylene or HDPE. Plastics such as HDPE have been implemented in numerous industries because of their unique material properties. The durability and reliability of a polymer-based product are determined by a number of factors inherent to the material itself (crystallinity and average molecular weight), to its processing (shear-induced degradation and process-induced thermal degradation), and to its service environment (temperature, humidity and the presence of vibrations) (Sunderland 2000).

Polyethylene is the highest-volume polymer in the world. Its high toughness, ductility, excellent chemical resistance, low water vapor permeability, and very low water absorption, combined with the ease with which it can be processed are major benefits for application in the bottling industry (Harper 2006). High-density polyethylene has also added considerable breadth to the design capabilities of plastic bottles; most importantly bottles can now be made with greater stiffness and less weight (Lee 1998).

Polymer Microstructure

In its simplest form a polyethylene molecule consists of a long backbone of an even number of covalently linked carbon atoms with a pair of hydrogen atoms attached at each carbon. The chain ends of HDPE are terminated by methyl groups (Peacock 2000). A schematic of polyethylene’s backbone structure is shown in Figure 2-2 below.

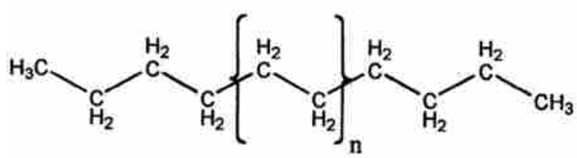

Figure 2-2 Polyethylene backbone schematic (Peacock 2000)

There are various different forms of polyethylene which are distinguished by their variations in branch structure. HDPE is chemically closest to pure (or virgin) polyethylene. It consists primarily of unbranched molecules with very few flaws to alter its linearity. With an extremely low level of flaws to hinder organization, a high degree of crystallinity can be achieved, resulting in resins that have a high density compared to other grades of polyethylene (Peacock 2000). In addition, polyethylene particles act as stress acceptors, which will absorb the impact energy and improve the impact strength of the material (Choi 1989). The schematic representation of polymer microstructures can be seen in Figure 2-3.

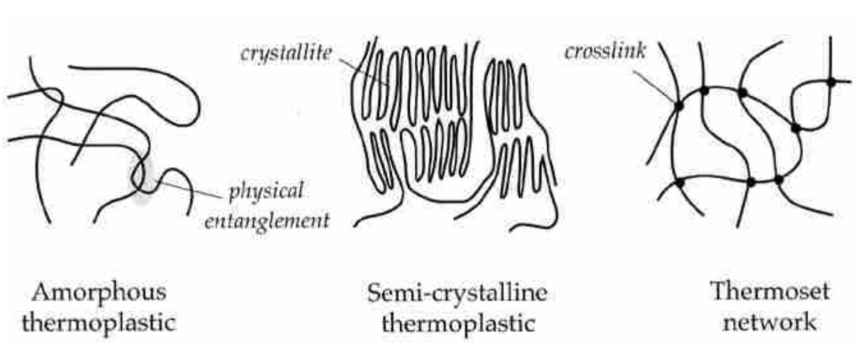

Figure 2-3 Schematics of various polymer microstructures (Peacock 2000)

HDPE is classified as a semi-crystalline thermoplastic, with crystallinity varying based on resin grade and processing conditions. The degree of crystallinity of HDPE ranges from 55 to 77% (Peacock 2000).

Mechanical Properties

The stress-strain relationship of a plastic shows a continuously decreasing stiffness with larger strain. This is because, in contrast to metals, plastics do not undergo complete instantaneous recovery upon unloading (Bonilla 2003). Figure 2-4 displays images of the polymer cross section during various phases of a tensile test, which is summarized in chapters three and four of the research. This diagram represents the generalized force versus elongation curve for polyethylene and associated tensile phenomena.

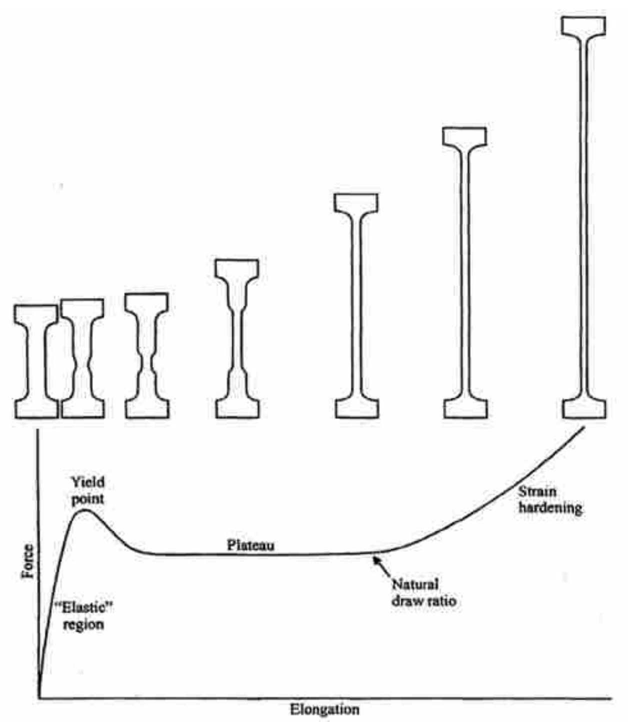

Figure 2-4 Stress/strain diagram for High Density Polyethylene (Peacock 2000)

HDPE's tensile strength in MPa (psi) is approximately 20-38 MPa (3100-5500 psi) (Richardson 1989). This is relatively low in comparison to other engineering plastics such as polycarbonate (PC), commonly used in Nalgene™ bottles (Strong 2006), or acrylonitrile butadiene styrene (ABS). This low tensile strength provides added toughness to the material, giving it a high amount of impact resistance. It also increases the likelihood of material deformation under extreme pressures and loads.

Thermal Properties

In the blow molding and hot filling applications seen in Figure 2-1, temperature has a major influence on the behavior of the polymer used to create the bottle. The hypothesis of the research is focused on the effects of the hot filling temperature of the liquid on HDPE bottle properties. The effect of temperature on plastics is not well understood in the plastics industry and was poorly defined in the literature. An explanation of the defining thermal parameters of polyethylene will be provided here.

Melting Range

Semi-crystalline polymers do not exhibit melting points in the classic sense, i.e., as a sharply defined transition from the solid to the liquid state occurring at a discrete temperature. Thus, polyethylene undergoes a transition from the semi-crystalline to the molten state that takes place over a temperature range that can span from less than 10°C up to 70°C. As it passes through this transition the semi-crystalline morphology gradually takes on more of the characteristics of the amorphous state at the expense of the crystalline regions. The melting range is broad because it consists of a series of overlapping melting points that correspond to the melting of lamellae (a layered plate like structure comprising the plastic) of various thicknesses. Thicker lamellae have higher melting points (Peacock 2003).

Polyethylene can vary based on the degree of chain branching among resin grades and the type of processing parameters. There is a definite correlation between melting point and degree of chain branching in polyethylene (Van Kimmenade 2006). A higher melting point is associated with a higher degree of chain branching in the polymer structure.

There is no exact way to quantitatively relate melting point or softening point to long term polymer properties except to note that the upper service temperature, even short-term, must be well below melting or softening temperature (Belofsky 152). This lack of a definite temperature in which plastics will fail has lead the researcher to determine the “trigger range” in which the HDPE bottles involved in the study will experience deformation during the hot filling process. High-density polyethylene’s melting point ranges from 125-132°C (Sunderland 2000). This creates a potential problem for some manufacturing companies who have no way of determining the appropriate liquid filling temperature while simultaneously taking into consideration the bottle design and corresponding resin grade.

Glass Transition Temperature (Tg)

Unlike the melting range, the glass transition temperature occurs at a definite point when there is an abrupt change in the degree of freedom experienced by chains in the disordered region. Thus, the chain segments comprising the disordered regions of a polymeric sample exhibit very little freedom of motion below its glass transition temperature, whereas above this temperature, chain segments are free to move to a limited extent (Peacock 2000). The following formula represents the approximate relationship between glass transition temperature and melting temperature. The glass transition temperature will always lie below the peak melting temperature. Equation 2-1 demonstrates this relationship.

$$Tg = (0.5\text{-}0.8)\ X\ Tm \qquad (2\text{-}1)$$

Melting of a semi-crystalline polymer or any material is a first-order transition because it is a clear cut change of phase from solid to liquid. The glass transition of a material is a second-order transition – no change of phase is involved, and the phenomenon is a little harder to observe. Glass transition temperature (Tg) is popularly defined as that temperature below which the polymer is “glassy” and above which it is “rubbery” (Belofsky 1995).

The glass transition temperature of polyethylene has been assigned to a wide variety of temperatures, ranging from -110°C to -130°C. The location of the Tg of polyethylene depends on the testing procedure by which it is determined. In general, the more rapid the test, the higher the temperature at which the Tg will appear. The glass transition of polyethylene occurs at such low temperatures that it is very rarely encountered in commercial applications. This effectively means that polyethylene samples remain in the ductile state at all service temperatures (Peacock 2000). Understanding this concept is important to determine the behavior of the material at all temperature ranges, but at the high temperatures applied in the hot filling operation, the Tg will have no influence.

Melt Flow Index

The melt flow index test is used to monitor the quality of plastic materials. The quality of the material is indicated in this test by the melt flow rate through a specified die under prescribed conditions of temperature, load, and piston position in the barrel as timed measurement is being made. The melt flow rate through a specified capillary die is inversely proportional to the melt viscosity of the material if the melt flow rate is measured under constant load and temperature (Bonilla 2003). The melt flow index of a specified polymer will dramatically influence its moldability.

Heat Deflection Temperature

A common reference figure for the effect of temperature on stiffness of plastics is the heat deflection temperature which is described by the ISO 75 (ASTM D 648) test (Belofsky 1995). The test sample in ASTM D 648 is a molded or machined rectangular bar, 5 inch long by 0.50-inches wide and any thickness from 0.125 inches to 0.50 inches, which is mounted on rollers spaced 4 inches apart, and loaded in the center by another roller to give line contact at three points. This three-point bend test has a centrally located sliding weight such that there is a maximum bending stress of either 66 or 264 psi at the midpoint of the span.

The sample is immersed in a controlled temperature bath of mineral oil or silicone oil and the temperature is raised at a constant rate or 2 °C/min. The HDTUL is recorded when a deflection of 0.010 in is measured on the dial indicator attached to the loading weight. The 66 psi load level is used for low stiffness plastics, and the higher 264 psi level for engineering plastics and thermosets. The reported HDTUL depends on the stress level used (Belofsky 1995). The testing setup can be seen in Figure 2-5 below

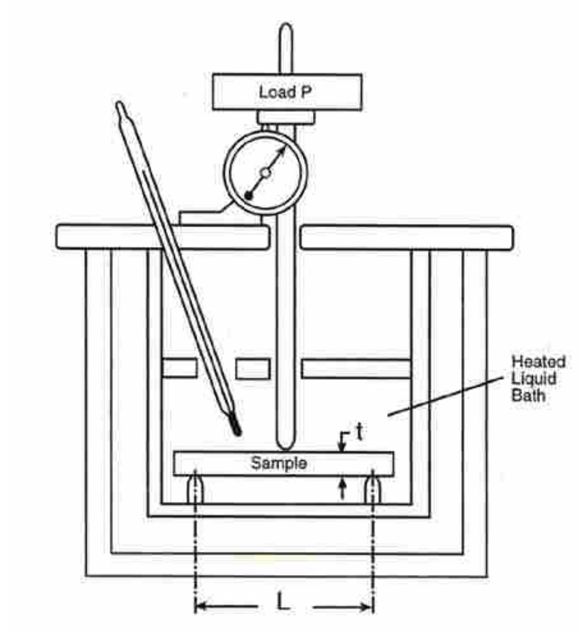

Figure 2-5 Three-point bend test used to determine the HDTUL of plastics (Belofsky 1995)

This test is intended to identify the short term behavior of the polymer under a specified load at elevated temperatures. Heat deflection temperature provides a simple measure of melting transitions.

At some temperatures the plastic will become so pliable and so easily distorted under load that it may not perform the function intended, especially if that function is structural. The temperature at which this happens varies widely among different plastics and among different applications (Strong 2006). The HDT of HDPE ranges from 82 to 91°C at 66 psi (Peacock 2000). This range is relatively narrow in comparison to other polymers, but the temperature mean is much lower than other engineering plastics used for structural applications.

In the case of polyethylene, samples become more deformable as the temperature rises, primarily for three reasons: (1) The disordered regions become more flexible due to increased thermal motion; (2) the proportion of relatively rigid crystalline regions decreases as thinner crystallites melt; and (3) the translation of chain segments through crystallites becomes easier (Peacock 2000). In general, the heat deflection temperature increases as the degree of crystalinity and lamellar thickness increases (Belofsky 1995). Increasing the wall thickness or adding supportive fillers to the material will increase the HDT. These are common methods employed by bottle producers to add structural integrity to the products that are hot filled.

2.2 Extrusion Blow Molding

Blow molding is the preferred manufacturing process to produce hollow, plastic containers and bottles using thermoplastics. The popularity of blow molding increased with the development of low-density polyethylene (LDPE) in the mid 1940's by ICI of England (Lee 1998). The development of LDPE and other grades of polyethylene (PE) allowed designers and producers to create more complex designs for products, while lowering cost of materials. The properties and associated benefits of PE revolutionized the bottling industry and caused an increase in the production of plastic containers to replace glass containers. This transition has occurred throughout the ever expanding bottling industry in an effort to reduce costs to both consumers and produces while increasing efficiency.

The basic extrusion blow molding process has two fundamental phases. First, a parison of hot plastic resin in a somewhat tubular shape is created. Second, a pressurized gas, usually air, is used to expand the hot parison and press it against a female mold cavity and pressure is held until the plastic cools (Lee 1990). The mold is machined to have the negative contour of the final desired finished part. The mold, typically split into two halves, opens after the part has cooled to the extent that the dimensions are stable (Harper 2006). Once the plastic cools, the part is ejected from the mold, excess flash is removed by hand or cut, and bottles are packaged.

The parison size is controlled by the dimensions of the die cavity. During the parison extrusion, the die gap is regulated or “programmed” to effect wall thickness changes in the parison and thereby increase or decrease thickness in the blow molded part (Peters 1983). Figure 2-6 is an illustration of the continuous extrusion blow molding process.

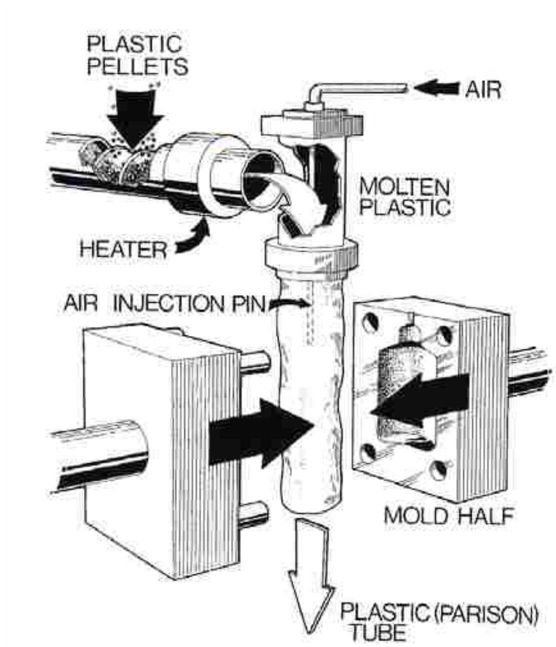

Figure 2-6 Extrusion blow molding process (Lee 1998)

Blow molding is a very complex process because a large number of properties are balanced and interdependent. It seems simple to melt some plastic, make a parison shape, close a mold around it, and inflate a hollow object, but consistency is the whole key to blow molding at production speeds. Achieving consistency is a matter of paying attention to all of the details involved in running the blow molding machine (Belcher 75).

The primary input variables in this process include material temperature and condition, die temperature and design, extrusion rate of the parison injection air temperature and pressure, mold temperature and design, clamping pressure, cooling rate and time. All of these variables affect the outcome of the part and must be carefully controlled.

Advantages of Blow Molding

Some of the obvious advantages of the blow molding process are its ability to make one-piece hollow parts with high productivity, little subsequent finishing, and high surface quality. Since it is a low pressure process, products have low residual stresses which provide adequate environmental stress-cracking resistance. Because of the stretching that accompanies internal blowup of the part in the mold, biaxial orientation is easily achieved, and with modification, uniaxial orientation, if preferred. Control over orientation gives better mechanical strength with lighter parts (Belofsky 1995). The improvements to the blow molding process have also inspired the development of new products such as squeeze bottles, tanks, toys, refrigerators and furniture (Belcher 1999).

Types of Blow Molding

There are various forms of blow molding which include extrusion blow molding, co-extrusion blow molding, injection blow molding, and stretch blow molding. Each method varies based on the condition of the parison (or preform) and the processing method. The research will focus on extrusion blow molding.

Extrusion blow molding uses a molten parison instead of a solid preform (created using an injection molding machine as a separate process prior to the blow molding operation). In both processes air is injected through one end of the parison or preform and the material is stretched to fill the mold cavity.

Bottle Geometry

Some potential sources of variation that are inherent in the blow molding process include varying wall thickness, bottle shape, amount of flash and the presence of a parting line.

Blow molding creates inherent fluctuations in wall thickness due the nature of the process itself. The bottle wall thickness is affected by the amount of air injected into the bottle, the bottle geometry, and the design of the parison die. Usually the bottom location of the bottle is the thickest and most rigid because the material is blown simultaneously outward and down, making contact with the bottom of the mold first, which prevents further stretching and thinning.

Wall thickness can be controlled by ensuring that there is consistent air pressure flowing into the parison. Inconsistent air pressure may create ripples in the interior of the bottle wall or in the neck of the bottle. It is ideal to have perfectly uniform wall thickness in the blow molding process, but this is often very difficult to achieve for many companies.

The geometry of the bottle also influences consistency of wall thickness. The parison die controls the shape of the parison as it leaves the barrel of the extruder. The die must be perfectly aligned at the end of the extrusion barrel in order for the parison to release uniformly from the die.

The parting line or seam is an observable line of plastic occurring along the outer perimeter of products that require the use of a two part mold for creation. During extrusion blow molding, the closing of the two part mold cuts off the parison and leaves the characteristic weld (or parting) line on the bottom of many bottles as evidence of the pinch-off (Harper 2006). This pinch creates a small degree of flash on the bottom of the bottle which is removed after the blow molding process occurs. In most instances it falls off automatically due to the intense pressure of the mold compressing on the parison. Other times the flash will be removed in post processing phases. The parting line also extends along the vertical axis of the bottle as a result of where the two halves of the mold join.

Extrusion Blow Molding Material

Material selection for blow molding applications is based on the end use of the product. In general, blow molding requires that plastic has high stiffness, good impact properties, good environmental stress cracking resistance (ESCR), and process consistently (Belcher 1999). Containers designed to carry reactive chemicals or other active liquids will require the use of a material with high chemical resistivity. The material used in blow molding must also have sufficient strength to allow time for the mold to close and good welding in the pinch off areas of the blow mold (Lee 1990).

Part consistency also relates to the material used in the blow molding process and its properties. Polymer properties change over time. Being organic materials, most synthetic polymers are sensitive to light, oxygen, moisture, heat, and other aggressive environments (Sunderland 2000). It is important to maintain consistency of properties in the material because any variation can lead to changes in material behavior and part shape. This research highlights the influence of heat on the bottle properties.

The development of blow molding was fueled by the introduction of high molecular weight polyethylene. Because of its wide ranges in density, melt indexes, and other basic characteristics, the material has a corresponding wide variation in possible end properties. Flexible bottles are best made from low or medium density polyethylene. High-density plastics are more suitable for rigid bottles such as the bottles used in this study.

The properties common to all items blown from polyethylene include light weight, toughness (even at low temperature), resistance to attack and penetration from chemicals, resistance to cracking under stress when holding liquids (environmental stress crack resistance), and excellent moldability (Lee 1998). The most common material used in blow molding today is high-density polyethylene because of its stiffness and chemical resistance. Two of the most common resins used in blow molding today, HDPE (processed using a parison) and PET (processed using a preform), are used for 90% of the container market (Belofsky 1995). This research focuses entirely on a HDPE bottle created for hot liquid applications.

2.3 Liquid Hot Filling

Hot filling is the process of heating a liquid to a desired temperature and filling the liquid directly into a container of choice. The container is then capped and the cap is sealed through an induction process. Liquid products that must maintain strict health and safety standards are generally hot filled into glass and plastic containers. The hot filling process prevents the formation of bacteria and enzymes in the liquid and increases the shelf life of the product. Hot filling is performed for containers used in a variety of industries, and the distribution of such products accounts for a large percentage of manufacturing worldwide.

During the filling process the fill liquid passes through a closed system. When filling, the fill nozzle lowers into the molded container (See Figure 2-7) and fills it with the desired dosing quantity (Oschmann 1999). The filling takes place under conditions of constant product pressure and precisely adjustable filling times, and thus leads to high filling accuracies.

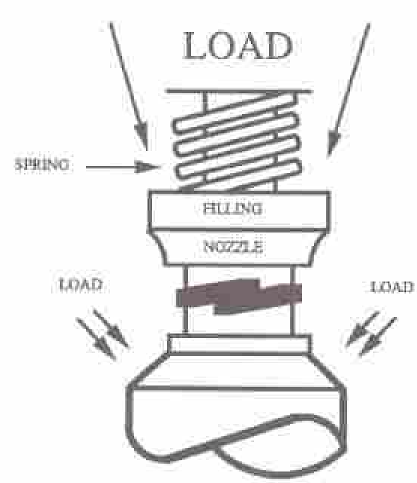

Figure 2-7 Schematic of hot filling nozzle (Oschmann 1999)

See Figure 2-8 for a simplified schematic of the time-pressure-dosing system used in most filling applications. Besides the high filling accuracy, the system is practically free from wear and the adjustment of the filling volume can be easily performed by the operator through the machine operation panel (Oschmann 1999).

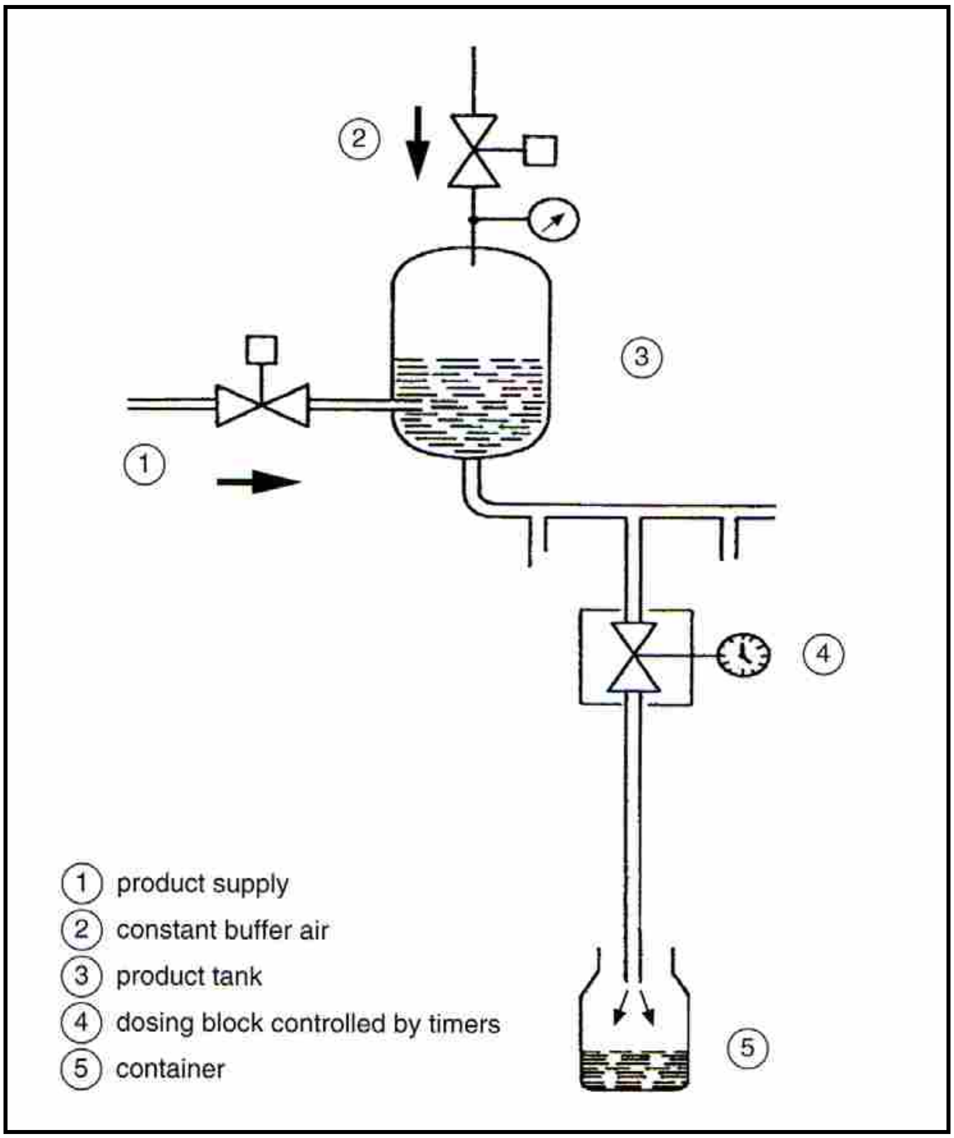

Figure 2-8 Simplified scheme of time-pressure-dosing system (Oschmann 1999)

Liquid Products

A variety of liquids are hot filled into plastic bottles to increase the overall shelf-life and quality of the product. Many foods and drinks must undergo heat treatments such as pasteurization and sterilization in order to kill pathogens and different spoiling microorganisms (Ophir 2004). Pasteurization is normally carried out at temperatures below 100°C, and sterilization is carried out above 120°C. Given a filling temperature, specific container dimensions are required in order to achieve a target volume average pasteurization value for the most thermal resistant microorganism or spoilage enzyme (Silva 1997).

Pasteurization and sterilization are time-temperature dependent: the higher the temperature, the shorter the time required for the destruction of the microorganisms. This phenomenon explains why many companies choose to fill their bottles at the highest temperature possible to decrease cycle time and to ensure lower levels of microorganisms in the liquid.

During heat treatment of the liquid, beneficial components of the food such as vitamins, nutrients, and flavor compounds may also be destroyed. However, the destruction of microorganisms is more temperature-dependant than is destruction of beneficial components. The research focuses primarily on the heat treatment and hot filling of fruit purees using the pasteurization method at temperatures below 100°C.

The hot fill treatment must target the microbial and enzyme inactivation, while maintaining the original organoleptic and nutritive fruit characteristics. The severity of the heat treatment and the resulting shelf-life are determined mostly by the fruit pH (Silva 353). This will vary depending on the type of fruit puree being processed. Although the pH level is of critical value to the product quality, many filling companies choose a filling temperature based on other filling specifications as a benchmark, disregarding pH level.

Ideally the temperature of the hot fill should be determined based on the liquid content, bottle material and bottle design, but these variables are often overlooked during the filling process. Overlooking any one of these variables can lead to costly defects.

During the hot filling process, liquid temperature is constantly monitored with heating charts to ensure adequate sterilization (See Appendix D).

The liquid will cycle through the system until it has reached a steady fill temperature. Fluctuations in filling temperature result from the temperature regulator. These fluctuations are constantly occurring throughout the hot filling procedure, creating a dramatic source of variation in filling temperature and overall product consistency and quality. See Appendix D for sample temperature logs from Fillco Custom Bottling, LLC.

Hot Filling of HDPE

The linear nature of HDPE permits the development of high degrees of crystallinity, which endow it with the highest stiffness and lowest permeability of all the types of polyethylene. This combination makes it suitable for many small, medium, and large liquid containment applications, such as milk and detergent bottles, pails, drums, and chemical storage tanks (Peacock 2003).

The relationship between thermal shrinkage of HDPE during the hot filling process and molecular or crystallization rearrangements taking place when oriented products are heated above the Tg is not completely understood (Ophir 2004). Studies have shown that for air cooling of commercial bottles a hot filling temperature near

110°C should not be used to avoid over processing. There is a conflict with this rule in that final quality of liquids is greatly improved when using higher filling temperatures (Silva 1997). That is the dilemma with most hot filling companies that are pushing the hot filling temperature higher and higher to ensure adequate pasteurization of liquids. With HDPE bottles, high filling temperatures are not recommended, but many companies have been able to get away with filling at temperatures approaching 110°C. Others experience high levels of defects related to deformation near this temperature and a variety of lower temperatures.

2.4 Summary

Although there are many of phases and variables to consider throughout the bottle production process, we have limited our discussion to blow molding and hot filling of HDPE bottles. The liquid hot filling phase (See Figure 2-1) was identified in the early phases of the research as a variable of interest in the study. The emergence of defects occurred after the hot filling phase, capping and sealing phases of production (See Figure 2-1). This summary of blow molding, hot filling, and the material used to create the bottles observed in this study (HDPE) provide a sufficient background to understand the tests and results that will follow in the next three chapters of the thesis.

3 Methodology

3.1 Introduction

This segment of the thesis explains the data collection methods and tests used to understand the possible root causes of the defective bottles and how these variables potentially influence final bottle shape after the hot filling stage of production has been completed.

The methodology of the research is largely based on the scientific method and root cause analysis. Root cause analysis is the process of drilling down from symptoms, to problem definition, to possible causes, to actual cause(s) (Okes 2005). Doing so is an iterative process that combines divergent and convergent thinking. Several common process analysis tools can be useful throughout the process, such as:

- Flowcharts – Aids in understanding the operation steps involved in the process, and where data could be collected in order to identify the major contributions to problems.
- Brainstorming - Provides a mechanism for identifying all possible causes, from which those least likely can be eliminated based on logic, with data collection then focusing on the most likely.

- Ishikawa Fishbone Diagram - A tool for breaking down the system/process into functional and subsystems/components, identifying the logical cause and effect relationships.
- Run charts - Enable analysis of data over time to look for trends/patterns that may indicate root cause.
- Histograms - Unlike run charts, histograms group all the data into one distribution, the shape of which might indicate other patterns worth investigating.
- Pareto diagrams - Can be used to analyze categorical information on root causes to identify major contributors.
- Statistical tests - While graphical tools such as run charts and histograms are good ways to analyze data, they are not as sensitive to small differences that might exist between sources of variation. Statistical tests such as the t-test, F-test, analysis of variance (ANOVA), and chi-square test can detect small differences based on desired levels of confidence (Okes 2005).
- The following analysis methods were applied to the research to determine the

possible symptoms of the defects in the study:

- Process Flowcharts
- Brainstorming
- Ishikawa Fishbone Diagram
- Pareto diagrams
- Statistical testing (chi-square testing and ANOVA)

Two additional tests, which were validated using statistical testing, were conducted based on the results of early analysis and data collection.’/The tests included a hot filling simulation and tensile testing of various locations on the bottles. The details of these tests will be discussed later in this section.

The variables involved in all phases of the manufacturing process will be summarized with an Ishikawa (cause and effect) diagram. Additional analysis tools and testing methods will then be summarized with a proper explanation and reasoning behind their implementation. The development of additional tests was based on the results of the experimentation and identification of key variables that have the most dramatic influence on the final outcome of the product. These variables were identified through the results of the review of literature and the collaboration of the data obtained from industry professionals and experts in the field of plastics and plastics engineering.

This process began with a review of prior work by Bruce and Andrew Strong of Sonic Plastics to eliminate existing defects. A Pareto Analysis of existing defective bottles from previous runs was then conducted and trends related to the description, location and number of defects were observed and documented (See Appendix A). The results of this analysis led to a series of hot filling simulations to test and observe the effect of filling temperature on the final bottle shape using virgin bottles of the same design as those observed in the Pareto Analysis. The simulations were then followed by a series of tensile tests on various locations of a sample of bottles.

3.2 Definition of a Defect

A defect will be defined in this research as morphological shape change (deformation) to the original bottle shape. The research will consider only those defects related to the compression or expansion of the sidewall of the Boston Round HDPE bottles used in the study.

3.3 Previous Industry Efforts and Experimentation

Sonic Plastics, specializing in blow molding, and Fillco Custom Bottlers, specializing in hot filling, have partnered in the research to gain a greater understanding of the source of these common defects. There is a current dispute between these two companies related to the source of defects that they have experienced after hot filling has occurred.

Sonic Plastics has worked cooperatively with Fillco to make changes to the bottle design in an attempt to solve the problem of post hot filling bottle deformation. Each change was performed independently to either the bottle design or some element of the production process. The individual changes and results will be discussed in this section.

These changes occurred during a one year period following the original discovery of a batch of defective bottles. The defects were discovered following the hot filling of the bottles at Fillco's manufacturing facility. Other filling customers of Sonic Plastics who have used the same bottle have had no complaints related to defective bottles.

Wall Thickness

The original assumption regarding the source of defects was the idea that variation in wall thickness was leading to weaknesses in various locations of the bottle. An additional concern expressed by Fillco was that there are microscopic weaknesses in the bottle that decrease the overall structural integrity of the bottle

Variations in wall thickness lead to a lack of overall concentricity of the bottle. Sonic Plastics obtained measurements on random samples of bottles that were sent to Fillco and validated that their wall thicknesses were within 0.020" (See Appendix B).

The standards that Sonic Plastics and Fillco had agreed to are as follows: "The standards for the outside of the bottle need to stay within 0.020" when comparing 'top seam' to 'bottom seam' and then comparing '90° from seam top' to '90° from seam bottom'. The standards for the wall thickness need to stay above 0.040". The wall thickness should also have only 0.020" difference (one side could potentially be much thicker, which can be tough to change)." Wall thickness measurements were taken from the end of October 2006 to the end of January 2007 (See Appendix B). All thickness measurements taken during this period were within the specification outlined above and are within 15 to 20 thousandths of an inch difference.

Sonic Plastics made the claim that they cannot make a bottle with greater uniformity due to inherent variation that exists within their blow molding process. They have sufficiently met their goal to be within 20 thousandths of an inch or less.

Despite the results of these tests, Sonic Plastics altered their process and increased the bottle wall thickness from the original 91 gram standard bottle to a 115 gram design. This change increased the sidewall thickness and overall stability of the bottle, allowing

Fillco to fill at an elevated temperature without the occurrence of defects. As stated in the research, an increase in bottle weight leads to an increase in the HDT of the material. This created a slight increase in costs, but greatly improved the durability and performance of the bottle under high filling temperatures.

Blow Molding Material

After this change in wall thickness, Fillco continued to experience defects which emerged after their hot filling process. Fillco requested that the bottle resin be upgraded to a material with a higher HDT. The original grade of HDPE was Bapolene® (HD0760 Blow Molding Resin produced by Bamberger Polymers International Corp.) and was later changed to Paxon™ (AD60-007 Blow Molding Resin produced by Exxon Mobil Chemical) (See Appendix C for resin data sheets). Sonic Plastics also experimented with Polypropylene (PP) which has a slightly higher HDT than HDPE (5° to 10° increase) and is a higher stressed resin. Despite the resin changes, the emergence of defects continued.

Summary of Changes

As stated, these changes occurred over a period of approximately one year from October 2006 to December 2007. The defects have continued to occur despite the numerous changes that have taken place. The research will explore other variables involved in the production process in an attempt to discover those variables that could be influencing the final shape of the bottles.

The problem of defects has created financial losses for both firms and dissatisfaction among customers in relation to quality. The research related to blow molding, hot filling and thermal properties of HDPE will be applied to this situation.

3.4 Root Cause Analysis

The major focus of this research and testing is to implement analysis tools to determine the root cause of defects occurring in a specific HDPE bottle after the blow molding, hot filling and sealing processes have taken place.

Based on previous industry data related to defects in HDPE bottles and the results of additional tests performed observing multiple variables involved in the production of these bottles, hypotheses have been formed to justify additional tests and experimentation of other related variables. This reiterative process of developing hypotheses and proving or disproving them is based on experimentation and is a vital part of any investigative or analytical process. Ultimately, the process will conclude with the identification of physical, human, and latent root causes (Latino 5).

The basic steps involved in finding the root cause are:

- Understand the process, including the structure of the system (and subsystems) involved, and how it performs over time.
- Identify all possible sources of errors or variation in the process, and select those sources that require further analysis based on your current understanding of the problem.
- Collect and analyze quantitative and/or qualitative data, and match the findings to the sources identified in the previous step that can actually produce the outcomes observed (Okes 2005).

3.5 Process Flowcharts

In order to gain an understanding of the possible variables that may be influencing the creation of defects, the blow molding and hot filling processes were observed in great detail. These observations were used to create an Ishikawa, or cause and effect, diagram of the potential variables influencing final bottle shape. The defects emerged just after the hot filling phase of the process, so both blow molding and hot filling were observed.

Process Flowchart: Blow Molding

- Blow molding resin provided by Exxon Mobile Chemical
- Resin loaded into hopper system at Sonic Plastics
- Resin dried if necessary and heated in the extrusion barrel of the blow molder until it reaches a molten state
- Resin and colorant loaded into each blow molding machine for heating
- Resin is extruded into the parison form
- Parison drops into the blow mold
- The blow mold clamps onto the bottom of the parison, the parison is cut, and air is injected into the top of the parison until the parison is expanded
- Air pressure is held until the bottle cools
- The blow mold opens and the bottle is ejected from the mold onto a conveyer
- Excess flash is removed from the bottom of the bottle
- The top neck of the bottle is cut
- Bottles are packaged

Process Flowchart: Liquid Hot Filling

- Pre-Filling
  - Liquid arrives in a tanker and is unloaded at ambient temperature
  - Liquid is pumped to the balance tank. Solids are suspended during transportation so the liquid is agitated
  - Liquid travels through the heat exchanger and temperature controller
  - Liquid advances to a series of thermocouples which measure the temperature as it leaves the pin
  - The last thermocouple gathers a reading before the liquid advances to the filler. If the temperature is acceptable it passes to the filler. If it is rejected it transferred to the balance tank to be re-tested (The steam value of the system maintains the product at 178°F)
  - Virgin bottles are washed with ozonated water to disinfect any potential bacteria
- Hot Filling of Liquid
  - Bottles are sorted by hand or machine and loaded on the conveyer
  - The filler will allow the liquid to enter the filler from 170-190°F
  - Bottles advance to the filling station and the filler lowers onto the top of the bottle and sprays a measured amount of liquid into the container while the bottle moves through the system

- Nitrogen Filling
    - A predetermined (metered in milliseconds) dose of liquid nitrogen is added to the top of the bottle to eliminate the presence of oxygen prior to capping and sealing (oxygen is a food and juice contaminator)
- Bottle Capping
    - Bottle cap is twisted onto the bottle via a roller system
- Induction Sealing
    - The capped bottle advances under a induction sealing and the seal within the cap is activated
- Water Cooling (air cooling is also common)
    - Three cascading waterfalls cool the bottles as they advance through the system. The water temperature gradually decreases as the bottles advance through the system

3.6 Ishikawa Fishbone (Cause and Effect) Diagram

The variables discovered during process flowcharting are summarized using a method known as the Ishikawa Fishbone Diagram. This diagram, also known as a cause and effect diagram, visually outlines which variables effect which phase of the process. This method was used to help the researcher determine which variables influence which phases of the production process for filled HDPE bottles.

Typically, fishbone analysis plots four major classifications of potential causes (i.e., human, machine, material, and method) but can include any combination of categories (Mobley 1999). Iterations of these four categories are listed below in the Ishikawa Fishbone Diagram (See Figure 3-1).

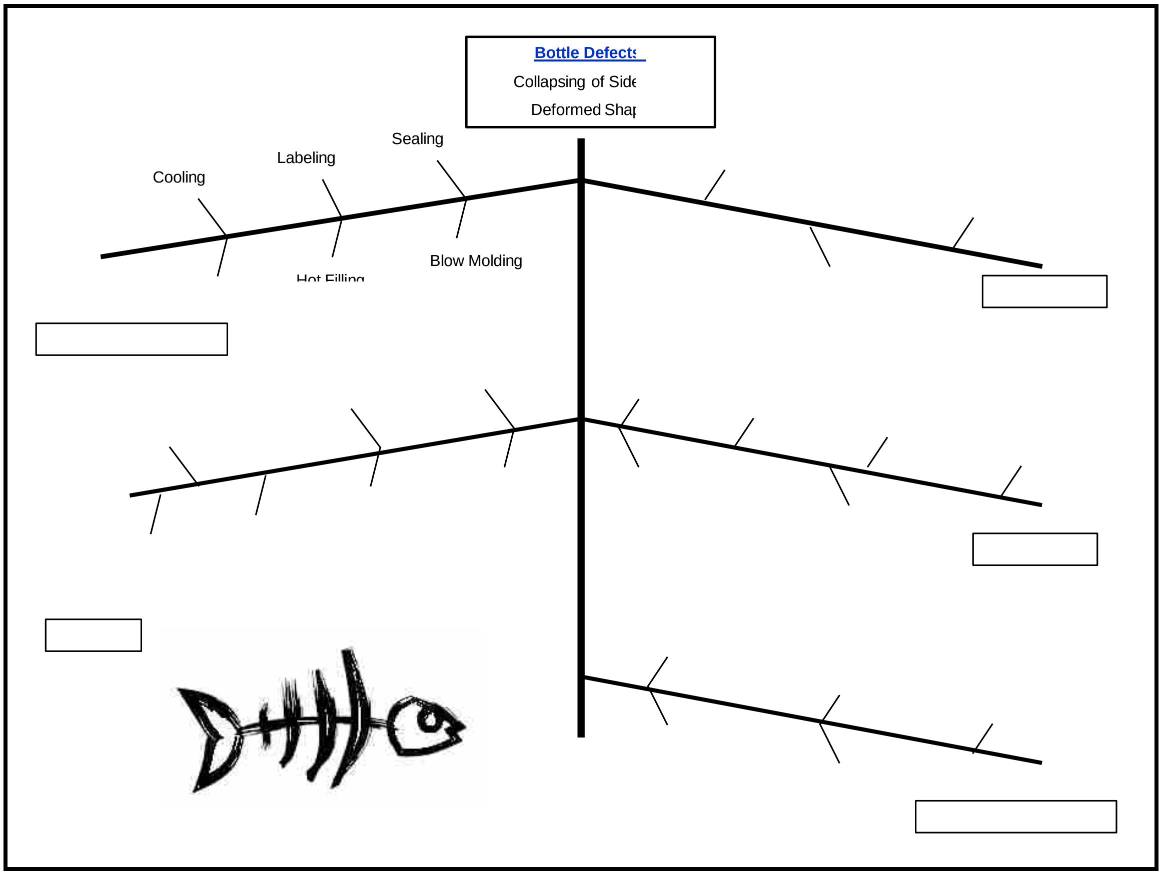

Figure 3-1Ishikawa Fishbone diagram for defective HDPE bottles (terrain.org)

The advantage of the fishbone diagram is that it forces the investigator to logically group each of the factors identified during the investigation. This process may automatically eliminate some factors and uncover other issues that must be addressed (Mobley 1999).

Through this process the variable of "Fill Temperature of the Liquid" in the Hot Filling and Sealing category was identified as a primary variable that could lead to morphological shape changes if filling temperature were to exceed the heat deflection temperature of the resin used to produce the bottle. Other related variables include "HDT of HDPE," "Internal/External Pressure Changes," and "Internal Vacuum." "Material," "Resin Grade of HDPE," "Sidewall Thickness," and "Nitrogen Content" have all been tested by Sonic Plastics. The other variables listed have been determined to be less critical to the final shape of the bottles by the researcher.

3.7 Pareto Analysis

Pareto Analysis is a quality systems tool used to determine trends in data. Pareto charts are simple to construct and interpret, and they can provide important insights for problem solving and process improvement. Typically, Pareto charts portray the frequency of occurrence of a variable of interest in various categories, arranged in order of descending frequency. Attention is then focused on the category that has the highest frequency of occurrence (Stevenson 2000).

According to the Pareto principle, named after Italian economist Vilfredo Pareto (1848-1923), a few factors or causes will account for a disproportionately high percentage of the occurrences of some event. In a study of the Italian economy, for example, Pareto found that 80% of the wealth was held by 20% of the people. This came to be known as the 80/20 principle. Typically in a Pareto analysis, about 80% of the occurrences will fall into 20% of the categories. A Pareto analysis is intended to distinguish the 'vital few' factors from the 'trivial many' factors, allowing the allocation of resources for addressing the vital few, where they can be expected to have the greatest impact (Stevenson 2000).

This tool was implemented to conduct an introductory analysis of a collection of defective bottles from two production runs. In this analysis the defective bottles were analyzed visually for morphological shape changes to any are of the bottle. In this test the defects were organized according to their location on the bottle and the severity of the defect. The severity was determined by the amount of shape change or deflection that occurred on the surface of the bottle.

Almost all companies use Pareto analyses-either formally or informally. It is commonly believed the principle is simple and very effective, since by solving the top 20 percent of quality issues, the defect rate is reduced 80 percent. As pointed out by Bhote, however, 90 percent of the companies in this country are unable to solve their chronic quality problems. The problem with this 90 percent is they apply the Pareto principle indiscriminately. Like any other tool, the Pareto principle comes with a set of limitations and assumptions. If these constraints are not adhered to, the tool gives erroneous results (Velury 1997).

In the world of problems solving, the Pareto principle is king. We all have experienced that 90 percent of the business comes from 10 percent of the customers, and that 80 percent of the complaints come from 20 percent of the customers. The typical problem-solving process consists of collecting all data into one bucket, sorting in descending order, and picking the top few problems. It is that simple. When complaints don't go away, the problem-solving cycle repeats, sorting in descending order (Velury 1997).

In the Pareto Analysis notes (See Appendix A) bottles that had no visible shape change were characterized by the phrase “no change.” Bottles with a minor shape change characterized by any deviation from concentricity of the original unfilled bottle were characterized as “minor defects.” Bottles with major indents in any location on the bottle or major deviations from concentricity were characterized as “major defects.” The defects were then observed and documented based on their location and ranked. The results were charted on a Pareto spread with “defect description and location” on the x-axis and “# of defects in the sample” on the y-axis. The defect description and location with the largest number of occurrences was listed on the left of the Pareto chart with additional defects charted to the right in descending order. The frequency of defects was ranked with the largest volume on the left.

3.8 Hot Filling Simulation

Based on the results of the Pareto analysis, a hot filling simulation was designed to recreate the defects observed from two early samples and to prove the likelihood of their occurrence.

The hot filling simulation replicated the hot filling process in a contained environment in which filling temperatures could be changed without interruption to full scale production.

Twenty blow molded bottles were randomly selected to perform two hot filling simulations which testing ten bottles each. The filling temperatures were determined based on the heat distortion (deflection) temperature (HDT) of HDPE ranging from 82 to 91°C at 66 psi. The heat distortion temperature of a polymeric sample is the temperature at which it begins to show appreciable deformation under load in the short term (Peacock

2000). The load in this simulation is generated by the pressure created in the bottle after it is hot filled and sealed.

The filling range of the simulation consisted of the following temperatures (°C): 72.5, 75, 77.5, 80, 82.5, 85, 87.5, 90, 92.5, and 95. In the first simulation the filling order was randomized. In the second simulation the filling occurred in descending order based on temperature.

Liquid temperature was regulated using two temperature controlled baths containing water. The temperature baths were used to heat beakers of juice to a predetermined temperature. Each beaker was filled with 950 ml of juice, which is the exact amount currently used in production. The temperatures of the juice and the controller baths were carefully monitored using thermocouples linked to a data acquisition system in Lab View.

When the juice reached the desired temperature it was poured into the selected bottle with an identical filling process, hand capped and carefully placed on a conveyer containing an induction sealer that activated a magnetic seal inside the cap. The induction sealer was set at 60% of its maximum power level based on current production specifications. Bottles were then air cooled and monitored for morphological shape changes.

The bottles were observed 48 hours after the simulation and bottle diameter was measured at various regions of the bottle for positive or negative deflection.

To quantify the visual shape changes or defects observed in the initial Pareto Analysis and hot filling simulation, the bottle was divided into four regions labeled A, B, C and D as seen in the figure below.

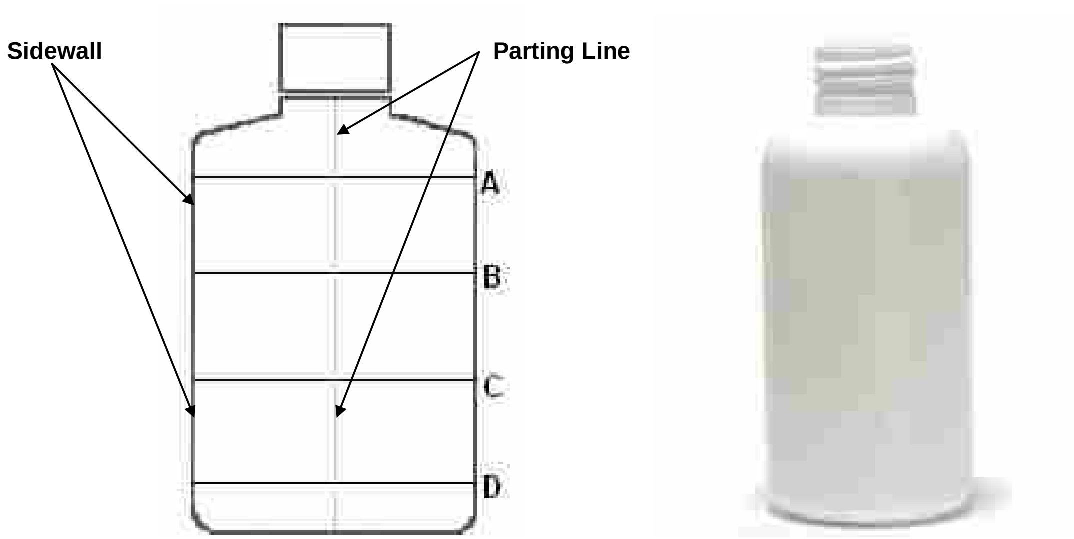

Figure 3-2 Hot filling simulation testing regions with bottle descriptions (left) and HDPE

The simulation was designed to observe shape changes anywhere on the bottle. Measurements were taken before and after the hot filling simulation in eight locations. Four diameter measurements were taken in regions A-D along each bottle's parting line or seam, which runs along the vertical axis of the bottle and is labeled in grey in the Figure 3-4. Four additional diameter measurements were taken along each bottle's sidewall, the center of which is located 90° from the parting line in both directions. The difference in diameter or shape change is the quantifiable value for the defect in that location used to provide a numerical comparison of deflection.

One additional measurement was taken for each of the four regions. The measurements for the parting line and the sidewall of each region were averaged to create an overall estimate for that region of the bottle. The bottle was then hot filled and the minimum diameter for that region was then measurement. The difference was used to represent the overall bottle shrinkage for that region versus shrinkage along the parting line and sidewall alone.

3.9 Tensile Testing

Based on the observations of the hot filling simulations, it was determined that the emergence of defects along the bottle sidewall could be related to structural weaknesses in the defective region. In order to compare the strength of the various regions of the bottle, a series of tensile tests were performed on segments of a sample of blow molded bottles that had not been filled.

Tensile dog bones were created with the center of the sample on the parting lines and sidewalls of the bottles in the rectangular regions shown in Figure 3-3. Five bottles were selected to test the bottle parting line and five bottles were selected to test the bottle sidewalls. Each bottle contained four dog bone shaped samples in compliance with ASTM D638 for a total of 40 samples for the tensile testing. A total of five samples were created for each region of the bottle.

Each bottle was cut into sections and the HDPE was flattened prior to milling the tensile profile. The dog bone profile was programmed into a CNC end mill and tensile samples were cut. This is a non-standard test created to capture the mechanical properties of the material in the each bottle section.

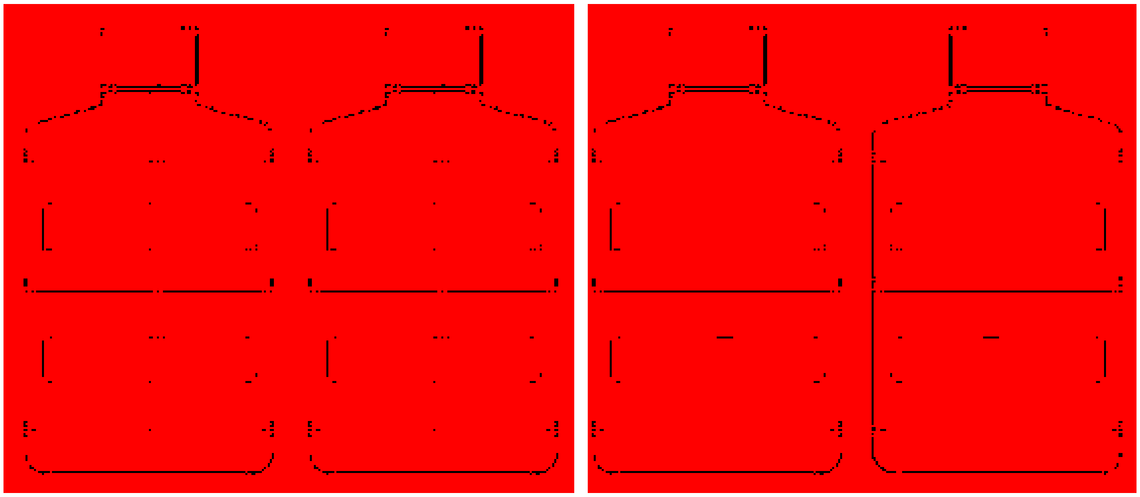

Figure 3-3 Tensile testing locations on the bottle parting line (left) and sidewall (right)

To record the results of each tensile test, a naming system was created to describe the bottle location that each tensile sample was cut from. Table 3-1 provides abbreviations and corresponding descriptions for each of the bottle region

Table 3-1 Bottle region abbreviations and corresponding descriptions

Region	Description
UTSW	Upper Top Sidewall
LTSW	Lower Top Sidewall
UBSW	Upper Bottom Sidewall
LBSW	Lower Bottom Sidewall
URPL	Upper Right Parting Line
LRPL	Lower Right Parting Line
ULPL	Upper Left Parting
LLPL	Lower Left Parting Line

The words “Top” and “Bottom” were used to designate two hemispheres of the bottles that were being tested along the parting line. Figure 3-4 is a photograph of the bottom of the HDPE bottles used in all of the tests. The upper region of the bottle is the half of the bottle above the parting line. The lower region of the bottle is the half of the bottle below the parting line.

Figure 3-4 HDPE bottle bottom

The “Upper Top Sidewall” is in the region above the parting line in Figure 3-8, in the upper region of the bottle along the vertical axis. The “Upper Bottom Sidewall” is in the region above the parting line in Figure 3-8, in the upper region of the bottle along the vertical axis.

The terms “Left” and “Right” were used to describe the halves of the bottles used for tensile testing of the bottle sidewall. The “Upper Left Parting Line” is the region to the left of the bottom parting line seen in Figure 3-8, in the upper region of the bottle along the vertical axis. The “Lower Left Parting Line” is the region to the left of the bottom parting line seen in Figure 3-8, in the lower region of the bottle along the vertical axis. Figure 3-5 and 3-6 are graphical representations of each testing location on the bottle.

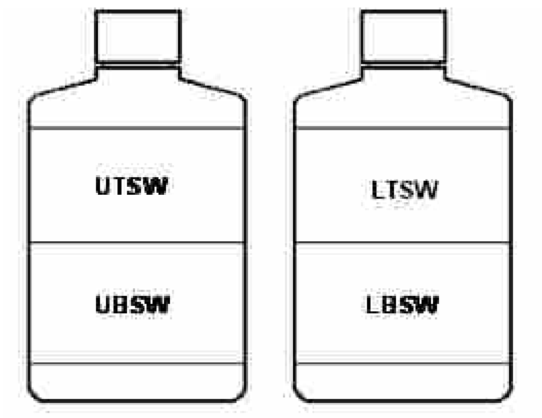

Figure 3-5 Tensile testing location designations for the bottle sidewall

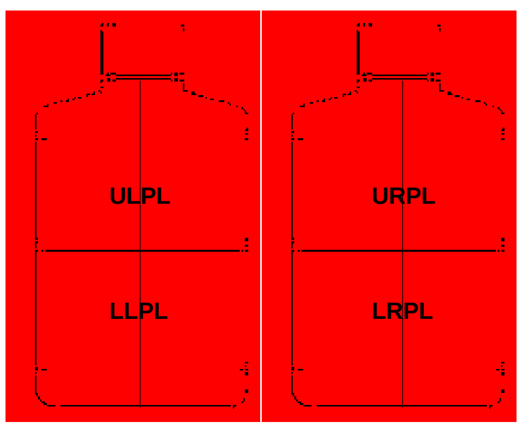

Figure 3-6 Tensile testing location designations for the bottle parting line

The tensile tests were performed according to ASTM D638 standards. The dimensions for the tensile dog bone used in the testing can be seen in Figure 3-7.

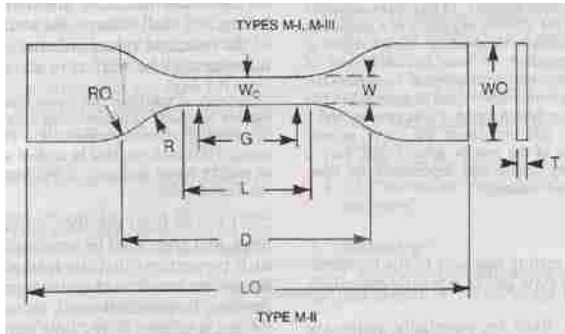

FIG. 1 Tension Test Specimens

D 638M

Specimen Dimensions for Thickness, *T*, mm[D]

Dimensions (see drawings)	10 or Under	4 or Under		Tolerances
	Type M-I	Type M-II	Type M-III	
W—Width of narrow section[A,B]	10	6	2.5	±0.5[H]
L—Length of narrow section	60	33	10	±0.5
WO—Width of overall, min[E,F]	20	25	10	±0.5
LO—Length overall, min[F,G]	150	115	60	no max
G—Gage length[C]	50	—	7.5	±0.25
G—Gage length[C]	—	25	—	±0.5
D—Distance between grips	115	80	25	±5
R—Radius of fillet	60	14	15	±1
RO—Outer radius (Type II)	—	25	—	±1

[A] The width at the center W_c shall be plus 0.00 mm, minus 0.10 mm compared with width *W* at other parts of the reduced section. Any reduction in *W* at the center shall be gradual, equally on each side so that no abrupt changes in dimension result.

[B] For molded specimens, a draft of not over 0.15 mm may be allowed for Type M-1, 4 mm in thickness, and this should be taken into account when calculating width of the specimen. Thus a typical section of a molded Type M-I specimen, having the maximum allowable draft, could be as follows:

Figure 3-7 ASTM D638 tensile dog bone (ASTM 1993)

An Instron tensile testing machine was used to pull the samples. The load cell was calibrated and customer gripers were added to the machine made for plastic samples. Samples were placed in the grips of the Instron tensile testing machine with a slight bend and the bend was removed as the sample was clamped into the grips.

For ASTM D638 the test speed is determined by the material specification. For ISO 527 the test speed is typically 5 or 50mm/min for measuring strength and elongation and 1mm/min for measuring modulus. Temperature was also tracked for each test and noted. See Appendix E for the detailed results of tensile tests.

The primary variable to be observed and compared to other samples was the maximum load in foot pounds that the sample would experiences before necking. After necking takes place in the sample the load measurement drops dramatically due to the decreasing cross section size of the sample.

4 Results

4.1 Introduction

The sequential results of the following tests and statistical evaluations of each test have been included in this section:

- Pareto Analysis
- Hot Filling Simulations
- Tensile Testing

The results of the tests illustrate a strong correlation between hot filling temperature and bottle deformation. Another trend observed is the occurrence of defects and a lower maximum tensile load along the bottle parting line compared to the bottle sidewall. A detailed overview of these results can be found in the following sections.

4.2 Pareto Analysis

The results of the Pareto analysis for two different samples of defective bottles can be found in Tables 4-1 and 4-2. Table 4-1 lists the type and frequency of defects that occurred in a sample of 36 defective bottles. Table 4-2 lists the type and frequency of defects that occurred in a sample of 9 defective bottles. The corresponding Pareto Charts for these results can be found in Figures 4-1 and 4-2 respective. A detailed written description of the defects can be found in Appendix A.

Table 4-1 Pareto analysis results of defective bottles (n=36)

(6 Cases x 6 Bottles = 36 Bottles)	# Defects
Major Indent Top Sidewall	2
Minor Indent Top Sidewall	2
Major Indent Bottom Sidewall	1
Minor Indent Bottom Sidewall	1
Major Indent Right Parting Line	11
Minor Indent Right Parting Line	10
Major Indent Left Parting Line	4
Minor Indent Left Parting Line	5

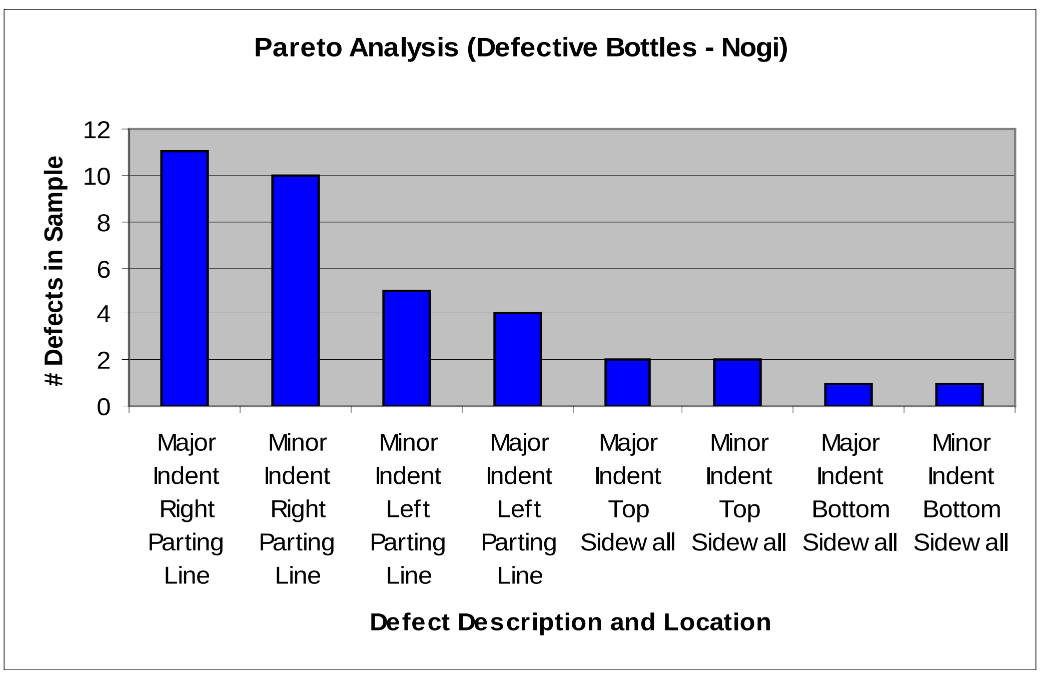

Figure 4-1 Pareto diagram of defective bottles (n=36)

Figure 4-1 illustrates an early trend observed in this study. Of 36 bottles, 21 of the bottles had noticeable deformation along the right parting line of the bottle. Of the remaining 15 bottles, 9 had visible deformation on the left parting line.

Table 4-2 Pareto analysis results of defective bottles (n=9)

(9 Individual Bottles)	# Defects
Major Indent Top Sidewall	0
Minor Indent Top Sidewall	0
Major Indent Bottom Sidewall	0
Minor Indent Bottom Sidewall	0
Major Indent Right Parting Line	0
Minor Indent Right Parting Line	0
Major Indent Left Parting Lin	6
Minor Indent Left Parting Line	3

Figure 4-2 illustrates a similar trend to that of Figure 4-1. All 9 of the defective bottles had observable deformation along the left parting line only.

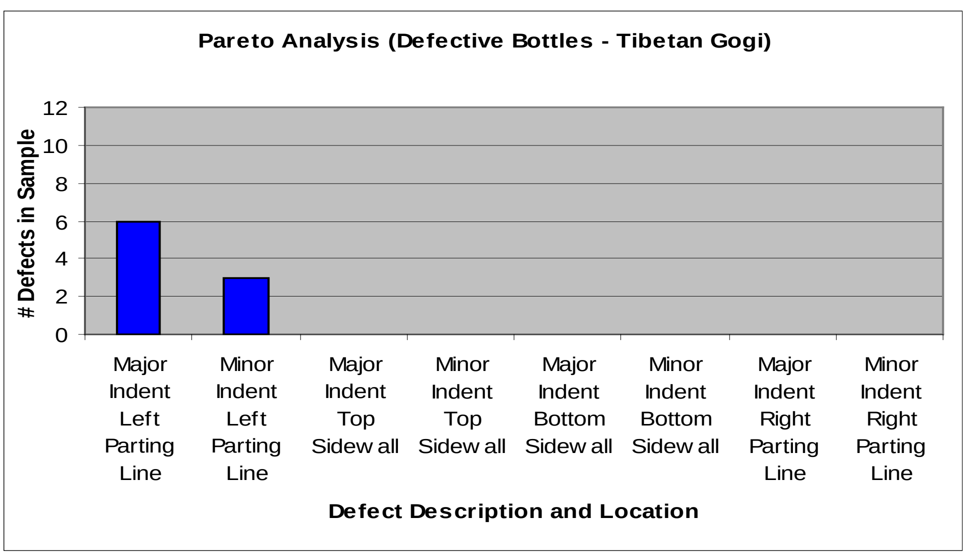

Figure 4-2 Pareto diagram of defective bottles (n=9)

A chi square test of significance was performed on the results of the Pareto analysis to test the hypothesis that there are differences in occurrence of the 8 types of defects. If each type of defect were equally likely, we expect 4.5 (36/8).We observed 11, 10, 5, 4, 2, 2, 1, 1 defects. For the category of "Major Indent Right Parting Line" there were 11occurrences. For the category of "Minor Indent Right Parting Line" there were 10 occurrences. For the category of "Minor Indent Left Parting Line" there were 4 occurrences. For the category of "Major Indent Left Parting Line" there were 5 occurrences. For both types of categories on the sidewall there were 2 occurrences (4 total). For both types of occurrences on the bottom sidewall there were 2 occurrences (4 total). These occurrences were used in the calculation of the chi square statistic as the observed inputs.

$$X^2 = \Sigma \text{ (Observed – Expected)}^2 / \text{(Expected)} = 24.44 \qquad (4\text{-}1)$$

With a p-value of 0.0019, we can conclude that the occurrence of defects among the bottle samples is not equally likely. Assuming the sample is representative of all bottles, the parting line of the bottles is more susceptible to defects than any other region of the bottle. The first sample of 36 defective bottles contained 30/36 bottles with defects along the parting lines. The second sample of 9 defective bottles contained 9/9 with defects along the left parting line.

Through these observations it was concluded that there is a spatial weakness along the bottle's parting line that is exploited above certain threshold temperatures.

4.3 Hot Filling Simulation

An initial hot filling simulation was conducted in an attempt to determine if defects would emerge at filling temperatures close to the HDT of HDPE, which ranges from 81 – 92°C. In the first simulation, the bottles were filled with juice at multiple liquid temperatures, sealed, and cooled for 48 hours. The bottles were then visually inspected for occurrences of deformation similar to those observed in the initial Pareto Analysis. The results of this simulation are described in Table 4-3.

Table 4-3 Initial hot filling simulation results

Temperature	Date Filled	Time Filled	Cooling Rate	Bottle Temp. Before Fill	Seal Status
75° C (167° F)	23-May-07	4:15 p.m.	4:51 p.m. = 132.9° F	73.8° F	Sealed
77.5° C (171.5° F)	23-May-07	4:33 p.m.	4:51 p.m. = 146° F	73.0° F	Sealed
80° C (176° F)	23-May-07	4:41 p.m.	4:51 p.m. = 156.1° F	71.3° F	Sealed
82.5° C (180.5° F)	23-May-07	4:56 p.m.	5:06 p.m. = 160.8° F	71.1° F	Sealed
85° C (185° F)	23-May-07	5:07 p.m.	5:20 p.m. = 161.1° F	70.7° F	Sealed
87.5° C (189.5° F)	23-May-07	5:23 p.m.	5:33 p.m. = 162.5° F	70.0° F	Sealed
90° C (194° F)	23-May-07	5:38 p.m.	5:51 p.m. = 164.5° F	70.0° F	Sealed
92.5° C (198.5° F)	23-May-07	6:24 p.m.	6:35 p.m. = 165° F	69.6° F	Sealed
95° C (203° F)	23-May-07	6:44 p.m.	NA	NA	Sealed
	24 Hours After Hot Fill				
75° C (167° F)	No Change				
77.5° C (171.5° F)	No Change				
80° C (176° F)	No Change				
82.5° C (180.5° F)	Very minor paneling along the parting line to the left of the number 2				
85° C (185° F)	Very minor paneling along the parting line to the right of the number 2				
87.5° C (189.5° F)	Very minor paneling along the parting line to the left of the number 6				
90° C (194° F)	Minor paneling along the bottle sidewall above the number 5. Square bottle shape to the left and right of this defect approximately 45°				
92.5° C (198.5° F)	Major paneling 20° to the right of the parting line to the right of the number 5. Indent in the top 3/4 of the bottle and diminishes to the bottom				
95° C (203° F)	Major paneling along the parting line to the right of the number 4				

Table 4-3 demonstrates that bottle deformation begins to emerge at a threshold temperature of 82.5°C. As the filling temperature increases, the degree of deformation becomes more dramatic.

This initial hot filling simulation provided validation that there is a positive correlation between hot filling temperature and bottle deformation.

Two additional hot filling simulations were conducted to determine the “trigger range” of temperatures through which the selected HDPE bottles would experience defects after the initial trial simulation was proven

These simulations were performed to further validate the correlation between filling temperature and bottle deformation. The filling order was randomized as described in Chapter 3 of the thesis to eliminate any statistical noise that could potentially bias the results. The filling of each bottle was repeated using a standardized procedure. This procedure is also described in Chapter 3 of the thesis.

The order of the first simulation was randomized and the order of the second simulation occurred in descending order from highest to lowest filling temperature (the opposite order of the trial hot filling simulation).

In an effort to quantify the degree of deformation that occurred after the bottles were hot filled, a series of diameter measurements were taken along various circumferences of the bottle. As outlined in Chapter 3, each bottle was divided into four regions labeled A through D as seen in Figure 4-3. The region was represented by a line drawn around the circumference of the bottle upon which all measurements were taken.

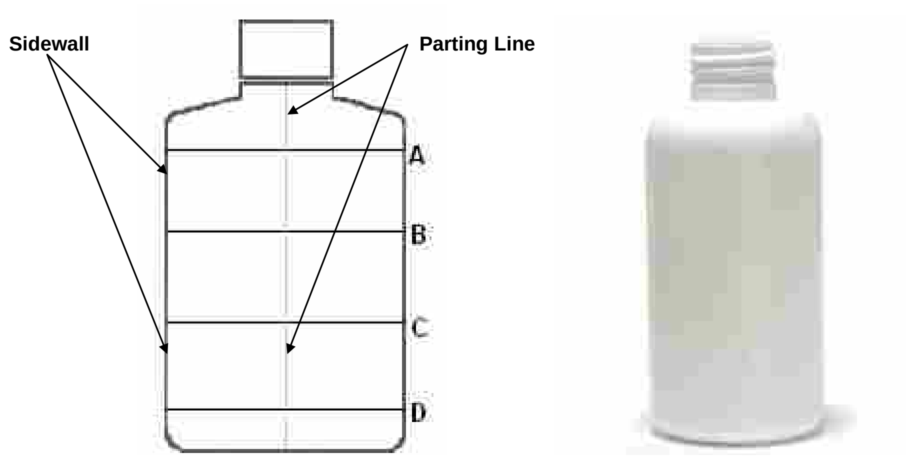

Figure 4-3 Hot filling simulation testing regions with bottle descriptions (left) and HDPE

Region A is located in the top quarter section of the bottle and the additional regions comprise the remaining quarter sections of the bottle, descending in alphabetical order from top to bottom.

In region A the researcher measured the diameter from parting line to parting line and sidewall to sidewall. These two diameter measurements were then averaged to create a diameter measurement that is intended to represent the entire bottle in that region. After the bottles were hot filled, sealed, and cooled for a period of 48 hours, the bottles were then observed for occurrences of deformation. A contraction of the bottle would result in a smaller diameter from side to side. Any expansion of the bottle would increase the diameter from side to side.

If the bottles experienced any deformation there would be a resultant change in the diameter of the bottle in that region. Instead of recording the diameter from parting line to parting line and sidewall to sidewall after hot filling, the researcher located the smallest diameter regardless of its location. This diameter measurement was then compared to the original diameter average for that region and the difference in diameter represented deformation. If the final diameter minus the initial diameter was negative a contraction of the bottle had occurred and vice versa.

This procedure was repeated for each bottle region, A through D. A total of 20 bottles were used in this study, 10 for each hot filling simulation. Each bottle contained four regions from which diameter measurements were taken, for a total of 40 data points per simulation.

Table 4-4 contains all of the data recorded during Hot Filling Simulation #1. The filling temperature, filling time and number of each bottle are recorded three left columns of the table. The table also includes the diameter measurements from parting line to parting line (Parting Line Diameter), sidewall to sidewall (Sidewall Diameter), and the average of both measurements. The minimum diameter of that region was also recorded and the resulting amount of deformation in the two right columns respectively. All diameter and deformation measurements are recorded in inches.

From the data contained in Table 4-4, four scatter plots (See figures 4-4 to 4-7) were generated to visually illustrate the amount of deformation that occurred for each region of the bottle. On these scatter plots the deformation of each filled bottle is located on the y-axis, and the liquid filling temperature in degrees Celsius is listed on the x-axis. Each individual scatter plot contains 10 measurements, one for each filling temperature.

Table 4-4 Hot Filling Simulation # 1 Data

Bottle #	Fill Temp. (°C)	Fill Time	Region A Parting Line Diameter	Region A Sidewall Diameter	Region A Average Diameter	Region A Minimum Diameter	Deformation (Inches)
5	95	3:44 p.m.	3.639	3.657	3.648	3.42	**0.228**
1	92.5	2:29 p.m.	3.642	3.657	3.6495	3.419	**0.2305**
9	90	4:18 p.m.	3.642	3.658	3.65	3.396	**0.254**
8	87.5	4:05 p.m.	3.646	3.657	3.6515	3.603	**0.0485**
10	85	4:24 p.m.	3.645	3.655	3.65	3.589	**0.061**
6	82.5	3:48 p.m.	3.639	3.653	3.646	3.608	**0.038**
2	80	2:55 p.m.	3.641	3.652	3.6465	3.609	**0.0375**
7	77.5	3:57 p.m.	3.644	3.658	3.651	3.611	**0.04**
3	75	3:11 p.m.	3.638	3.654	3.646	3.621	**0.025**
4	72.5	3:20 p.m.	3.636	3.655	3.6455	3.614	**0.0315**
Bottle #	**Fill Temp. (°C)**	**Fill Time**	**Region B Parting Line Diameter**	**Region B Parting Line Diameter**	**Region B Average Diameter**	**Region B Minimum Diameter**	**Deformation (Inches)**
5	95	3:44 p.m.	3.648	3.657	3.6525	3.19	**0.4625**
1	92.5	2:29 p.m.	3.649	3.658	3.6535	3.188	**0.4655**
9	90	4:18 p.m.	3.644	3.652	3.648	3.172	**0.476**
8	87.5	4:05 p.m.	3.651	3.657	3.654	3.559	**0.095**
10	85	4:24 p.m.	3.642	3.655	3.6485	3.537	**0.1115**
6	82.5	3:48 p.m.	3.64	3.657	3.6485	3.58	**0.0685**
2	80	2:55 p.m.	3.648	3.656	3.652	3.602	**0.05**
7	77.5	3:57 p.m.	3.64	3.657	3.6485	3.593	**0.0555**
3	75	3:11 p.m.	3.641	3.651	3.646	3.611	**0.035**
4	72.5	3:20 p.m.	3.635	3.655	3.645	3.607	**0.038**

Table 4-4 Hot Filling Simulation #1 Data Continued

Bottle #	Fill Temp . (°C)	Fill Time	Region C Parting Line Diameter	Region C Parting Line Diameter	Region C Average Diameter	Region C Minimum Diameter	Deformation (Inches)
5	95	3:44 p.m.	3.648	3.647	3.6475	3.332	**0.3155**
1	92.5	2:29 p.m.	3.645	3.647	3.646	3.343	**0.303**
9	90	4:18 p.m.	3.642	3.654	3.648	3.324	**0.324**
8	87.5	4:05 p.m.	3.641	3.653	3.647	3.564	**0.083**
10	85	4:24 p.m.	3.635	3.653	3.644	3.546	**0.098**
6	82.5	3:48 p.m.	3.645	3.655	3.65	3.587	**0.063**
2	80	2:55 p.m.	3.645	3.655	3.65	3.608	**0.042**
7	77.5	3:57 p.m.	3.637	3.655	3.646	3.594	**0.052**
3	75	3:11 p.m.	3.644	3.648	3.646	3.61	**0.036**
4	72.5	3:20 p.m.	3.639	3.65	3.6445	3.616	**0.0285**
Bottle #	**Fill Temp . (°C)**	**Fill Time**	**Region D Parting Line Diameter**	**Region D Parting Line Diameter**	**Region D Average Diameter**	**Region D Minimum Diameter**	**Deformation (Inches)**
5	95	3:44 p.m.	3.645	3.648	3.6465	3.571	**0.0755**
1	92.5	2:29 p.m.	3.647	3.648	3.6475	3.564	**0.0835**
9	90	4:18 p.m.	3.644	3.651	3.6475	3.558	**0.0895**
8	87.5	4:05 p.m.	3.644	3.648	3.646	3.608	**0.038**
10	85	4:24 p.m.	3.637	3.644	3.6405	3.593	**0.0475**
6	82.5	3:48 p.m.	3.644	3.648	3.646	3.617	**0.029**
2	80	2:55 p.m.	3.649	3.651	3.65	3.624	**0.026**
7	77.5	3:57 p.m.	3.642	3.651	3.6465	3.616	**0.0305**
3	75	3:11 p.m.	3.645	3.65	3.6475	3.628	**0.0195**
4	72.5	3:20 p.m.	3.642	3.648	3.645	3.631	**0.014**

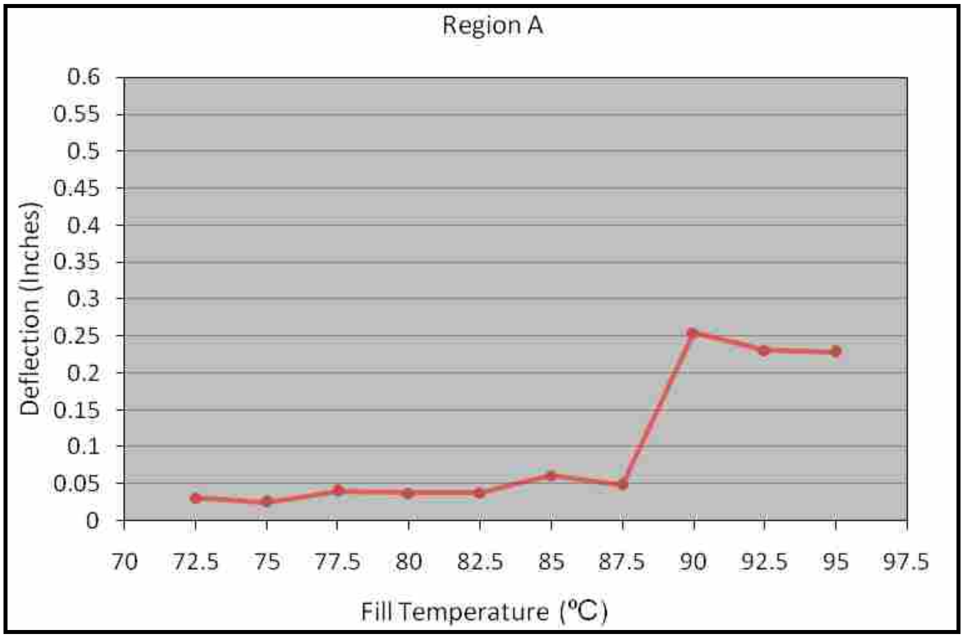

Figure 4-4 Region A: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

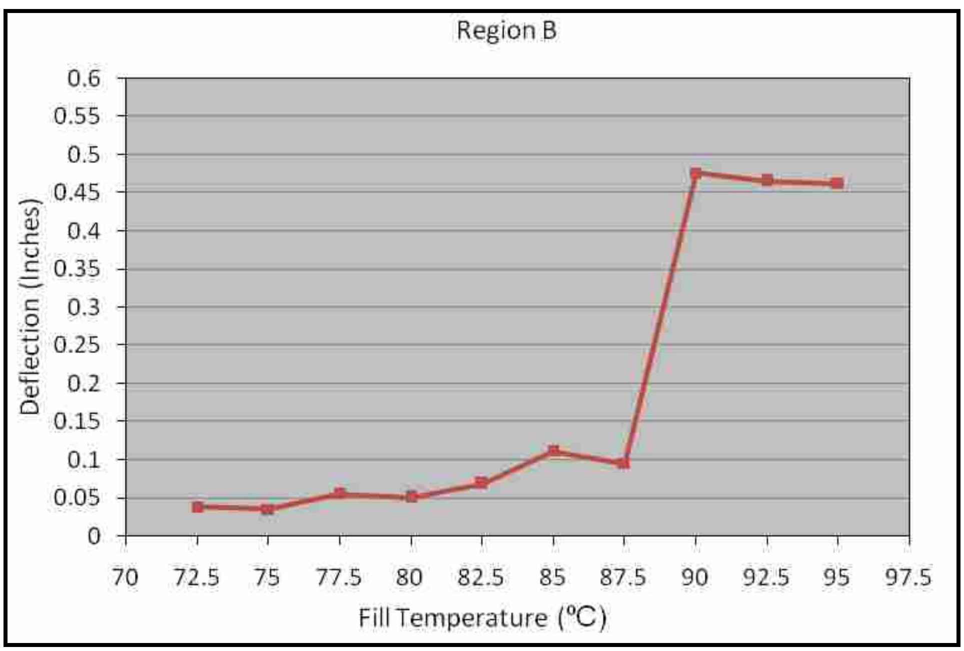

Figure 4-5 Region B: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

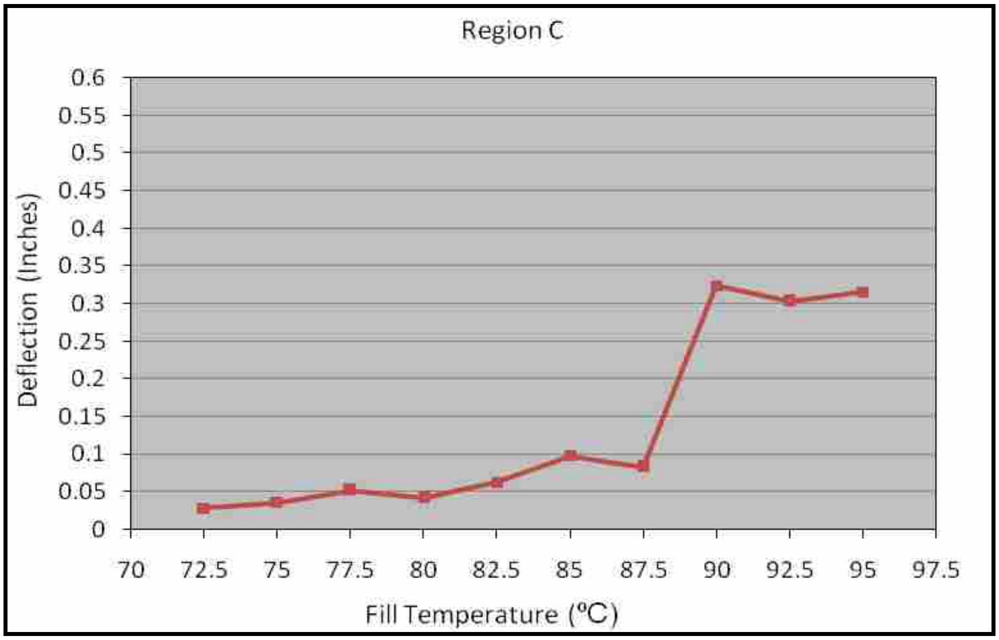

Figure 4-6 Region C: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

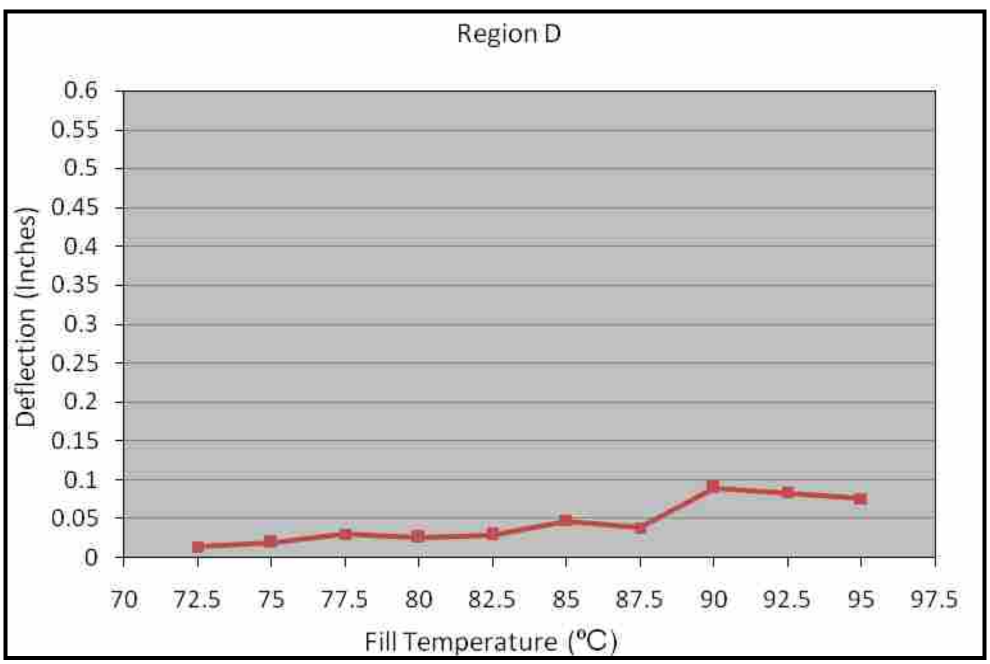

Figure 4-7 Region D: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

These scatter plots demonstrate a positive correlation between liquid hot filling temperature and degree of deformation. As the hot filling temperature rises, the degree of deformation increases. There also appears to be a threshold temperature of 87.5º C after which a dramatic amount of deformation begins to occur indefinitely.

Tables 4-5 and Figures 4-8 through 4-11 represent the data and scatter plots for Hot Filling Simulation #2.

Table 4-5 Hot Filling Simulation # 2 Data

Bottle #	Fill Temp. (°C)	Fill Time	Region A Parting Line Diameter	Region A Sidewall Diameter	Region A Average Diameter	Region A Minimum Diameter	Deformation (Inches)
11	95	4:45 p.m.	3.642	3.655	3.6485	3.464	**0.1845**
12	92.5	5:06 p.m.	3.638	3.656	3.647	3.398	**0.249**
13	90	5:29 p.m.	3.639	3.655	3.647	3.416	**0.231**
14	87.5	5:44 p.m.	3.642	3.652	3.647	3.481	**0.166**
15	85	5:55 p.m.	3.642	3.651	3.6465	3.586	**0.0605**
16	82.5	6:08 p.m.	3.646	3.656	3.651	3.559	**0.092**
17	80	6:13 p.m.	3.644	3.653	3.6485	3.602	**0.0465**
18	77.5	6:22 p.m.	3.639	3.654	3.6465	3.609	**0.0375**
19	75	6:25 p.m.	3.639	3.658	3.6485	3.613	**0.0355**
20	72.5	6:36 p.m.	3.635	3.652	3.6435	3.603	**0.0405**
Bottle #	**Fill Temp. (°C)**	**Fill Time**	**Region B Parting Line Diameter**	**Region B Sidewall Diameter**	**Region B Average Diameter**	**Region B Minimum Diameter**	**Deformation (Inches)**
11	95	4:45 p.m.	3.647	3.65	3.6485	3.21	**0.4385**
12	92.5	5:06 p.m.	3.64	3.656	3.648	3.153	**0.495**
13	90	5:29 p.m.	3.641	3.657	3.649	3.274	**0.375**
14	87.5	5:44 p.m.	3.638	3.657	3.6475	3.327	**0.3205**
15	85	5:55 p.m.	3.649	3.651	3.65	3.563	**0.087**
16	82.5	6:08 p.m.	3.647	3.655	3.651	3.527	**0.124**
17	80	6:13 p.m.	3.638	3.653	3.6455	3.578	**0.0675**
18	77.5	6:22 p.m.	3.644	3.656	3.65	3.578	**0.072**
19	75	6:25 p.m.	3.649	3.66	3.6545	3.604	**0.0505**
20	72.5	6:36 p.m.	3.639	3.652	3.6455	3.598	**0.0475**

Table 4-5 Hot Filling Simulation # 2 Data Continued

Bottle #	Fill Temp . (°C)	Fill Time	Region C Parting Line Diameter	Region C Sidewall Diameter	Region C Average Diameter	Region C Minimum Diameter	Deformation (Inches)
11	95	4:45 p.m.	3.645	3.651	3.648	3.396	**0.252**
12	92.5	5:06 p.m.	3.637	3.654	3.6455	3.195	**0.4505**
13	90	5:29 p.m.	3.644	3.653	3.6485	3.485	**0.1635**
14	87.5	5:44 p.m.	3.638	3.653	3.6455	3.436	**0.2095**
15	85	5:55 p.m.	3.65	3.649	3.6495	3.59	**0.0595**
16	82.5	6:08 p.m.	3.65	3.647	3.6485	3.567	**0.0815**
17	80	6:13 p.m.	3.638	3.65	3.644	3.568	**0.076**
18	77.5	6:22 p.m.	3.643	3.651	3.647	3.595	**0.052**
19	75	6:25 p.m.	3.641	3.655	3.648	3.597	**0.051**
20	72.5	6:36 p.m.	3.641	3.653	3.647	3.605	**0.042**
Bottle #	**Fill Temp . (°C)**	**Fill Time**	**Region D Parting Line Diameter**	**Region D Sidewall Diameter**	**Region D Average Diameter**	**Region D Minimum Diameter**	**Deformation (Inches)**
11	95	4:45 p.m.	3.643	3.648	3.6455	3.571	**0.0745**
12	92.5	5:06 p.m.	3.641	3.65	3.6455	3.531	**0.1145**
13	90	5:29 p.m.	3.645	3.651	3.648	3.603	**0.045**
14	87.5	5:44 p.m.	3.643	3.648	3.6455	3.57	**0.0755**
15	85	5:55 p.m.	3.647	3.644	3.6455	3.614	**0.0315**
16	82.5	6:08 p.m.	3.642	3.648	3.645	3.607	**0.038**
17	80	6:13 p.m.	3.641	3.65	3.6455	3.616	**0.0295**
18	77.5	6:22 p.m.	3.644	3.649	3.6465	3.622	**0.0245**
19	75	6:25 p.m.	3.643	3.649	3.646	3.625	**0.021**
20	72.5	6:36 p.m.	3.643	3.648	3.6455	3.624	**0.0215**

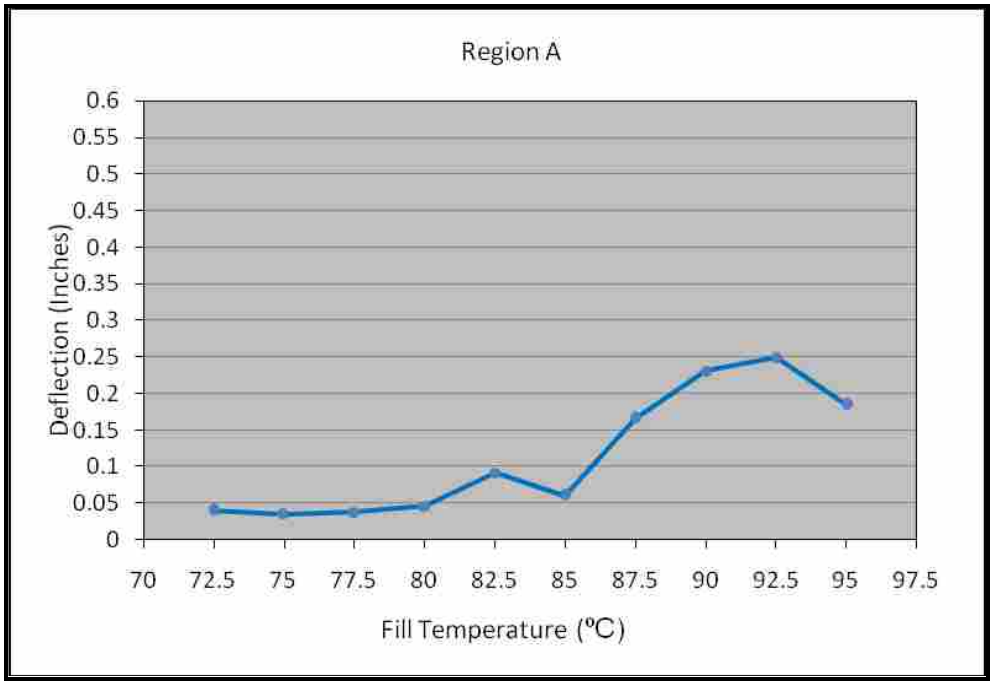

Figure 4-8 Region A: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

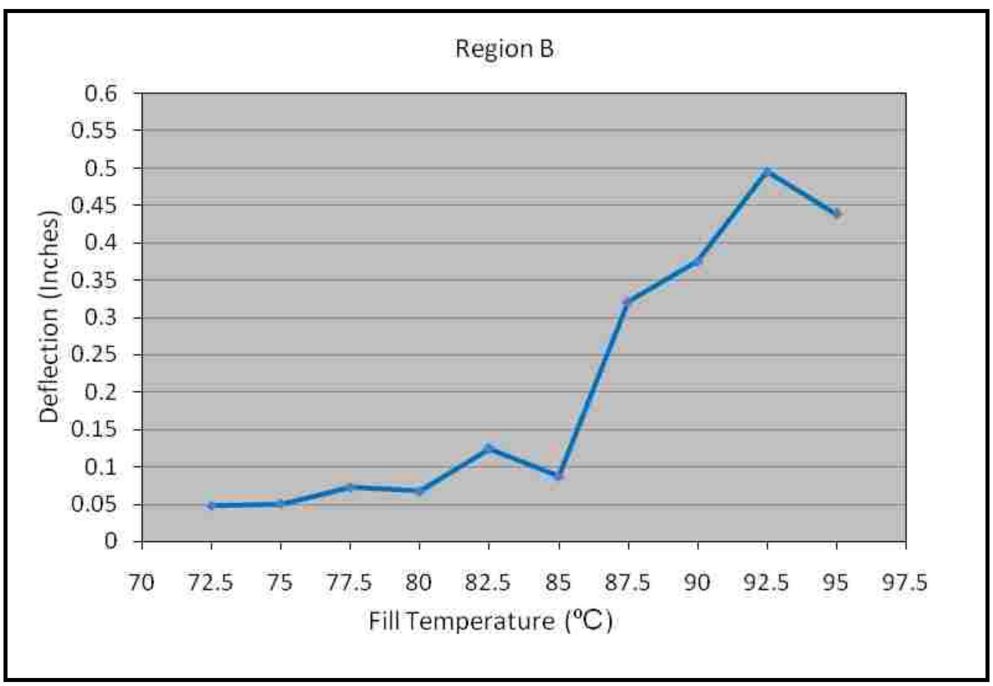

Figure 4-9 Region B: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

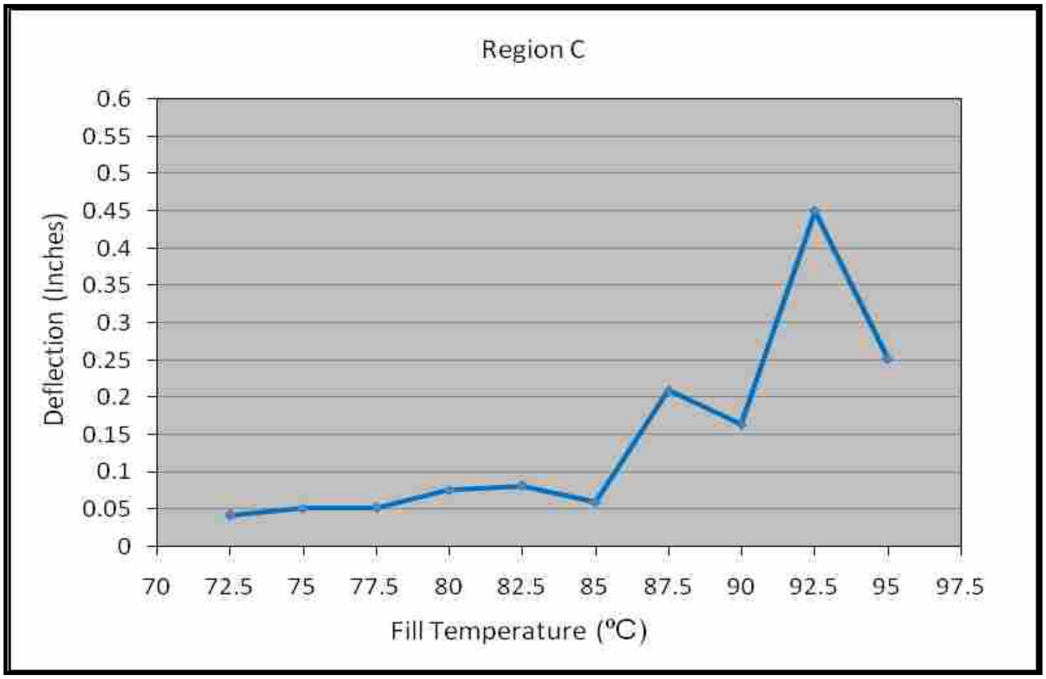

Figure 4-10 Region C: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

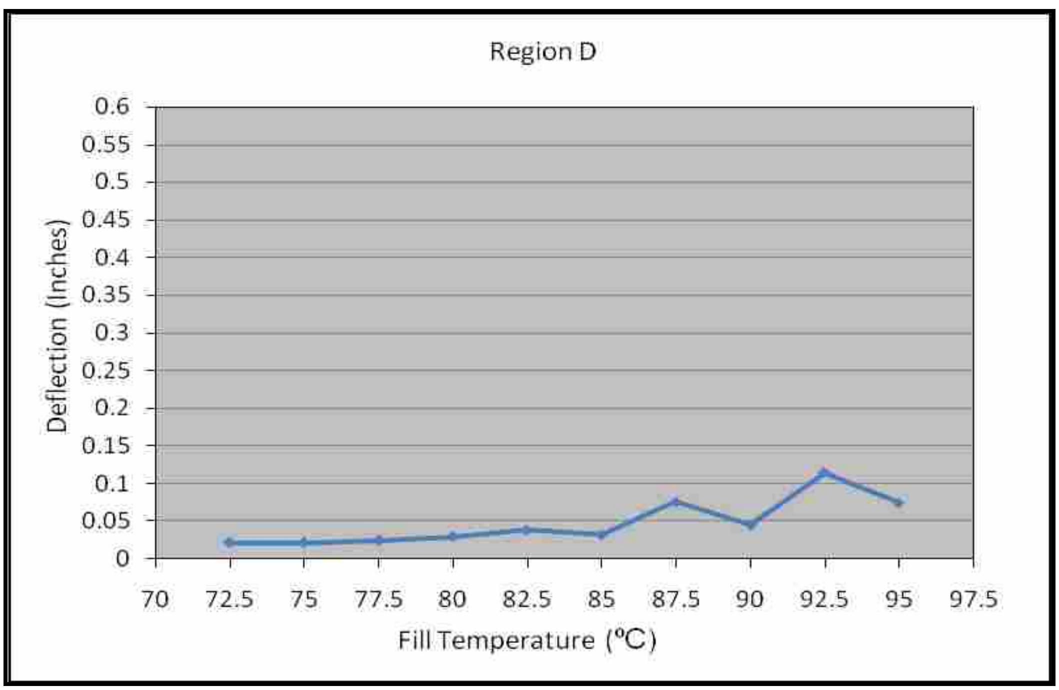

Figure 4-11 Region D: Filling Temperature (ºC) vs. Bottle Deformation (Inches)

Figures 4-8 though 4-11 demonstrate a similar trend to Figures 4-4 through 4-7, there is a positive correlation between liquid hot filling temperature and degree of deformation.

Through these simulations it was proven that bottle deformation would occur in a trigger range between 85 and 95° C. It was also observed that these shape changes continued to occur along the parting lines of the bottles.

It was observed during various hot filling simulations and the initial Pareto analysis, that there is a spatial weakness in the bottle along the parting line. This weakness is present but not exploited until hot filling temperature is increased to fall within the bottles trigger range. These results lead to an additional test to determine possible mechanical and structural differences between the parting line and sidewall of the bottle**.**

4.4 Tensile Testing

The purpose of performing the tensile tests on these bottles was to determine if the mechanical properties of the various regions of the bottle are different between bottle locations and to quantify these differences. The results of these tests demonstrated the inherent betrayal properties in the sidewall versus the parting line of the bottle and confirmed the existence of a spatial weakness along the parting line of the bottle. The bottles used in this test were unfilled virgin HDPE bottles.

The maximum elongation and maximum yield were recorded for samples cut from various regions of the bottle with the center of the test specimen on either the center of the bottle sidewall or the bottle parting line, as shown in Figure 3-3.

Table 4-7 shows the maximum load for every sample that was tested. Tensile runs 1 through 4 represent bottles that were tested along the bottle sidewall. Tensile runs 5 through 8 represent bottles that were tested along the bottle parting line. Table 4-8 lists averages that were calculated for each tensile run and for the bottle regions combined.

Table 4-6 Tensile testing results (maximum load of each bottle location in lbf)

Tensile Run	Location	B #	Max Load (lbf)	B #	Max Load	B #	Max Load	B #	Max Load	B #	Max Load
1	UTSW	1	46.2818	2	48.8052	3	47.3019	5	46.5234	6	47.7046
2	LTSW	1	57.7717	2	55.1676	3	58.9529	5	55.3824	6	53.8254
3	UBSW	1	46.0938	2	52.8858	3	45.5301	5	53.9059	6	54.2817
4	LBSW	1	58.9797	2	60.0804	3	58.577	5	60.0804	6	60.0267
5	URPL	1	39.5167	2	39.3019	3	38.2012	5	38.0133	6	44.6173
6	LRPL	1	50.3891	2	48.6979	3	47.812	5	47.7851	6	51.9462
7	ULPL	1	41.0469	2	38.5771	3	39.5972	5	42.9529	6	40.8321
8	LLPL	1	52.1878	2	47.9193	3	53.1812	5	57.2616	6	50.6039

Table 4-7 Summary of tensile results

Tensile Run	Location	Ave Max Load (lbf)
1	UTSW	47.32338
2	LTSW	56.22
3	UBSW	50.53946
4	LBSW	59.54884
Sidewall Average Max Load = 53.4079		
5	URPL	39.93008
6	LRPL	49.32606
7	ULPL	40.60124
8	LLPL	52.23076
Parting Line Average Max Load = 45.522		

The average maximum load of the bottles that were tensile tested with the parting line in the center of the dog bone was 45.522 lbf. The average maximum load of the bottles that were tensile tested with the center of the sidewall in the center of dog bone was 53.4079 lbf. The difference between the two results is 7.8859 lbf. Therefore, in general, the sidewall has higher mechanical strength with an average maximum tensile load of 53.54884 lbf.

Following the tensile tests an analysis of variance (ANOVA) was performed on the data to show this difference statistically. This ANOVA table in 4.9 summarizes the effect of the interaction, and the differences between bottles. “The Sidewall vs. The Parting Line” and “Upper vs. Lower.”

There is a statistically significant difference between measurements on the sidewall and parting line (pvalue < .001). The tensile strength is higher for the average of the tensile samples with the sidewall in the center of the dog bone.

There is a statistically significant difference between measurements on the upper and lower bottle locations (pvalue<.001). As mentioned in Chapter 2, the upper regions of the bottle are generally thinner than the lower regions of the bottle. It can be observed that this influences the maximum tensile strength of the sample depending on the sample location.

There is not a significant interaction, meaning that measurements are consistent with the main effects. A significant interaction would have indicated that say measurements on the upper parting line differed from the lower parting line but there was no difference between upper and lower measurements on the sidewalls.

The last factor in the ANOVA table is entitled "Bottle," which if its p-value is significant would indicate a difference among bottles used to create tensile samples. Since the p-value of 0.5973 is not significant, the bottles appear to be a homogeneous sample.

Table 4-8 Analysis of Variance (ANOVA) table

Response: y	**Df**	**Sum Sq**	**Mean Sq**	**F Value**	**Pr(>F)**
Sidewall or Parting Line	1	621.87	621.87	78.5232	<.001
Upper or Lower	1	947.29	947.29	119.6133	<.001
Location Interaction	1	6.08	6.08	0.7680	0.3874
Bottle	4	22.19	5.55	0.7006	0.5973
Residuals	32	253.43	7.92		

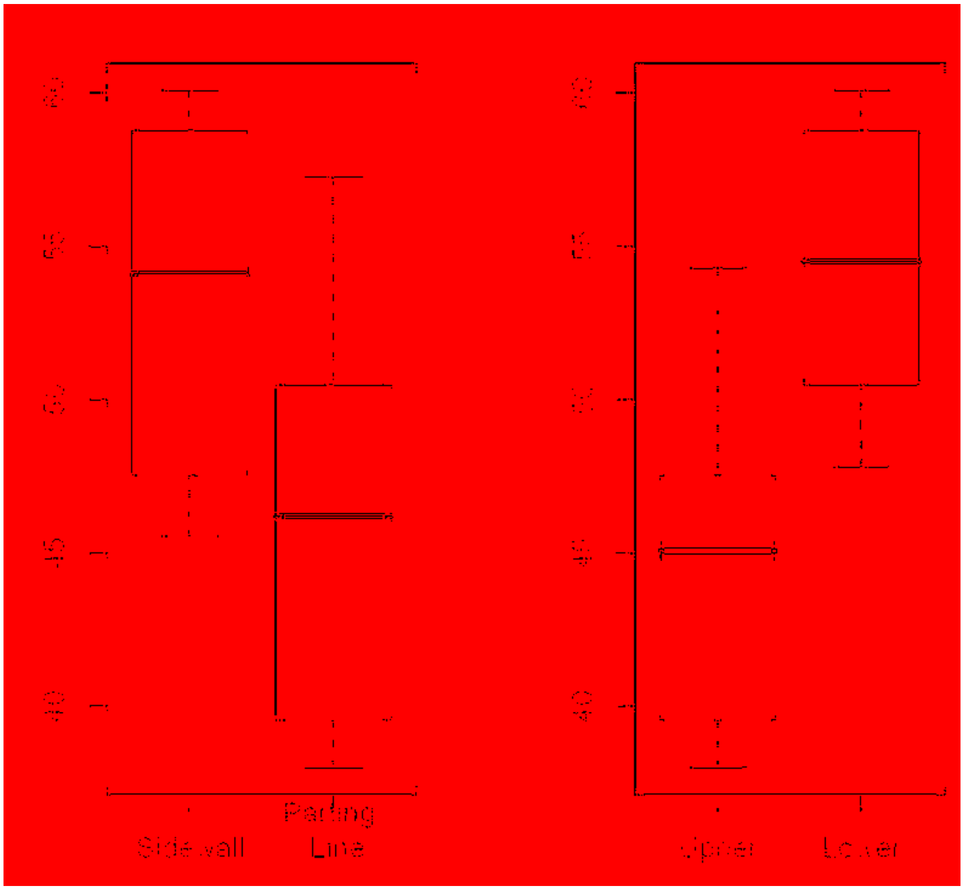

Figure 4-12 Box Plots of ANOVA results

The ANOVA results in Figure 4-12 display the dramatic differences between the characteristics of the “Sidewall” vs. the “Parting Line,” and the “Upper” region of the bottle vs. the “Lower” region of the bottle. The difference between the box plots locations demonstrates the statistical difference.

5 Conclusions and Recommendations

5.1 Purpose of Research

The occurrence of deformation in plastic bottles is a common problem in the bottling industry where bottles are blow molded, hot filled at high temperatures and sealed. The root cause of this deformation is a process problem, which is unknown to most companies who have experienced such defects

The purpose of the research was to observe the blow molding and hot filling processes and to determine which variables in those processes influence bottle shape. In earlier tests the both the blow molding resin and bottle thickness were changed in an effort to eliminate defects. The result of changing these variables did not create a decrease in defects. The use of an Ishikawa fishbone diagram identified hot filling temperature a major variable that influences final bottle shape.

5.2 Hot Filling Simulations

The effect of hot filling temperature on final bottle shape was tested with two hot filling simulations. These simulations identified the temperature, or “trigger range”, at which the bottles start to experience dramatic shape changes. In the first simulation the filling order was randomized. In the second simulation the filling order occurred from the highest temperature (95ºC) to the lowest temperature (72.5ºC).

Figures 5-1 through 5-4 illustrate the results of both hot filling simulations for each of the four bottle regions, regions A through D. The orange vertical line marks the point at which major deflection begins to occur in both simulations.

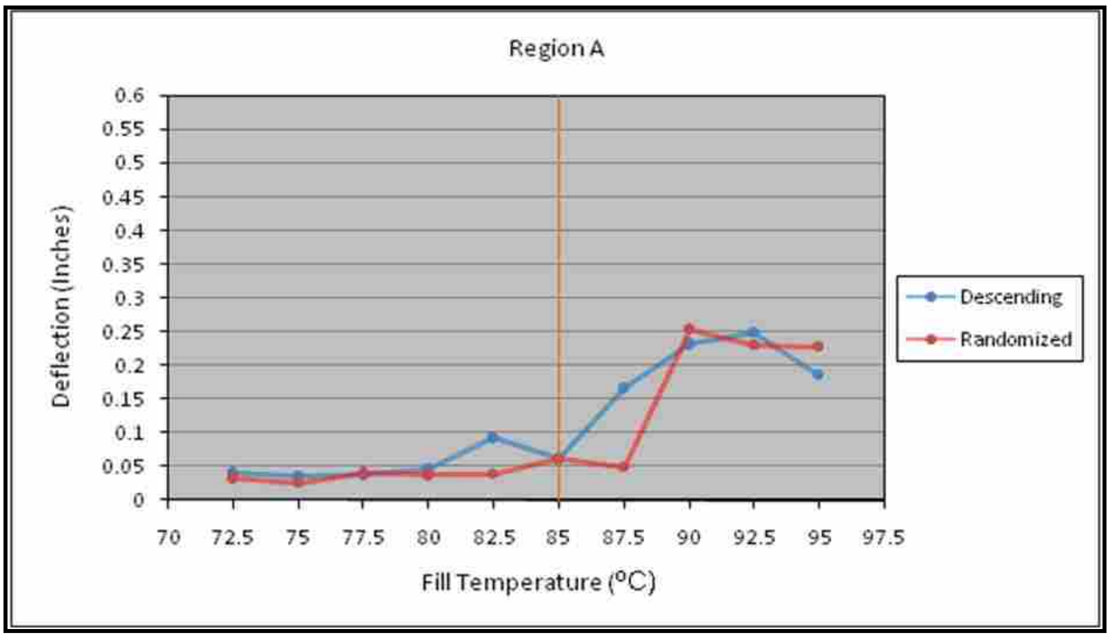

Figure 5-1 Combined Hot Filling Simulation Results for Region A

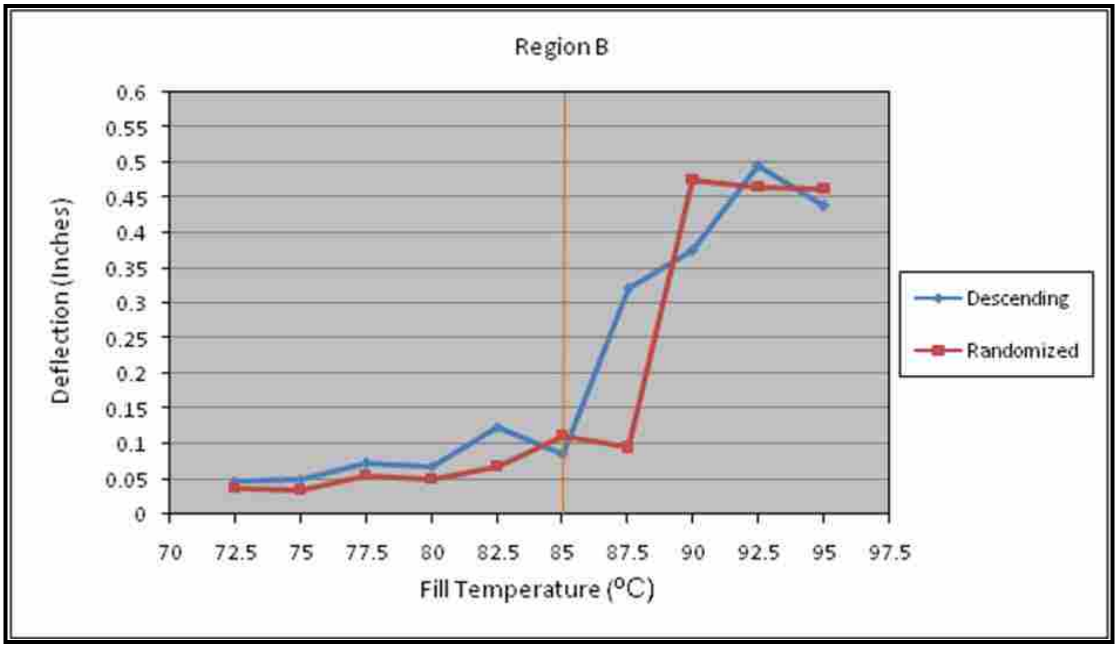

Figure 5-2 Combined Hot Filling Simulation Results for Region B

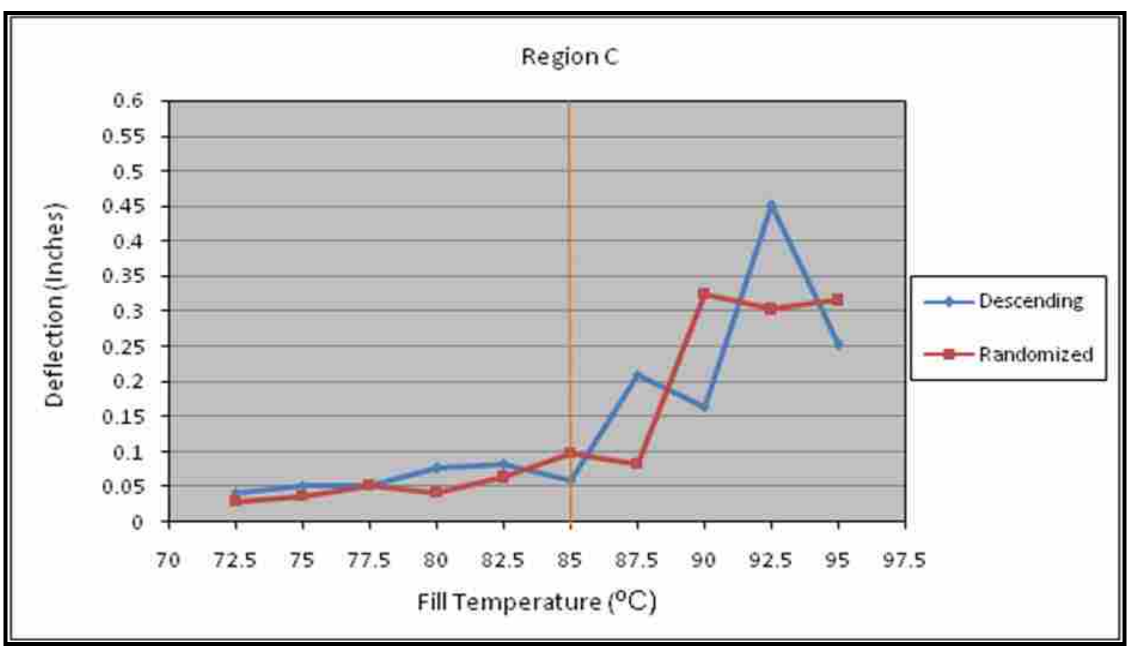

Figure 5-3 Combined Hot Filling Simulation Results for Region C

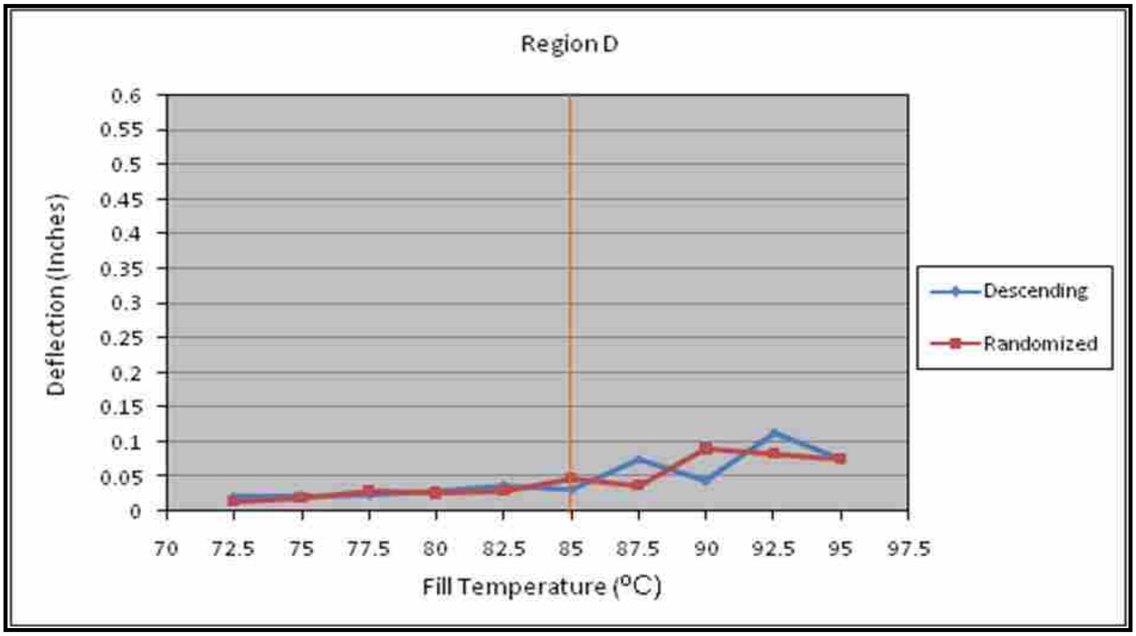

Figure 5-4 Combined Hot Filling Simulation Results for Region D

The Heat Deflection Temperature (HDT) of HDPE ranges from 82ºC to 91°C (Peacock 2000). When the hot filling temperature begins to approach the lowest temperature of the HDT range, bottle fillers should be aware that dramatic defects will begin to emerge.

In addition, the results of these simulations demonstrate a positive correlation between liquid hot filling temperature and the degree of deflection observed on the surface of the bottles. The data from both hot filling simulations show that after 85ºC the overall magnitude of deflection begins to increase dramatically. This is particularly true in Region B of the bottle (See Figure 5-2)

5.3 Pareto Analysis and Tensile Testing

An early Pareto Analysis of defective bottle showed that bottle defects are more likely to occur along the bottle's parting line. In fact, in a sample of thirty six defective bottles, thirty of those bottles experienced deflection near the vicinity of the parting line. In another sample of nine defective bottles, all nine bottles had defects on the parting line. A chi square test of significance was performed on the results of the Pareto analysis to test the hypothesis that there are differences in occurrence of the 8 types of defects. If each type of defect were equally likely, we expect 4.5 (36/8).We observed 11, 10, 5, 4, 2, 2, 1, 1 defects.

With a p-value of 0.0019, we can conclude that the occurrence of defects among the bottle samples is not equally likely. Assuming the sample is representative of all bottles, the parting line of the bottles is more susceptible to defects than any other region of the bottle. his analysis discovered a possible spatial weakness located along the regions of the parting line.

A series of tensile tests were performed to test the difference in strength among the different bottle regions. The following values were generated from these tensile tests:

- Sidewall average max load = 53.4079
- Parting line average max load = 45.522

An ANOVA was performed which determined the following:

- There is a statistically significant difference between measurements on the sidewall and parting line (pvalue < .001).
- There is a statistically significant difference between measurements on the upper and lower bottle locations (pvalue<.001).

- Measurements on the upper parting line differed from the lower parting line (pvalue>.001).
- The bottles appear to be a homogenous sample (pvalue>.001).

The overall results of the tensile testing and ANOVA further confirm that the area of the bottle containing the parting line is more susceptible to deformation when exposed to high temperature liquids.

The reason for this significant difference in strength is a difference in sample thickness. Figure 5-1 shows thickness measurements for all the samples used for the tensile tests (See Appendix E). The average thickness measurements for each bottle region are included at the bottom of each column.

Table 5-1 Tensile Sample Thickness Measurements

Tensile Sample Thickness (Inches)				
	UTSW	**LTSW**	**URPL**	**ULPL**
	0.0640	0.0770	0.0470	0.0495
	0.0600	0.0690	0.0480	0.0480
	0.0630	0.0755	0.0455	0.0495
	0.0595	0.0685	0.0445	0.0525
	0.0595	0.0685	0.0555	0.0500
Average	**0.0612**	**0.0717**	**0.0481**	**0.0499**
	UBSW	**LBSW**	**LRPL**	**LLPL**
	0.0580	0.0735	0.0580	0.0640
	0.0650	0.0725	0.0600	0.0565
	0.0565	0.0700	0.0575	0.0645
	0.0650	0.0750	0.0550	0.0640
	0.0655	0.0745	0.0610	0.0580
Average	**0.0620**	**0.0731**	**0.0583**	**0.0614**

From this table we can see that the parting line regions in the upper portion of the bottle are much thinner than the other regions of the bottle. At filling temperatures above 85ºC this thinner region becomes the weakest area of the bottle. The research shows that defects are more likely to occur at these weaker areas.

It is impossible to have a perfectly uniform bottle because the blow molding process inherently creates fluctuations in wall thickness. To permanently fix the problem of defects, bottle producers should refine their blow molding process to minimize fluctuations in wall thickness between locations along the same horizontal circumference. Bottle producers should also maintain their two-part molds so that the appearance of the parting line on the surface of the bottle is less visible.

5.4 General Conclusions

The null hypothesis of this thesis was that liquid hot filling temperature has no effect on the final shape of the bottle. During the course of the research the null hypothesis was rejected and it was concluded that liquid hot filling temperature significantly affects the final bottle shape.

As a result of the research, the participants could conclude that problems experienced with deformation were a result of excessive filling temperatures. For this particular HDPE bottle, deformation will inevitably occur in a trigger range between 85 and 95° C. Therefore manufacturers using this particular bottle should ensure that filling temperatures do not exceed 85° C to prevent possible defects.

Another important characteristic of these bottles was discovered during the initial Pareto analysis and the hot filling simulations. It was observed that there was an inherent spatial weakness in the bottle along the parting line. This weakness was present but not exploited until hot filling temperature is increased to fall within the bottles trigger range.

The results of the tensile testing supported this claim showing that there was a significant difference in maximum tensile load between the parting line of the bottle and the sidewall. This difference of 7.8859 lbf was found to be statistically significant through an ANOVA. We further concluded that this difference is due to differences in wall thickness between regions, not wall thickness overall.

5.5 Recommendations for Manufacturers

The results of the research provide numerous insights for manufacturers who use the hot filling process to fill HDPE bottles. In order to get better performance and decrease the defect rate that occurs in bottles, manufacturers should consider both bottle design and bottle material before determining the appropriate hot filling temperature.

It is often common to determine an arbitrary hot filling temperature based on the HDT of the bottle's material. The research shows that there may be other variables that contribute to the emergence of defects and that the filling temperature can be affected by these variables. Bottle producers cannot increase the hot filling temperature without considering the consequences of this change. Figures 5-1 through 5-4 show that small changes in filling temperature lead to dramatic fluctuations in deflection on the outer surface of the bottle.

The research process described in this thesis can be used by bottle manufacturers and hot filling companies to minimize the occurrence of post hot filling bottle defects. By following the steps outlined in this thesis and performing hot filing simulations, the hot filling trigger range for a unique bottle design can be determined and manufacturers can reduce the occurrence of defects dramatically.

This recommendation is particularly relevant for companies that hot fill bottles not designed for hot filling applications. For these companies it is even more important to determine the appropriate hot filling temperature through simulation.

For manufacturers already experiencing defects, the researcher recommends the same approach as for those companies who are trying to prevent defects. It would also be valuable for these manufacturers to progress through the entire process explained in the thesis to discover additional root causes that may be affecting the process.

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APPENDICES

Appendix A. Pareto Analysis Notes

PRODUCT NO: 27-16812
LOT NO: 16031071
EXP DATE: 4/2008
DESCRIPTION: NOJI
CASE QTY: 6
INNER PACKS: 1
UNIT SIZE: 32 OZ. 946 ML

DEFECT DESCRIPTION – CASE #1

Bottle #	Defect Description
1	Indent not visible because seal has been broken. Indent originally along the parting line to the left of the number 6, very minor.
2	Major indent along the parting line to the right of the number 4. Occurred at the top half of the bottle sidewall and diminishes toward the bottom.
3	Major indent along the sidewall below the number 3. Extends the length of the bottle.
4	Minor indent along the parting line to the right of the number 5. Slightly offset to the left of the parting line 20°. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom.
5	Major indent along the parting line to the right of the number 4. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom. No labeling.
6	Very minor indent along the parting line to the left of the number 6. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom.

DEFECT DESCRIPTION – CASE #2

Bottle #	Defect Description
1	Indent not visible because seal has been broken. Indent originally along the parting line to the right of the number 5, very minor.
2	Very major indent along the parting line to the left of the number 1. Indent occurs in the top ¾ of the bottle and diminishes toward the bottom.
3	Major indent along the parting line to the right of the number 4. Indent

	occurs in the top ¾ of the bottle and diminishes toward the bottom.
4	Very minor indent along the bottle parting line to the right of the number 2. Very minor indent 45° to the right of the other parting line.
5	Major indent 30° to the left of the parting line to the left of the number 2. Indent occurs in the top ¾ of the bottle and diminishes toward the bottom.
6	Very minor indent along the parting line to the right of the number 3.

DEFECT DESCRIPTION – CASE #3

Bottle #	Defect Description
1	Slight indent along the bottle sidewall between the parting lines. Indent along the side with the number (above number). To the right and left of the indent (which is centrally located between the parting lines), there is a hard spot where the bottle is not concentric.
2	Minor indent along the parting line to the right of the number 3. 90° from this indent there is an additional minor indent. Directly between these two indents and 45° to left of the parting line to the right of the number 3 there are squared sections which are not concentric. Labeling along defective regions is wrinkled.
3	Major indent along the sidewall 60° to the left of the parting line to the left of the number 3 on the bottom of the bottle. The indent is primarily in the upper ¾ of the bottle (top to bottom). Corresponding hard region to the left of the defect, lack concentricity.
4	Two minor indents located on the parting line to the right of the number 3 and 120° to the right of the first defect. The first defect is less pronounced than the second, which extends along the entire sidewall of the bottle.
5	Minor defect along the parting line to the right of the number 3. Corresponding hard spot 45° to the right of the defective line.
6	Major defect on the parting line to the right of the number 1. Defect along the top half of the bottle.

DEFECT DESCRIPTION – CASE #4

Bottle #	Defect Description
1	Very major indent along the parting line to the right of the number 4. Corresponding hard lines to the left and right of the parting line indent where there is lack of concentricity.
2	Very minor indent along the parting line to the right of the number 4. I would pass as acceptable.
3	Very minor indent along the bottle sidewall above the number 6. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom.
4	Very minor (questionable) indent along the parting line to the right of the number 3. I would pass as acceptable.
5	Very minor indent along the parting line to the left of the number 6. Corresponding hard spot (not concentric) 45° to the right of the indent.
6	Very minor indent along the parting line to the right of the number 2.

DEFECT DESCRIPTION – CASE #5

Bottle #	Defect Description
1	Minor indent along the parting line to the left of the number 6. Corresponding hard lines to the left and right of the parting line indent where there is lack of concentricity.
2	Major defect along the parting line to the right of the number 2. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom.
3	Major defect along the parting line to the right of the number 4. Defect extends approximately ½ of the bottle from top to bottom and gradually diminishes toward the bottom.
4	Major defect along the sidewall above the number 6. Defect extends approximately ½ of the bottle from top to bottom and gradually diminishes toward the bottom.
5	Major defect along the parting line to the right of the number 2. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom.
6	Minor indent along the parting line to the left of the number 1. Corresponding hard lines to the left and right of the parting line indent where there is lack of concentricity.

DEFECT DESCRIPTION – CASE #6

Bottle #	Defect Description
1	Major defect along the parting line to the left of the number 1. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom. No label.
2	Major defect along the parting line to the right of the number 5. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom. No label.
3	Major defect along the parting line to the right of the number 2. Defect extends along the entire parting line of the bottle from top to bottom and gradually diminishes toward the bottom. No label.
4	Major defect along the parting line to the left of the number 6. Defect extends along the entire parting line of the bottle from top to bottom and gradually diminishes toward the bottom. No label.
5	Major defect along the sidewall above the number 6. Defect extends approximately ¾ of the bottle from top to bottom and gradually diminishes toward the bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. No label.
6	Major defect along the parting line to the right of the number 6. Defect extends approximately ½ of the bottle from top to bottom and gradually diminishes toward the bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. No label.

PRODUCT INFORMATION

PRODUCT NO: 27-16812
LOT NO: 16031071
EXP DATE: 4/2008
DESCRIPTION: TIBETAN GOGI
UNIT SIZE: 32 OZ. 946 ML

DEFECT DESCRIPTION – INDIVIDUAL BOTTLES

Bottle #	Defect Description
1	Very minor indent along the parting line to the left of the number 2 (defect almost not present). LOT NO: 16305061 EXP: 01/2008
2	Major indent along the parting line to the left of where the number would normally be (no number on this bottle). Defect extends approximately ½ of the bottle from top to bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. LOT NO: 16305061 EXP: 01/2008
3	Major indent along the parting line to the left of the number 5. Defect extends approximately ½ of the bottle from top to bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. LOT NO: NA EXP: NA
4	Major indent along the parting line to the left of the number 5. Defect extends approximately ¾ of the bottle from top to bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. LOT NO: 16305061 EXP: 01/2008
5	Very minor indent along the parting line to the left of the number 5 (defect almost not present). LOT NO: NA EXP: NA
6	Major indent along the parting line to the left of the number 5. Defect extends approximately ¾ of the bottle from top to bottom. LOT NO: NA EXP: NA
7	Major indent along the parting line to the left of the number 2. Defect extends along the entire parting line of the bottle from top to bottom and gradually diminishes toward the bottom. LOT NO: NA EXP: NA

8	Minor indent along the parting line to the left of the number 6. Defect extends approximately ¾ of the bottle from top to bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. LOT NO: NA EXP: NA
9	Major indent along the parting line to the left of the number 6. Defect extends along the entire parting line of the bottle from top to bottom and gradually diminishes toward the bottom. Corresponding hard lines to the left and right of the indent where there is lack of concentricity. LOT NO: 062081 EXP: 07/2008

Appendix B. Sonic Plastics Wall Thickness Measurements

32oz Boston Round (115g)

Person who Measured: Drew Strong
Time Bottles Pulled: 5:00 p.m.

Date: 31 Oct 2006
Time of Measurements: 6:50 p.m.

	Standards	**Bottle 1**	**Bottle 2**	**Bottle 3**	**Bottle 4**	**Bottle 5**	**Bottle 6**
Top Seam	**3.655**	3.653	3.658	3.654	3.655	3.652	3.657
90° From Seam Top	**3.660**	3.66	3.66	3.662	3.664	3.658	3.662
Bottom Seam	**3.655**	3.638	3.644	3.649	3.643	3.646	3.644
90° From Seam Bottom	**3.660**	3.653	3.655	3.656	3.657	3.654	3.657
Width inside neck	**0.775**	0.92	0.904	0.916	0.916	0.93	0.918
Weight	**115g**	114	114	112	112	112	116
Check the neck if straight	**Good/Bad**	GOOD	GOOD	GOOD	GOOD	GOOD	GOOD
Thickness Seam (top)	**>0.045**	0.054	0.054	0.053	0.05	0.049	0.058
Thickness Seam (top)	**>0.045**	0.057	0.051	0.049	0.058	0.057	0.059
Thickness 90° from Seam (top)	**>0.045**	0.053	0.056	0.05	0.062	0.059	0.056
Thickness 90° from Seam (top)	**>0.045**	0.06	0.055	0.05	0.057	0.051	0.055
Thickness Seam (bottom)	**>0.045**	0.062	0.056	0.046	0.057	0.053	0.052
Thickness Seam (bottom)	**>0.045**	0.053	0.046	0.058	0.056	0.052	0.06
Thickness 90° from Seam (bottom)	**>0.045**	0.047	0.051	0.06	0.048	0.057	0.054
Thickness 90° from Seam (bottom)	**>0.045**	0.055	0.057	0.057	0.048	0.05	0.056

The standards for the wall thickness need to stay above about **0.045**. The wall thickness should also have only about **0.020** difference. (One side could potentially be much thicker, which can be tough to change).

Note: I took a while measuring these, seeing that we often have rejects with one side caving in. These measurements reflect the lowest points I could find. However, I never found an entire side with measurements below even 0.050" down the whole side. They were grouped in a small section, than the measurements increased. Bottle was quite uniform; usually approximately 0.050. Nothing ever below 0.040." I took a hot bottle off the line to feel for a soft side. I felt one on number 5. I then measured the number 5 side and at 180° of measurement (covering entire side) and never found measurement below 0.05. The soft side was the seam left of the number. I used ultrasonic gage and micrometer. All > 0.050." (Drew Strong)

Hourly Instant Production

When the bottle neck is wobbling, check the wall thickness in the neck by cutting the top of the bottle off and cutting the throat of the bottle in half 90° to the seam.

The standards for the outside of the bottle need to stay within **0.020** when comparing 'top seam' to 'bottom seam' and then comparing '90° from seam top' to '90° from seam bottom'. The numbers shown are rough estimates of what the measurements should be.

Appendix C.   Blow Molding Resin Data Sheets

ExxonMobil Chemical

PAXON™

High Density Polyethylene

AD60-007 Blow Molding Resin

Description

AD60-007 is a medium molecular weight distribution high density polyethylene homopolymer. It possesses excellent processing uniformity and produces bottles with excellent appearance and rigidity. AD60-007 offers the maximum in barrier properties available in high density polyethylene, and imparts minimum odor and taste to the packaged product.

Applications

- Liquid food containers for milk, water, and juices.

[redacted]

Resin Properties	Test Based On	Unit SI (English)	Typical Value[1]
Melt Index, 190/2.16	ASTM D 1238	g/10 min	0.73
Density	ASTM D 4883	g/cm^3 (lbs/ft^3)	0.963 (60.2)
Molded Properties[2]			
Mechanical (23°C, 50% relative humidity, unless otherwise noted)			
Tensile Strength at Yield	ASTM D 638	MPa (psi)	32 (4700)
Tensile Strength at Break	ASTM D 638	MPa (psi)	12 (1800)
Elongation at Yield	ASTM D 638	%	7.0
Elongation at Break	ASTM D 638	%	350
Tensile Modulus	ASTM D 638	MPa (psi)	2620 (380,000)
Flexural Modulus[3]	ASTM D 790	MPa (psi)	1720 (215,000)
Tensile Impact	ASTM D 1822	$joules/cm^2$ ($ft\ lbs/in^2$)	13 (60)
Impact Brittleness Temperature	ASTM D 746	°C (°F)	<-76 (<-105)
Environmental Stress Crack Resistance[4]	ASTM D 1693	hrs	10
Thermal			
Vicat Softening Temperature	ASTM D 1525	°C (°F)	127 (260)
Heat Deflection Temperature, 66 psi	ASTM D 648	°C (°F)	79 (175)
Coefficient of Linear Thermal Expansion	ASTM D 696	cm/cm/°C (in/in/°F)	$1.1x10^{-4}$ ($6x10^{-5}$)
Processing			
Typical Melt Temperature		°C (°F)	191 (375)

1. Values are typical and should not be interpreted as specifications. Values may change with future development.
2. All molded properties were measured on compression molded plaques.
3. Method 1, Procedure A (1"x3"x0.125"), Tangent calculation.
4. Condition B, 100% Igepal.
5. AD60-007 has NSF and UL recognition. Contact your ExxonMobil Chemical representative for details.

All high density polyethylene polymer grades can - in principle - be used in food contact applications in the USA (FDA). Migration or use limitations may apply. Please contact your ExxonMobil Chemical representative for more detailed information and/or actual compliance certification documents for the specific grade of interest.

FDA Status:

This resin meets all the requirements of the FDA for olefin polymers to be used as articles or components of articles for contact with food as set forth in 21 CFR 177.1520 (c) 3.1 and 3.2a.

Revised January 2006

[redacted]

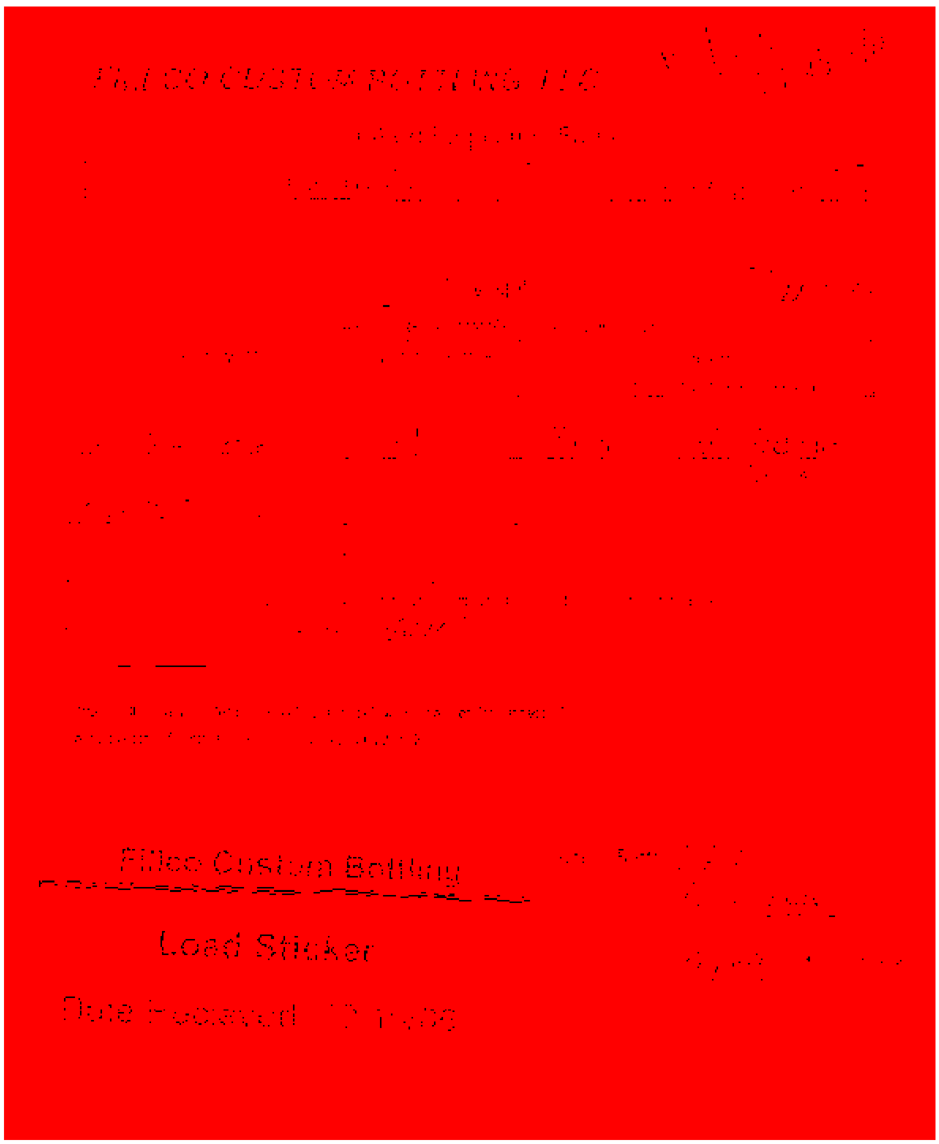
product	recommended for applications that require high top load strength and good stiffness. This meets FDA standards for food contact applications.

[redacted]

Bamberger Polymers International Corp., does not guarantee the applicability or the accuracy of the information contained herein, nor the suitability of the products described herein for any particular purpose. No warranties of any kind, either express or implied, are made with respect to the products described herein or with respect to the use of the products described herein. The user assumes all risk and liabilities in connection with such usage.

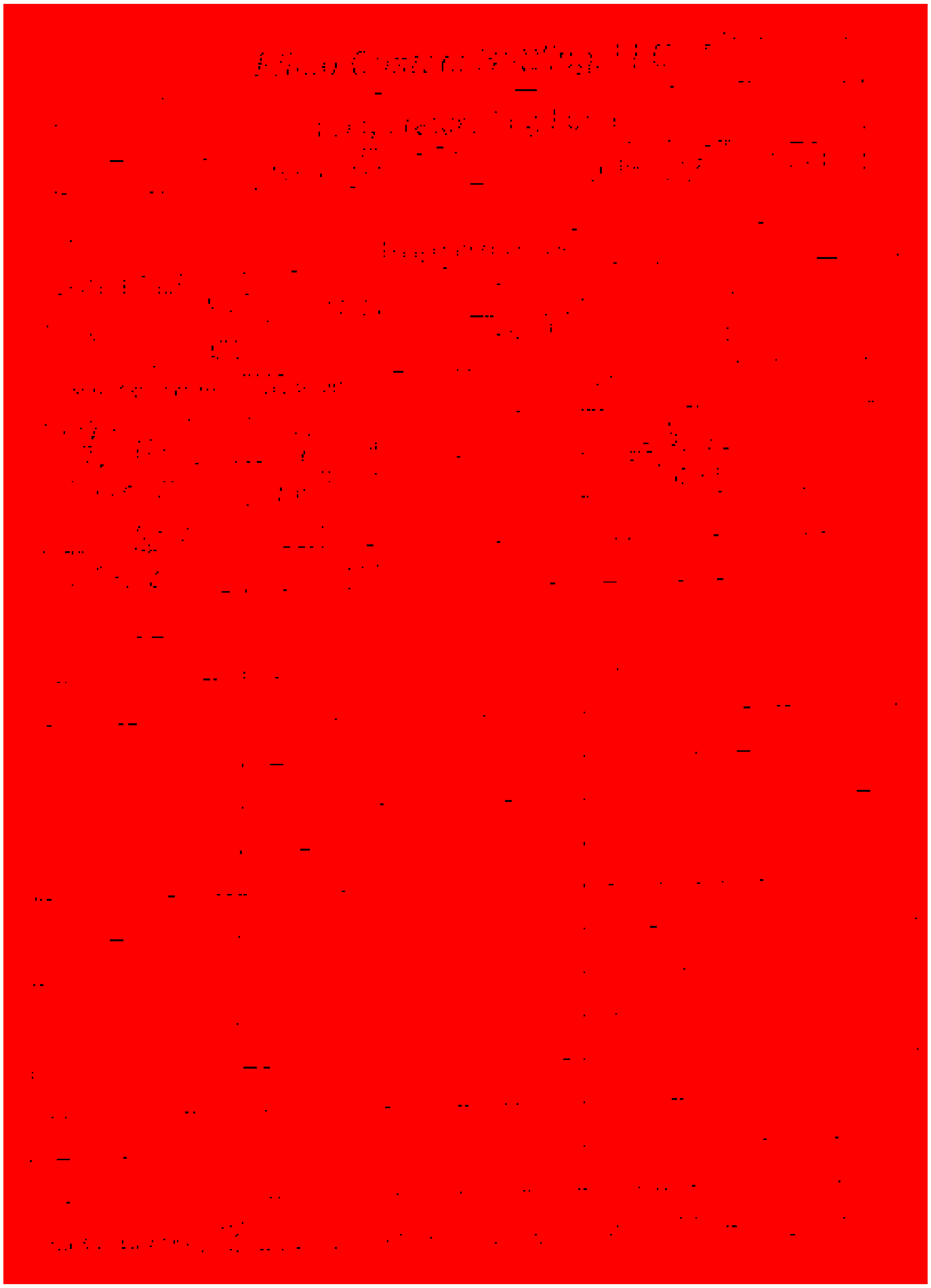
[redacted]

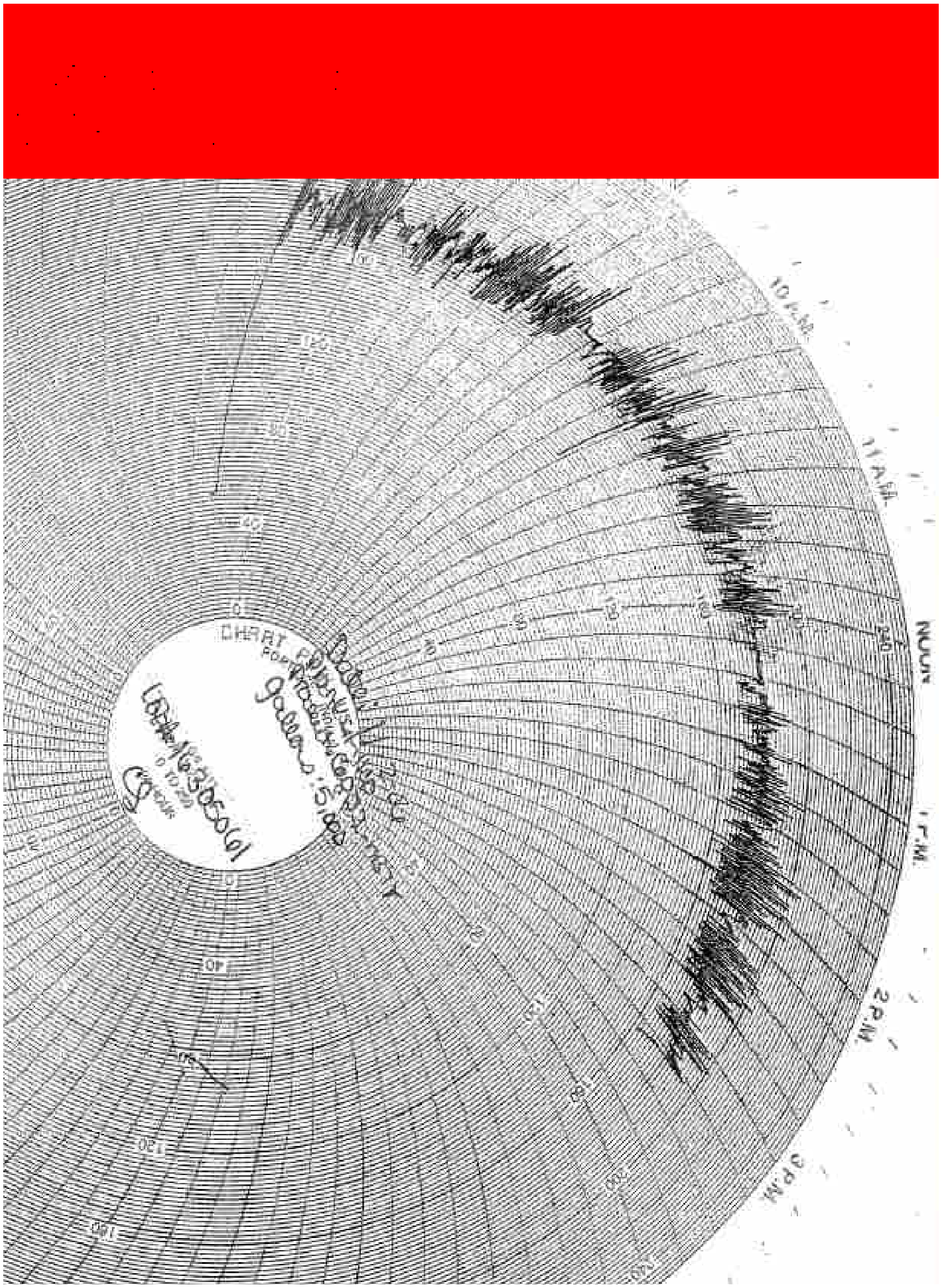
Appendix D. Fillco Custom Bottlers Temperature Logs

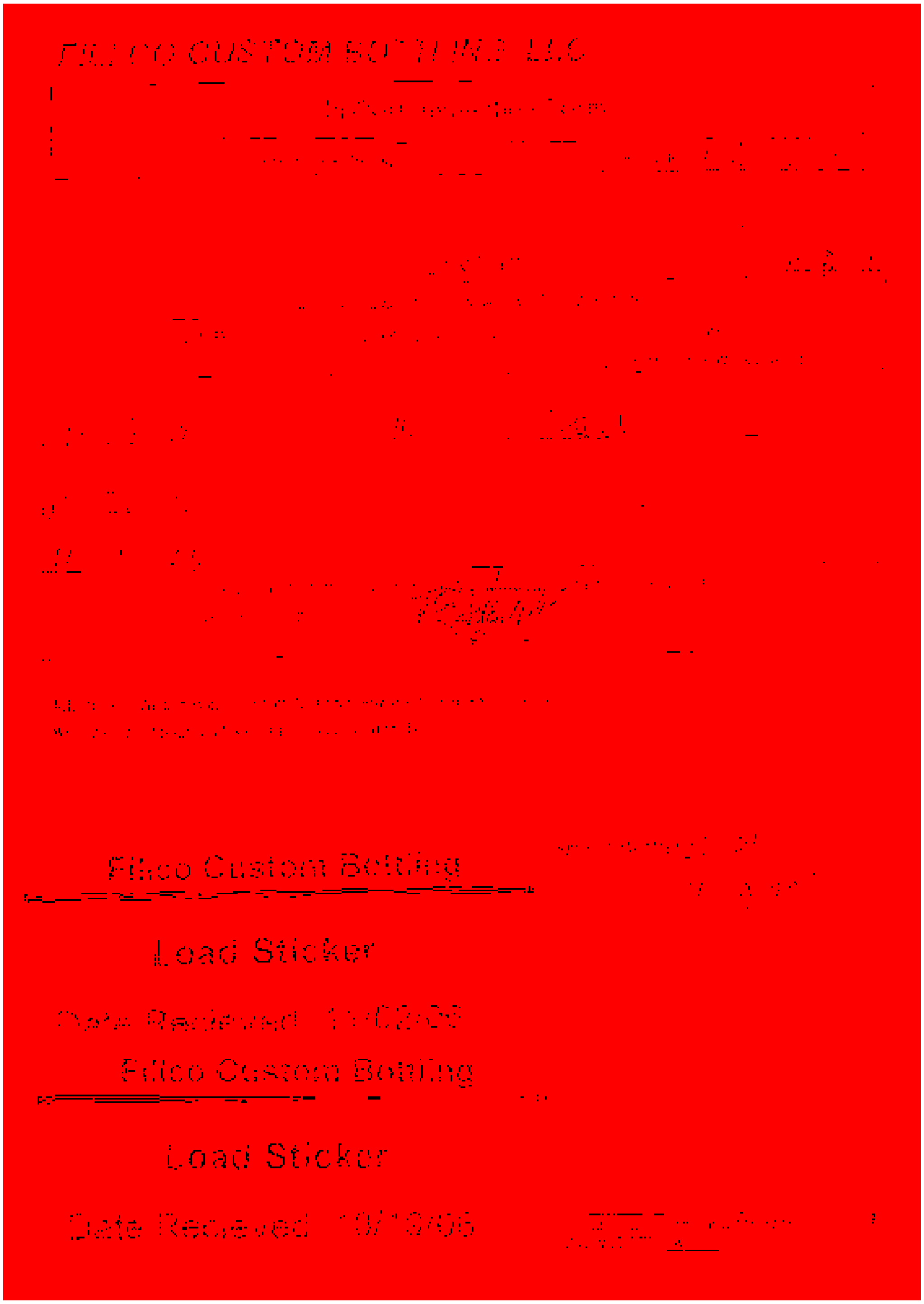

Fillco Custom Bottling

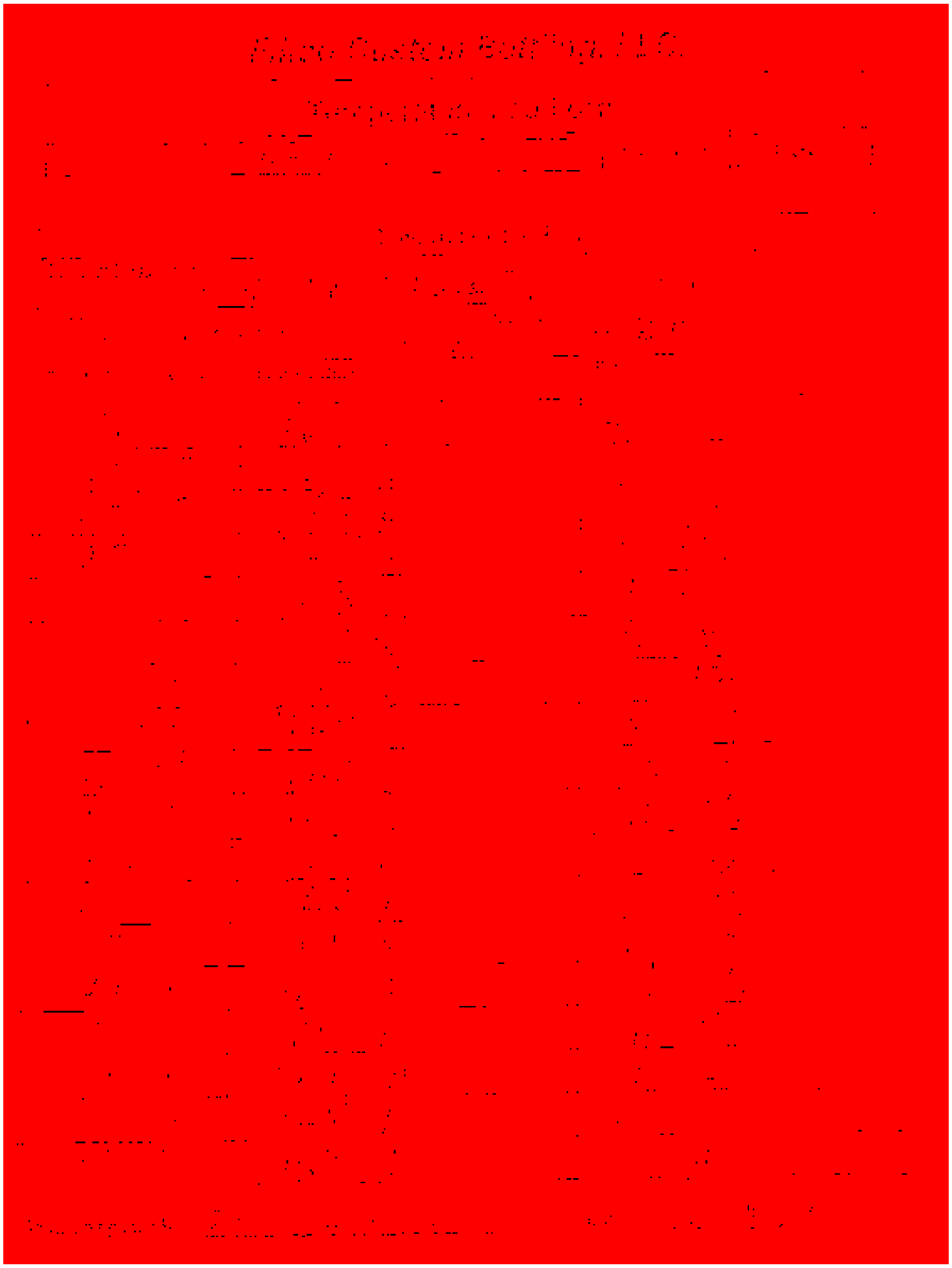
Load Sticker

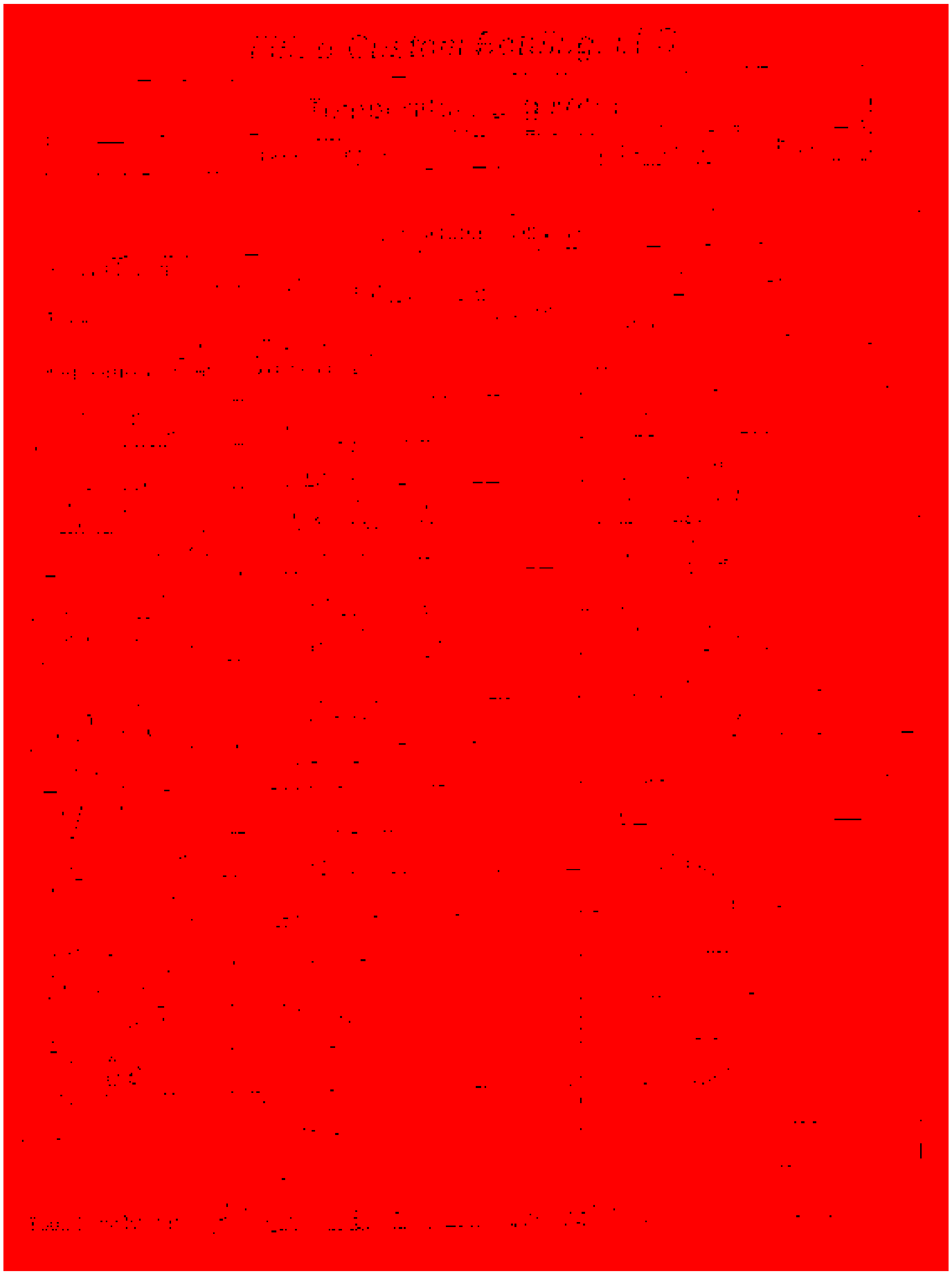
Date Received

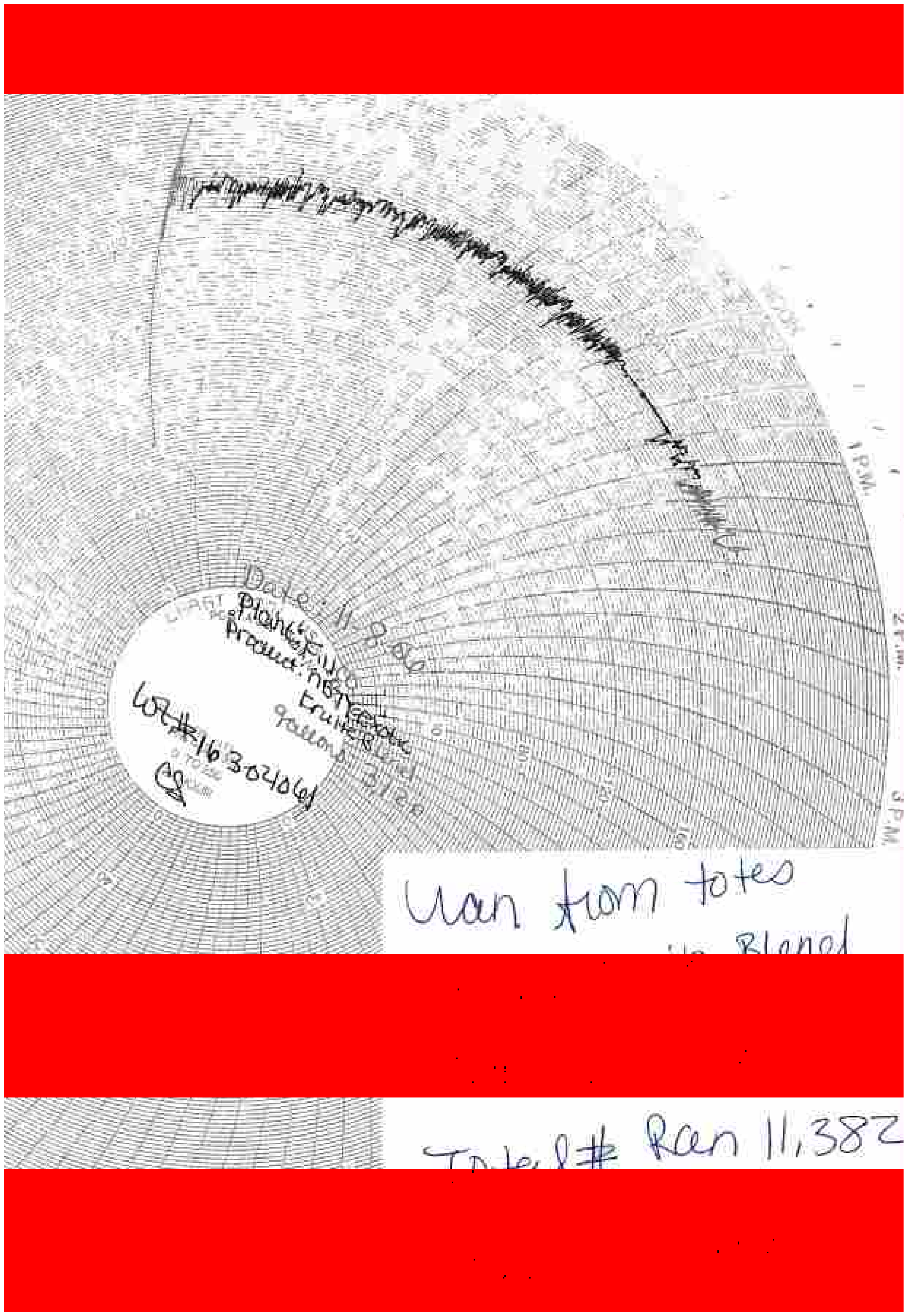



10 A.M.
11 A.M.
NOON
1 P.M.
2 P.M.
3 P.M.


Filco Custom Bottling
Load Sticker
Filco Custom Bottling
Load Sticker



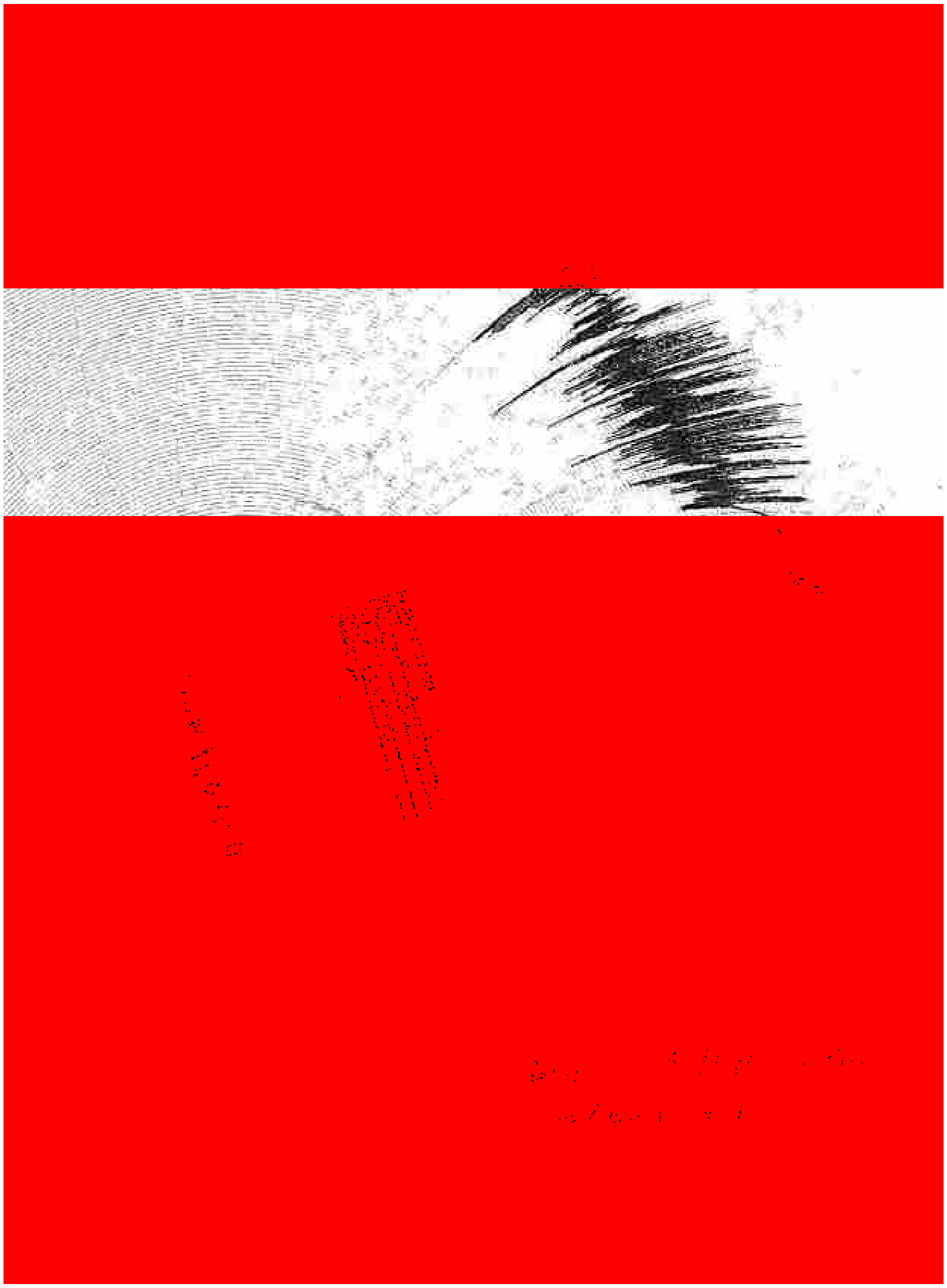



1 P.M.
2 P.M.
3 P.M.
Ran from totes
Ran 11,382

Filico Custom Bottling, LLC.

Temperature Log Form

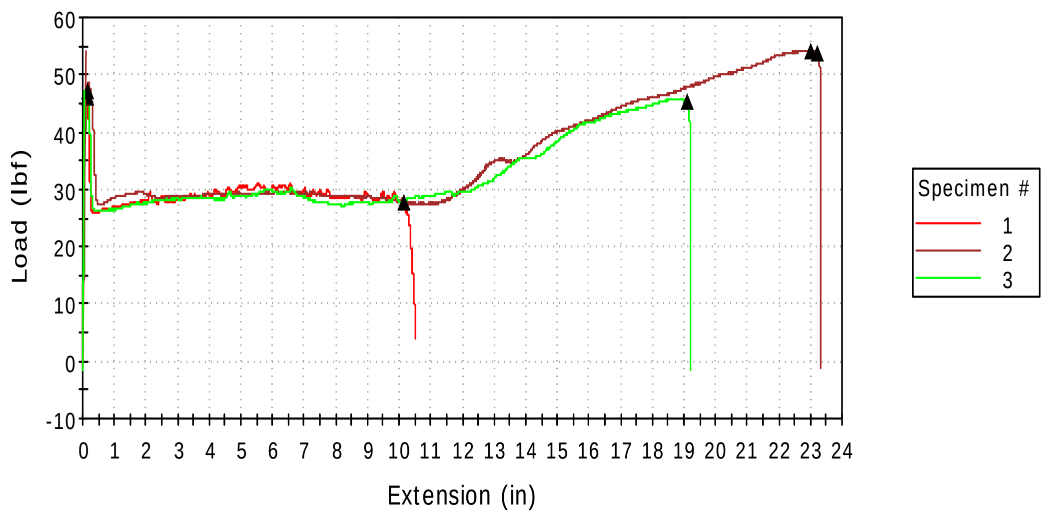

Fillco Custom Setting, LLC

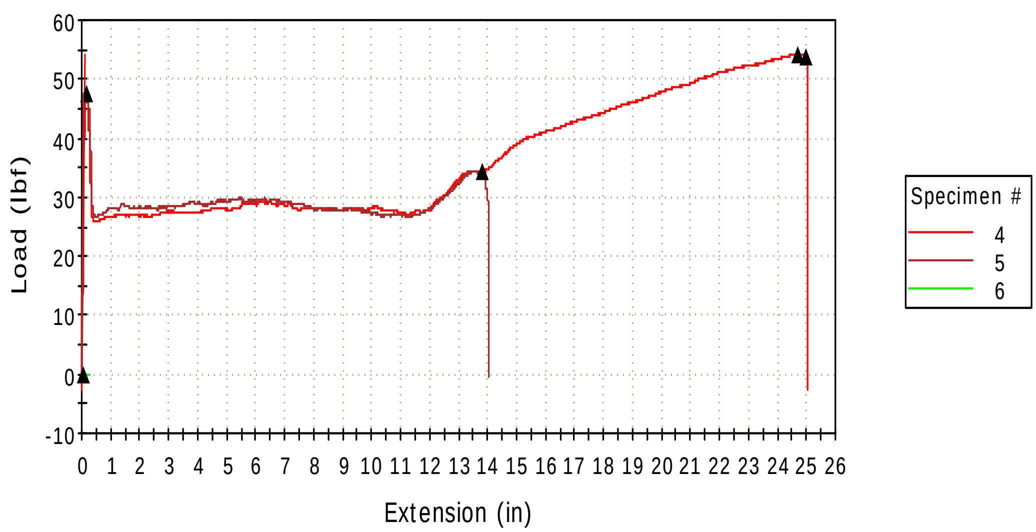


Appendix E. Tensile Testing Data

Upper Top Sidewall

Sample 1-1: Has shortened grip end on the top end of the dog bone. Samples all will be loaded with right side facing up in the grips.





Upper Top Sidewall

	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 10:27:15	UTSW 1-1	Sample necking initiation occurred at bottom end of the dog bone (if looking at it from left to right, it started on the left hand side).	1.29921
2	7/23/2007 10:44:21	UTSW 2-1	Necking began at the right end of the sample, but broke at the bottom end into the curvature approaching the gripping section. The extension may have been extra long due to loose gripping?	1.29921
3	7/23/2007 11:16:45	UTSW 3-1	Neck at the left end, break at the right end	1.29921
4	7/23/2007 11:30:30	UTSW 5-1	Neck .25 inches from left, break in the center of the sample with fraying.	1.29921
5	7/23/2007 11:52:44	UTSW 6-1	Neck at left end, break at left end	1.29921

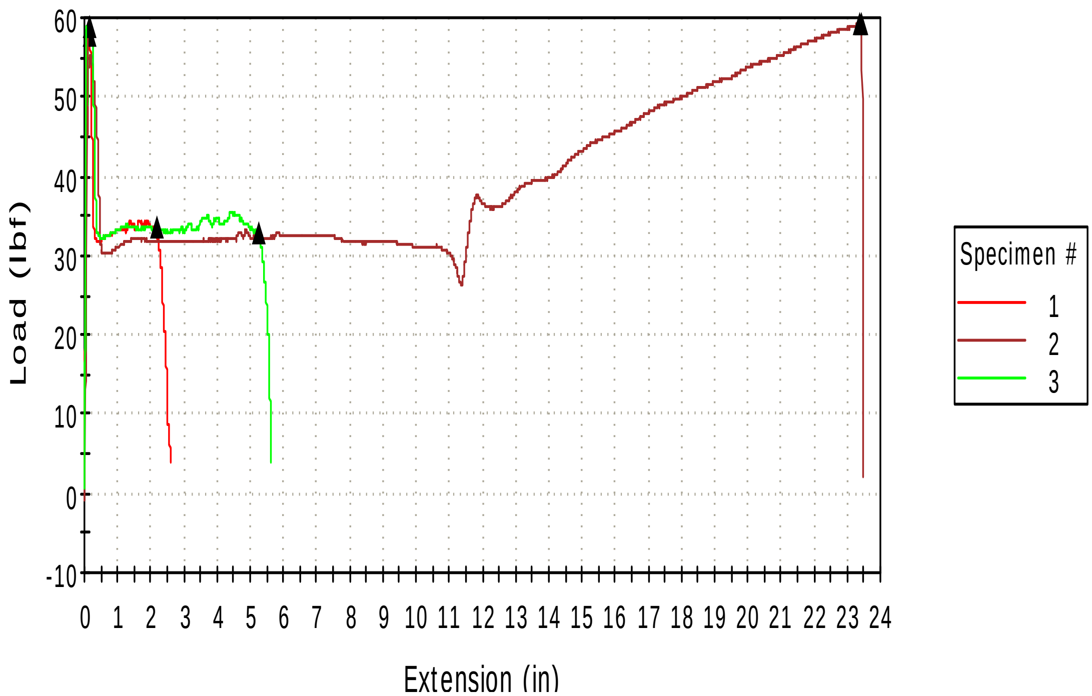
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.06400	0.21950	0.01405	63.12516
2	0.06000	0.23300	0.01398	66.44441
3	0.06300	0.21800	0.01373	66.12107
4	0.05950	0.22700	0.01351	64.45723
5	0.05950	0.22500	0.01339	66.45679

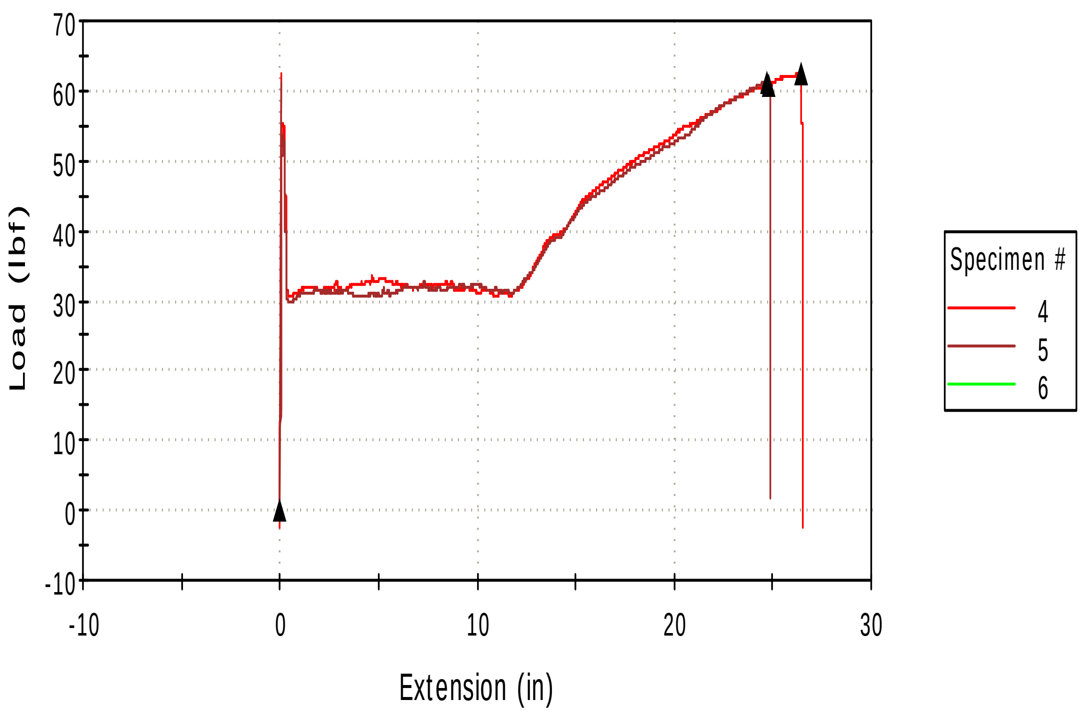
	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.29263	780.97919	46.28000	0.00304
2	3.87705	1786.09541	54.20000	0.00530
3	3.44415	-----	47.30000	-----
4	4.00701	1924.39733	54.17000	0.00432
5	3.56136	1063.06844	47.70000	0.00418

	Extension at Break (Standard) (in)
1	10.14960
2	23.21043
3	19.09270
4	25.00628
5	13.81567

Lower Top Sidewall

Sample 1-2: Has a small mill mark out of the right side, will not affect length of sample





Lower Top Sidewall

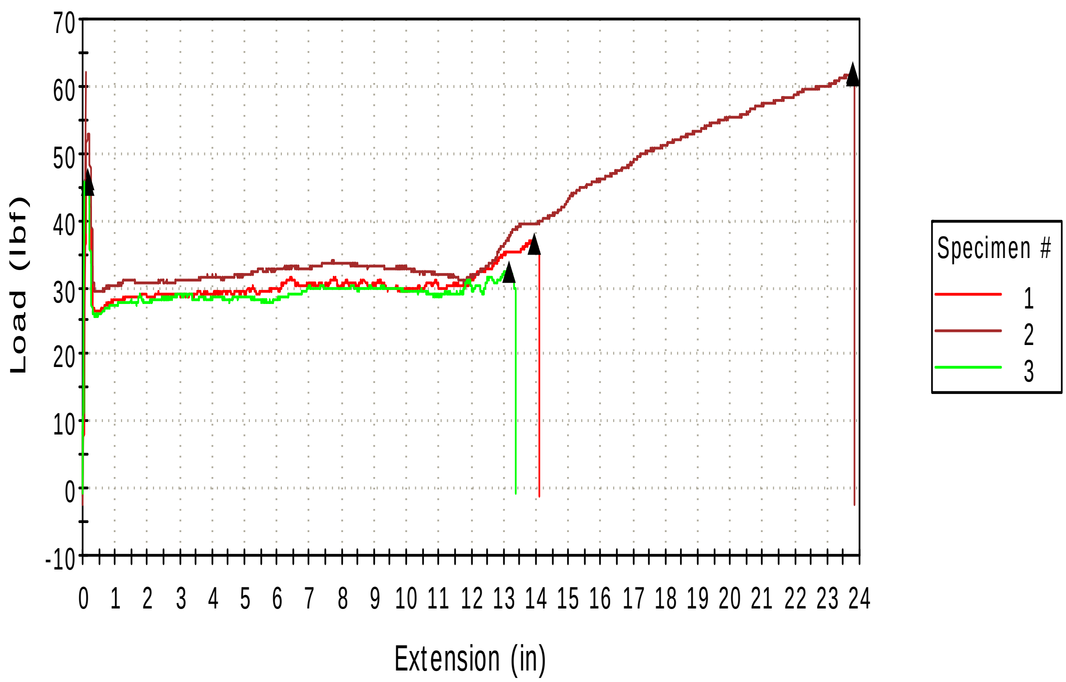
	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 12:28:57	LTSW 1-2	neck at left, break at right, 85.1° F	1.29921
2	7/23/2007 12:33:59	LTSW 2-2	neck right, break center, 86.1° F	1.29921
3	7/23/2007 12:50:54	LTSW 3-2	neck left, break right, 85.9° F	1.29921
4	7/23/2007 12:57:24	LTSW 5-2	neck left, break center, 85.6° F	1.29921
5	7/23/2007 13:17:38	LTSW 6-2	neck left, break center, 86.2°F	1.29921

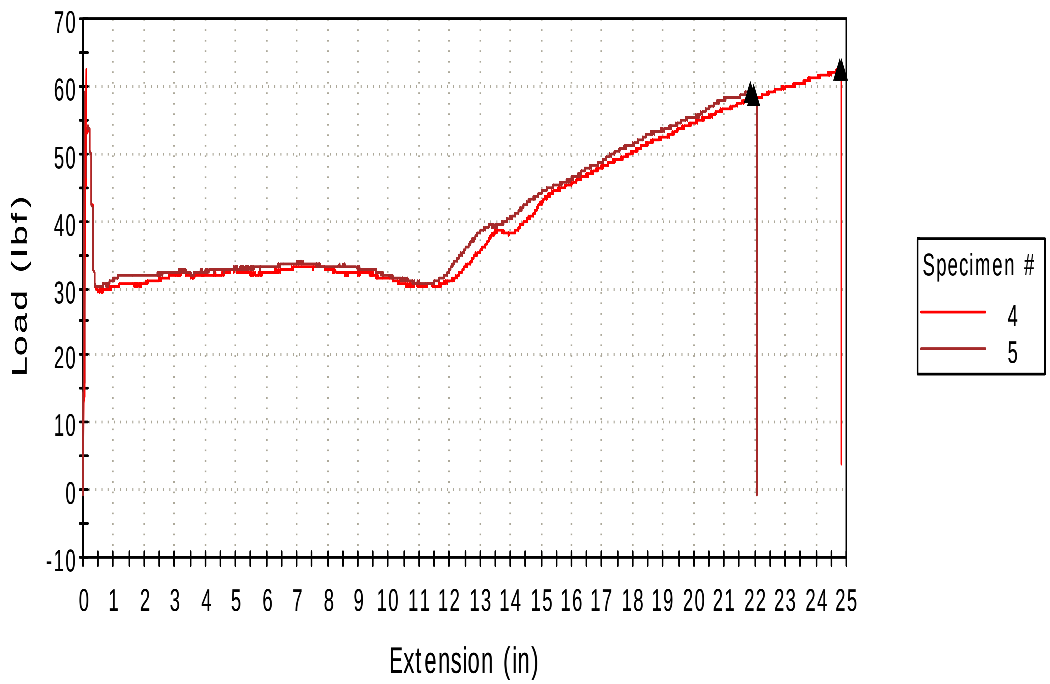
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.07700	0.22300	0.01717	62.17519
2	0.06900	0.23300	0.01608	60.37786
3	0.07550	0.21900	0.01653	65.64434
4	0.06850	0.23850	0.01634	61.05220
5	0.06850	0.22550	0.01545	64.98495

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.36449	-----	57.77000	-----
2	3.68361	1803.59948	59.28000	0.00409
3	3.56545	-----	58.95000	-----
4	3.82047	-----	62.42000	-----
5	3.97467	-----	61.40000	-----

	Extension at Break (Standard) (in)
1	2.16871
2	23.43663
3	5.27065
4	26.43190
5	24.83250

Upper Bottom Sidewall





Upper Bottom Sidewall

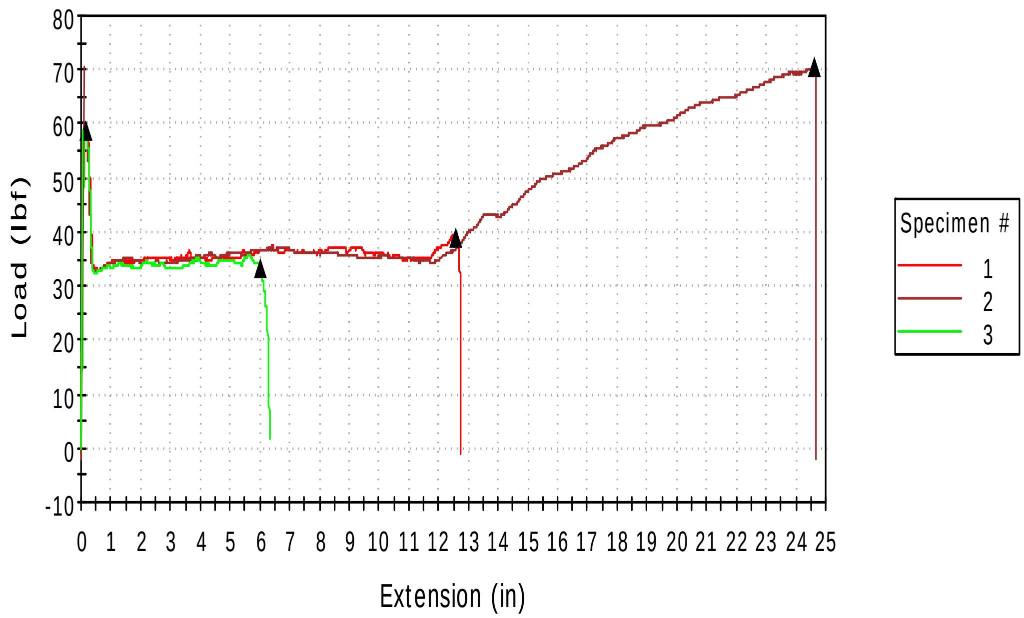
	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 18:03:15	UBSW 1-3	neck right, break right, 86.6° F	1.29921
2	7/23/2007 18:19:17	UBSW 2-3	neck right, break right, 84.5°F	1.29921
3	7/23/2007 18:34:54	UBSW 3-3	neck right, break left, 85.5° F	1.29921
4	7/23/2007 18:47:58	ULPL 5-3	neck right, break right, 84.4° F	1.29921
5	7/23/2007 19:11:05	UBSW 6-3	neck right, break right, 83.8° F	1.29921

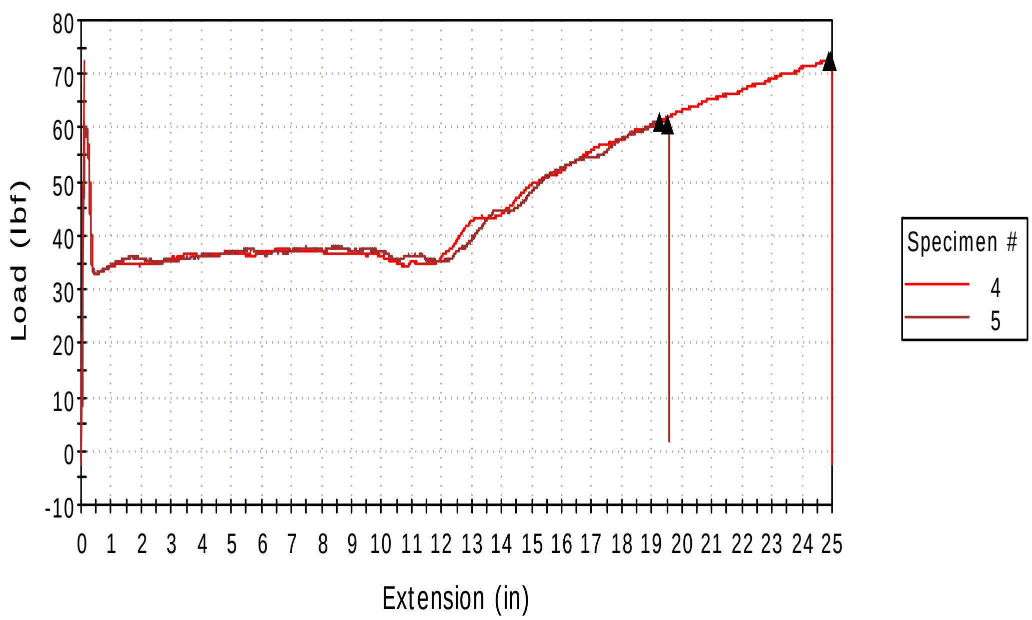
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.05800	0.22450	0.01302	68.22582
2	0.06500	0.22550	0.01466	68.14520
3	0.05650	0.22050	0.01246	71.25800
4	0.06500	0.22800	0.01482	70.33193
5	0.06550	0.22600	0.01480	70.38869

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.53790	1075.21273	46.09000	0.00695
2	4.21251	1832.70840	61.93000	0.00462
3	3.65030	-----	45.53000	-----
4	4.20436	1910.22564	62.44000	0.00333
5	3.99700	1690.94500	59.19000	0.00288

	Extension at Break (Standard) (in)
1	13.97622
2	23.81535
3	13.18927
4	24.82117
5	21.97181

Lower Bottom Sidewall





Lower Bottom Sidewall

	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 16:06:42	LBSW 1-4	Neck right, break left, 86.4° F	1.29921
2	7/23/2007 16:16:14	LBSW 2-4	Neck right; break right, 82.3° F.	1.29921
3	7/23/2007 16:32:01	LBSW 3-4	Neck right, break left, 82.2° F	1.29921
4	7/23/2007 16:40:41	LBSW 5-4	Neck right, break just left of the right end, 82.7° F	1.29921
5	7/23/2007 16:57:27	LBSW 6-4	Necked right, dramatic break at right into the curved section. 86.8° F.	1.29921

	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.07350	0.22350	0.01643	64.41803
2	0.07250	0.22500	0.01631	69.92404
3	0.07000	0.22150	0.01551	70.14901
4	0.07500	0.22700	0.01703	63.50271
5	0.07450	0.22650	0.01687	65.39578

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.58872	967.55251	58.98000	0.00414
2	4.31668	-----	70.50000	-----
3	3.77794	-----	58.58000	-----
4	4.26375	1913.73888	72.64000	0.00348
5	3.62570	1502.00942	61.32000	0.00674

	Extension at Break (Standard) (in)
1	12.57468
2	24.63559
3	6.03871
4	24.86697
5	19.52100

Upper Right Parting Line

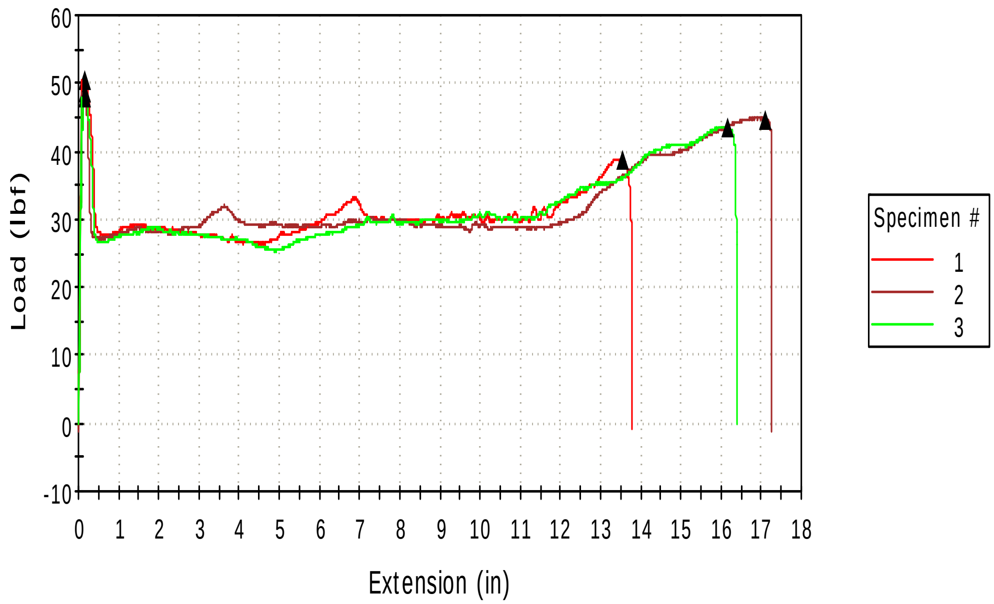
	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 15:06:45	URPL 1-5	neck 1/4" left of PL (neck on center of sample), break left end. 86.6° F.	1.29921
2	7/23/2007 15:15:49	URPL 3-5	neck 0.25" right of PL (the PL was in the center), break to the right of the center. 84.4° F.	1.29921
3	7/23/2007 15:33:16	URPL 3-5	Neck 3/8" right of PL (PL centered), break left end, 82.1 F	1.29921
4	7/23/2007 15:42:35	URPL 5-5	Neck on PL (PL centered), break right, 86.5 F	1.29921
5	7/23/2007 15:53:10	URPL 6-5	Neck on the left (1" left of PL), Break at center, 83.0° F.	1.29921

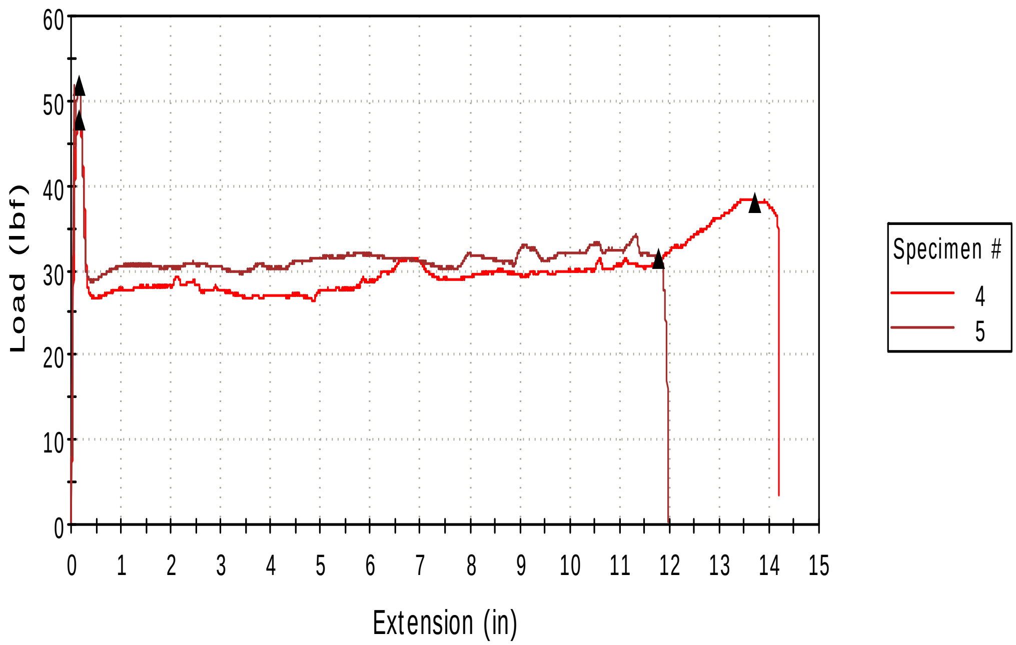
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.04700	0.22500	0.01058	78.02961
2	0.04800	0.22500	0.01080	75.86257
3	0.04550	0.22300	0.01015	80.70058
4	0.04450	0.22600	0.01006	77.94016
5	0.05550	0.22450	0.01246	73.10251

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.73680	-----	39.52000	-----
2	4.41460	-----	47.79000	-----
3	3.76232	-----	38.20000	-----
4	3.77979	-----	38.01000	-----
5	3.77914	-----	47.28000	-----

	Extension at Break (Standard) (in)
1	11.92089
2	22.05822
3	11.84227
4	13.78946
5	18.51045

Lower Right Parting Line





Lower Right Parting Line

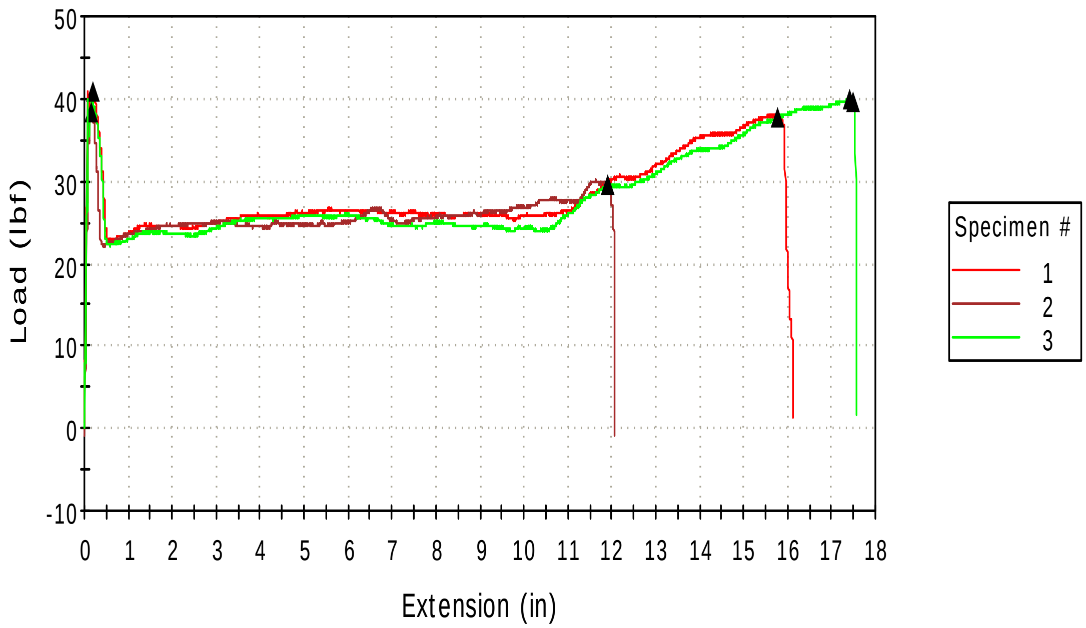
	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 21:50:28	LRPL 1-6	Neck on PL (center of sample), break left, 83.2° F	1.29921
2	7/23/2007 22:02:31	LRPL 2-6	Neck 0.25" above PL (right of the sample), break left, 84.3° F.	1.29921
3	7/23/2007 22:23:34	LRPL 3-6	Neck 0.25" above PL (right of sample), right of sample, break right, 85.7° F.	1.29921
4	7/23/2007 22:35:26	LRPL 5-6	Neck on the PL (center of sample), break right, 86° F	1.29921
5	7/23/2007 22:45:02	LRPL 6-6	neck on left end, 1" left of PL, break right, 86.4° F	1.29921

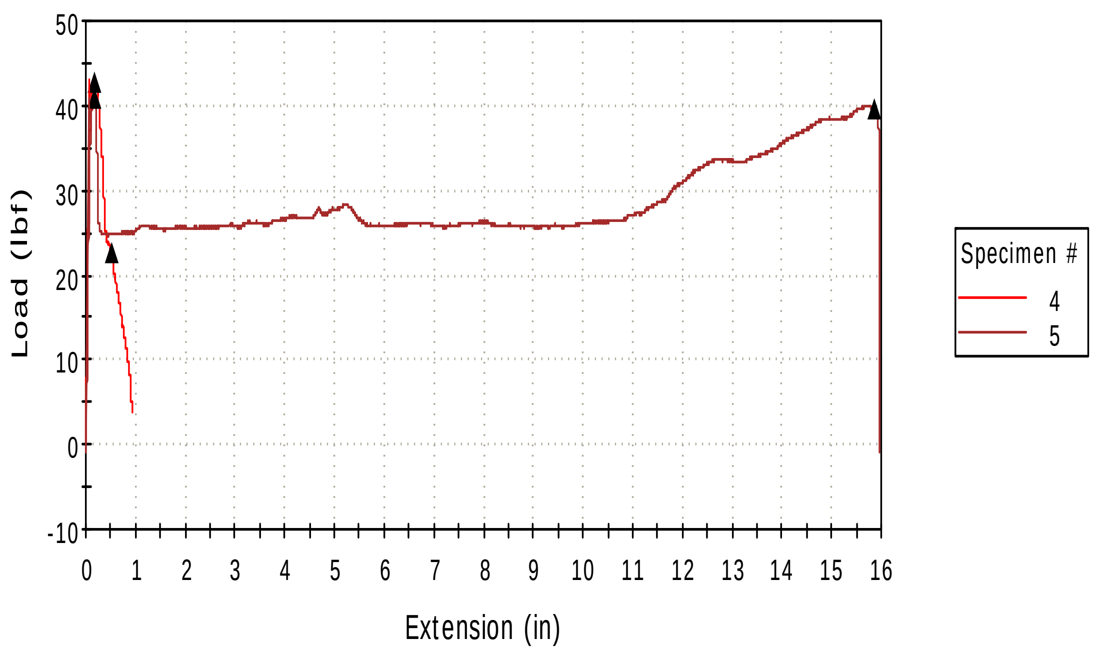
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.05800	0.22550	0.01308	76.67806
2	0.06000	0.22700	0.01362	70.21541
3	0.05750	0.22500	0.01294	70.84926
4	0.05500	0.22600	0.01243	77.50888
5	0.06100	0.22550	0.01376	75.01819

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.84857	-----	50.39000	-----
2	3.57547	1316.90102	48.70000	0.00196
3	3.69561	-----	47.81000	-----
4	3.84434	-----	47.79000	-----
5	3.77054	-----	51.95000	-----

	Extension at Break (Standard) (in)
1	13.53918
2	17.11127
3	16.16967
4	13.71886
5	11.78159

Upper Left Parting Line





Upper Left Parting Line

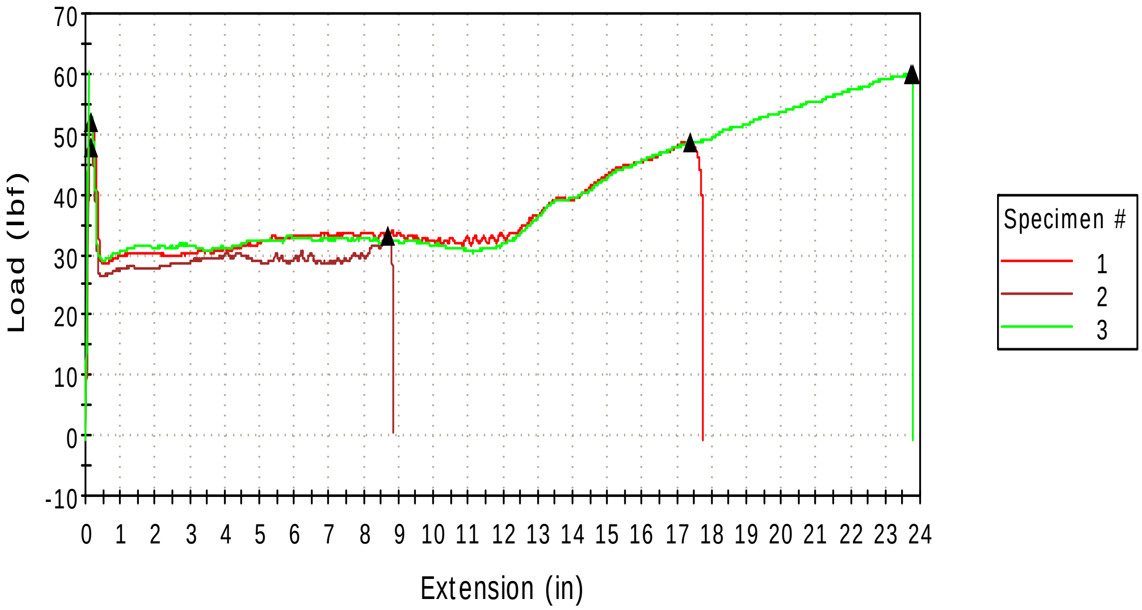
	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 17:11:49	ULPL 1-7	Neck 3/8 right of PL (necking at right end), break left end. 83.9° F.	1.29921
2	7/23/2007 17:24:12	ULPL 2-7	Neck on PL (centered, but slightly right), break dramatic on left end. 82.6° F.	1.29921
3	7/23/2007 17:33:17	ULPL 3-7	neck on right end (1/2" right of PL), break at left end. 82.7° F.	1.29921
4	7/23/2007 17:48:14	ULPL 5-7	Neck on right end (1/2" right of PL), break right, 84.8° F	1.29921
5	7/23/2007 17:50:55	ULPL 6-7	neck on PL (located 1/4" right of the center), break on left, 83.2° F	1.29921

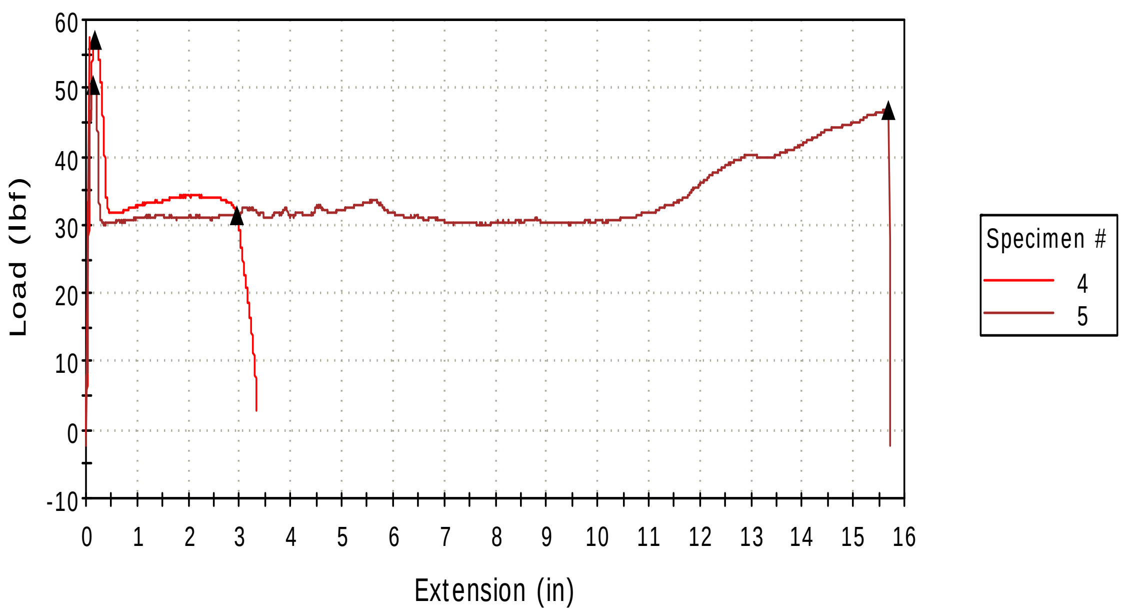
	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.04950	0.22750	0.01126	70.25308
2	0.04800	0.22750	0.01092	69.09677
3	0.04950	0.22650	0.01121	68.24703
4	0.05250	0.23150	0.01215	71.08438
5	0.05000	0.22750	0.01138	70.84811

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.64258	-----	41.05000	-----
2	3.52532	-----	38.58000	-----
3	3.55570	-----	39.87000	-----
4	3.52750	-----	42.95000	-----
5	3.58492	-----	40.83000	-----

	Extension at Break (Standard) (in)
1	15.78717
2	11.90271
3	17.47771
4	0.51520
5	15.85328

Lower Left Parting Line





Lower Left Parting Line

	Start Date	Specimen Number	Specimen note 1	Reduced Section (Gage) Length (in)
1	7/23/2007 13:38:20	LLPL 1-8	neck right (0.25" above parting line), break left, 89.3° F	1.29921
2	7/23/2007 14:00:22	LLPL 2-8	neck middle (1/16" left of parting line), break right, 86.3° F	1.29921
3	7/23/2007 14:14:54	LLPL 3-8	neck right (3/8" above PL) break center with Y-shaped fraying. 84.8° F	1.29921
4	7/23/2007 14:45:52	LLPL 5-8	Neck on PL (more to left of specimen) and break at same point, basically in the center but a little left.	1.29921
5	7/23/2007 14:50:34	LLPL 6-8	neck on PL, 1/4" right of the center. Break 2" left of right. 88.7° F	1.29921

	Thickness (in)	Width (in)	Area (in^2)	Modulus (E-modulus) (ksi)
1	0.06400	0.22850	0.01462	62.17285
2	0.05650	0.22800	0.01288	69.78851
3	0.06450	0.22850	0.01474	67.24310
4	0.06400	0.23700	0.01517	71.92922
5	0.05800	0.22850	0.01325	77.99385

	Ultimate Tensile Stress (ksi)	% Elongation	Max Load (lbf)	Extension Offset at Slack Correction (Channel Value 0 lbf) (in)
1	3.56680	1337.30986	52.19000	0.00361
2	3.71778	-----	47.92000	-----
3	4.07831	-----	60.21000	-----
4	3.77162	227.25780	57.26000	0.00547
5	3.81627	-----	50.60000	-----

	Extension at Break (Standard) (in)
1	17.37807
2	8.68101
3	23.76623
4	2.95803
5	15.70170