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DETERMINATION OF FUSION TEMPERATURE OF
POLYETHYLENE FIBER SHEETS BY MEASURING
TEMPERATURE OF THE MELTING SURFACE (MTMS)
METHOD

presented by

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has been accepted towards fulfillment
of the requirements for the

Master of
Science

degree in

Packaging

A handwritten signature in cursive script, reading "Hugh E. Lockhart".

Major Professor's Signature

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**DETERMINATION OF FUSION TEMPERATURE OF POLYETHYLENE FIBER
SHEETS BY MEASURING TEMPERATURE OF THE MELTING SURFACE
(MTMS) METHOD**

By

Sony Agrawal

A THESIS

**Submitted to
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In partial fulfillment of the requirements
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ABSTRACT

DETERMINATION OF FUSION TEMPERATURE OF POLYETHYLENE FIBER SHEETS BY MEASURING TEMPERATURE OF THE MELTING SURFACE (MTMS) METHOD

By

Sony Agrawal

MTMS stands for **M**asuring **T**emperature of the **M**elting **S**urface. It is a systematic approach to find the strongest peelable heat seal temperature for any two sealable materials. It involves analysis of the time-temperature profile, obtained by means of a thermocouple, to determine the temperature at which the strongest peelable heat seal can be obtained. This temperature is the fusion temperature, represented by an inflection point in the time-temperature profile. Previously, different flexible materials like HDPE, LDPE and OPP have been analyzed and successfully sealed using MTMS. Tyvek[®] is DuPont's trade name for material made up of pure fibers of spun bonded high density polyethylene. Prior attempts to evaluate the inflection point for Tyvek[®] were not successful, partly due to the effect of heat during the sealing process resulting in fiber destruction and ultimately decreased seal strength. This study found an inflection point for Tyvek[®] and a strong peelable heat seal for easy opening of the Tyvek[®] based packaging. In this study inflection point was evaluated using MTMS instrument. The strongest peelable heat seal was obtained near the inflection point, which is less than the melting point of the Tyvek[®] being sealed. This technique was successfully implemented to obtain the temperature required to obtain a strong peelable heat seal for Tyvek[®] 1073B.

DEDICATION

My parents Namoji Agrawal and Sangita Agrawal,

My beloved brother Lavish Agrawal

Thank you for all the support, everlasting encouragement

and

Vipin Yadav

Thank you for your constructive criticism and believing in me

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CHAPTER 1

INTRODUCTION

Measuring Temperature of the Melting Surface (MTMS) method is a systematic approach to determine the strongest possible peelable heat seal temperature for any two sealable materials. This method was introduced to the U.S by Kazuo Hishinuma, a consulting engineer from Kawasaki, Japan. The MTMS instrument can provide a high degree of precision and laboratory validation for designing heat sealable packages for blister and sterile packaging products. The strongest peelable heat seal can be obtained at a temperature which is at or slightly above the “fusion temperature” for the surface of the material being sealed. This fusion temperature is represented as the inflection point on the time temperature data profile. At the inflection point the second derivative of temperature with respect to time changes from a negative to a positive value due to change in heat flow rate as a result of melting of the film surface. This data point can be determined by means of the following methods:

- i) The MTMS method, based on the second derivative of temperature with respect to time (Hishinuma 2001).
- ii) The Table curve method, based on non-linear regression of the second derivative of temperature with respect to time (Aithani et al 2006).

This study was carried out to determine the inflection point and sealing temperature at which the strongest possible peelable heat seals can be obtained for uncoated spun bonded polyethylene fiber sheets (Tyvek® 1073B). Earlier attempts to determine the inflection point (fusion temperature) for Tyvek® 1073B

were unsuccessful. Thus, different approaches were used to obtain an inflection point for Tyvek® 1073B using MTMS instrument. Since MTMS shows a high degree of precision in measuring temperatures for use in obtaining a strong peelable heat seal for many materials, it was used for the analysis.

1.1 Background

In today's life, every product in the market exists in some form of packaging e.g. packaging in a bag, wrap or box, separately or in combinations. Packaging is the third largest industry in the United States and is booming in the rest of the world (Hines 2005). Flexible packaging is a widely used form of packaging. It includes packaging of simple grocery bags to sophisticated barrier structures for foods, pharmaceuticals, medical devices, cosmetics and beverages. The flexible packaging industry constitutes 20% of the packaging industry in the United States and utilizes approximately 29% of the total thermoplastic resins manufactured (Research studies 2003). An important aspect of good packaging is seal integrity. Seal integrity plays a very important role in packaging as it is important for protecting and maintaining the sterility of packaged product and to increase its shelf life

The shelf life and sterility of the packaged products can be maintained by ensuring excellent seal integrity, failing which can lead to leakage and deterioration of the product under environmental stress and handling. The package can be sealed by either heat sealing process or cold sealing process, and in both processes maintenance of the seal integrity is important. The process

by which two or more plastics films or sheets are fused together by external application of heat and pressure at a particular area is called heat sealing (Smillie 2006). This type of seal can be achieved by using different kinds of sealing methods and instruments (for example; bar sealing, impulse sealing, and ultrasonic sealing). By this application, peelable and non-peelable seals can be obtained for product packaging. Peelable seals are commonly utilized in flexible packaging. A peelable seal is obtained by sealing two polymer films at semi-molten stage (Meka et al 1994). Peelable seals are mostly used in packaging of food, pharmaceutical and medical device products. The temperature, pressure and sealing time (dwell time) play an important role in seal integrity of the peelable packaging. At present, industry makes use of a wide range of sealing temperatures to obtain a good seal at desired machine speed. Hence, by knowing the interface temperature and the sealing time, it's possible to obtain a good seal for the package. It is difficult to know the correct interface temperature for achieving a peelable seal because the interface temperature is usually not measured as a part of the process of designing or accomplishing heat seals.

This study is aimed at determining the correct interface temperature by finding the fusion temperature, which occurs at the inflection point in the time-temperature curve for sealing of the material, in order to obtain a strong peelable heat seal for Tyvek® 1073B grade.

1.2 Objective and Reasoning

The inflection point (fusion temperature) for some flexible materials like LDPE (low density polyethylene), HDPE (high density polyethylene), and OPP (oriented polypropylene) have been analyzed using MTMS (Aithani et al 2005). But it was difficult to obtain a strong peelable heat seal for spun bounded polyethylene fiber sheets (Tyvek® 1073B), laminated with PE/PET. Tyvek® to Tyvek® sealing is possible by heat seal process but it is difficult to obtain a strong seal because the melting of Tyvek® destroys its fiber structure and hence reduces its seal strength and flexibility in the seal area. Some uncoated grades of Tyvek® like 1073B and 1059B, having undergone no corona treatments can be used and sealed by heat sealing (DuPont 2007). This study was carried out by using two lots of an uncoated grade of Tyvek® called as Tyvek® 1073B. The two lots are referred to as lot 1 and lot 2. Lot 1 was obtained from School of Packaging Lab and lot 2 was obtained from Tolas Healthcare Packaging Company (Feasterville, PA).

The main objective of this thesis was to determine the inflection point of uncoated grade of Tyvek® 1073B and thus obtain the strongest peelable heat seal (fusion temperature) in the laboratory using MTMS and to determine whether these sealing conditions are independent of the source of Tyvek® 1073B (i.e. lot 1 or lot 2).

1.3 Hypotheses

This study is based on the following hypotheses:

- 1) Tyvek® 1073B contains areas having high and low density of fibers.
- 2) The differential distribution of fibers (high and low density areas) results in variation in the physical properties such as melting temperature.
- 3) Inflection point of Tyvek® 1073B can be determined using MTMS.
- 4) Strong peelable heat seals can be obtained when sealing Tyvek® 1073B to Tyvek® 1073B.

CHAPTER 2

LITERATURE REVIEW

Sealing is important to maintain the integrity of a package in the consumer environment, especially in pharmaceutical and food products packaging applications, because seal failure is a more common cause of product deterioration than the package itself. A highly controlled seal is the most required goal as it provides a higher production rate and less downtime together with better final package performance. Sealing is an important step in packaging of a variety of products, e.g. flexible packaging and blister packaging.

2.1 Heat Sealing

2.1.1 Introduction

Sealing of materials by application of heat is widely used in the packaging industry to join polymer films. Many studies have been done on heat sealing methods, process variables and effects on seal testing and properties (Dodin 1981, Theller 1989, Stokes 1989). Heat sealing is a process of welding surfaces of thermoplastic polymers in order to produce strong seals to withstand stresses in the distribution and consumer environment. Many different techniques (e.g. jaw-type bar sealers, rotary sealers, band rotary sealers, impulse sealers, bead sealers, hot knife or side-weld sealers) can be used for this process (Meka et al 1994, Muller et al 1998). The basic principle involved in these processes is that of forcing the two semi-molten polymer films to come into close contact hence

resulting in welding of the polymers. As described by Athani et al (2005), when the surfaces are pressed together for a sufficient period of time, the polymer chains diffuse across the interface and result in formation of connection bridges. The bridge formation depends upon the properties of the materials being sealed, such as molecular weight, material composition and thermal conductivity. Hence it's very important to select the right material for sealing.

2.1.2 Types of seals

One very important factor that decides the nature of the desired seal is the requirement of the consumer. There can be two types of basic sealing choices, weld (non-peelable) and peelable sealing. Weld seal is obtained by strongly sealing the two polymer films together, so that the material fails before the seal, such as shrink wraps (Whittemore and Ponzo 2002, Ivey 1999). Peelable seals can be obtained by using peelable films or HSC (heat seal-coated) materials and by controlling the sealing temperature. The basic principle behind peelable sealing is incompatibility between polybutylene and polyethylene resulting in weak bonding. This is referred to as polybutylene-polyolefin technology (Petrie 2000, Athani et al 2005). HSC technologies are widely used to develop peelable packages. The basic principle involves cohesive failure of adhesives whose cohesive strength is less than the bond strength between adhesive and sealed material (Tetsuya et al 2005).

2.1.3 Sealing parameters

Main sealing parameters that determine the type and quality of the seal are the interfacial temperature, sealing pressure and dwell time. A proper combination of these three parameters is very important for sealing. As described by Yuan et al (2007), dwell time is the duration of time that the laminated film is brought into contact by the heated bars during the heat-seal cycle. The greater the heat-flow rate the shorter is the dwell time required. It was earlier shown in a review by Doden (1981) that seal properties are determined by the maximum temperature achieved at the interface surface. By controlling the sealing temperature of the films in contact one can possibly achieve either a weld or peelable seal. Prior work by Meka et al (1994), described the experimental technique for rapidly measuring the changing interfacial temperatures between two films during sealing. Heat sealable films can be used for sealing over a wide range of temperatures; hence one can raise the sealing temperature to speed up the packaging process. But this increase in temperature can affect the nature of the seal; hence, to obtain the desired seal, the process must be optimized for sealing temperature and dwell time. Aithani et al (2006) showed that “easy open” packaging can be obtained by evaluating the mechanical and physical properties of the seal. In their study the inflection points on the temperature-time sealing profile were obtained and determined to be equal to the fusion temperature. They claimed that the highest peel seal strength was obtained at a temperature near the fusion point, but below the melting point. The effect of pressure has been previously studied by Theller (1989) and Meka et al (1994) and it was shown that

the pressure has limited effect on the sealing properties of the sealed films. As described by Meka et al (1994), slight pressure is helpful in bringing two microscopically uneven film surfaces into intimate contact, but higher pressure has no beneficial influence on seal properties. However, increased pressures and dwell times at temperatures above the final melting point of the polymer are detrimental to seal appearance due to material deformation in the sealing area. Hence optimization of sealing temperature and dwell time can raise production rates.

Dwell time, sealing temperature, pressure and contaminations can affect the seal strength of the film or pouch. As described by Kharas (1981), increase in dwell time leads to increase in the seal strength, if pressure and temperature are maintained constant. Similarly, increase in pressure or temperature results in increasing the seal strength, if other parameters are maintained as constants (e.g. dwell time).

Seal integrity can also be affected by contamination (Kharas 1981). The source of contamination can be the product that is placed in the pouch or the anti-static/anti-slip material applied on the film. It is difficult to quantify the effect of these contaminants but it can be overcome by simple techniques. For example, liquid contaminants, if any, in the seal area can be squeezed out to obtain a good fusion seal. Seal integrity is also affected by the machine variables, such as uneven transfer of heat from sealing bar to the film, incorrect temperature setting, uneven pressure due to wear and tear of the sealing bar rubber pads and inadequate cooling of the sealing jaw and seal interface area.

2.2 Spun Bonded Olefins

In 2001, the global production of spunbonded nonwoven fabrics reached a record 1,400,000 metric tons with an annual growth rate of between 6 and 8% (Wirtz 2002). Spunbonded fabrics are filament sheets made using an integrated process of spinning, attenuation, deposition, bonding, and winding into roll goods. The fabrics are made up to 5.2 m wide and usually not less than 3.0 m wide in order to facilitate productivity (Smorada 2004). Most spunbonded processes yield a sheet having planar–isotropic properties owing to the random laydown of the fibers. Unlike woven fabrics, spunbonded sheets are generally nondirectional and can be cut and used without concern for higher stretching in the bias direction or unraveling at the edges. The structure of traditional woven and knit fabrics permits the fibers to readily move within the fabric when in-plane shear forces are applied, resulting in a fabric that readily conforms in three dimensions. Because calender bonding of a spun web causes some of the fibers to fuse together, thus giving the sheet integrity, the structure has a relatively stiff hand or drape compared to traditional textile fabrics (Smorada 2004). A few examples of spun bonded fabrics are Typar, Tyvek[®], Accord, Bidim, Cerex etc.

2.2.1 Tyvek[®]

Tyvek[®], registered and trade named by DuPont for spun bounded fibers of pure high density polyethylene (HDPE) was introduced to the market in 1967 (Dupont 2007, Tyvek[®] Handbook 2004). It is made from high density polyethylene fibers; Tyvek[®] is an extremely versatile material, offering a balance

of physical characteristics that combine the best properties of paper, film and cloth. Tyvek® is strong, lightweight, flexible, smooth, low-linting, opaque and resistant to water, chemicals, abrasion and aging. Its unique combination of properties makes it ideal for a broad range of applications.

2.2.2 Tyvek® production

The initial step required to form Tyvek® sheets involves continuous spinning of very fine strands of interconnected fibers (average diameter~4 µm) of 100% high density polyethylene. These fibers are then distributed randomly in a moving bed to form a web-like structure after the flash spin and then they are bonded together by heating and pressure without the use of binders or fillers (DuPont 2007). Flashspun high density polyethylene fabrics have been commercial since the early 1960s. The process of producing spunbonded webs by flash spinning is a radical departure from the conventional melt spinning approach. In melt spinning, a molten polymer is typically extruded through a spin plate containing 20,000 tiny holes (Smorada 2004).

2.2.3 Types of Tyvek®

By the method of flash spinning 2 types of Tyvek® structures can be produced, soft and hard. Type 10, a “hard,” area-bonded product, is a smooth, stiff nondirectional paper-like substrate with good printability in both sheet and roll form. The hard type of Tyvek® is used for making peelable and non-peelable pouches for the medical device industry and for many other commercial

applications. Types 14 and 16 are “soft,” point-bonded products with an embossed pattern, providing a fabric-like flexible substrate with good printability and tear resistance. Like Type 10, they have high opacity, excellent whiteness and good surface stability. Sewing, gluing, and, to a limited extent, ultrasonic seaming and heat sealing may be used in fabricating these styles. Soft types are used for making protective garments such as, protective suits for workers in radiation plants (DuPont™ 2007 and Tyvek® Handbook 2004).

2.2.4 Applications of Tyvek®

Tyvek® has wide range of applications and is used in almost every sterile packaging process in the medical device industry. Examples are, top cover for trays in form-fill seal systems (MacKenzie et al 1998), packaging for powdered or granular desiccants (McKedy 1993), breathable strips (Pilaro 1973) for pouch packaging, serving as easy open features or sterilization vents (O’ Brien 1990 & Selke et al 2004). Although the most commonly known application is Tyvek®HomeWrap®, Tyvek® is also used in protective garments, envelopes, tags and labels, indoor and outdoor signs and banners, sterile medical and industrial packaging as well as bags, maps and car covers (DuPont™ 2007 and Tyvek® Handbook 2004).

2.2.5 Limitations of Tyvek®: Printing and Sealing

2.2.5.1 Printing

Most spunbonded olefin that will be printed on is corona (discharge) treated to improve ink and coating adhesion (Laughlin et al 1990). This treatment oxidizes the surface and increases the wettability of the surface to inks, coatings and adhesives. This treatment lasts more than 20 years. To reduce the buildup of static electricity during sheet and roll handling operations, some styles are also coated with an antistatic agent (Babinec et al 1990). Spunbonded olefin destined for use in the packaging of sterile medical devices is not corona treated nor antistated. Hence It is difficult to print on uncoated Tyvek® grade for medical packaging.

2.2.5.2 Sealing

Although it is possible to fuse spunbonded olefin to itself using only heat, it is difficult to obtain strong seals this way because melting the material destroys its fiber structure, reducing both flexibility and tear strength in the seal area. Non-corona-treated, non-antistated styles of spunbonded olefin are preferred for heat-sealing spunbonded olefin to itself. The molecular film of oxide and antistat on the surface of corona-treated/antistated spunbonded olefin causes a discontinuous melt to form, thus reducing the seal strength (DuPont™ 2007). However, the preferred method is to apply a coating with a melting point below that of spunbonded olefin, such as branched polyethylene. With such a coating, high seal strengths can be achieved using hot-bar or impulse technique (Theller

1989). Spunbonded olefin, like polyethylene film, cannot be dielectrically sealed by conventional methods. However, commercial proprietary processes have been developed that allow spunbonded olefin to be dielectrically sealed using conventional radio-frequency equipment. Uncoated Tyvek® to Tyvek® heat sealing is possible but it is difficult to obtain a strong seal. Another limitation is that Tyvek® is made of Polyethylene and hence its use is limited to lower temperatures (O'Brien 1990).

2.3 MTMS

The method for measuring temperature of melting surface (MTMS) instrument was developed by Kazuo Hishinuma, a consulting engineer from Kawasaki, Japan. This instrument deals with the theoretical handling of heat sealing technique. The temperature of the sealing surface is measured precisely by means of a thermocouple. Utilizing MTMS makes the determination of melting surface sealing temperature much easier and hence helps to maintain and achieve a good seal with great precision.

MTMS is used to design the process for producing peelable seals by control of the seal temperature (Hishinuma 2001). The largest problem in the measurement of the melting surface temperature is the integrity of performance of thermometry in the very small space at the interface between two materials during the sealing process. This is overcome by inserting thermocouple sensor of 10-40 μm in the welding plane. The temperature of the welding plane is measured directly, and taking this information as an element enables all analysis of the heat sealing and development of the control method. This method makes it

possible to achieve optimum sealing conditions by identification of temperature windows for peel seal and tear seal. Most of the other methods used to obtain sealing temperature are based on trial and error based analysis. MTMS gets rid of this trial and error problem (Hishinuma 2004). MTMS has been used to determine the fusion temperature for obtaining the highest peelable seal strength in flexible packaging materials like LDPE, HDPE, LLDPE etc (Athani et al 2005). The conventional methods (e.g. heat jaw system) could only control temperature of the heat block. By grasping the relationship between melting surface temperature of packaging material and surface temperature of the heat block, "MTMS" removes an element of variation in the heating control of the heat block (Hishinuma 2004).

The conventional measuring system makes use of sensors that control the temperature of the heat sealing jaw. On the other hand, MTMS instrument, senses the relationship between the melting surface temperature of the packaging material and surface temperature of the heating block, and removes the variations that were caused due to the heating block, thus resulting in a precise control of sealing temperature. MTMS can simulate the performance of commercial heat sealers, that are capable of controlling the temperature on either side of the seal, and thus MTMS can be used to design the sealing parameters for commercial users.

CHAPTER 3

METHODOLOGY

3.1 Materials

In this study, uncoated grade of Tyvek[®] 1073B was used. This grade of Tyvek[®] 1073B was obtained from two different sources referred to as lot 1 and lot 2. The list of the material and related information is given in Table 1.

Table 1. List of Material Samples

Lot #	Film	Thickness- mil	Supplier	Remarks
1	Tyvek [®] 1073B	7±3 mil	School of Packaging	Uncoated Tyvek [®] 1073B
2	Tyvek [®] 1073B	8±3 mil	Tolas Healthcare Packaging	Uncoated Tyvek [®] 1073B

Tyvek[®] is a spun bonded olefin, made of 100% pure fibers of high density polyethylene (HDPE). The thickness of the fibers generally ranges from 5-10 μm (Tyvek[®] Handbook 2004). Tyvek[®] has a swirl characteristic, as it is manufactured by spun bonding process, thus forming sheets with inconsistency in fiber distribution and thereby resulting in differences across the same sheet such as caliper and basis weight (Tyvek[®] Handbook 2004). When Tyvek[®] is viewed with the naked eye, uneven distribution of the fibers can be observed. These areas were divided into high density areas (having more accumulation of fibers) and low density areas (having low accumulation of fibers). Such distribution of fibers was random throughout the Tyvek[®] 1073B sheet. Thus, various methods were used to confirm the density differences in high and low density area samples

from either lot. Once the density differences were confirmed, the heat sealing properties of these two areas of Tyvek® were compared in order to determine if these differences play a role in Tyvek® to Tyvek® heat sealing process and in the determination of the fusion temperature.

3.2 Equipment and Methods

3.2.1 Melting Temperature (°C) of Tyvek® 1073B

A Differential Scanning Calorimeter (DSC) Q-100 (TA Instrument, New Castle, Delaware) was used for determining the melting point of the high and low density areas of Tyvek® 1073B using the ASTM standards D3418–04 (ASTM 1997). The basic principle underlying this technique is that, when the sample undergoes a physical transformation such as phase transitions, more (or less) heat will need to flow to it than the reference to maintain both at the same temperature. For example, as a solid sample melts to a liquid it will require more heat flowing to the sample to increase its temperature at the same rate as the reference. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters help determine the melting point of the sample (Brennan 1976).

When Tyvek® 1073B is viewed closely by the naked eye, differential distribution of the HDPE fibers throughout the sheet is observed. Multiple testing by various techniques was performed to confirm this observation and hence determine whether uneven distribution of HDPE fibers was the cause for prior problems in Tyvek® to Tyvek® sealing.

The first step in the analysis was to determine the difference in the melting points of the high and low fiber density regions of Tyvek® 1073B, if any. This was done by utilizing DSC (Differential Scanning Calorimeter) instrument. Samples were compared between lot 1 and lot 2. Six samples each, from high and low density areas (from both of lots 1 & 2) were prepared and placed in aluminum pans. The samples (~5 mg), were heated from room temperature to 160°C at rate of 10°C/min. The DSC melting points for the first heating cycle were analyzed using the Universal Analysis Software version 3.9 (TA Instrument, New Castle, Delaware). All the samples showed double peaks (Figure 13, Figure 14, Figure 16 and Figure 17). Since the DSC analysis displayed two peaks, it was difficult to analyze the melting point. Thus, a second heating cycle was required. The samples were cooled and analyzed as described above. In the second heating cycle, a single peak was obtained, and hence a melting point was determined for each sample. This procedure was practiced for both lots of Tyvek® 1073B samples.

3.2.2 Light Transmission

In order to confirm the visual analysis, that Tyvek® 1073B consists of areas with differential distribution of HDPE fibers, the UV/Vis Spectrometer (Perkin Elmer Lambda – 25, Perkin Elmer Instrument, Waltham, MA) was used. This instrument measures the percentage of UV light transmission through the Tyvek® 1073B samples containing the high or the low density areas. Samples from lot 1 and lot 2 were analyzed in this experiment. This spectrometer consists

of a light source, a compartment in which samples were kept, a diode – array detector, and a data computing computer. The Tyvek® 1073B samples were placed between the light source and the detector. The spectrometer measures the amount of ultraviolet and visible light transmitted through the sample. The percentage of the light, in the range of 190 nm-800 nm, transmitted by the sample was represented by T %. Six samples from each lot, 1 inch wide, containing high or low density areas were analyzed. The objective of this experiment was to determine if there was any difference in transmission of light in high and low density areas in the two lots of Tyvek® 1073B (Figure 5 ,Figure 6, Figure 8 and Figure 9) (Appendix 1, Table 3 and Table 4).

3.2.3 Brightness Values

A colorimeter (Hunter Lab, Hunter Associates laboratory, Inc. Reston, Virginia) was also used to confirm the visual analysis that Tyvek® 1073B consists of areas with differential distribution of HDPE fibers. The colorimeter was used to find the difference in reflectance between samples containing high or low density areas of Tyvek® 1073B. Reflectance values for six samples, from each lot, 1 inch wide, containing high or low density areas were analyzed. The brightness value for the samples was determined by analyzing the $L^*a^*b^*$ values, where L stands for the light reflectance, which represents whiteness of the material when the L value is close to 100 and blackness when it is close to zero. Thus, by using this analysis the differences in brightness among the high and low density areas containing samples were determined (Appendix 1, Table 6 and Table 7).

3.2.4 Optical Microscopy

An optical microscope (Olympus BHT; Olympus Optical Co. Ltd., Tokyo, Japan) was used to view the high and low density areas of the samples of Tyvek® 1073B. This technique was utilized as another way of confirming the presence of differential distribution of HDPE fibers across the Tyvek® 1073B sheet. The optical microscope magnifies an image by transmitting a beam of light through the sample with the aid of a set of lenses. The condenser lens in the microscope focuses the light on the sample and the objective lens in the microscope magnifies the beam, followed by transmission of the beam on to the projector lens so that the observer can see the image. This was utilized to observe area density differences in Tyvek® 1073B (high and low in Tyvek® 1073B). Samples having high or low density areas were observed under 20X magnification (Figure 3 and Figure 4). Samples from each defined area (one sample from high and low density areas) were observed under the optical microscope for differences in the two areas, if any.

3.2.5 MTMS Instrument

MTMS instrument (from Hishinuma Consulting Engineering Office, Tokyo, Japan), available in the School of Packaging laboratory, was used for this study. This instrument was used to find the strongest peelable heat seal temperature by indication of inflection point. This instrument consists of three components; temperature control unit, heat sealing unit (with fixed and movable jaw) and time-temperature recorder unit. The temperature control unit controls the temperature

of the upper and the lower jaws. The lower heat sealing jaw is fixed and the upper heat sealing jaw is movable by hand. The time-temperature recorder unit contains an oscilloscope, in which the data profile is obtained, and a 50 μm thermocouple (K type), that senses the heat sealing temperature between the materials at the interface. It is held firmly by the heat sealing jaws. The data from the oscilloscope is transferred to the computer for further analysis (Aithani et al 2005). MTMS instrument was used to determine the inflection point and to seal the Tyvek[®] 1073B samples for further tests.

Definition of the terms used in this thesis:

- a) Inflection Point: the point on time–temperature profile where the second derivative changes from negative to positive values. This is based on logic that there is a change in heat flow rate when the film surface starts to melt.
- b) Fusion Temperature: it is the temperature which is represented by an inflection point in time–temperature profile for sealing plastics materials.
- c) Interface Temperature: the temperature at the interface of the two film layers which are sealed together. This is identified by placing a thermocouple in the interface of the sealing surfaces of the two materials.
- d) Set Temperature: is the temperature which is set on the heat sealing jaws by using the temperature control in the machine. This temperature is 1- 2°C higher than the interface temperature.

e) Sealing Temperature: is the temperature of the heat sealing jaw surfaces. This temperature helps in effective and an acceptable joining of the two materials.

3.2.6 Determination of Inflection Point (Fusion Temperature)

Inflection point is a point on the time-temperature profile at which the second derivative of the temperature with respect to time changes from negative to a positive value for a material being sealed (Figure 1).

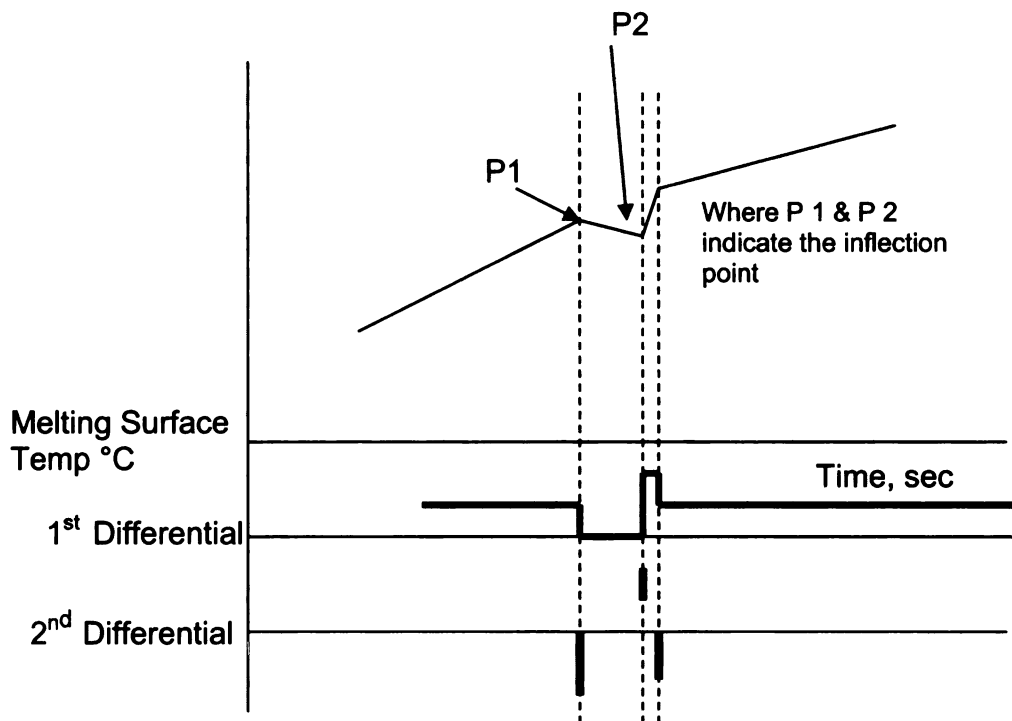


Figure 1. Determination of Inflection Point

Adopted from: Aithani et al 2005

This is due to the change in rate of heat flow during melting of the film. The time temperature profile is obtained using the MTMS instrument. The sealing temperature (refer to section 3.2.5, p 22 in this thesis), set on the MTMS

temperature control unit, is kept 1°C less than the melting point (i.e. 135°C). Thus a temperature of 134°C was chosen to seal two layers of Tyvek[®] 1073B. The interface temperature (refer to section 3.2.5, p 21 of this thesis) was recorded as a function of time by the time-temperature recorder unit by means of a thermocouple (Figure 2).

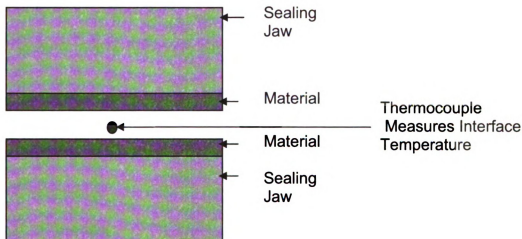


Figure 2. Location of Interface Temperature

In this study, the interface temperature was measured to be 133°C , 1°C less than set temperature (refer to section 3.2.5, p 21 of this thesis). Thus the interface temperature is verified to be $1 - 2^{\circ}\text{C}$ less than the set temperature. Six samples, each of high and low density areas (from each lot of Tyvek[®] 1073B), were sealed. The time temperature data profile was monitored by Memory 8855 Hicorder (Tokyo, Japan), and transmitted to computer for further analysis. The analysis of inflection point was done by two methods

- i) MTMS method, based on second derivative of temperature with time (Hishinuma 2001).

- ii) Table curve method based on non-linear regression. This method was helpful as an extra technique in order to confirm the MTMS analysis (Aithani et al 2006).

The analysis of inflection point by MTMS was carried out on Microsoft Excel work sheet in which the time-temperature profile data points were utilized to determine the inflection point.

2nd differential values (d2) were calculated using the following equation:

$$D2(n) = d1(n+2) - d1(n)$$

Here,

$$d1(n) = T(n+2) - T(n)$$

D1 = 1st differential

D2 = 2nd differential

d2(n) = 2nd differential for time order n,

T = temperature

n = order number in time-temperature profile data.

Table 17, Table 18, Table 19 and Table 20 in Appendix 1 (all show the array of data points and the selected temperature).

At the inflection point, the change in heat conductivity results in change in the rate of heat flow thus changing the temperature at the inflection point. The 1st and 2nd differential curve is used to identify inflection point by means of change in slope and curve during transition from a negative to a positive value, respectively. Once the 2nd differential values were obtained, the value at which the negative to positive transition takes place was considered as the inflection

point for that temperature. This was followed by plotting 2nd differential values against temperature. This shows that the 2nd differential is a better method for inflection point identification as it involves sign change hence making the calculation and determination of inflection point easier and more accurate (Figure 20, Figure 21, Figure 22 and Figure 23). Six samples of high and low density areas (each from two lots of Tyvek® 1073B) were sealed, and respective inflection points determined. The inflection points were compared among the two groups in order to understand if the inflection point is affected by differential distribution of HDPE fibers across Tyvek® 1073B sheets.

The second method used to find the inflection point of Tyvek® 1073B is the Table Curve™ Method 2D v5.0 (AISN Software, Mapleton, OR). This method was used to confirm the MTMS analysis. In this method, the second derivative function was plotted with respect to time. The inflection point was identified by noting the temperature that corresponds to time when the curve crosses the zero line at the x-axis (negative to positive transition) (Figure 24, Figure 25, Figure 26 and Figure 27) (Hishinuma 2001) (Aithani et al 2005).

3.2.7 Heat Sealing

Prior to the sealing process, samples of Tyvek® 1073B (from both the lots) were cut in the form of 1 inch wide strips and conditioned for 48hrs at 23°C/ 50%RH. Sealing temperature (refer to section 3.2.5, p 22 of this thesis) was set on MTMS instrument using temperature control unit. The temperature at the interface was verified by placing a thermocouple in between the sealing surfaces

of the material; this temperature was monitored on the Memory Hicorder. Sealing of samples was carried out at eight different set temperatures 125°C, 129°C, 132°C, 134°C, 136.5°C, 139°C, 141°C and 145°C. These set temperatures resulted in temperature at the interface of 124°C, 128°C, 131°C, 133°C, 135.5°C, 138°C, 140°C and 144°C respectively.

3.2.8 Seal Strength

The unsupported test procedure of ASTM F88–00: Standard method of testing for seal strength of flexible barrier materials (ASTM 2000) was followed for the testing of peel strength. Six samples of Tyvek® 1073B (from both the lots) were cut in the form of 1 inch wide strips and conditioned for 48hrs at 23°C/ 50%RH. These samples were then sealed at eight different sealing temperatures close to the inflection point and tested for peel strength by using a universal testing machine (model # 5565, Instron Corporation, Canton, MA). The Instron utilizes a crosshead–movement based mechanism with grips used to hold the samples (Mulet 2004). The grips were separated by 10 mm (0.4 in) and the crosshead travelled at a speed of 10in/min (25.4cm/ min). For each sample, the peel strength, shrinkage, extension and type of seal failure were noted at the breakage point (Figure 29 and Figure 30).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Hypothesis: Tyvek® 1073B contains areas that have regions of high and low density of fibers.

When Tyvek® 1073B is viewed with the naked eye, uneven distribution of the fibers can be observed. These areas were divided into high density areas (having more accumulation of fibers) and low density areas (having low accumulation of fibers). This distribution of fibers was random throughout the Tyvek® 1073B sheet. Thus, various techniques involving microscopy and spectroscopy were used to confirm the density differences in high and low density areas from either lot.

4.1.1 Optical Microscopy

The visually classified high and low density areas were located, isolated and analyzed using the optical microscope. When Tyvek® 1073B samples viewed through optical microscope, did show presence of high and low density regions in the respective high and low density samples. But, this difference was not prominent enough to confirm the existence of differential distribution of fiber distribution (Figures 3 and Figure 4).

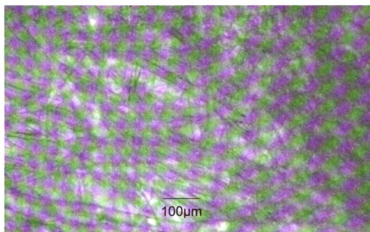


Figure 3. 20x Magnification View of Low Density Area for Tyvek® 1073B

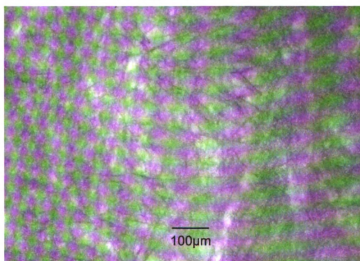


Figure 4. 20x Magnification View of High Density Area of Tyvek® 1073B

4.1.2 UV/Vis Spectrometer

UV/Vis Spectrometer was used to test the differential distribution of fibers in the Tyvek® 1073B samples, because the optical microscope failed to confirm the differential distribution. UV/Vis spectroscopy was utilized to determine if there was any significant difference in the light transmission for high and low density regions in two different lots of Tyvek® 1073B. A large difference in light

transmission was found between the high and low density regions for both lots of Tyvek® 1073B samples (Figure 5, Figure 6, Figure 8 and Figure 9).

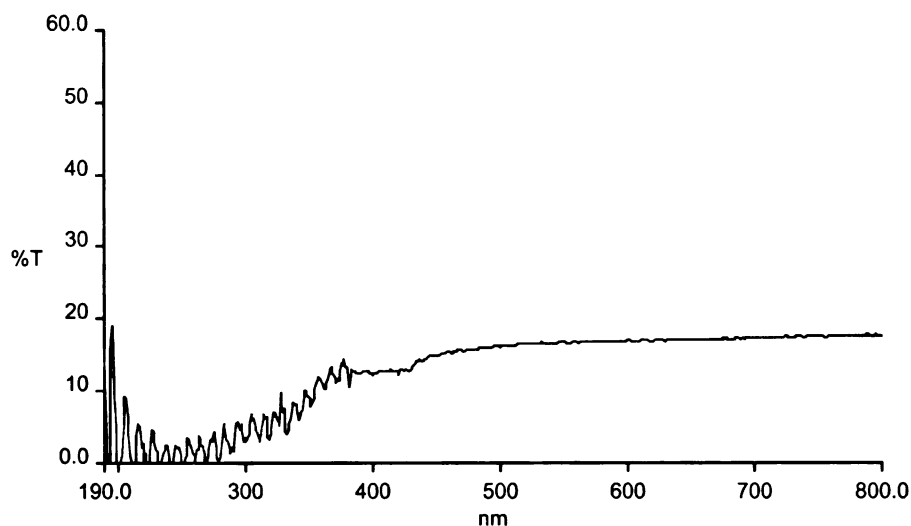


Figure 5. Percentage of Light Transmission by UV/Vis Spectrometer in High Density Area of Lot 1 Tyvek® 1073B

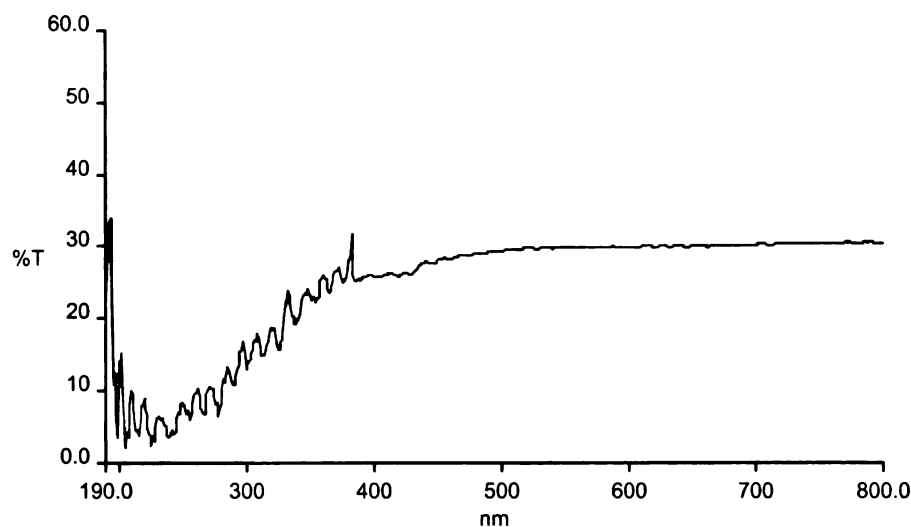


Figure 6. Percentage of Light Transmission by UV/Vis Spectrometer in Low Density Area of Lot 1 Tyvek® 1073B

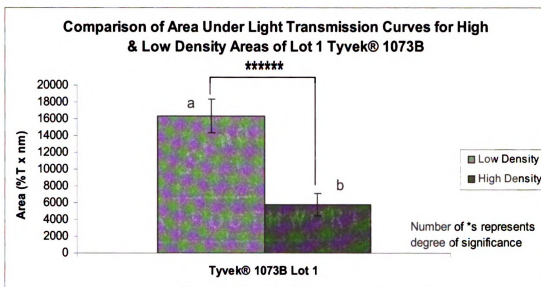


Figure 7. Comparison of Area Under Light Transmission Curves for High & Low Density Areas of Lot 1 Tyvek® 1073B

Note: Bars labelled by different letters are statistically different at $p \leq 0.05$

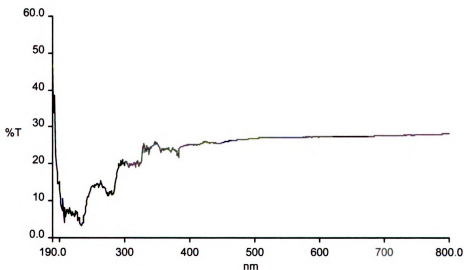


Figure 8. Percentage of Light Transmission by UV/Vis Spectrometer in Low Density Area of Lot 2 Tyvek® 1073B

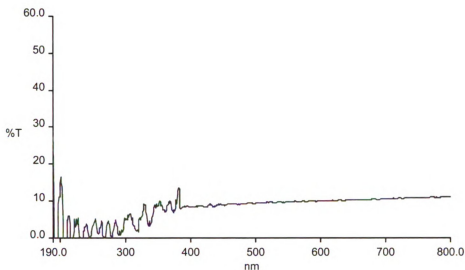


Figure 9. Percentage of Light Transmission by UV/Vis Spectrometer in High Density Area of Lot 2 Tyvek® 1073B

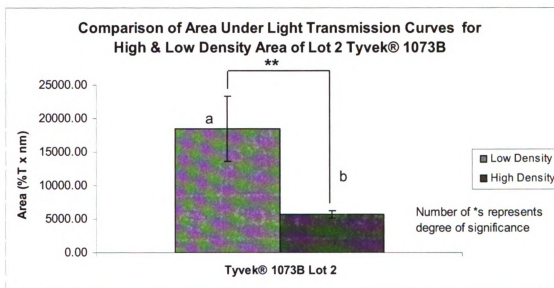


Figure 10. Comparison of Area Under Light Transmission Curves for High & Low Density Areas of Lot 2 Tyvek® 1073B

Note: Bars labelled by different letters are statistically different at $p \leq 0.05$

The areas under the curves were calculated for all the Figures (Figure 5, Figure 6, Figure 8 and Figure 9). These areas were used for statistical analysis. The student's t-test was conducted on the calculated areas. A significant difference in the percentage of light transmission between high and low density areas for both lots of Tyvek® 1073B was found (Figure 7 & Figure 10). A similar statistical analysis was carried out to compare the distribution of fiber density among samples from lots 1 & 2. This analysis showed that lots 1 and 2 did not differ in light transmission. Similar results were obtained for light transmission between lots 1 and 2 low densities of Tyvek® 1073B (Appendix 1, Table 3 & Table 4). Because the lots 1 and 2 did not differ in light transmission, high density samples from both the lots were combined as one set and similarly low density samples from both lots were merged for further analysis. The student's t-test analysis was carried out for light transmission values for the merged high density and low density Tyvek® 1073B samples. This analysis showed that there is a statistically significant difference between the high and low density regions of the Tyvek® 1073B (Appendix 1 Table 5, Figure 11).

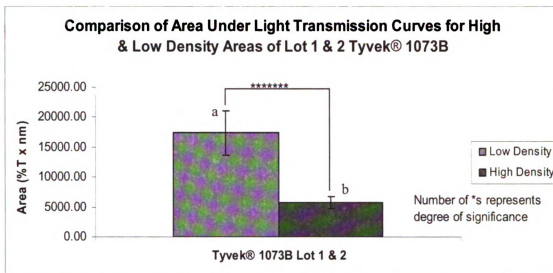


Figure 11. Comparison of Area Under Light Transmission Curves for High & Low Density Area of Lot 1 & 2

Note: Bars labelled by different letters are statistically different at $p \leq 0.05$

This analysis correlated with the visual inspection. Hence, this analysis confirmed that there is a fiber density difference in the high and low density areas samples from either lot.

4.1.3 Colorimeter

Colorimetric analysis was utilized to determine if there was any real significant difference in color of fiber density among high and low density regions containing Tyvek® 1073B samples from either lots, and if this analysis confirms the earlier analysis by light spectroscopy and visual inspection.

Colorimetric data is noted as the $L^*a^*b^*$ values (corresponds to CYELAB systems). For this analysis, only L values were considered. L values describe the amount of lightness and darkness. Student's t-test analysis was carried out to analyze the L values of the high and low density regions from both the lots. There

was significant difference in brightness values for high and low density areas containing Tyvek® 1073B samples. The brightness values for high density area containing samples were higher than samples with low density of fiber, hence signifying that high density area samples are brighter than low density area samples (Appendix 1 Table 6, Table 7 & Table 8). The differences in brightness between high and low density areas were statistically significant. More brightness signifies more density. Hence the colorimetric analysis confirms the previous results using UV/Vis spectrometer and visual inspection.

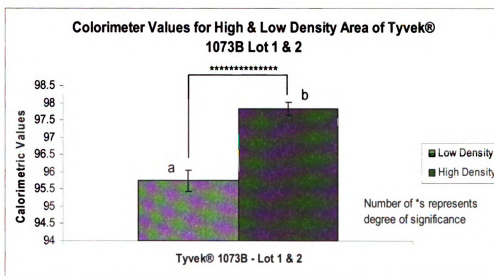


Figure 12. Colorimetric Values for High & Low Density Area of Tyvek® 1073B Lot 1 & 2

Note: Bars labelled by different letters are statistically different at $p \leq 0.05$

Thus, the first hypothesis was proved to be true that two regions (high and low density regions) in Tyvek® 1073B do exist, as shown by visual, reflectance values and %transmission values Table 2.

Table 2. Results of the Test Performed for High and Low Density Regions of Tyvek® 1073B

Type of Test	Results	Property
% Transmission	Significantly Different	Sheet Property
Reflectance	Significantly Different	Sheet Property
Optical Microscope	Not Prominent	Sheet Property
Visual	Significantly Different	Sheet Property

The Table 2 results are the combination summary for lots 1 and 2 results.

4.2 Hypothesis: The differential distribution of fibers (high and low density areas) results in variation of the melting temperature.

4.2.1 Melting Temperature of Tyvek® 1073B

The melting points of Tyvek® 1073B samples (n = 6 for each density for each lot) were analyzed using DSC (Differential Scanning Calorimeter). Samples with high and low density of fibers were analyzed for difference in melting temperature, if any. The melting point for high and low density areas of Tyvek® 1073B are shown in Figure 13, Figure 14, Figure 16 and Figure 17 (the figures are all 1 example for each lot of density). These were calculated in accordance with the procedures as described by ASTM D3418-97. In the above mentioned figures two peaks were obtained, whereas a single peak is needed to calculate the melting point of the sample. Thus in order to obtain a single peak for the melting point, the samples (six of high and low density areas, each from lot 1 & 2) were reheated to obtain the 2nd cycle DSC curve (Figure 15 and Figure 18). In

the 2nd cycle, DSC samples (both the high and low density areas from lot 1 & 2) showed approximately similar melting peak, thereby signifying similar melting points.

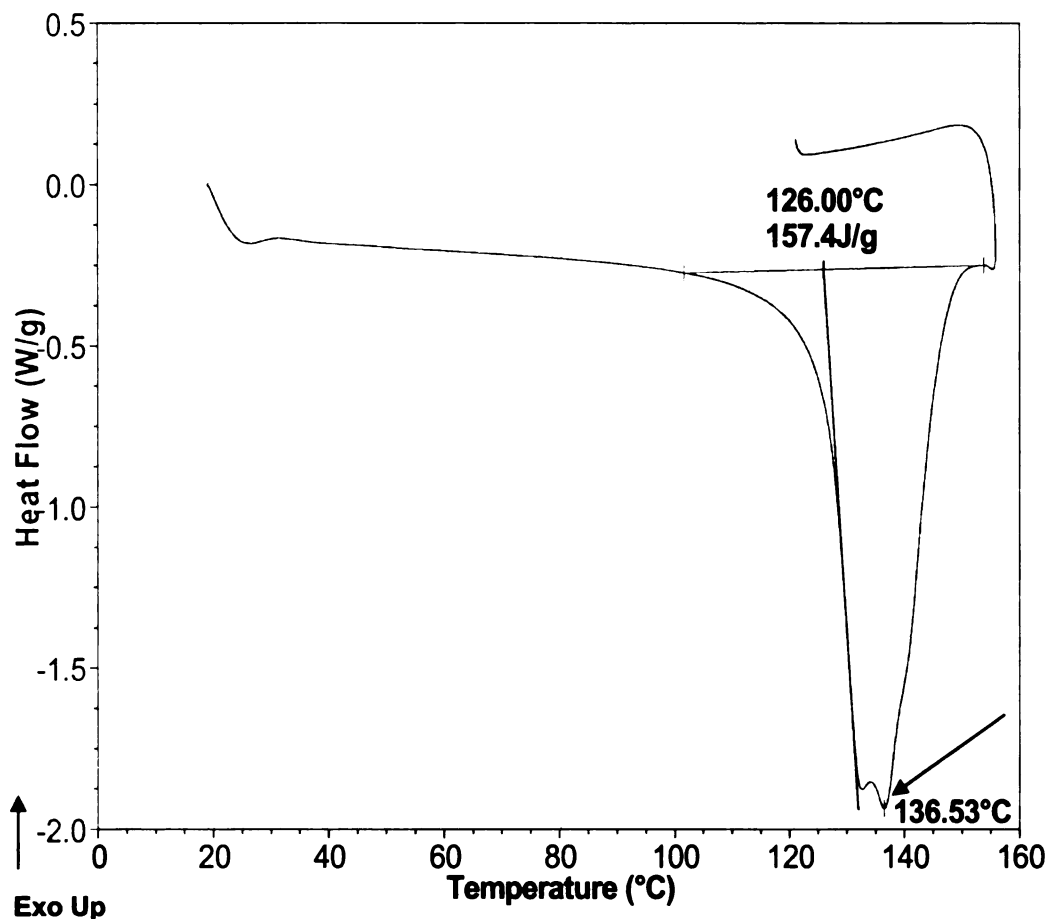


Figure 13. Melting Point for High Density Area of Lot 1Tyvek® 1073B - First Cycle Showing Two Melting Point Peaks

The melting peak in the first heating cycle for Tyvek® 1073B lot 1 is 136.53°C

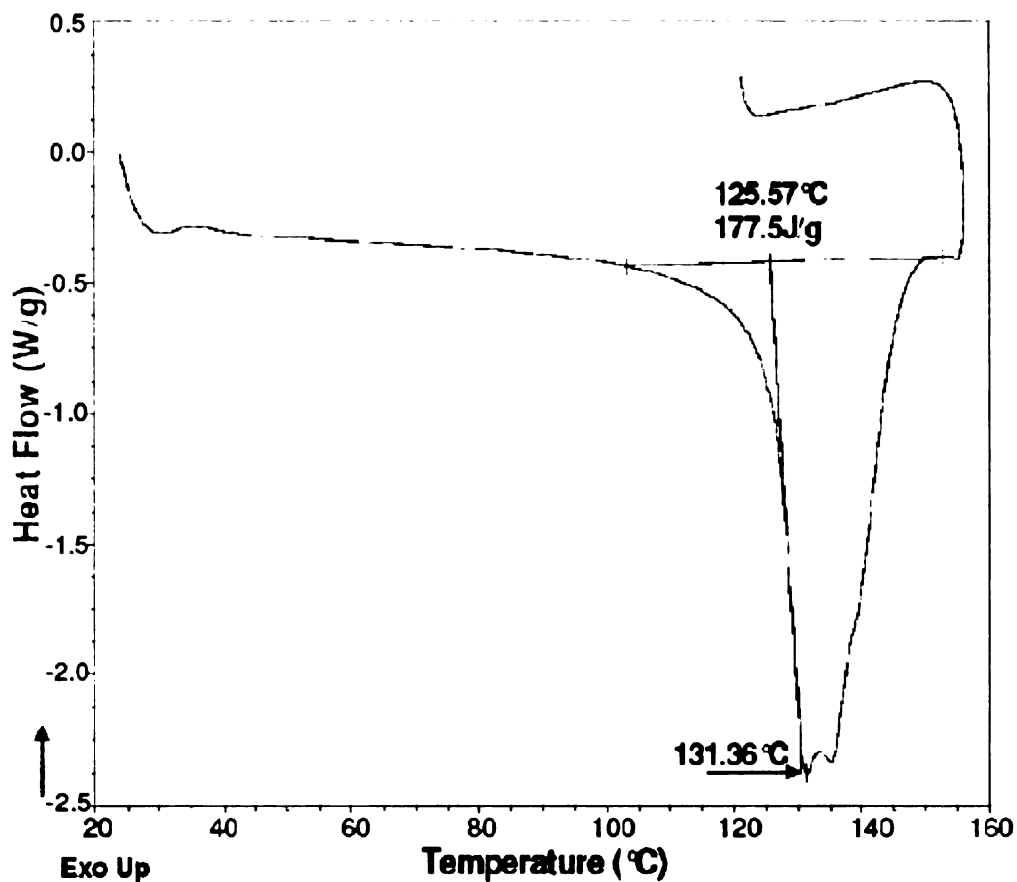


Figure 14. Melting Point for Low Density Area of Lot 1 Tyvek® 1073B - First Cycle Showing Two Melting Point Peaks

The melting peak in the first heating cycle for Tyvek® 1073B lot 1 is 131.36°C

Note: The reverse in the peaks is noticed because of the difference in heating of the high and low density areas of Tyvek® 1073B

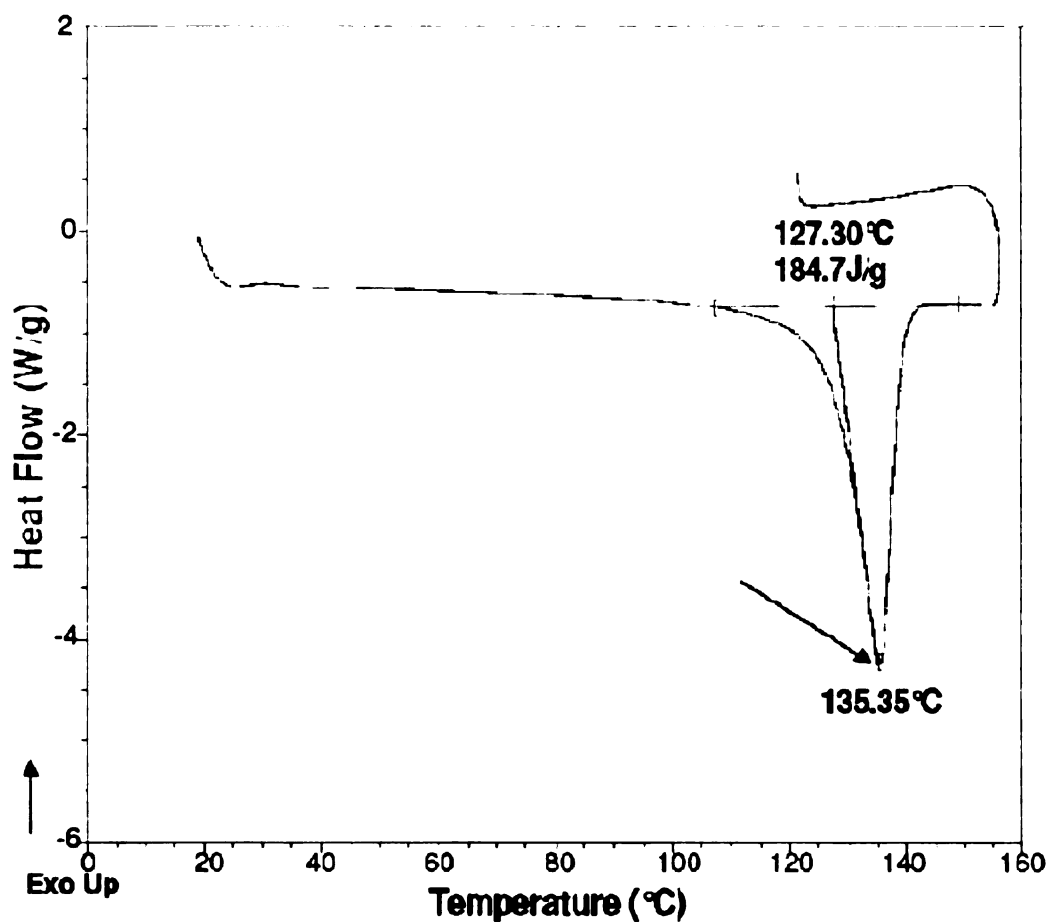
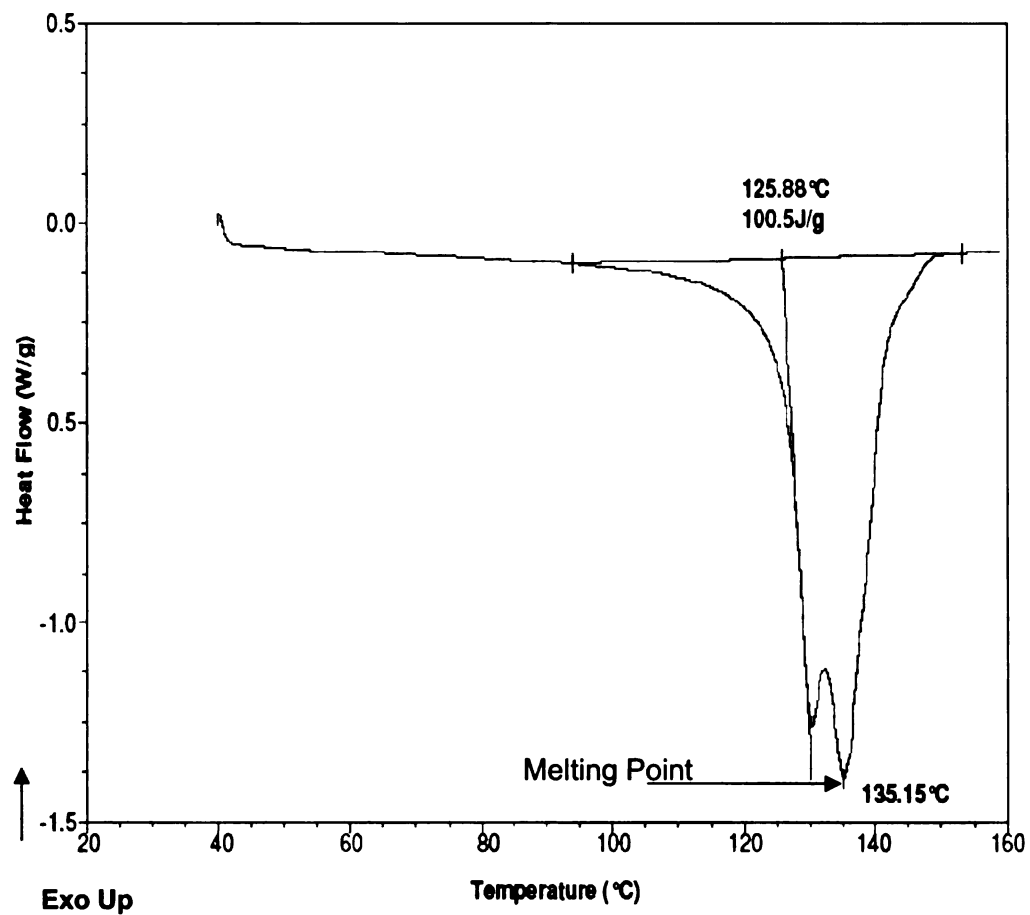


Figure 15. Melting Point for Lot 1 Tyvek® 1073B One Common Peak for High and Low Density Areas

The melting peak in second heating cycle Tyvek® 1073B lot 1 is 135.35°C



**Figure 16. Melting Point for High Density Area of Lot 2 Tyvek® 1073B
- First Cycle Showing Two Melting Point Peaks**

The melting peak in the first heating cycle for Tyvek® 1073B lot 2 is 135.15°C

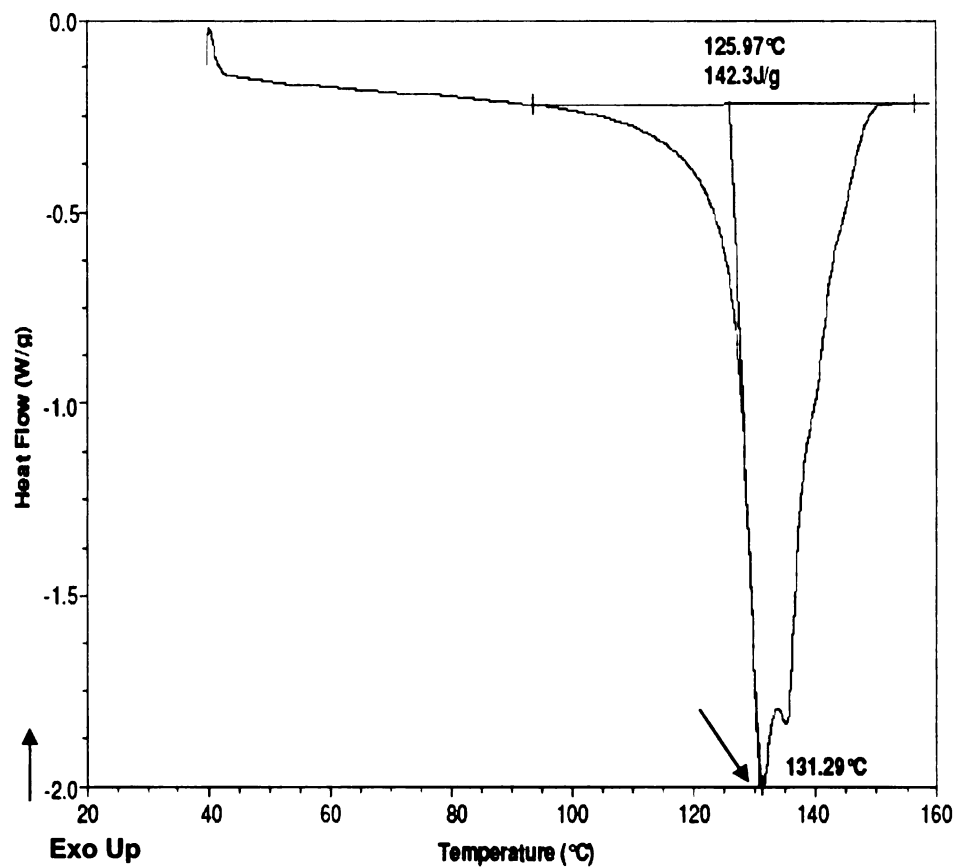


Figure 17. Melting Point for Low Density Area of Lot 2 Tyvek® 1073B - First Cycle Showing Two Melting Point Peaks

The melting peak in the first heating cycle for Tyvek® 1073B lot 2 is 131.29°C

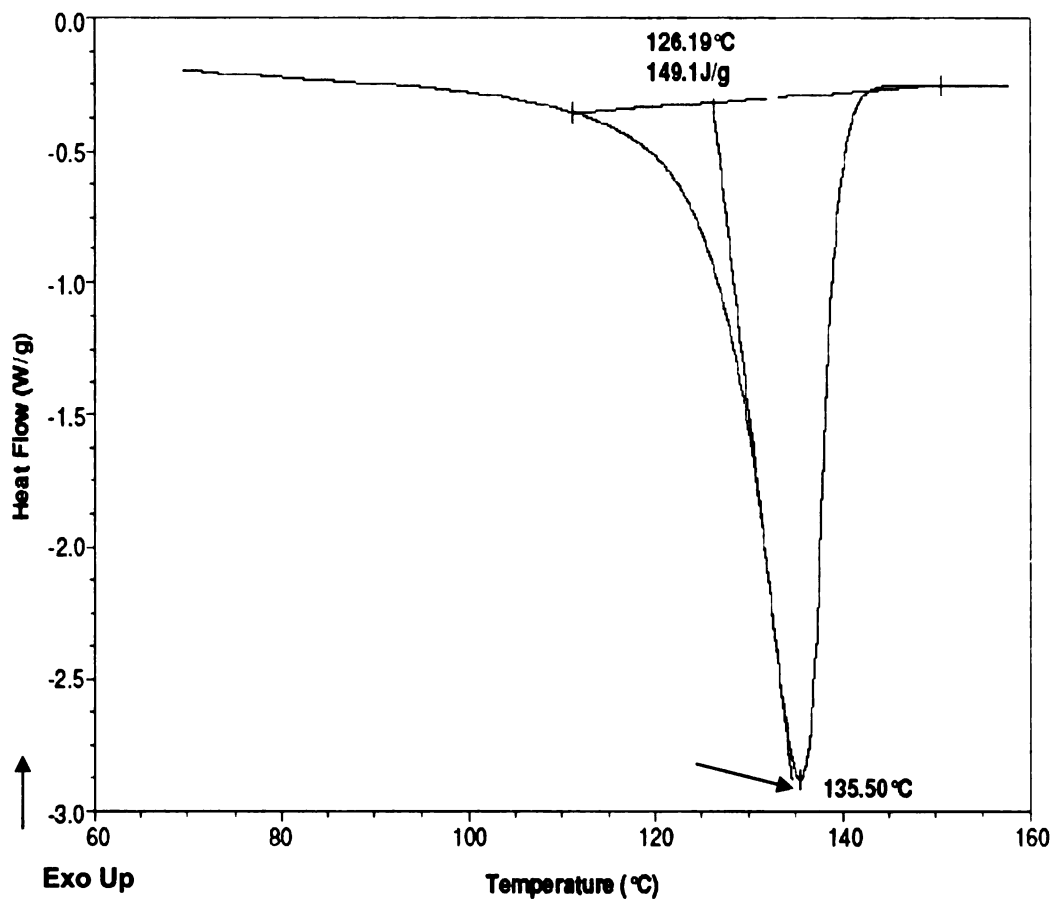


Figure 18. Melting Point for Lot 2 Tyvek® 1073B One Common Peak for High and Low Density Areas

The example graphs that are shown in Figures 13 to 18 are plotted with temperature on the x-axis and heat flow on the y-axis. The heat flow indicates the amount of heat required for this exothermic reaction in order to melt the material. Student's t-test was performed on the second cycle test values to find if there was any significant difference between the melting points for high and low density areas of lots 1 & 2 of Tyvek® 1073B. The t-test analysis did not show any significant difference in melting point for the two areas (Appendix 1, Table 9 and Table 10).

Since there was no statistically significant difference among the melting point values of high and low density areas for lots 1 and 2, the lots were combined for further analysis. A t-test analysis, performed to compare the combined melting point values of high density (lots 1 & 2) and low density (lots 1 and 2), showed no significant difference in melting point between the high and low density sample (Figure 19 and Appendix 1, Table 11).

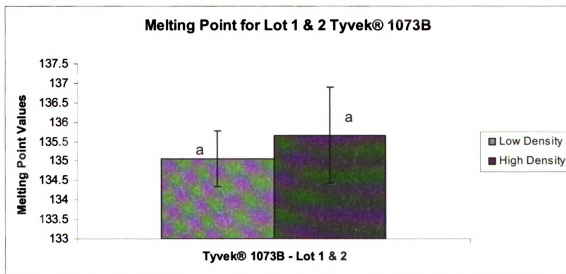


Figure 19. Melting Point of Lot 1 & 2 Tyvek® 1073B for High and Low Density Areas

Note: Bars labelled by the same letters are not statistical significantly different at $p \leq 0.05$

Because the results did not support the hypothesis, the finding that density difference does not affect the melting point is quite interesting; it confirmed the expectation. The next question was to determine whether the inflection point can be determined for regions of high and low density Tyvek® 1073B samples using MTMS.

4.3 Hypothesis: Inflection point of Tyvek® 1073B can be determined using MTMS.

4.3.1 Determination of Inflection Point (Fusion Temperature)

In order to determine the inflection point, six samples (1 inch wide) from each area (high and low density areas from lot 1 & 2) of Tyvek® 1073B were cut and analyzed. The sealing temperature (refer section 3.2.5, p 22 of this thesis), set on the MTMS temperature control unit, is kept 1°C less than the melting point (i.e. 135°C). This temperature of 134°C was chosen to seal two layers of Tyvek® 1073B. The interface temperature was recorded as a function of time by the time-temperature recorder unit by means of a thermocouple. Once the actual sealing temperature at interface was noted, sealing of six samples (each of high and low density areas from lot 1 & 2) were carried out by placing a thermocouple in between them. The time-temperature profile data was obtained on Memory Hicorder, which was then transferred to the computer. Analysis of the inflection point was determined at a set temperature of 134°C by the two earlier described methods (refer to section 3.2.6, p 22 of this thesis for detail).

As described in Figure 20, Figure 21, Figure 22 and Figure 23, the first bar that undergoes transition from a negative value to a positive value signifies the inflection point. This was confirmed by the Table Curve analysis described below. The inflection points for lot 1 Tyvek® 1073B high & low density areas are 125°C and 125°C and for the lot 2 Tyvek® 1073B, the value for high & low density areas are 125°C and 124°C respectively.

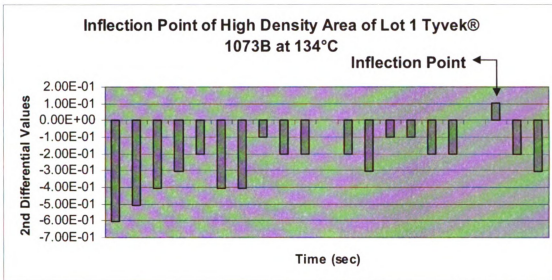


Figure 20. Inflection Point of High Density Area of Lot 1 Tyvek® 1073B at 134°C by MTMS Method

Point 19 is the Inflection Point

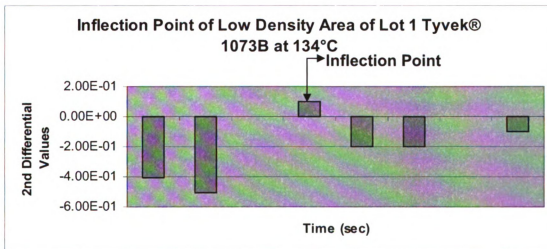


Figure 21. Inflection Point of Low Density Area of Lot 1 Tyvek® 1073B at 134°C by MTMS Method

Point 4 is the Inflection Point

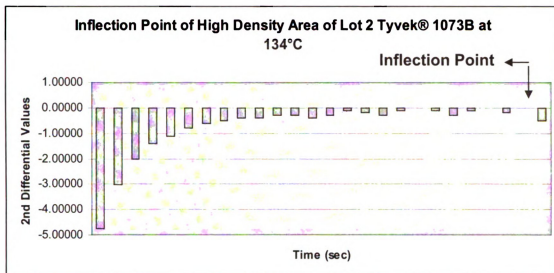


Figure 22. Inflection Point of High Density Area of Lot 2 Tyvek® 1073B at 134°C by MTMS Method

Point 25 is the Inflection Point

Note: Since the D2 value for point 25 bar is small (+ 0.00010), it is not seen in the chart

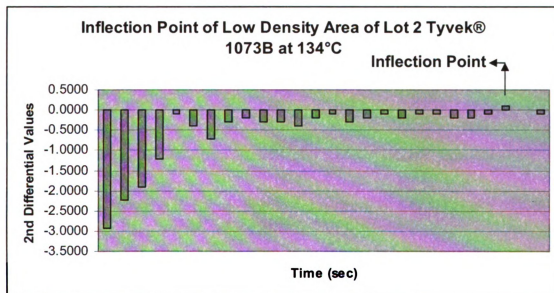


Figure 23. Inflection Point of Low Density Area of Lot 2 Tyvek® 1073B at 134°C by MTMS Method

Point 24 is the Inflection Point.

Another way to determine inflection point is by Table curve method Figure 24, Figure 25, Figure 26 and Figure 27 (these figures are all 1 example of each lot and density). In Figure 24 the 2nd derivative of temperature is plotted as a function of time. The point when the curve crosses the zero line at the x axis (negative to positive transition) is the inflection point. Thus, the inflection point was estimated to be 127.2°C for Figure 24.

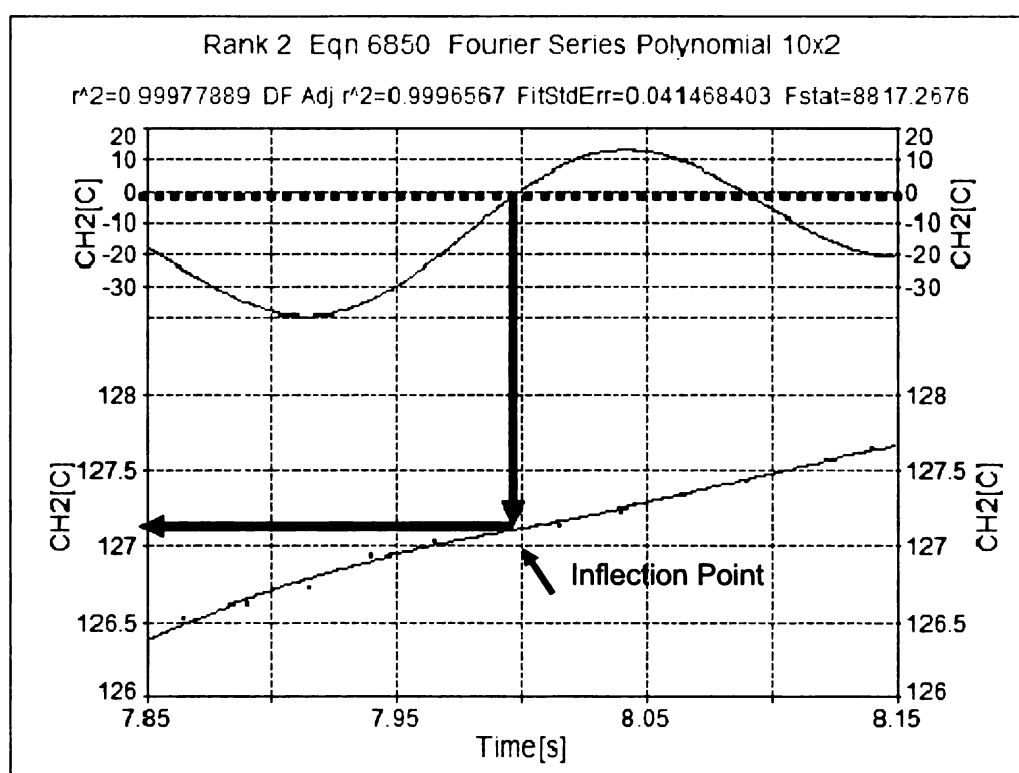


Figure 24. Inflection Point of High Density Area of Lot 1 Tyvek® 1073B at 134°C by Table Curve Method

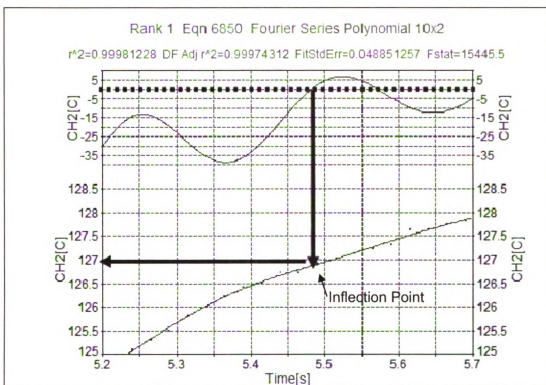


Figure 25. Inflection Point of Low Density Area of Lot 1Tyvek® 1073B at 134°C by Table Curve Method

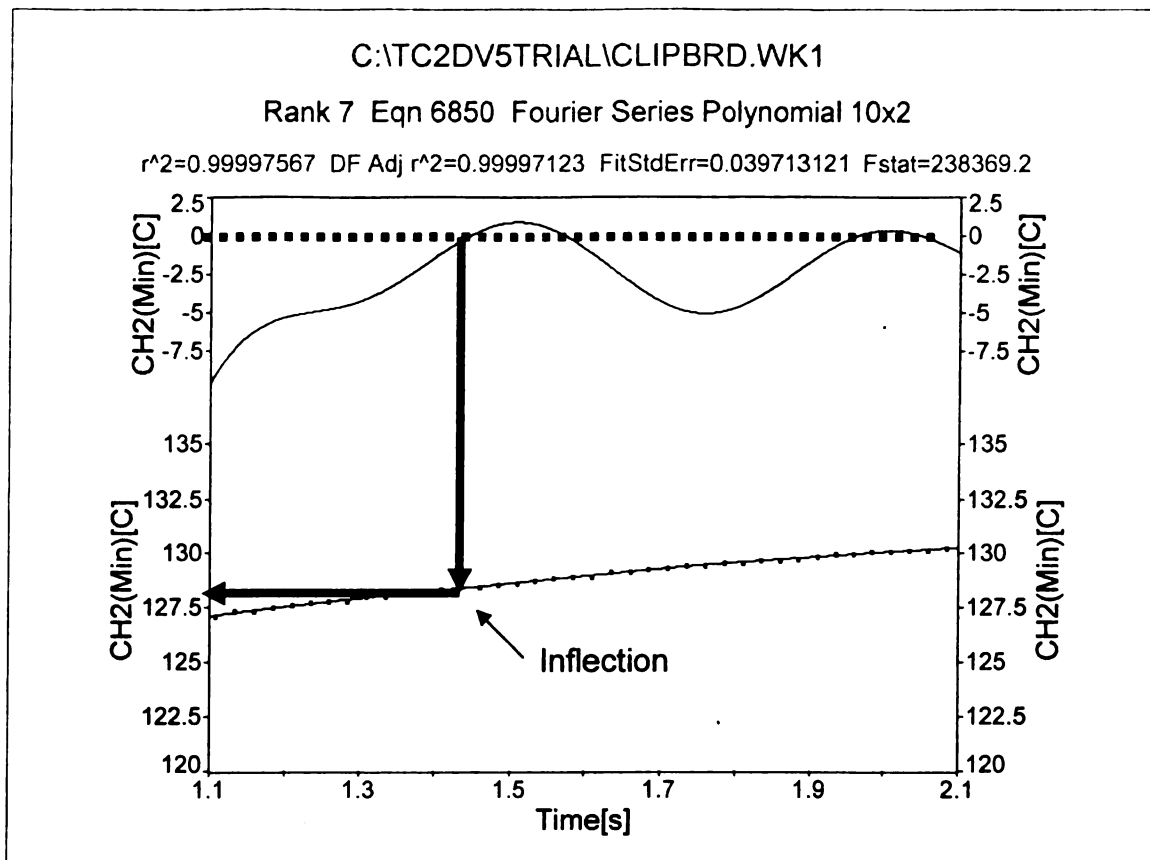


Figure 26. Inflection Point of High Density Area of Lot 2 Tyvek® 1073B at 134°C by Table Curve Method

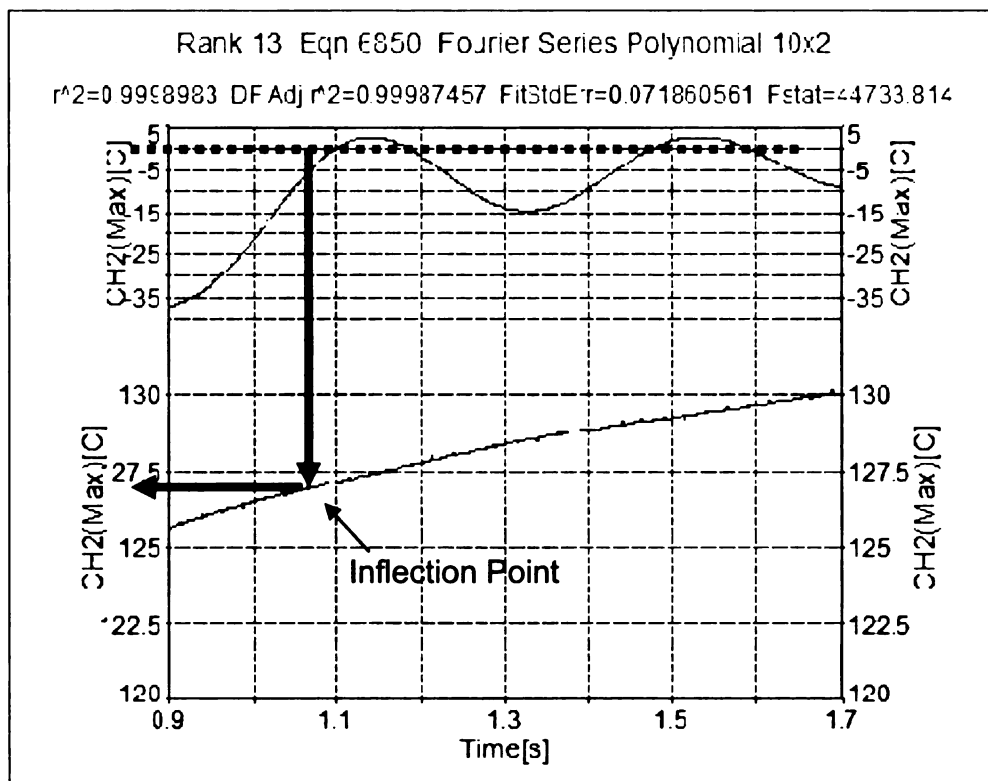


Figure 27. Inflection Point of Low Density Area of Lot 2 Tyvek® 1073B at 134°C by Table Curve Method

Thus, it was shown that it is possible to obtain an inflection point of Tyvek® 1073B, and it was determined to be similar for the two lots of Tyvek® 1073B, 125°C and 124°C for lots 1 and 2 respectively. The inflection point was found to be 10°C less than the melting point (~135°C) for both lots of Tyvek® 1073B (Appendix 1, Table 12, Table 13, Table 14 and Table 15).

As the inflection points of high and low density areas of lots 1 & 2 Tyvek® 1073B showed no significant difference, the values of the two lots were combined together and analyzed. The combined values for high density area (MTMS and Table curve for lots 1 & 2) and low density area (MTMS and Table curve for lot 1 & 2) also showed no significant difference between them.

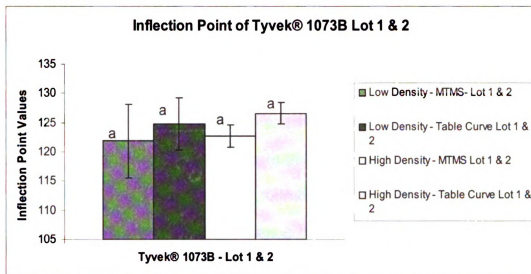


Figure 28. Inflection Point of Tyvek® 1073B Lot 1 & 2

Note: Bars labelled by the same letters are not statistical significantly different at $p \leq 0.05$

Thus the inflection point for Tyvek® 1073B upon lot combination was estimated to be approximately 125°C for high and low density areas of Tyvek® 1073B (Appendix 1, Table 16 and Figure 28).

4.4 Hypothesis: Strong heat peelable seal can be obtained for sealing Tyvek® 1073B to Tyvek® 1073B using MTMS analysis.

Once the inflection point was determined for Tyvek® 1073B, it was hypothesized that a strong peelable heat seal temperature on Tyvek® 1073B can be obtained near the inflection point. The samples (from lot 1 & 2) were cut into 1" wide strips and conditioned for 48 hours at 23°C at 50% RH before sealing. The temperature on MTMS temperature control unit was set approximately 1-2°C higher than the sealing temperature. The temperature at the interface was

monitored by placing a thermocouple in between the sealing jaws and was verified during trial seals. The temperature was displayed on the Memory Hicorder. Samples of lot 1 & 2 were sealed at set temperature of 125°C, 129°C, 132°C, 134°C, 136.5°C, 139°C, 141°C and 145°C. Using the universal machine, peel strength of seal at each temperature (for six samples each of lot 1 & 2) was tested. At 125°C, there was no seal formation for both lots of Tyvek® 1073B, however at 129°C, tack seal was obtained in both the lots of Tyvek® 1073B. At 132°C a good peelable seal with slight shrinkage (5% of shrinkage) was obtained in both the lots of Tyvek® 1073B, at 134°C a good peelable heat seal with shrinkage of 10% was noted in lot 1 & 2 of Tyvek® 1073B, at 136°C, 139°C, 141°C, 145°C there was sealing but with lots of shrinkage (approximately 30%-50% of shrinkage was seen) in the material in both the Tyvek® 1073B lot cases (Figure 29 and Figure 30). Thus, it was observed that as the temperature for sealing increased, the peel strength value went up and then dropped down.

It is possible to obtain a peelable heat seal at around set temperature of 136-139°C for Tyvek® 1073B to Tyvek® 1073B sealing. The sealing temperature was approximately 10-14°C higher than the inflection point of the sample as sealing could not take place at the inflection point (Appendix 1, Table 21 and Table 23).

Since, the two lots of Tyvek® 1073B values showed similar inflection point also sealed at the same temperatures, the values of peel strength, shrinkage and extension at break were merged (Figure 31 and Appendix 1, Table 25). Since, finally resulting in fourteen different values (7 values from lot 1 and 7 values of lot

2), the average of the two sets were taken and combined to analyze the strongest peel strength.

The strongest peelable heat seal was obtained at 139°C, but this value was not further considered because of high percentage of shrinkage in material during the sealing process. The tensile strength of Tyvek® 1073B is 4.6 N/mm (DuPont 2007) and the peel strength of Tyvek® 1073B obtained from the experiments was determined to be 1.16 N/mm.

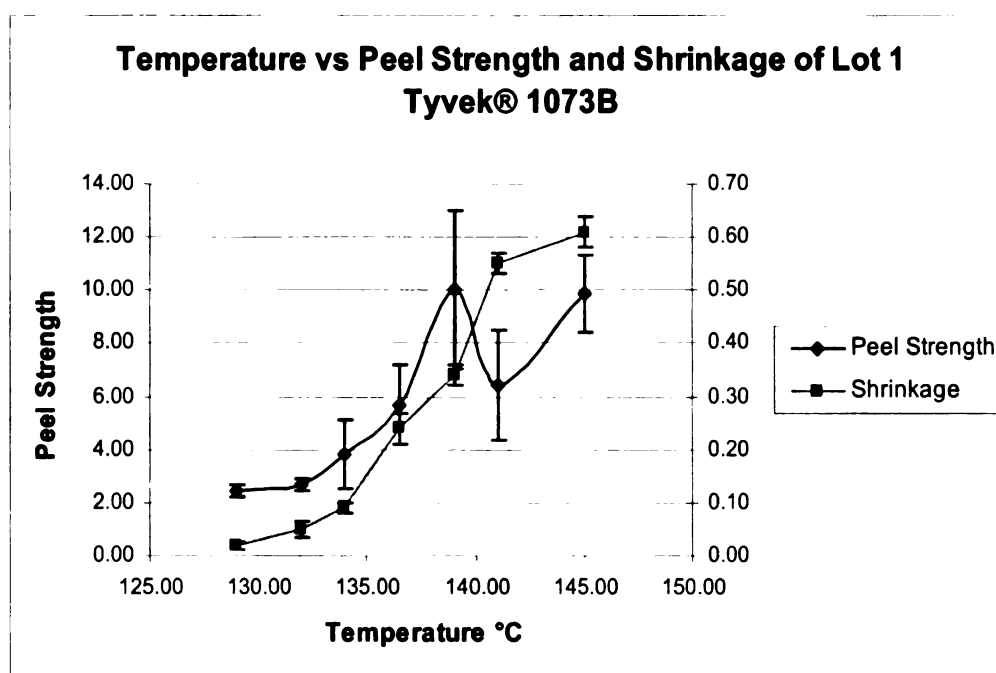


Figure 29. Peel Strength Values for Tyvek® 1073B at Different Temperatures for Lot 1 Tyvek® 1073B Values

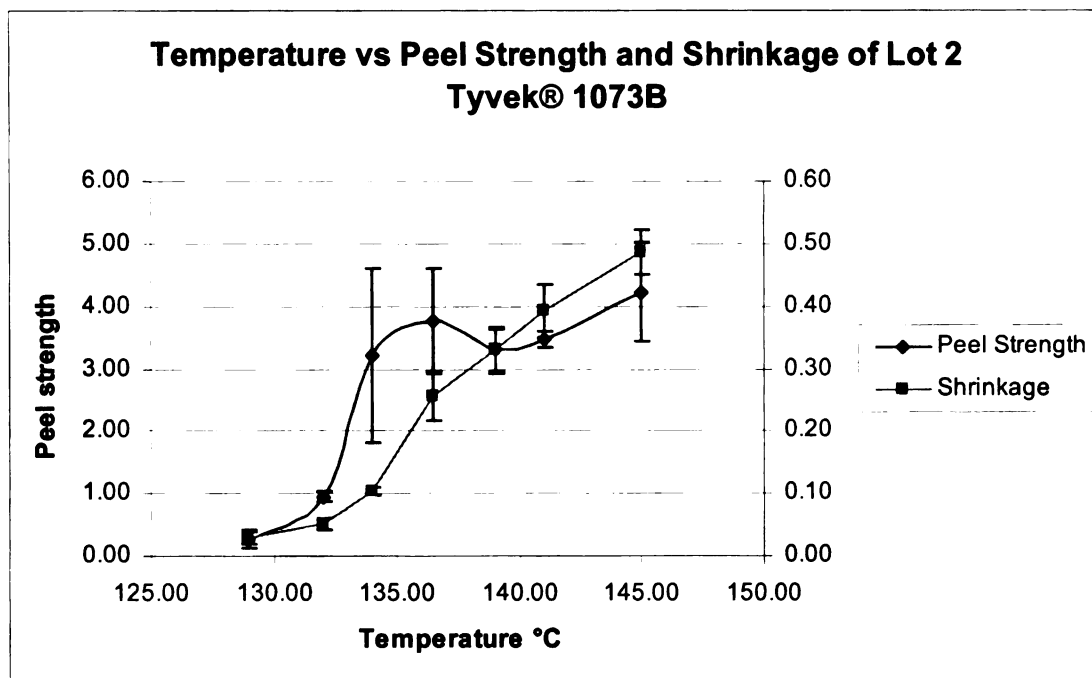


Figure 30. Peel Strength Values for Tyvek® 1073B at Different Temperatures for Lot 2 Tyvek® 1073B Values

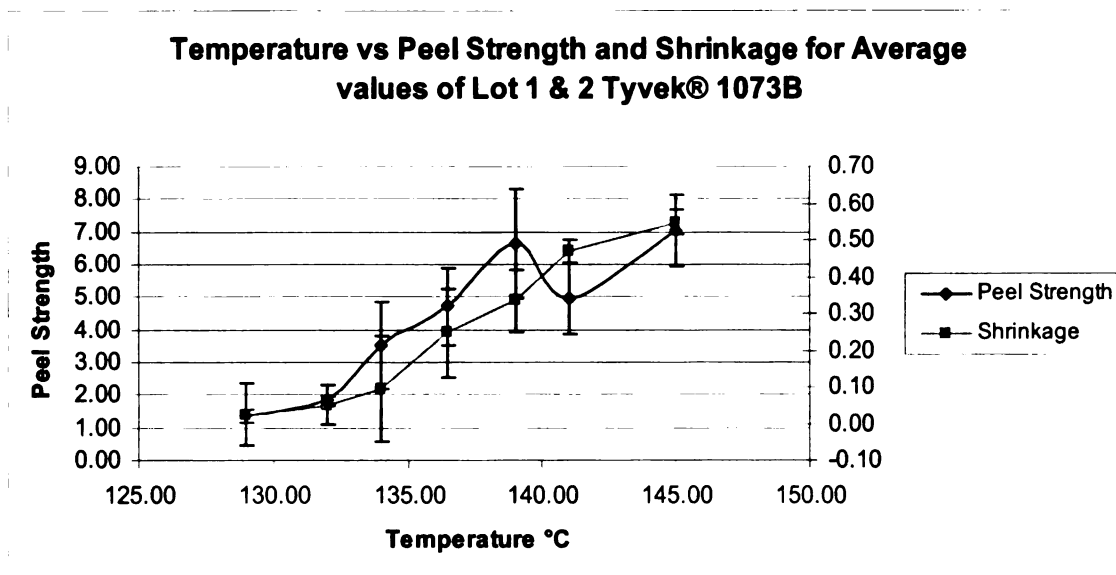


Figure 31. Peel Strength Values for Tyvek® 1073B at Different Temperatures for Lot 1 & 2 Tyvek® 1073B Values

The hypothesis that a strong peelable heat seal can be obtained is confirmed. However, it is accompanied by excessive heat shrinkage (30 – 35 %). So, as a matter of practical application, the strongest seal would not be used.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

Measuring temperature of the melting surface (MTMS) is a systematic approach to obtain the strongest possible peelable heat seal for any two materials. Thus, this study was aimed at determining this inflection point at which the strongest possible peelable heat seal temperature can be obtained for spun bonded polyethylene fiber sheets - Tyvek® 1073B. We were able to determine an inflection point for Tyvek® 1073B by MTMS instrument, that earlier could not be estimated. The inflection point for two lots of Tyvek® 1073B was found to be 125°C. It was also possible to obtain a peelable heat seal for Tyvek® 1073B to Tyvek® 1073B sealing. We also showed that Hishinuma's defined logic, that inflection point is the point at which the strongest possible peelable heat seal can be obtained, may not necessarily apply to all types of sealing materials as shown in the current study using Tyvek® 1073B samples from two different lots. Although strongest peelable seal was seen in Tyvek® 1073B at 139°C, it was not at the inflection point (125°C), instead there was no seal at inflection point; also the strongest seal displayed an unacceptable amount of shrinkage of the material. Since, high density polyethylene fibers are distributed randomly throughout the Tyvek® 1073B sheet; it was assumed that there might be some differences in physical properties, like melting point and inflection point, in high and low density areas of two lots of Tyvek® 1073B. The analysis showed that there was no significant difference in physical properties of Tyvek® 1073B at high

and low density areas in two lots of Tyvek® 1073B. This shows that, although there are high and low density regions in Tyvek® 1073B, they may not directly affect the inflection point or the seal strength of the Tyvek® 1073B. The failure to obtain a strong peelable heat seal at the inflection point can be due to other properties of the Tyvek® 1073B like the porosity of the material.

This study was done using uncoated grade of Tyvek® 1073B; thus, we were able to show that MTMS method can be used to determine the fusion temperature of uncoated Tyvek® 1073B and possibly for other grades that are available in the industry. Visual, spectroscopic and colorimetric analysis was carried out and showed the evidence for existence of differential distribution of fiber density in sheets of Tyvek® 1073B. However, the optical microscopic results did not confirm the same observation, hence the proposal is that more testing needs to be done to further confirm this. Other physical properties (porosity) of Tyvek® 1073B were not tested, hence, further testing on it is recommended. More types of plastic films should be tested using MTMS. This methodology can be adopted to find better sealing temperature for induction and blister sealing. Further work can be done towards determination of the fusion temperature for coated Tyvek® 1073B and a comparison of results with uncoated grades of Tyvek® 1073B would be very informative.

APPENDIX 1
RESULTS FROM TESTS (TABLES)

Table 3 .Total Area Under Light Transmission (T) Curve for Six Samples of Low and High Density Area of Tyvek® 1073B – Lot 1 (School of Packaging (SoP) - Lot of Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (SoP Lot)	High Density Area of Tyvek® 1073B (SoP Lot)
1	14548.36	6498.26
2	22778.18	5140.01
3	20847.80	6037.65
4	24396.28	4985.85
5	15670.51	5977.20
6	12646.38	5462.01
Average	18481.25	5683.50
Std Dev	4826.29	584.24

t-test for significant different between means: p = **2.39456E-06**

Table 4 .Total Area Under Light Transmission (T) Curve for Six Samples of Low and High Density Area of Tyvek® 1073B – Lot 2 (Tolas Healthcare Lot of Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (Tolas Healthcare lot)	High Density Area of Tyvek® 1073B (Tolas Healthcare Lot)
1	14959.45	4501.21
2	19633.69	6450.98
3	16982.28	6529.75
4	13820.82	4763.60
5	16097.78	7764.93
6	16660.76	4788.12
Average	16359.13	5799.76
Std Dev	1983.55	1311.65

t-test for significant different between means: p = **0.001196675**

Table 5 .Total Area Under Light Transmission (T) Curve for Twelve Samples of Low and High Density Area of Tyvek® 1073B

Combination of Lot 1 & 2 Tyvek® 1073B Values

Samples	Low Density Area of Tyvek® 1073B	High Density Area of Tyvek® 1073B
1	14548.36	6498.26
2	22778.18	5140.01
3	20847.80	6037.65
4	24396.28	4985.85
5	15670.51	5977.20
6	12646.38	5462.01
7	14959.45	4501.21
8	19633.69	6450.98
9	16982.28	6529.75
10	13820.82	4763.60
11	16097.78	7764.93
12	16660.76	4788.12
Average	17420.19	5741.63
Std Dev	3688.41	969.98

t-test for significant different between means: p = **1.28637E-07**

Table 6. Colorimeter Values for High and Low Density Areas of Tyvek® 1073B – Lot 1 (School of Packaging (SoP) -Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (SoP Lot)	High Density Area of Tyvek® 1073B (SoP Lot)
1	96.16	97.57
2	95.58	97.56
3	95.56	97.84
4	96.37	97.55
5	95.38	98.02
6	95.59	97.72
Average	95.77	97.71
Std Dev	0.39	0.19

t-test for significant different between means: p = **1.01365E-05**

Table 7. Colorimeter Values for High and Low Density Areas of Tyvek® 1073B – Lot 2 (Tolas Healthcare Lot of Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (Tolas Healthcare lot)	High Density Area of Tyvek® 1073B (Tolas Healthcare lot)
1	95.89	98.01
2	95.28	97.88
3	95.78	97.99
4	95.79	97.99
5	95.84	97.86
6	95.66	97.91
Average	95.71	97.94
Std Dev	0.22	0.06

t-test for significant different between means: $p = 5.18518E-07$

Table 8. Colorimeter Values for High and Low Density Areas of Tyvek® 1073B Combination of Lot 1 & 2 Tyvek® 1073B Values

Samples	Low Density Area of Tyvek® 1073B	High Density Area of Tyvek® 1073B
1	95.89	98.01
2	95.28	97.88
3	95.78	97.99
4	95.79	97.99
5	95.84	97.86
6	95.66	97.91
7	96.16	97.57
8	95.58	97.56
9	95.56	97.84
10	96.37	97.55
11	95.38	98.02
12	95.59	97.72
Average	95.74	97.83
Std Dev	0.31	0.18

t-test for significant different between means: $p = 9.51989E-14$

Table 9. Melting Point Values for High and Low Density Areas of Tyvek® 1073B – Lot 1 (School of Packaging (SoP) - Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (SoP Lot)	High Density Area of Tyvek® 1073B (SoP Lot)
1	135.65	135.54
2	136.26	135.35
3	135.76	138.65
4	135.66	135.61
5	135.33	137.39
6	134.99	136.12
Average	135.61	136.44
Std Dev	0.43	1.31

t-test for significant different between means: $p = 0.187726819$

Table 10. Melting Point Values for High and Low Density Areas of Tyvek® 1073B – Lot 2 (Tolas Healthcare Lot of Tyvek® 1073B Samples)

Samples	Low Density Area of Tyvek® 1073B (Tolas Healthcare Lot)	High Density Area of Tyvek® 1073B (Tolas Healthcare Lot)
1	134.44	134.89
2	134.02	134.96
3	134.25	135.50
4	135.04	134.48
5	135.08	134.45
6	134.17	134.97
Average	134.50	134.88
Std Dev	0.45	0.39

t-test for significant different between means: $p = 0.155209843$

**Table 11. Melting Point Values for High and Low Density Areas of Tyvek® 1073B
Combination of Lot 1 & 2 Values**

Samples	Low Density Area of Tyvek® 1073B	High Density Area of Tyvek® 1073B
1	134.44	134.89
2	134.02	134.96
3	134.25	135.50
4	135.04	134.48
5	135.08	134.45
6	134.17	134.97
7	135.65	135.54
8	136.26	135.35
9	135.76	138.65
10	135.66	135.61
11	135.33	137.39
12	134.99	136.12
Average	135.05	135.66
Std Dev	0.72	1.23

t-test for significant different between means: p = **0.158891**

**Table 12. Inflection Point of Low Density Area of Tyvek® 1073B at Set
Temperature of 134°C and Average Melting Point of 135°C
Lot 1- (School of Packaging (SoP) - Lot of Tyvek® 1073B Samples Values)**

Samples	Interface Temperature °C	MTMS °C	Table Curve °C
1	133.00	120.00	120.80
2	133.00	124.00	128.00
3	133.00	125.00	126.90
4	133.00	125.00	126.45
5	133.00	123.00	127.20
6	133.00	125.00	122.80
Average		123.67	125.36
Std Deviation		1.966384	2.872702

t-test for significant different between means: p = **0.264905**

**Table 13. Inflection Point of High Density Area of Tyvek® 1073B at Set Temperature of 134°C and Average Melting Point of 135°C
Lot 1- (School of Packaging (SoP) - Lot of Tyvek® 1073B Samples Values)**

Samples	Interface Temperature °C	MTMS °C	Table Curve °C
1	133.00	125.00	127.10
2	133.00	122.00	126.80
3	133.00	123.00	127.10
4	133.00	124.00	127.30
5	133.00	123.00	126.60
6	133.00	125.00	127.55
Average		123.67	127.08
Std Deviation		1.21106	0.340221

t-test for significant different between means: p = **0.000656**

**Table 14. Inflection Point of Low Density Area of Tyvek® 1073B at Set Temperature of 134°C and Average Melting Point of 135°C
Lot 2 - (Tolas Healthcare Lot of Tyvek® 1073B Samples Values)**

Samples	Interface Temperature °C	MTMS °C	Table Curve °C
1	133	126	130.10
2	133	102.7	113.05
3	133	125	127.40
4	133	123	125.50
5	133	121	126.10
6	133	123	123.88
Average		120.12	124.34
Std Deviation		8.71	5.91

t-test for significant different between means: p = **0.352084**

**Table 15. Inflection Point of High Density Area of Tyvek® 1073B at Set Temperature of 134°C and Average Melting Point of 135°C
Lot 2 - (Tolas Healthcare Lot of Tyvek® 1073B Samples Values)**

Samples	Interface Temperature °C	MTMS °C	Table Curve °C
1	133	125	128.00
2	133	120	130.20
3	133	122	125.90
4	133	120	124.50
5	133	123	124.00
6	133	120	123.60
Average		121.67	126.03
Std Deviation		2.07	2.59

t-test for significant different between means: p = **0.00967**

Table 16. Inflection Point of High and Low Density Area of Tyvek® 1073B at Set Temperature of 134°C and Average Melting Point of 135°C

Combination of Lot 1 & 2 for Tyvek® 1073B Samples Values

	Low Density	Low Density	High Density	High Density
Samples	MTMS	Table Curve	MTMS	Table Curve
1	126.00	130.10	125.00	128.00
2	102.70	113.05	120.00	130.20
3	125.00	127.40	122.00	125.90
4	123.00	125.50	120.00	124.50
5	121.00	126.10	123.00	124.00
6	123.00	123.88	120.00	123.60
7	120.00	120.80	125.00	127.10
8	124.00	128.00	122.00	126.80
9	125.00	126.90	123.00	127.10
10	125.00	126.45	124.00	127.30
11	123.00	127.20	123.00	126.60
12	125.00	122.80	125.00	127.55
Average	121.89	124.85	122.67	126.55
Std Deviation	6.30	4.46	1.92	1.85

t-test for significant different between means:

p value for MTMS vs Table Curve for Low Density is **0.19962**

p value for MTMS vs Table Curve for High Density is **4.66483E-05**

p value for MTMS for Low Density vs High Density is **0.6901**

p value for Table Curve method for Low Density vs High Density is **0.24039**

Table 17. Time-Temperature Data Obtained from MTMS for High Density Area of Lot 1 Tyvek® 1073B at 134°C

Time(Sec)	Temperature °C	1st Differential Value (D1)	2nd Differential Value (D2)
2.33E+01	1.11E+02	2.93E+00	-6.06E-01
2.33E+01	1.12E+02	2.63E+00	-5.05E-01
2.34E+01	1.14E+02	2.32E+00	-4.04E-01
2.34E+01	1.15E+02	2.12E+00	-3.03E-01
2.34E+01	1.16E+02	1.92E+00	-2.02E-01
2.34E+01	1.17E+02	1.82E+00	-4.04E-01
2.35E+01	1.18E+02	1.72E+00	-4.04E-01
2.35E+01	1.19E+02	1.41E+00	-1.01E-01
2.35E+01	1.20E+02	1.31E+00	-2.02E-01
2.35E+01	1.20E+02	1.31E+00	-2.02E-01
2.36E+01	1.21E+02	1.11E+00	0.00E+00
2.36E+01	1.22E+02	1.11E+00	-2.02E-01
2.36E+01	1.22E+02	1.11E+00	-3.03E-01
2.36E+01	1.23E+02	9.10E-01	-1.01E-01
2.37E+01	1.23E+02	8.08E-01	-1.01E-01
2.37E+01	1.24E+02	8.08E-01	-2.02E-01
2.37E+01	1.24E+02	7.08E-01	-2.02E-01
2.38E+01	1.25E+02	5.05E-01	1.01E-01
2.38E+01	1.25E+02	6.06E-01	-2.02E-01
2.38E+01	1.25E+02	6.06E-01	-3.03E-01
2.38E+01	1.26E+02	4.04E-01	1.00E-04
2.39E+01	1.26E+02	3.03E-01	3.03E-01
2.39E+01	1.26E+02	4.04E-01	-1.01E-01

The inflection point for High Density Area of Tyvek® 1073B lot 1 is **125°C**. The value in 2nd differential (D2), changes from negative to positive at the inflection point.

Table 18. Time - Temperature Data Obtained from MTMS for Low Density Area of Lot 1 Tyvek® 1073B at 134°C

Time(Sec)	Temperature °C	1st Differential (D1)	2nd Differential (D2)
4.84E+00	1.23E+02	1.11E+00	-4.04E-01
4.87E+00	1.23E+02	1.11E+00	-5.05E-01
4.89E+00	1.24E+02	7.07E-01	-1.42E-14
4.92E+00	1.25E+02	6.06E-01	1.01E-01
4.94E+00	1.25E+02	7.07E-01	-2.02E-01
4.97E+00	1.25E+02	7.07E-01	-2.02E-01
4.99E+00	1.26E+02	5.05E-01	0.00E+00
5.02E+00	1.26E+02	5.05E-01	-1.01E-01

The inflection point for Low Density Area of Tyvek®1073B lot 1 is **125°C**. The value in 2nd differential (D2), changes from negative to positive at the inflection point.

Table 19. Time - Temperature Data Obtained from MTMS for High Density Area of Lot 2 Tyvek® 1073B at 134°C

Time (Sec)	Temperature °C	1st Differential (D1)	2nd Differential (D2)
0.210	89.5392	10.9144	-4.74974
0.235	95.9059	7.9838	-3.03186
0.260	100.4536	6.1647	-2.02120
0.285	103.8897	4.9519	-1.41480
0.310	106.6183	4.1435	-1.11170
0.335	108.8416	3.5371	-0.80850
0.360	110.7618	3.0318	-0.60640
0.385	112.3787	2.7286	-0.50520
0.410	113.7936	2.4254	-0.40420
0.435	115.1073	2.2234	-0.40440
0.460	116.2190	2.0212	-0.30320
0.485	117.3307	1.8190	-0.30310
0.510	118.2402	1.7180	-0.40420
0.535	119.1497	1.5159	-0.30310
0.560	119.9582	1.3138	-0.10110
0.585	120.6656	1.2128	-0.20220
0.610	121.2720	1.2127	-0.30310
0.635	121.8784	1.0106	-0.10110
0.660	122.4847	0.9096	-0.00010
0.685	122.8890	0.9095	-0.10100
0.710	123.3943	0.9095	-0.30310
0.735	123.7985	0.8085	-0.10110
0.760	124.3038	0.6064	-0.00010
0.785	124.6070	0.7074	-0.20210
0.810	124.9102	0.6063	0.00010
0.835	125.3144	0.5053	0.00000
0.860	125.5165	0.6064	-0.20220

The inflection point for High Density Area of Tyvek® 1073B lot 2 is **124.9°C**. The value in 2nd differential (D2), changes from negative to positive at the inflection point.

Table 20. Time - Temperature Data Obtained from MTMS for Low Density Area of Lot 2 Tyvek® 1073B at 134°C

Time(Sec)	Temperature °C	1st Differential Value (D1)	2nd Differential Value (D2)
0.290	98.5335	7.5795	-2.9307
0.315	102.2727	6.0636	-2.2233
0.340	106.1130	4.6488	-1.9202
0.365	108.3363	3.8403	-1.2127
0.390	110.7618	2.7286	-0.1011
0.415	112.1766	2.6276	-0.4043
0.440	113.4904	2.6275	-0.7073
0.465	114.8042	2.2233	-0.3032
0.490	116.1179	1.9202	-0.2022
0.515	117.0275	1.9201	-0.3031
0.540	118.0381	1.7180	-0.3032
0.565	118.9476	1.6170	-0.4043
0.590	119.7561	1.4148	-0.2020
0.615	120.5646	1.2127	-0.1010
0.640	121.1709	1.2128	-0.3033
0.665	121.7773	1.1117	-0.2022
0.690	122.3837	0.9095	-0.1010
0.715	122.8890	0.9095	-0.2021
0.740	123.2932	0.8085	-0.1011
0.765	123.7985	0.7074	-0.1010
0.790	124.1017	0.7074	-0.2021
0.815	124.5059	0.6064	-0.2022
0.840	124.8091	0.5053	-0.1011
0.865	125.1123	0.4042	0.1011
0.890	125.3144	0.4042	0.0001
0.915	125.5165	0.5053	-0.1010

The inflection point for Low Density Area of Tyvek® 1073B lot 1 is **125.11°C**. The value in 2nd differential (D2), changes from negative to positive at the inflection point.

**Table 21. Seal Strength (Average Values) for Tyvek® 1073B
Lot 1 - (School of Packaging (SoP) - Tyvek® 1073B Samples Values)**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	2.46	0.02	1.28
132.00	2.70	0.05	1.37
134.00	3.82	0.09	1.96
136.50	5.66	0.24	1.57
139.00	10.01	0.34	2.20
141.00	6.43	0.55	0.55
145.00	9.86	0.61	0.61

The strongest seal strength value for Tyvek® 1073B lot 1 at **139°C**

**Table 22. Seal Strength (Std Deviation Values) for Tyvek® 1073B
Lot 1 - (School of Packaging (SoP) - Tyvek® 1073B Samples Values)**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	0.23	0.01	0.16
132.00	0.24	0.01	0.10
134.00	1.30	0.01	0.28
136.50	1.49	0.03	0.20
139.00	2.98	0.02	0.14
141.00	2.06	0.02	0.02
145.00	1.45	0.03	0.03

**Table 23. Seal Strength (Average Values) for Tyvek® 1073B
Lot 2 - (Tolas Healthcare Tyvek® 1073B Samples Values)**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	0.26	0.03	1.16
132.00	0.95	0.05	1.34
134.00	3.20	0.10	2.17
136.50	3.75	0.26	2.19
139.00	3.29	0.33	1.78
141.00	3.47	0.39	1.64
145.00	4.23	0.49	1.23

The strongest seal strength value for Tyvek® 1073B lot 2 at **136.5°C**

**Table 24. Seal Strength (Std Deviation Values) for Tyvek® 1073B
Lot 2 - (Tolas Healthcare Tyvek® 1073B Samples Values)**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	0.14	0.09	0.01
132.00	0.07	0.15	0.01
134.00	1.39	0.32	0.01
136.50	0.85	0.16	0.04
139.00	0.37	0.21	0.03
141.00	0.13	0.29	0.04
145.00	0.79	0.52	0.03

**Table 25. Seal Strength (Average Values) for Tyvek® 1073B
Combination of Lot 1 & 2 Tyvek® 1073B Values**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	1.36	0.03	1.22
132.00	1.83	0.05	1.36
134.00	3.51	0.10	2.06
136.50	4.71	0.25	1.88
139.00	6.65	0.33	1.99
141.00	4.95	0.47	1.10
145.00	7.04	0.55	0.92

The strongest seal strength value for Tyvek® 1073B lot 1 & 2 at **139°C**

**Table 26. Seal Strength (Std Deviation Values) for Tyvek® 1073B
Combination of Lot 1 & 2 Tyvek® 1073B Values**

Temperature °C	Peel Strength (lb/in)	Shrinkage (in)	Extension at Break (in)
129.00	0.184213	0.09	0.12
132.00	0.153834	0.06	0.13
134.00	1.347958	0.14	0.30
136.50	1.170727	0.12	0.18
139.00	1.674071	0.08	0.17
141.00	1.094898	0.03	0.16
145.00	1.118976	0.03	0.27

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