Characterising the Mechanical Properties of Polymers used in Sterile Barrier Systems

Thomas O'Brien

School of Engineering and Innovation, The Open University, UK

ABSTRACT

Polymers are widely used in the manufacture of sterile barrier systems for the healthcare sector. Sterile barrier systems are packaging systems utilised in medical, pharmaceutical and diagnostic applications where the content of the package requires post-packaging sterilisation. Products such as bags, pouches and thermoformed trays can be manufactured from thermoplastics such as polypropylene, polyethylene, polystyrene and polycarbonate. Prior to forming, single-layer or multi-layer sheet first undergoes extrusion and is then wound on a roll. The extruded roll-stock is then formed by a secondary process into final packaging components. The various stages in the production processes subject the materials to varying temperatures, pressures and stresses and the result of these variations in the production processes generally leads to variation in the mechanical properties of the final components and in particular the heat sealability of the components. Increased demands from regulatory authorities over concerns about patient safety have led manufacturers to seek new ways to improve their process technology and product quality with a view to reducing the variation in these mechanical properties. This paper discusses the use of polymer analysis to gain better understanding of some of these material properties and how they can influence product quality.

Keywords: Polymers, material properties, extruded film

1 Introduction

Stringent requirements in the healthcare industry demand tighter manufacturing tolerances for sterile barrier systems. Sterile barrier systems are used to maintain the products in acceptable condition prior to use, to allow sterilization and to keep the stored device free from contamination **[1]**. Polymer films have been widely used in medical packaging, however, due to high performance requirements, selecting the right material at high quality consistently can be challenging.

Polyethylene (PE) is one of the most versatile polymers available today and is currently widely used in healthcare packaging **[2,3]**. The wide variety of molecular chain architectures available in these polymers has made possible extensive combinations for end-user packaging applications. Low Density Polyethylene resins (LDPE) in particular have been used in the manufacture of a wide range of films within the medical packaging industry due to its low cost, relative toughness, flexibility, sealability and ease of processability. The increasing demand for higher speeds in packaging equipment has placed limitations on conventional LDPE films. However, the emergence of linear low density resins (LLDPE) over the years has resulted in films with the superior

thermal and mechanical properties required for use in higher speed packaging equipment.

Polyethylene terephthalate (PET) is commonly used in packaging due to its toughness, dimensional stability, chemical resistance, clarity and gas barrier properties **[4]**. However, it is rather costly and is therefore normally used in thinner gauges or combined with other materials. One of its disadvantages is its low melt strength, which makes conventional grade PET difficult to process by blown film extrusion. The lamination process enables PET to be combined with other material such as PE in order to create a single packaging structure, utilizing the best properties from each polymer.

Some film webs exhibit mechanical non-uniformities that originate from variation in resins and additives and/or changes during the film extrusion process. In blown film extrusion, several processing factors can contribute to these non-uniformities **[5]**. These may be related to the degree of orientation and stretching that the polymer melt experiences during bubble formation as it leaves the die, the bubble inflation into the air ring, the collapse from tube to flat web or the lay flat tube passing through the rest of the web handling process. These non-uniformities will add further challenges to downstream processes and final heat sealing processes can experience considerable deviations in the performance of the resulting seals.

This study uses material characterisation and analysis methods to investigate different samples of a blown film PET/EVA-PE laminate produced on a blown film line which was set-up to produce different consecutive batches by varying the blow-up ratio. The batches were selected on the basis that the processing equation factor was constant, the resulting batches met in-house inspection criteria and the polymers processed within the film line were of a consistent type and specification. Thermal and mechanical characterizations of the films were performed in order to better understand the effect of film properties on further downstream processes.

2 Methodology

Two samples of polyester laminate PET/EVA-PE, taken from different batches of blown film roll-stock, were used in this study. These films have an average basis weight of 67 g/m^2 and an overall thickness of around 63 μm. The layer distribution was in the region of 12 μm BOPET and 51 μm EVA-PE. Both films were extruded from the same production line with film A having a BUR of 1.3 and film B at 2.6.

Various characterization methods were used to identify the film properties.

a. **ATR-IR analysis**. Attenuated Total Reflection Infra Red (ATR-IR) test was performed using a Perkin Elmer Spectrum 1 ATR-IR with single bounce diamond crystal at 4000 – 600 cm^{-1} wavelength, 64 scans, 4 cm^{-1} resolution.

b. **DSC analysis**. In order to study the effect of extrusion conditions on film crystallinity, thermal characterization was performed using Perkin Elmer DSC-6

Modular Thermal Analyzer. In this analysis, 7 mg of samples were heated from 30˚C to 270˚C at a temperature scan rate of 10˚C/min.

c. **Tensile and shrinkage tests**. Tensile properties of the various films were measured according to the tensile test procedure in ISO 527. An Instron 4411 with a 5kN load cell was used to perform the measurements at an ambient room temperature of 22˚C. The crosshead speed was set at 300 mm/min with a gauge length of 50 mm. A minimum of ten specimens were tested for each condition in both machine and transverse directions. Shrinkage test was performed at 130˚C for 75 seconds on samples with diameter of 50 mm.

d. **Seal strength analysis**. Samples of various films were heat sealed using a Sencorp heat sealer model 12AS/2 along the transverse direction at a pressure of 60 psi, at a dwell time of 1.0 sec and various sealing temperatures from 90˚C to 170˚C. After sealing, the samples were removed from the heat sealer and allowed to cool under ambient conditions for at least 1 hour prior to testing. Subsequently the film heat seal performance was investigated using a T-peel test according to ASTM F88 using a Testometric Micro 350 at crosshead speed of 300 mm/min. A force vs. extension curve was obtained and the seal strength was defined as the force per unit width value obtained at displacement of 25 mm.

3 Results

Film samples were characterized and analysed using shrinkage and tensile properties of the films and the results are summarized in Table 1. The raw force-elongation behavior for both materials is displayed in Figure 1 and Figure 2 respectively. The results show that for both film samples, shrinkage in machine direction (MD) is higher than shrinkage in transverse direction (TD) due to preferential molecular orientation in the MD induced during blown film processing. In MD, Film A exhibited higher shrinkage than Film B. In TD, shrinkage was observed in Film B while expansion was observed in Film A instead.

		Film A	**Film B**
Shrinkage (%)	MD	84 ± 0.02	80 ± 0.00
	TD	-6 ± 0.17	7 ± 0.06
Tensile strength (N/mm^2)	MD	61.3 ± 1.0	52.2 ± 2.9
	TD	53.9 ± 2.5	58.5 ± 2.3

Table 1. Shrinkage and tensile properties of films with various BUR.

A similar trend was recorded for the film tensile properties. These results show that for the tensile strength and stress at break, Film A exhibited higher values in MD but lower in TD. These differences in tensile properties can be directly related to the increase in crystalline orientation along the MD axis with less orientation in the TD axis. The opposite pattern was observed for the strain at break values.

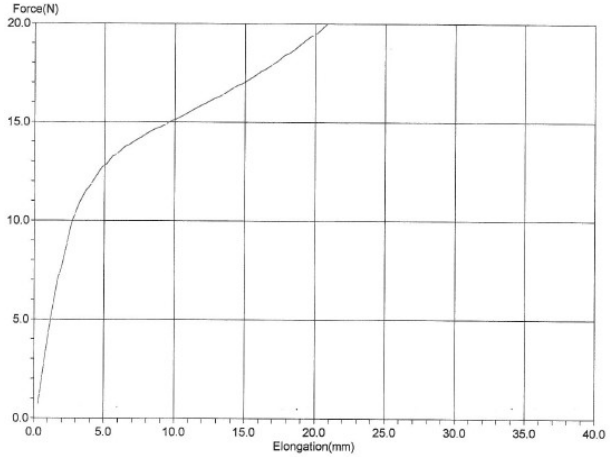


Figure 1. Force-elongation properties of low BUR Film A.

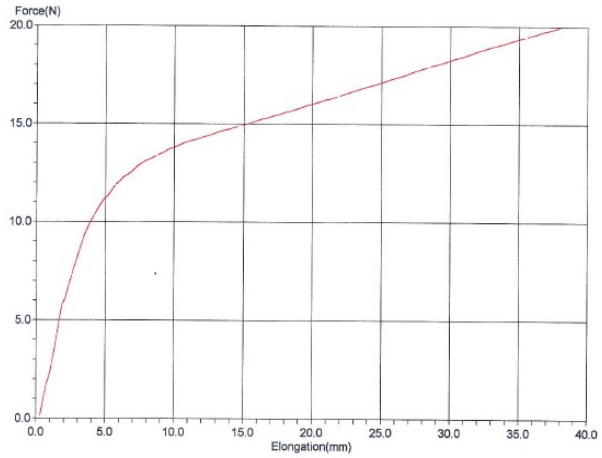


Figure 2. Force-elongation properties of high BUR Film B.

In order to investigate these effects in greater detail, ATR-IR analysis was performed on the various films in order to determine the degree of molecular orientation. The results are shown in Figure 3. These spectra show the characteristic IR absorbance bands associated with polyethylene. The difference between the two films was observed on the peaks at wave number 730 cm^{-1} and 718 cm^{-1}, which have been shown to represent elements within the chain structure whose transition moment are parallel to the crystalline a-axis and b-axis respectively **[6]**. Krishnaswamy **[5]** reported that on blown film this orientation shows good correlation between blow-up ratio and haul-off rates. The spectra of Film A show the appearance of peak at 730 cm^{-1} and slightly higher intensity peak at 718 cm^{-1}, meaning that there is more a-axis and b-axis orientation in this film. More optimized orientation study is necessary to fully understand these differences.

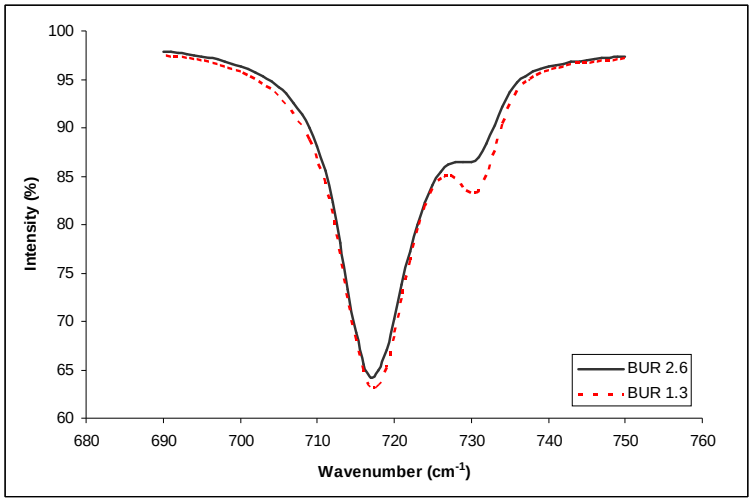


Figure 3. ATR-IR spectra for low (1.3) and high (2.6) BUR. Note the peaks at 730cm^{-1} and 718cm^{-1}.

DSC thermograms of the two films also show some significant differences. Figure 4 and Figure 5 show DSC thermograms for Film A and Film B respectively. Although the same melting temperature was observed for both films occurring at 104˚C, the overall shape of the endotherm peaks was different for the two films. The endotherm for Film A was lower and wider with a larger, additional shoulder being observed at the higher temperatures. Higher enthalpy value was also recorded for Film A, implying increased crystallinity. These findings from DSC analysis would also tend to confirm the ATR-IR analysis. Thus, variability during the blown film process could alter the chain orientation and result in changes to the crystalline structure of the films.

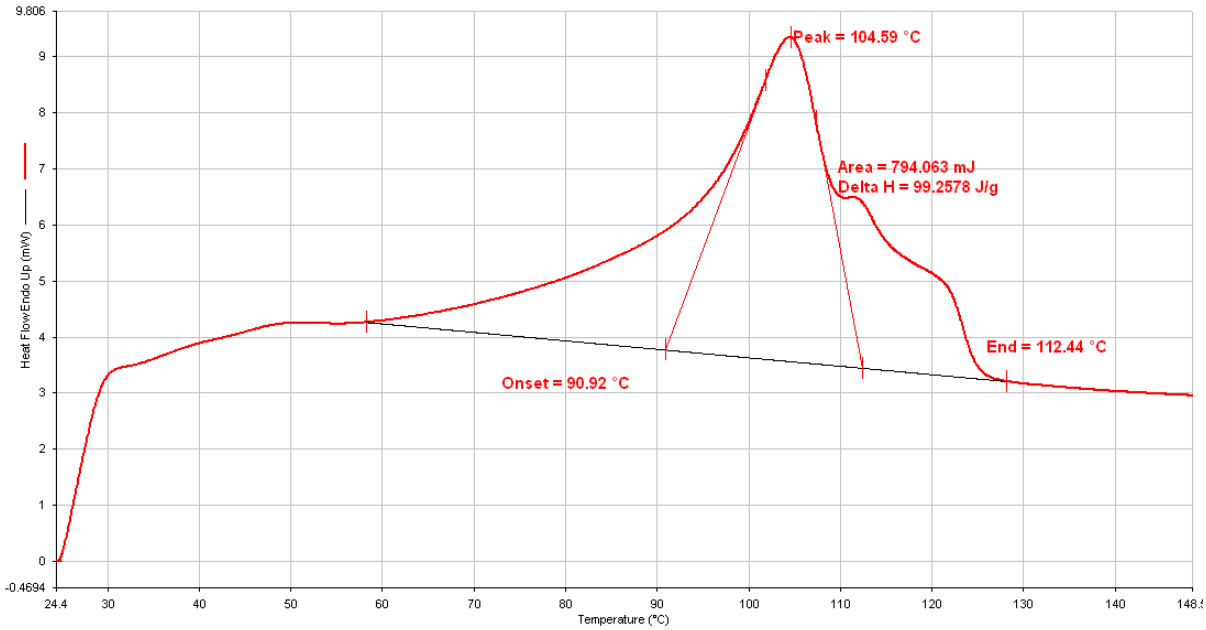


Figure 4. DSC thermogram for Film A.

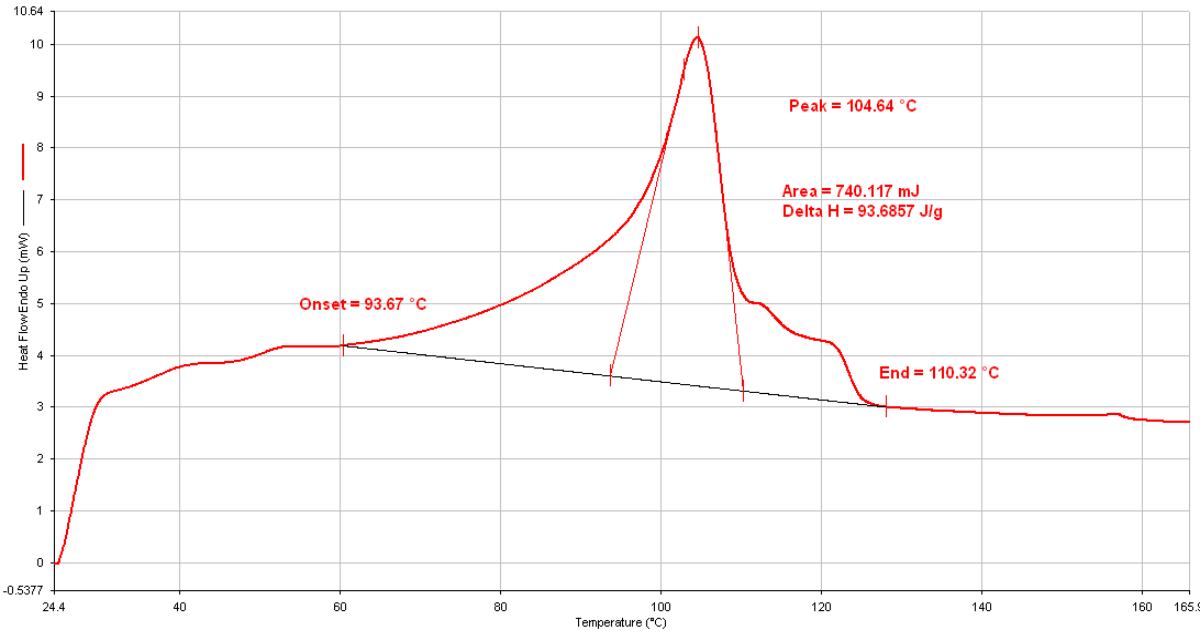


Figure 5. DSC thermogram of Film B.

Table 2 shows the seal strength for both films at various sealing temperatures. The results show that as the sealing temperature increases, the seal strength should increase due to intermolecular diffusion between chains at elevated temperature. However, comparing the two films, the seal strength of Film A was observed to be consistently higher and a greater difference was recorded at the elevated temperatures. This may be explained by the DSC analysis thermograms. The thermogram for Film A showed a lower onset to melting temperature (by 4˚C), compared to Film B. Therefore, as the temperature increased further, this would enable comparatively more molecular chain movements for Film A which would result in more intermolecular diffusion and a stronger seal **[7]**. In addition, the higher TD shrinkage for Film B would lead to excessive thinning of the film in the high temperature environment around the sealing jaws and may explain the weaker seal strength.

Seal temperature (°C)	**Seal strength (N/25mm)**	
	Film A	**Film B**
90	0.5	0.4
100	10.1	9.2
120	56.4	54.3
140	60.8	55
160	61.2	52.2
170	56.3	50.8

Table 2. Seal strength of films of various BUR at various sealing bar temperatures.

4 Discussion

The laboratory-based material characterisation in this study demonstrates a clear relationship between upstream processing parameters, specifically the Blow-Up Ratio (BUR), and downstream mechanical performance in sterile barrier laminates.

The structural and thermal deviations observed between Film A (BUR 1.3) and Film B (BUR 2.6) originate in the macromolecular physics of the blown film line. In blown film extrusion, the polymer melt experiences simultaneous longitudinal drawing (from the haul-off roll) and transverse stretching (from bubble inflation). At a low BUR of 1.3, the transverse stretching forces are minimized, causing the polymer chains to orient preferentially along the machine direction (MD) axis. This anisotropic orientation is validated by the ATR-IR spectra, which show elevated intensities for the crystalline a-axis and b-axis transition moments in Film A. This preferential alignment is responsible for Film A's significantly higher tensile strength in the MD (61.3 N/mm^2) relative to Film B (52.2 N/mm^2).

This crystalline orientation then dictates the thermal properties and heat sealability of the film structure. The DSC thermograms reveal that Film A possesses an expanded endothermic shoulder and higher melting enthalpy, indicating an increased overall level of crystallinity. However, its crystalline network undergoes a lower onset of melting (90.92°C) compared to Film B (93.67°C). This lower thermal onset facilitates accelerated molecular chain mobility at equivalent sealing temperatures, promoting deeper chain interdiffusion across the sealing interface and yielding consistently higher seal strengths across all tested temperatures above 90°C.

Conversely, Film B’s higher TD shrinkage (7%) introduces a structural liability inside the sealing equipment. When clamped between the high-temperature sealing jaws, the transverse relaxation forces cause local material thinning, reducing the cross-sectional mass of the seal zone and structurally compromising its load-bearing capacity. This demonstrates that optimizing seal performance cannot be achieved merely by adjusting jaw temperature or dwell time; it requires precise control of the structure embedded during the initial film extrusion stage.

Other studies have reported similar findings on the effect that orientation has on the mechanical performance of polymeric films [8, 9] and how mechanical properties influence the heat sealability of films [10].

5 Conclusion

This study confirms that variation in the blow-up ratio during upstream extrusion introduces significant variations in the crystalline morphology, tensile profiles, and heat-sealing integrity of PET/EVA-PE sterile barrier films. A low BUR of 1.3 optimizes MD mechanical properties and lowers the melting onset temperature, allowing enhanced chain interdiffusion and superior heat-seal strength compared to a higher BUR of 2.6.

For downstream packaging operations, these findings highlight the necessity of strict upstream process controls to stabilize mechanical tolerances and safeguard seal integrity. Future work could evaluate a broader range of BUR settings and investigate the long-term creep behaviour of the resulting seals under accelerated aging conditions.

Acknowledgments

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