

Flexible Packaging Division

Society of Plastics Engineers

June, 2015

Division 44

Building on the success of their 2012 experience, SPE and SPI co-operated to co-locate the National Plastics Exposition (NPE) with ANTEC March 23-27. More than 60,000 individuals representing 20,000 companies gathered in Orlando, Fla. to meet, connect and do business within our \$374-billion industry

On the following pages the papers selected as "best" by the division board are presented.

Those winning presenters are Dan Falla (Nova) and Patrick Thomas (PRThomas Technology)

The full list of presenters for the division's ANTEC sessions appears on the following page.

FlexPackCon 2015

The success of another partnership will produce a repeat performance: FlexPackCon 2014 convened with the AIMCAL Fall Technical Conference. This year, the Division will also co-host FlexPackCon 2015 with this very successful industry trade Association.

The conference will be held October 25-28 Naples FL at the Naples Grande Beach Resort. A variety of short courses and concurrent and technical sessions will complement opportunities for the attendees to network with industry experts and speakers and instructors from the sessions.

Conference Topics include:

- Package Forming & Converting
- Food Safety & Preservation
- Enhanced Shelf Presence
- Package Functionality & Consumer Benefits
- Material Advances and Their Benefits
- Sustainable Solutions

We are still working out details of the final technical program. Sponsorship exhibit details from da-vid.constant@basf.com ...Program details will come from chris.kersch@us.aschulman.com when they re available.

The Flexible Packaging Division was well represented at ANTEC 2015 with 10 papers presented at 2 sessions

Division Leadership ***Your board works for you!***

The following volunteers have been selected to the division leadership positions for the 2015-2016 term:

Officers

Chair: Paul Zerfas* Secretary: Wayne Morris*
Treasurer: John Wagner* Counselor: Dan Falla*

Board members

Henk Blom Andy Christie Jim Huang Barry Morris

Committee chairs

Membership chair Dan Falla*
FlexPackCon chair Donna Davis*
FlexPackCon program chair Chris Kersch*
Exhibitor/Sponsor chair Dave Constant*
Newsletter Editor Tom Dunn*

(* Also Board Members)

John Wagner received the SPE Honored Service Member Award at ANTEC, 2015. He was recognized for his voluntary contributions to the Flexible Packaging Division and the Extrusion Division of the Society. John currently serves as treasurer for the Flexible Packaging Division. He has authored and edited several books for the industry, he also serves as editor-in-chief of the *Journal of Plastic Film & Sheeting*.



FLEXIBLE PACKAGING DIVISION PAPERS PRESENTED AT ANTEC, 2015		
PEELABLE SEAL FILMS WITH ENHANCED MOISTURE BARRIER PROPERTIES FOR FLEXIBLE PACKAGING APPLICATIONS	Dan Falla	Nova Chemicals
PLASTIC PACKAGING MODELING: INTERACTIONS WITH FOOD. MODEL AND METHOD TO ESTIMATE THE SHELF LIFE OF OXYGEN-SENSITIVE FOOD PRODUCTS	Iván López	ICIPC*
LLDPE-EVOH HIGH BARRIER BLEND FILMS FABRICATED BY MULTIPLICATION EXTRUSION	Guojun Zhang	A. Schulman Inc
INFLUENCE OF A SUBSTRATE BIAS ON THE ADHESION OF SILICON ORGANIC PECVD-FILMS ON POLYPROPYLENE	Henrik Behm	Institute of Plastics Processing
CATALYTIC TECHNOLOGY AND CONTROLLED CHEMICAL RELEASE FOR POST-HARVEST PRESERVATION OF FRUITS AND VEGETABLES	Rajan Raje	Lifeline Technologies
STUDY OF SEALED PARTS OF FLUORINE FILM BY LASER ADVANCED WELDING OF PLASTICS METHOD	Kazushi Yamada	Kyoto Institute of Technology
PACKAGING AND FOOD INTERACTIONS MODELING: VALIDATION FOR COMPLIANCE WITH SPECIFIC MIGRATION REGULATIONS	Juan Estefan	ICIPC*
IMPACT PUNCTURE RESISTANCE OF MULTILAYER FLEXIBLE FOOD PACKAGES	Barry Morris	DuPont
HIGH PERFORMANCE PP/PE MULTILAYER FILMS ENABLED BY PP BASED OBC	Yushan Hu	The Dow Chemical Company
CORRELATING THE MELTING OF SEMI-CRYSTALLINE POLYMERS TO THE SHRINK WRAPPING PROCESS IN SHRINK-FILM PACKAGING APPLICATIONS	Bernard Obi	The Dow Chemical Company
UNDERSTANDING BLOWN POLYETHYLENE FILM DART STRENGTH VARIABILITY	Paul OConnell	Dow Chemical
PREDICTING PHYSICAL AND OPTICAL PROPERTIES OF CO-EXTRUDED BLOWN FILMS USING DESIGN OF EXPERIMENT BASED MODEL	Nitin Borse	NOVA Chemicals
LASER IMAGEABLE POLYMERIC FILM	Patrick Thomas	PRThomas Technologies. LLC
*Instituto de Capacitación e Investigación del Plástico y del Caucho, Medellín - Colombia		

Best paper selections by Dan Falla and Patrick Thomas follow:

PEELABLE SEAL FILMS WITH ENHANCED MOISTURE BARRIER PROPERTIES FOR FLEXIBLE PACKAGING APPLICATIONS

Dan Falla, NOVA Chemicals, Sarnia, Ontario, Canada

Abstract

Most cereal and cracker packages are designed to have the seal layer peel apart while being opened. Unfortunately, opening these packages often results in catastrophic failure of the film and spillage of the contents. In this study, various types of peelable seal layers were investigated. In addition, a new high moisture barrier sHDPE was evaluated.

2.0 Background

Peelable Seals

Many consumer foods require the package to be easy-open in order to access the contents. Unfortunately, consumer experience has shown that many products sold in “easy-open” packages are actually difficult to open and often result in the catastrophic destruction of the bag. Consumer Reports magazine (1) rated cereal bags as one of the five worst consumer packages due to problems with opening. The article showed that 75% of the bags tested tore while their examiner attempted to peel the bags open.

The need for an easy-open, peelable seal package has been studied previously. Various polymer technologies have been reported in the literature (2)(3)(4)(5)(6)(7). There are three generally accepted easy open, peelable seal failure modes: interfacial separation, delamination, and cohesive failure (4) (Figure 1-4).

- **Interfacial Separation:** The separation occurs at the seal interface. The seal strength is dependent on the sealing temperature
- **Delamination:** The seal separates at an internal interface (between internal layers or between the inside layers). Sealing temperature is one of the significant variables in determining seal strengths; however, the thickness of the internal layer and adhesion between the internal layers also play major roles.

- **Cohesive Failure:** The structure separates within the seal layer. The peel seal material’s inherent strength determines the strength of the seal.

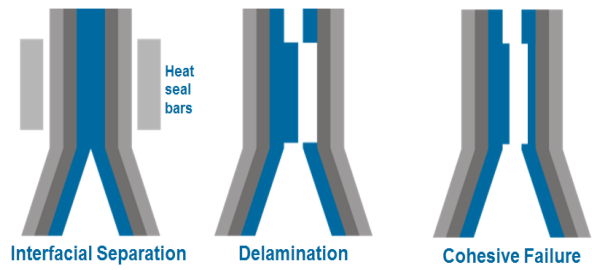


Figure 1: Fundamentals of Peelable Sealing (4)

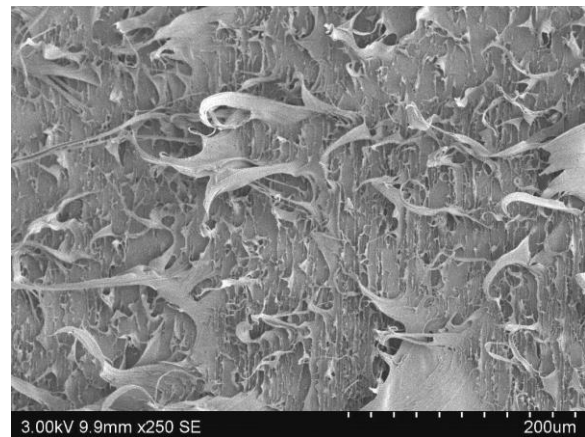


Figure 2: Interfacial Separation

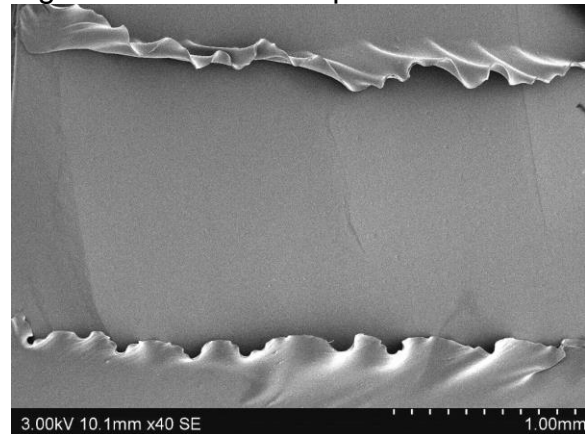


Figure 3: Delamination

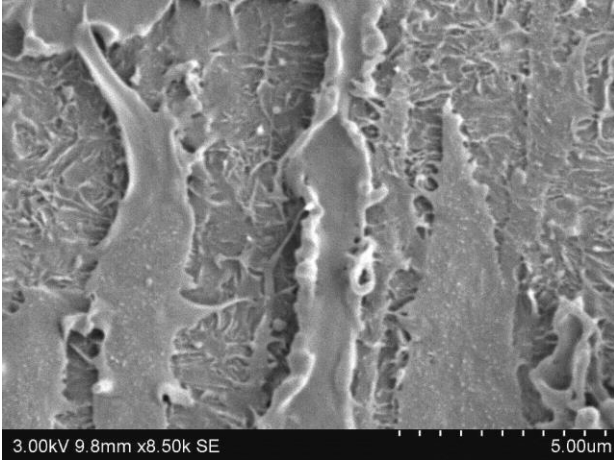


Figure 4: Cohesive Failure

Is this study films having peelable seal properties were studied along with an sHDPE having excellent moisture resistant properties. As previously stated, cohesive peel seal systems peel within the film layer while delamination fails at the internal interface. The cohesive seal results in a smooth, clean peel surface and offers “whitening” of the seal with added built in tamper evidence (5)(8) (Figure 5).

Peel mechanism: Cohesive Failure

Desire is to have system that peels **within** the film structure. This even applies to sealing to a PE or PP layer.

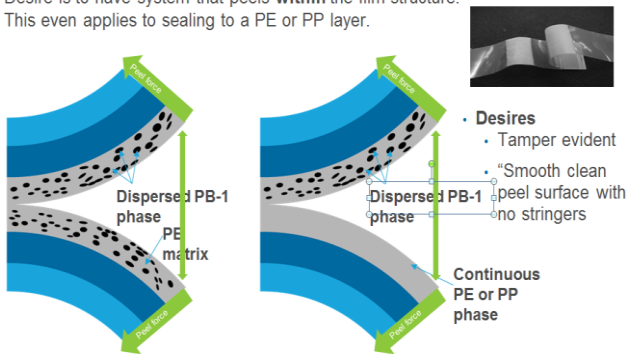


Figure 5: PB-1 Peel mechanism: Cohesive Failure

Three types of polymeric resin systems were used for peelable seal applications in this paper: polybutene-1 (PB-1), commercial ethylene propylene peel polymers (EP) and an ionomer specific to peel seal applications. The following is a brief background describing the performance of each of these resins.

Polybutene-1

PB-1 is a semi-crystalline, highly isotactic thermoplastic made from the polymerization of butene-1 using a Ziegler-Natta type catalyst. It works by being incompatible with polyethylene. In the seal layer, it creates a layer with PB-1 islands in the PE matrix as per Figure 5 & 6 and results in cohesive failure of the seal.

PB-1 is typically blended with 55% LDPE and 30% low seal initiation LLDPE. The PB-1 has more incompatibility with the LDPE than the LLDPE, hence the LLDPE should be chosen based on economics and seal initiation temperature desired. Slip and antiblock may be also required.

PB-1 makes flexible packaging “easy-open”

The Peelable concept is based up on the incompatibility between PB-1 and PE which causes a fracture intralayer

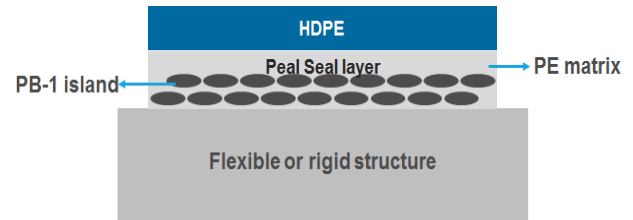


Figure 6: PB1 -How it works (5)

Ethylene Propylene Peel Polymers

The ethylene propylene peel polymer (EP) is a fully formulated copolymer (EP) that is commercially available. Seal failure may occur in a few ways: At lower temperatures, interfacial separation occurs at the seal / seal interface. At higher temperatures, the two seal layers “lock up” and failure occurs when the seal delaminates from the core layers. The strength of the bond at the HDPE / seal interface is significantly high enough that care must be taken on the packaging line to use enough heat to “just seal” the package.

Ionomer

Ionomers, are partially crosslinked acid copolymer resins. Ionomers can be formulated with PB-1 to fail cohesively or used alone in a delamination style peel seal where failure occurs between the ionomer sealant and the HDPE layer (8). Much like the EP peel polymer, care must be taken to not seal at too

high of temperature as seal lock-up is expected to occur.

Low Moisture Vapour Transmission Rate (MVTR) Film

A film that has low MVTR is a requirement for dry good packages, such as cereal and crackers, in order to maximize the shelf life of the product. Most cereal and cracker films are three-layer coextruded blown films that are comprised of a peelable seal layer and an HDPE core layer. The thickness of the HDPE typically determines the MVTR of the film. In this study, the MVTR properties of a high moisture barrier sHDPE (sHDPE) are compared to a conventional HDPE.

This study was done to:

1. Evaluate a number of different peelable seal formulations
2. Evaluate high moisture barrier sHDPE vs standard HDPE for moisture resistance

3.0 EXPERIMENTAL METHODS

Previous studies have limited the qualitative measurement of peelability to the laboratory cold seal strength test. Traditionally, 25.4mm (1”) wide samples were cut from the film and heat sealed in the transverse (TD) direction. The sealed samples were then pulled using a tensile tester to determine the seal strength vs. seal temperature. In this study, we used a combination of heat sealed film, hottack strength, and performance in a vertical form fill and seal (VFFS) machine.

Film Fabrication

Table 1 shows the resins used to make the 12 film structures shown in Table 2 (in Appendix). The films were fabricated using the 3 layer Brampton Coex line at NOVA Chemicals Centre for Performance Applications. The extrusion conditions were: 2.5:1 BUR, 10 cm (4”) die, 35-mil die gap, 45 kg/h (100 lbs/h) output rate, standard temperature profile, barrier screw with Maddock mixing head.

Name	Type	Melt Index dg/min	Density g/cc
PB-1	Polybutene-1	28	0.906
LDPE	LDPE	0.75	0.920
LLDPE	C8-LLDPE	1.00	0.920
sLLDPE	C-8 Single Site LLDPE	0.65	0.916
EP	EP copolymer	3.80	0.895
Ionomer	Zn - Ionomer	4.00	0.940
sHDPE	barrier HDPE	1.20	0.966
HDPE	HDPE	0.95	0.958

Table 1: Resins Used

All physical properties were tested at NOVA Chemicals Centre for Performance Applications. Test results were converted into absolute values for the purpose of comparing films of different thicknesses.

The films were subjected to the following physical tests to determine their various properties:

- MVTR, using Mocon test unit 100% RH
- Dart Impact, using ASTM D1709, Method A;
- Film Tear, using ASTM D1922;
- Puncture, using NOVA Chemicals Method
- 1% and 2% Secant Modulus, using ASTM D882;
- Tensiles, using ASTM D882;
- Hot Tack Strength; using method described below;
- Heat Seal Strength, using method described below;
- Vertical Form Fill and Seal (VFFS) described below;

Hot Tack Strength Test Method

The hot tack strength of the sample films were measured using the “J&B Hot Tack test method” which measures the force required to separate a heat seal before the seal has had a chance to fully cool. This simulates the filling of material into a pouch or bag before the seal has had a chance to cool.

The “J&B Hot Tack test method” used the following conditions:

Specimen Width:	25.4 mm
Sealing Time:	0.5 seconds
Sealing Pressure:	0.27 /mm/mm
Delay Time:	0.5 seconds
Peel Speed:	200 mm/seconds

Number of Samples/Temperature 5
Temperature Increments: 5°C
Temperature Range: 75°C - 150°C

Film Heat Seal Strength Test Method

The heat seal strength of the sample films measures the force required to separate a seal after the material has cooled to 23°C. In this study, the sealed samples were allowed to age for two weeks prior to testing. Again is a significant issue with some peel seal technologies, especially PB-1 blends.

Samples were tested using the following conditions:

Specimen Width: 25.4 mm
Sealing Time: 0.5 seconds
Sealing Pressure: 0.27 /mm/mm
Number of Samples/Temperature 5
Temperature Increments: 5°C
Temperature Range: 75°C - 150°C

The seal strengths were then determined using a Tensile Tester model according to the following test conditions:

Direction of Pull: 90° to seal
Crosshead Speed: 300 mm/minute
Full Scale Load: 5 kg

Vertical Film Form and Seal (VFFS)

The Rovema VFFS machine used in this study was a model VPI 260 with a Bozn DCS-3BC Auger Filler. It had a 165 mm forming set and Teflon® tape on both the vertical and cross sealing bars (Figure 7).



Figure 7: Rovema Vertical Form Fill and Seal Machine

19 cm x 16.5 cm pouches (seal to seal) were produced with no gusseting. The pouches were filled with 500 g of HDPE pellets to simulate the packaging of product in commercial applications. The 500 g of pellets only half filled the pouches so that they could be tested in the leak detector test.

A temperature window was selected using the hand squeeze method described below. All films were run from low temperature (where the seal easily opens) to a temperature where burn through occurred. Pellet filled pouches were submitted for leak testing while other pouches were emptied of the pellets aged for two weeks. Sample strips 24.5 mm (1”) were cut from the seal area and tested using a tensile tester.

Haug Pack-Vac Leak Detector Test

The purpose of this test is to evaluate the seal strength and general packaging integrity of pouches produced on the VFFS. A pouch was placed underwater in the leak detector tank and a vacuum was applied. If the seals fail then air from inside the pouch escapes through the failure and bubbles are noticed in the tank. Results from this test can be used to compare and rank different film structures at different equipment seal bar settings. Testing conditions were:

- Pouch volume filled 50% with pellets
- Line speed 20 bags / minute
- Replicates per temperature – 5
- Once good seals were achieved temperature increments were adjusted by 10°C increments
- Vacuum setting 20 inHg
- Hold for 30 seconds once targeted vacuum setting is reached

The Figure 8 & 9 illustrates pouches that have seal failure and burn through.

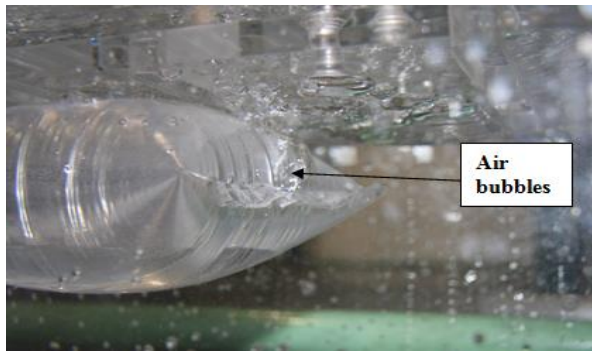


Figure 8: Seal Failure



Figure 9: Burn Through

Drop Test

10 pellet-filled pouches were made at each temperature and then dropped from a height of 1.8m (6') to simulate the packaging operation.

Compression Test

Five air-filled pouches were made at each temperature then tested under compression using a tensile tester to determine the load that the end seal failed at.

Pouch End Seal Strength

Five pellet-filled pouch samples were made at each temperature. The pellets were emptied and 25.4mm (1") wide samples were cut from the same seal region on each pouch and pulled using an Instru-Met 5-Head tensile tester.

4.0 RESULTS AND DISCUSSION

1) Commercial Film

Prior to the start of the project, samples of a commercial cereal liner were purchased at a local grocery store and tested to determine typical film properties. The analysis (Figure 10-12) indicates

that the film was a three layer co-ex with EP peel polymer in the seal layer and two HDPE layers. Physical properties of the film have been added to analysis of this study (Table 3). No VFFS work was done with the competitive due to the small sample size.

The heat seal curve of the film shows the classic curve expected for EP peel polymers. Note the dramatic increase in seal strength at the higher temperatures as the seal "locks up" into cohesive failure (Figure 13).

2) Fabricated Film Physical Properties

Table 3 and Figures 14 to 34 show the film physical properties, all of which can be found in the appendix.

Film Properties

1) MVTR (Figure 14)

The use of sHDPE was found to dramatically lower the MVTR of the films. As expected, thicker films were also found to have lower MVTR. The commercial film had MVTR properties similar to the films having conventional HDPE in the core.

2) Dart Impact (Figure 15)

Not enough sample was available to determine the dart impact properties of the commercial film.

The films with sHDPE in the core had slightly lower dart impact properties than films made with HDPE. Overall, films with the ionomer had the highest impact resistance.

3) Film Tear

3.1) TD Tear (Figure 16)

The films with EP peel polymer and ionomer had TD tear values that were less than half of the commercial film and films with PB-1. TD tear is not expected to play a significant role in this application.

The use of sHDPE resin did not appear to have an effect on the TD tear properties.

3.2) MD Tear (Figure 17)

In cereal bag applications, high machine direction (MD) tear resistance is a very desirable attribute

once the bag is opened. Figure 17 shows the MD tear for the films tested. All of the films tested had higher MD tear resistance than the commercial film. The ionomer films had the highest tear resistance, about double the commercial film.

4) Puncture (Figures 18 & 19)

There was not enough commercial film available to test puncture resistance. Overall, the films had similar puncture resistance when comparing normalized results

5) 1% and 2% Secant Modulus (Figures 20 & 21)

High film stiffness is a desirable attribute for these films for faster packaging speeds. Figures 20 and 21 show that films made with sHDPE in the core had higher 1% and 2% secant modulus. This was expected since the HDPE had a density of 0.958 g/cc vs. 0.966 g/cc for the sHDPE. The results were almost 20% higher than the commercial film. Higher stiffness could allow the film to be downgaged while still maintaining stiffness relative to the competitive film. It may also allow the packaging line to run at a faster rate.

6) Tensile (Figure 22 to 24)

No significant differences were seen with the tensile properties except the 2.3 mil commercial film had lower elongation than the other films tested.

7) Hot Tack (Figure 25)

Three distinct groupings were seen in the testing. The ionomer films had the lowest hot tack initiation temperature along with a broad, flat profile. EP films initiation temperature was only slightly higher than the Ionomer but rose quickly to the highest hot tack strength and the narrowest profile. PB-1 hot tack initiation temperature was approximately 25°C – 30°C higher than the other two types of sealant.

8) Film Heat Seal Strength (Figure 26)

Three distinct groupings were also seen with the films tested. The ionomer films had a sharp rise in seal strength in the interfacial separation phase followed by a drop in strength in the delamination phase. The maximum strength at higher temperatures was lower than the commercial films which appeared to “lock-up” beyond 130°C. EP

film had a wide, flat sealing profile. Testing was stopped at 155°C.

PB-1 had the highest sealing initiation temperature of the films tested and the narrowest sealing window. The seal strength continued to increase as the sealing temperatures increased. Unlike the other films, no plateau was observed.

As expected, the PB-1 films made with higher levels of LDPE had lower overall seal strengths. As previously stated, LDPE is more incompatible with the PB-1 than LLDPE.

VFFS

1) Hand Squeeze Test

In this study, the VFFS processing window was determined by hand squeezing the pouches. The seal initiation temperature (SIT) was defined first by the dry fill staying in the pouch once the seal bars opened, then the pouches were lightly squeezed by hand. The SIT was defined when the pouches retained internal pressure (no sign of leaks). The end processing temperature was determined by hand squeezing of the pouches again but this time, failure occurred when burn thru of the end seal was observed.

2) Haug Leak Tester

All pouches produced that retained the dry fill (pellets) passed at 10 in.Hg, but leaked at 13 to 15 in.Hg. This off-line test did not differentiate the different film structures and temperature profile (same results from initial seal to burn thru seal). The commercial cereal packages all failed at 5 in.Hg. A small individual sized cereal package failed @ 1 in.Hg.

Based on these results, the Haug test does not appear to be a reliable test for this application.

3) 6' Drop Test

The 6' drop test also did not provide any conclusive data to differentiate between film structures and temperature profile. Dry fill (pellets) were found to cause pin-holes failures on the side on the pouch when dropped giving a false positive.

4) Pouch End Seal Strength (Figures 27 to 32)

25.4mm (1") wide samples were cut from the end seals and allowed to age two weeks before testing. Similar to the film heat seal strength test, three groupings were also seen (Figure 27).

4.1) PB-1

Figure 28 depicts two different groupings of seal curves. The difference between the two groups is the amount of LDPE used in the blend. The higher heat seal strength grouping contained 30% LDPE vs 55% LDPE for the stronger seal group. The PB-1 producer reports that the amount LDPE plays a critical role the peel seal strength (5).

The PB-1 films all had higher heat seal initiation temperatures and lower, overall seal strength of the films tested.

4.2) Ionomer

Three distinct phases of sealing can be seen in Figure 29. At lower temperatures, interfacial separation occurred wherein the seal simply peeled apart. From about 95°C to about 130°C delamination occurred where the seal pulled away from the HDPE layer. Cohesive failure occurred from 130°C to 150°C.

4.3) EP Peel Polymer

The samples were tested after two weeks. The resin manufacturer claimed that with time, the seal strength decreases when the EP peel polymer is used in the seal layer (9). Figure 30 shows a classic seal curve with the 2.25 mil film having higher heat seal strength.

4.4) Comparison of Film Heat Seal Strength to VFFS Pouch Heat Seal Strength

Figures 31 & 32 compare the measured film heat seal strength to the measured strength of the pouch end seal. The figures show a significant correlation between the two. Heat seal initiation and burn through were almost identical. This indicates that the film heat seal strength test should be a suitable laboratory test method to predict performance in a VFFS unit.

5) Compression Test (Figure 33 to 36)

Five pouch samples from each temperature were tested under compression using a tensile tester.

Overall, the pouch seal performance under compression correlated with the pouch end seal testing.

5.1) PB-1 (Figure 34)

PB-1 seal initiation temperature was significantly higher than either the ionomer or EP peel polymers. Seal strength also continued to increase until cohesive failure was observed.

Lower compression strength was also observed with LDPE blends.

5.2) EP Peel Polymer (Figure 35)

The end seal strength with the EP peel polymer increased with temperature until cohesive failure occurred. In the cohesive failure region, the film was observed to fail vs. the seal peeling apart.

5.3) Ionomer (Figure 36)

Like the pouch end seal strength, the ionomer films showed three distinct regions; interfacial separation, delamination and cohesive failure (Figure 32). It was noted that during cohesive failure, the film failed vs. the seal peeling. The ionomer films had a very low and flat strength curve until cohesive failure.

5.0 CONCLUSIONS

- 1) The sHDPE significantly decreased the MVTR versus a conventional HDPE. It also resulted in films with the highest 1% and 2% secant modulus (20% higher than a commercial film).
- 2) Films made with ionomer in the seal layer had the best overall physical properties.
- 3) Ionomer was also found to have broadest peel seal window along with the lowest peel force.
- 4) EP peel polymers produced films with the highest hot tack strength.
- 5) Seal layer thickness did not appear to have a significant impact on the properties of the films tested.
- 6) The amount of LDPE blended with the PB-1 was found to affect the end seal strength of the films. Higher amount of LDPE resulted in lower seal strengths.

Overall, films made with either the Ionomer or EP Peel Polymer in the seal layer and a barrier HDPE

would produce an effective peelable seal film with enhanced barrier properties. Also, the higher film stiffness with a barrier sHDPE would allow downgauging of the film.

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	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9	Run 10	Run 11	Run 12
	1.75 PB-1 sHDPE 20% Skin	2.25 PB-1 HDPE 20% Skin	2.0 EP sHDPE 15% Skin	2.0 EP HDPE 15% Skin	2.25 EP HDPE 15% Skin	2.0 Ionomer sHDPE 15% Skin	2.0 Ionomer HDPE 15% Skin	2.25 Ionomer HDPE 15% Skin	2.0 10% PB-1 HDPE 20% Skin	2.0 15% PB-1 HDPE 20% Skin	2.0 Ionomer HDPE 10% Skin	2.0 EP + sHDPE LLDPE 15% Skin
Thickness, mil	1.8	2.3	2.0	2.0	2.3	2.0	2.0	2.3	2.0	2.0	2.0	2.0
Skin -												
PB-1	15%	15%							10	15		
LDPE	55%	55%							30	30		
sLLDPE	30%	30%							60	55		
EP			100%	100%	100%							100%
Ionomer						100%	100%	100%			100%	
Core												
sHDPE	100%		100%			100%						80%
HDPE		100%		100%	100%		100%	100%	100%	100%	100%	
LLDPE												20%
Skin												
HDPE	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%	100%

Table 2: Film Structures

PROPERTY	Commercial	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	Run 8	Run 9	Run 10	Run 11	Run 12
Thickness	2.3	1.8	2.0	2.0	2.3	2.0	2.0	2.3	2.0	2.0	2.0	2.0	2.0
UNITS													
BARRIER H2O FILM	0.205	0.087	0.199	0.195	0.187	0.0638	0.2066	0.1691	0.0602	0.216	0.1953	0.2051	0.079
FILM THICKNESS 2	2.3	1.6	2.0	2.1	2.2	2.0	2.0	2.4	2.0	2.1	2.2	2.0	2.0
Dart	Not Tested	37	44	42	39	23	44	46	36	59	55	58	27
Dart		64.8	88.0	84.0	87.8	46.0	88.0	103.5	72.0	118.0	123.8	116.0	54.0
1% Secant Mod MD	576	893	713	668	694	958	693	723	928	723	654	763	725
1% Secant Mod MD	0.91	1.08	0.98	0.92	1.08	1.32	0.96	1.12	1.28	1.00	1.01	1.05	1.00
2% Secant Mod MD	463	675	570	538	543	738	553	569	717	564	522	588	570
2% Secant Mod MD	0.73	0.81	0.79	0.74	0.84	1.02	0.76	0.88	0.99	0.78	0.81	0.81	0.79
1% Secant Mod TD	840	1152	904	869	886	1182	920	924	1117	874	868	923	883
1% Secant Mod TD	1.33	1.39	1.25	1.20	1.37	1.63	1.27	1.43	1.54	1.21	1.35	1.27	1.22
2% Secant Mod TD	671	892	707	692	689	911	722	725	867	675	672	728	700
2% Secant Mod TD	1.06	1.08	0.98	0.95	1.07	1.26	1.00	1.13	1.20	0.93	1.04	1.00	0.97
Tear MD	16	28	26	24	25	27	23	24	36	32	43	44	28
Tear MD	36.8	49.0	52.0	48.0	56.3	54.0	46.0	54.0	72.0	64.0	96.8	88.0	56.0
Tear TD	814	203	278	277	283	78	120	69	152	120	109	129	80
Tear TD	1872.2		556.0	554.0	636.8	156.0	240.0	155.3	304.0	240.0	245.3	258.0	160.0
Tensiel Break MD	42.5	32.3	42.2	41.4	38.2	35.6	40	44.3	34	36.7	39.8	40.9	36.7
Tensiel Break MD	0.067	0.039	0.058	0.057	0.059	0.049	0.055	0.069	0.047	0.051	0.062	0.056	0.051
Tensiel Elong MD	600	613	689	687	718	722	728	786	738	737	832	785	735
Tensiel Yield MD	20.2	26.8	23.6	23.3	22.9	26.8	23.8	24.4	27.8	24.3	23.2	24.9	24.7
Tensiel Yield MD	0.032	0.032	0.033	0.032	0.036	0.037	0.033	0.038	0.038	0.034	0.036	0.034	0.034
Tensiel Break TD	20.9	28.7	36.6	35.9	36.2	41.7	41.7	38.2	28.3	40	38.3	42.2	35
Tensiel Break TD	0.033	0.035	0.050	0.050	0.056		0.058	0.059	0.039	0.055	0.059	0.058	0.048
Tensiel Elong TD	459	751	786	795	855	873	873	835	755	848	829	871	837
Tensiel Yield TD	20.2	25.3	24.7	25.3	25.6	28.4	28.4	26.6	28.4	27.3	26.4	27.2	26.3
Tensiel Yield TD	0.032	0.031	0.034	0.035	0.040		0.039	0.041	0.039	0.038	0.041	0.038	0.036
Tensiel Break TD	23.4	25.3	24.7	25.3	25.6	28.4	28.4	26.6	28.4	27.3	26.4	27.2	26.3
Tensiel Break TD	0.037	0.031	0.034	0.035	0.040		0.039	0.041	0.039	0.038	0.041	0.038	0.036
Puncture	Not Tested	23	22	21	23	19	22	20	23	22	22	24	20
Puncture	40.3	44.0	44.0	42.0	51.8	38	44	45	46	44	49.5	48	40.0

Table 3: Physical Test Results

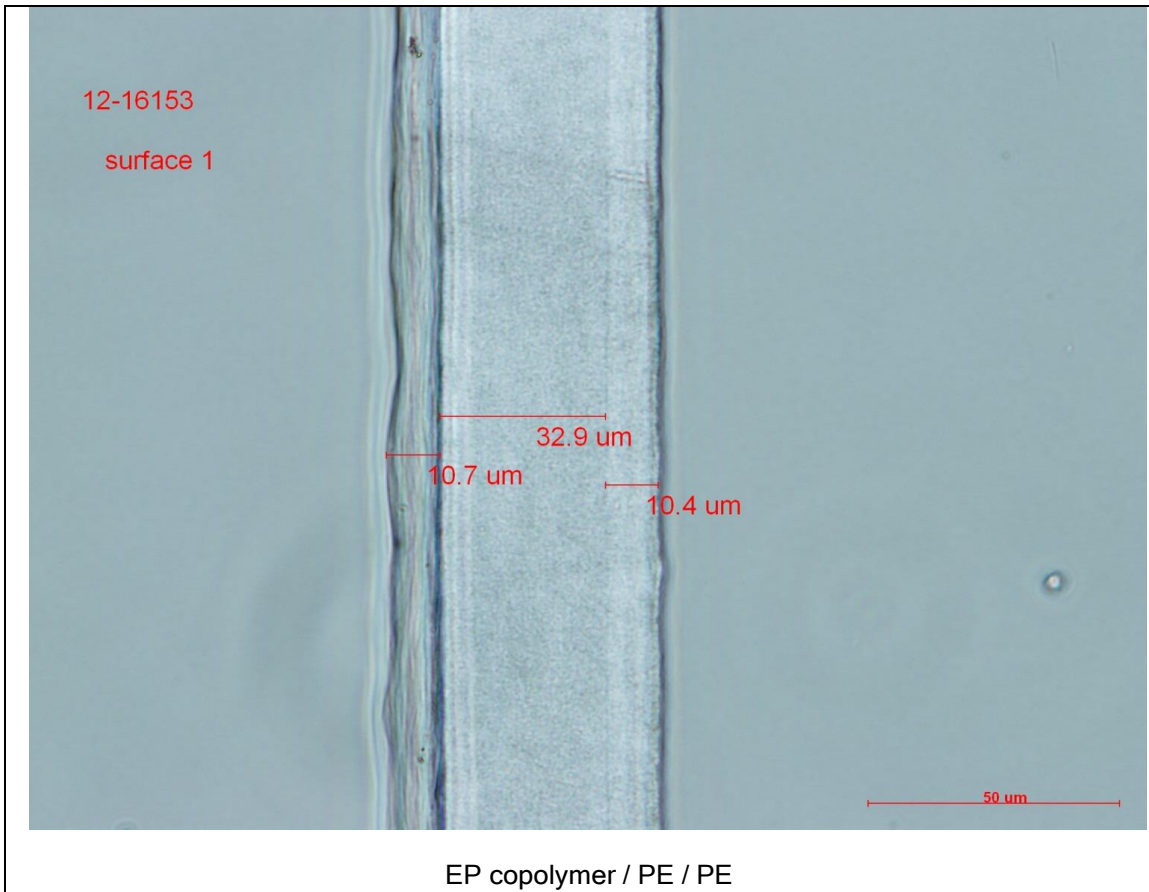


Figure 10: Commercial Cereal Film Micrograph

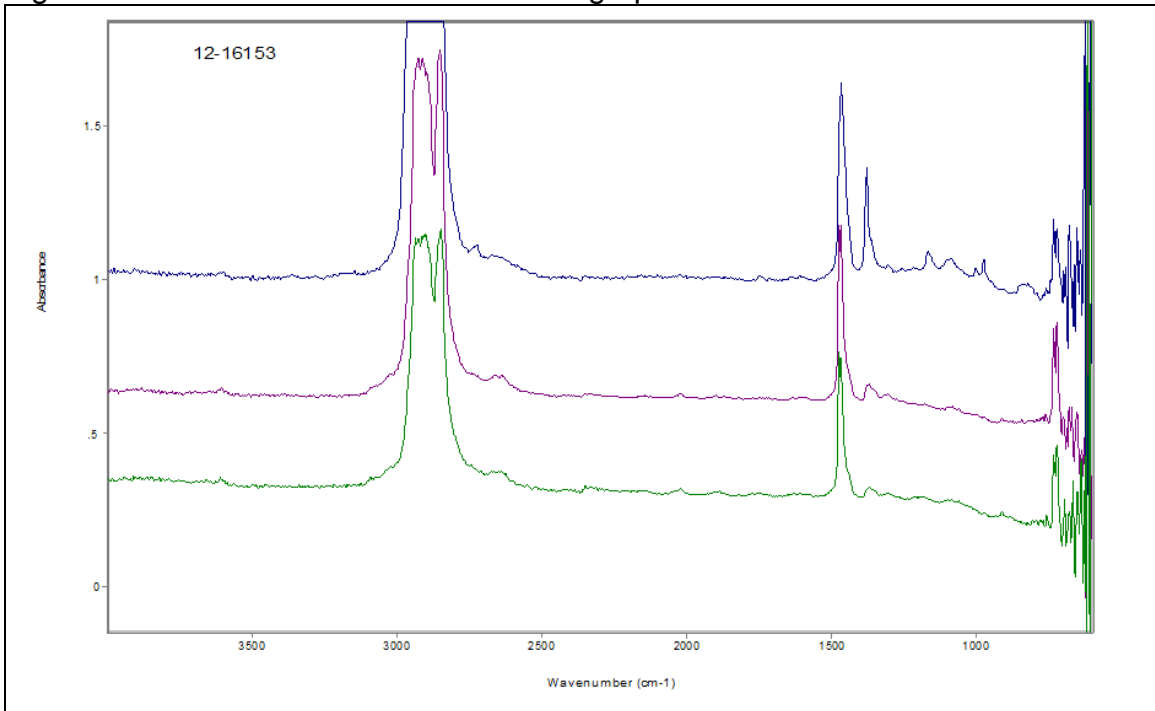


Figure 11: FTIR scan of all three layers indicating EP Peel Polymer in the seal layer

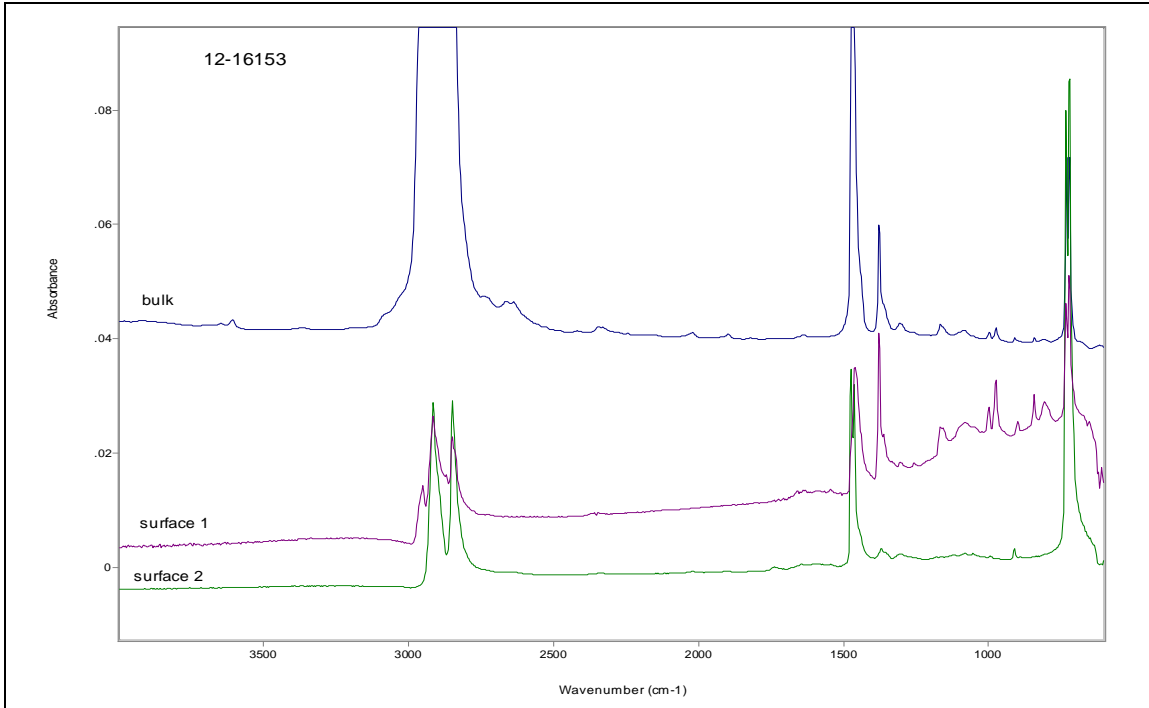


Figure 12: FTIR of seal layer

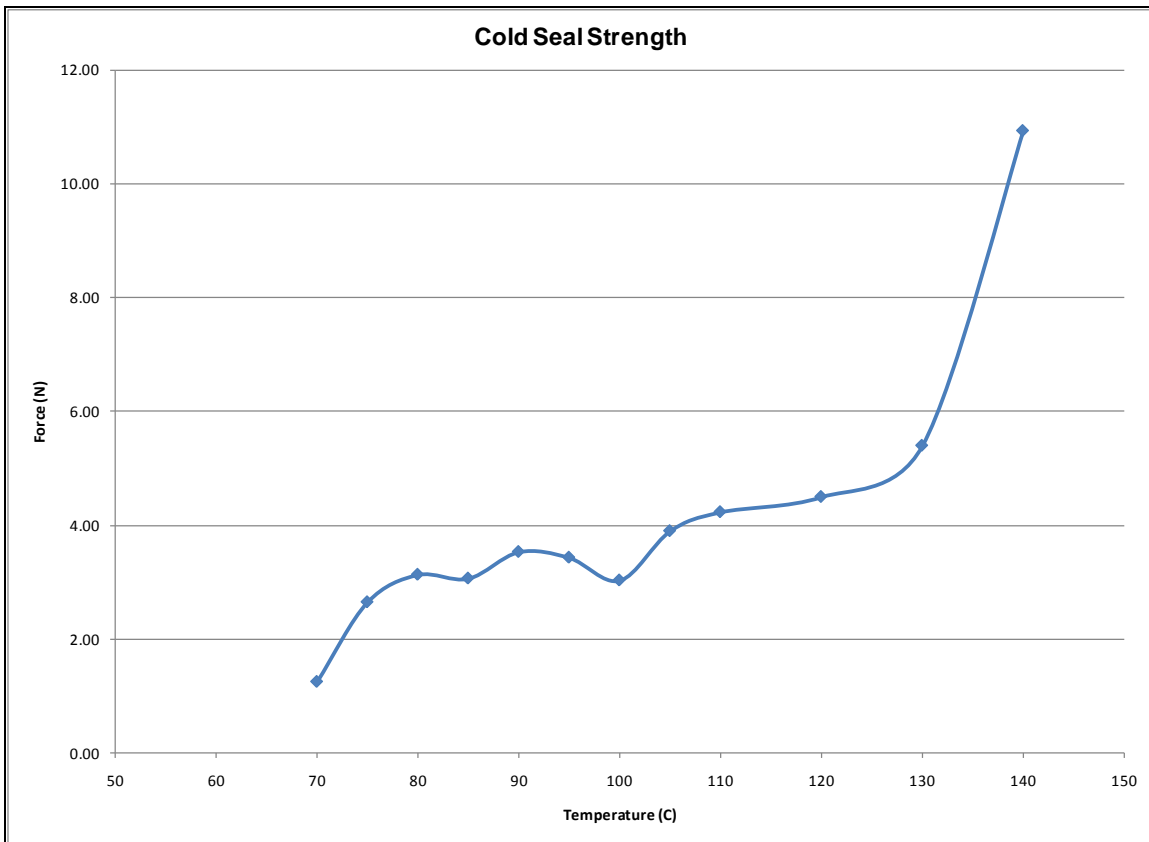


Figure 13: Commercial Cereal Film - Heat Seal Strength

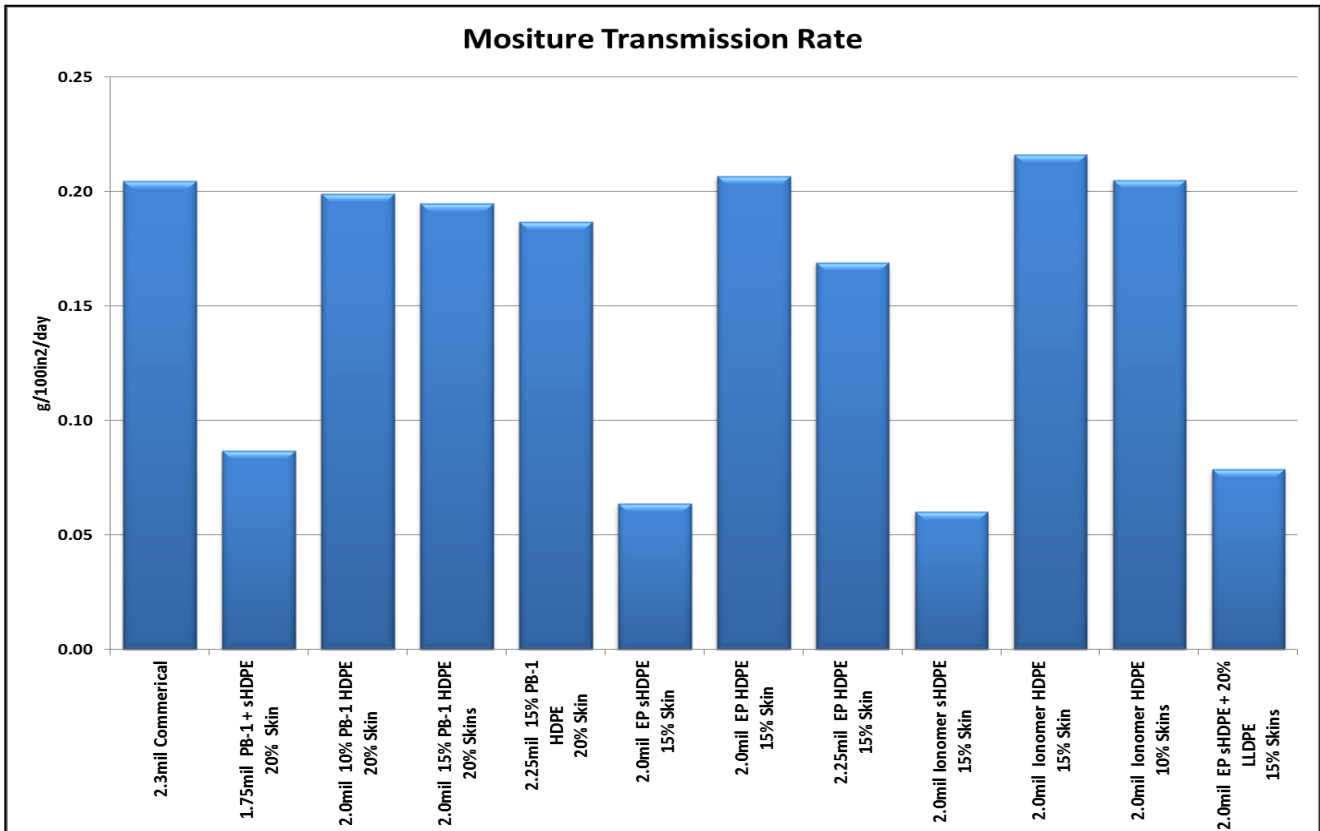


Figure 14: Mositure Vapour Transmission Rate

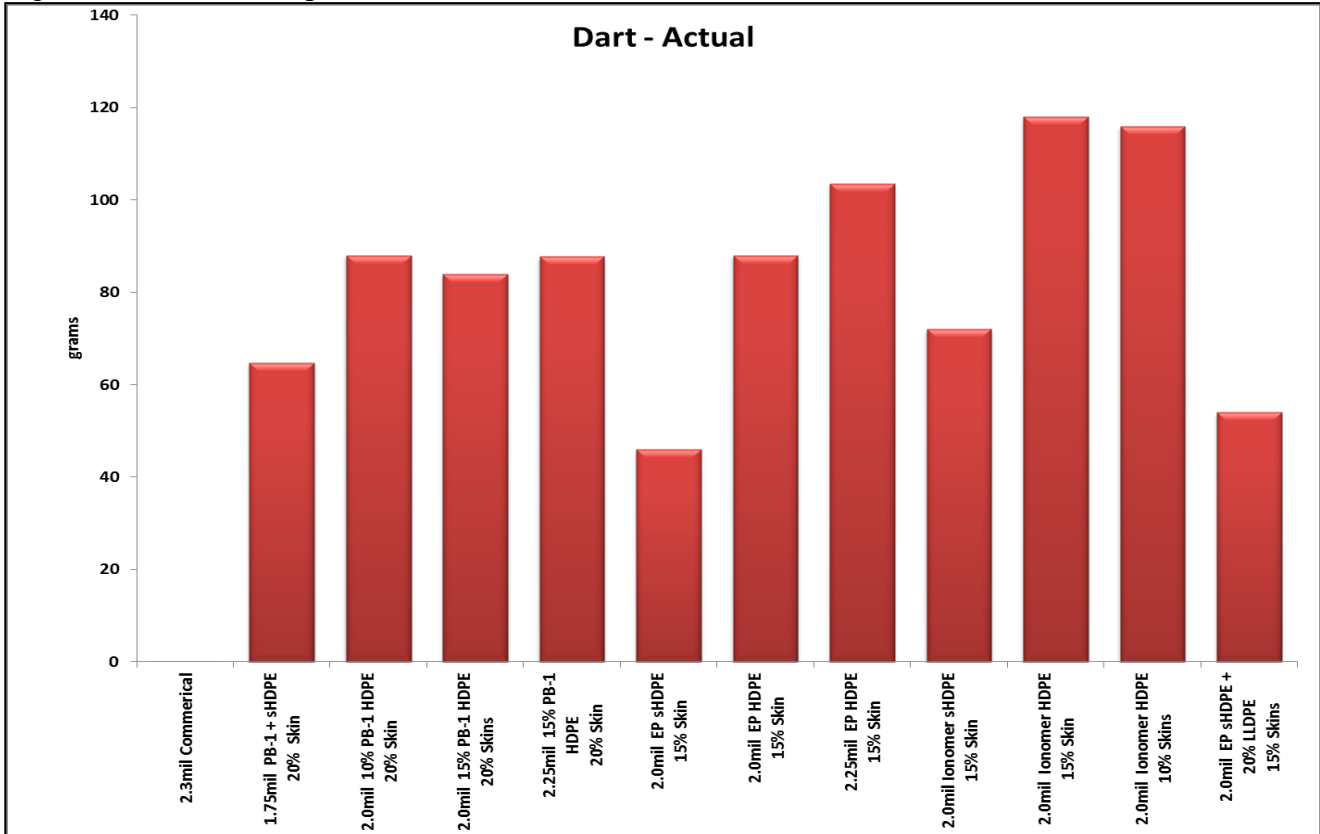


Figure 15: Dart Impact

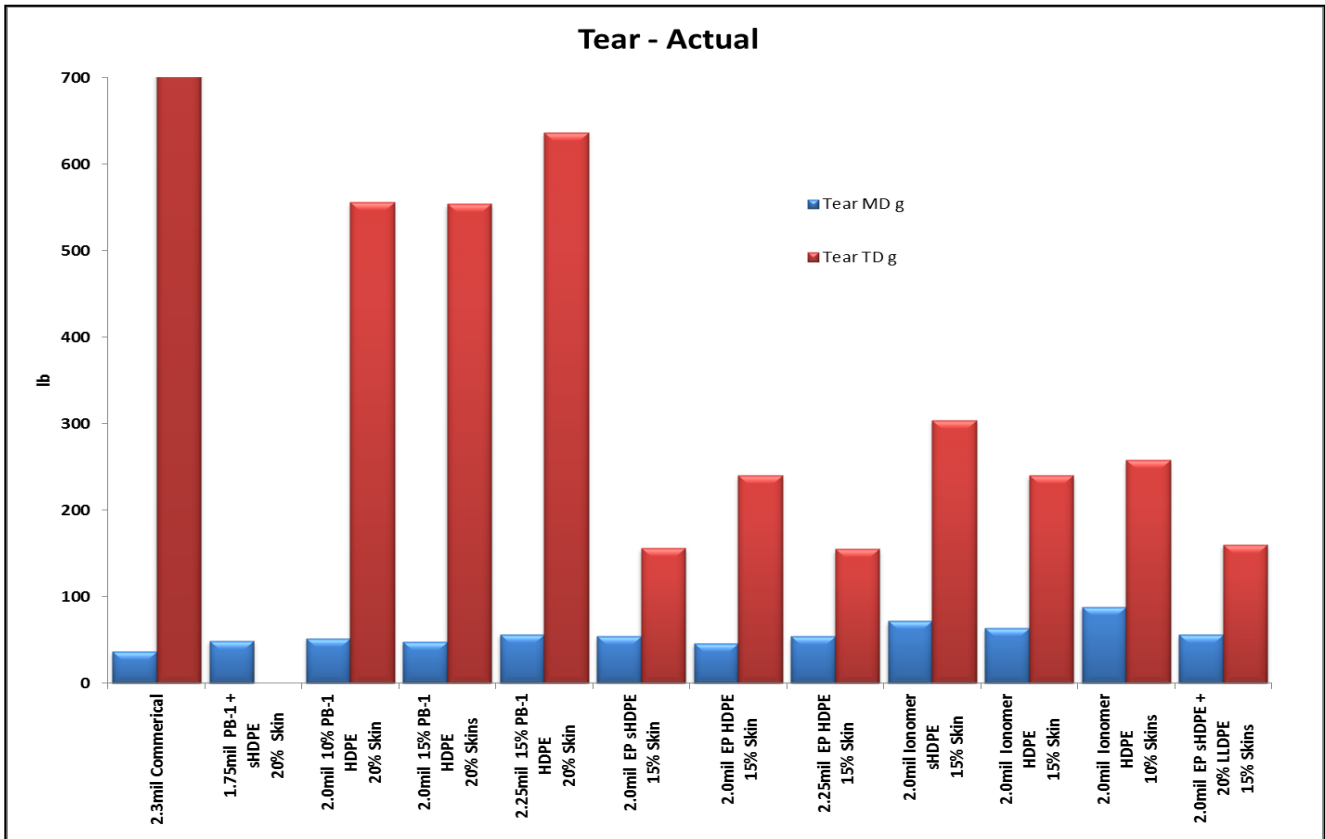


Figure 16: Elmendorf Tear

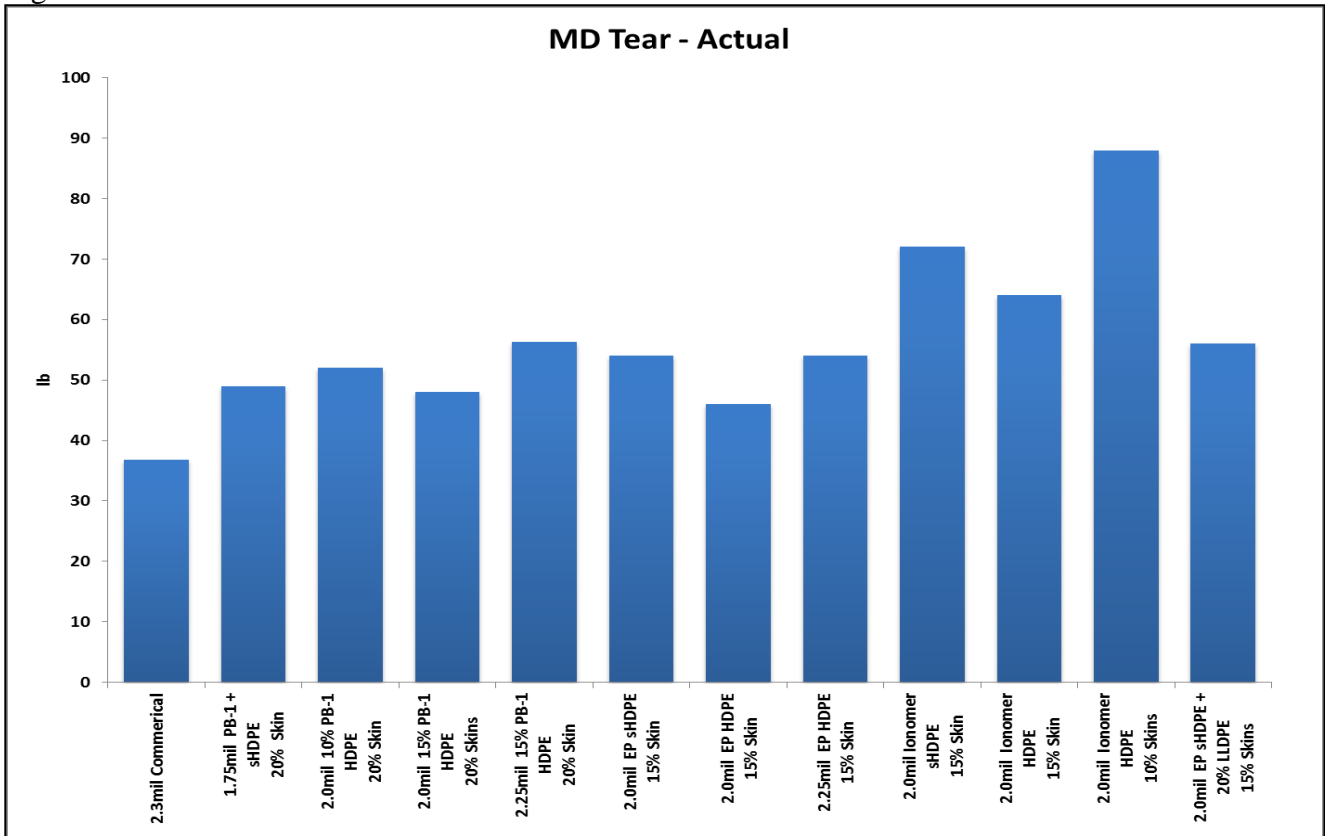


Figure 17: Elmendorf Tear – Machine Direction

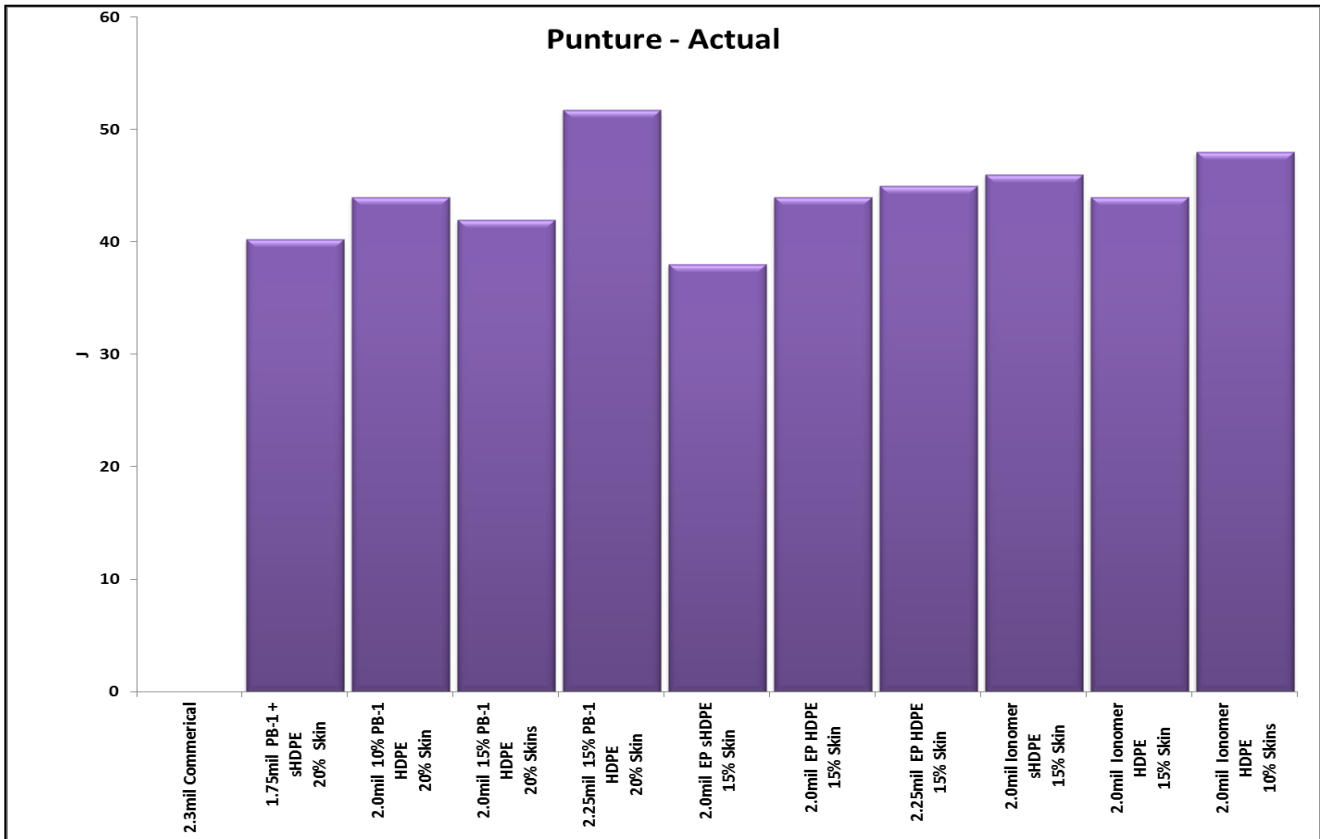


Figure 18: Puncture Resistance

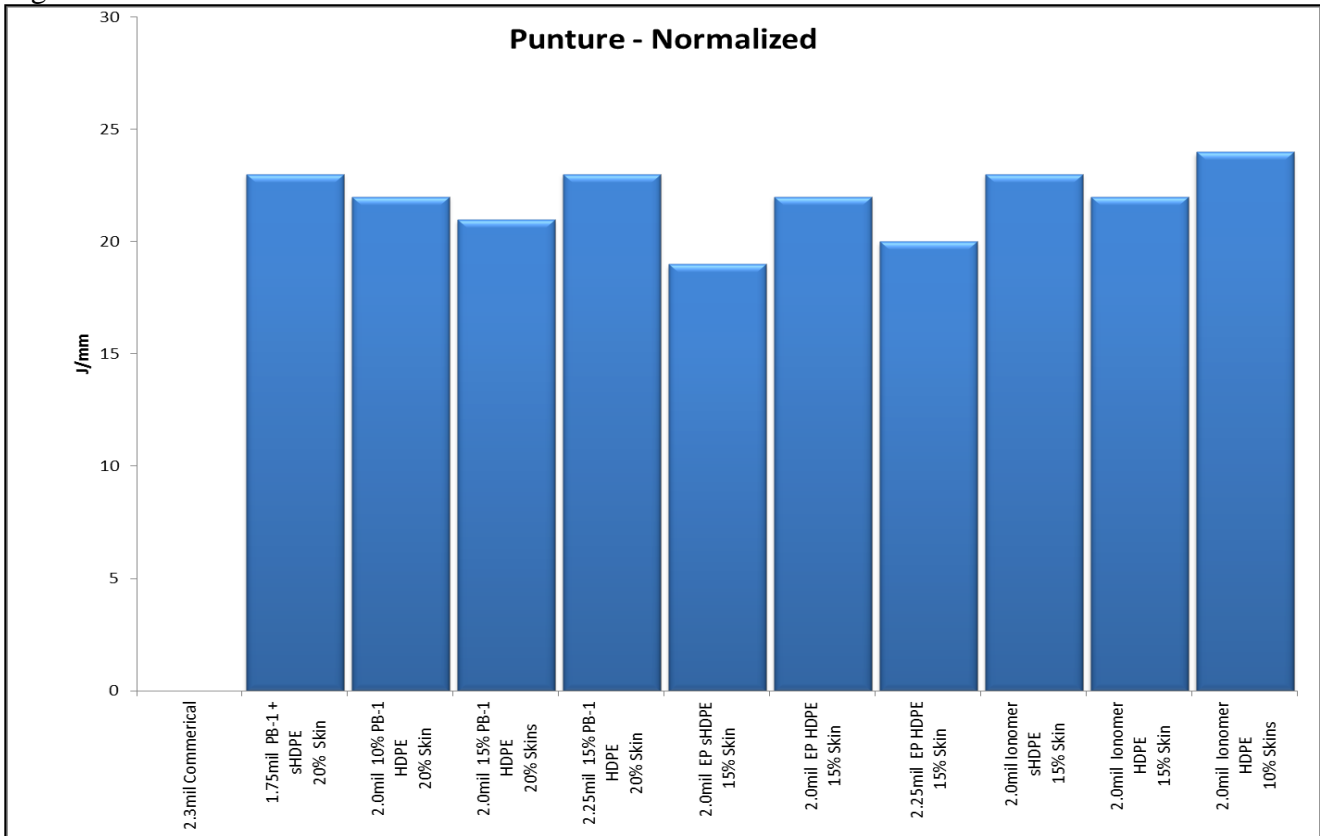


Figure 19: Normalized Puncture Resistance

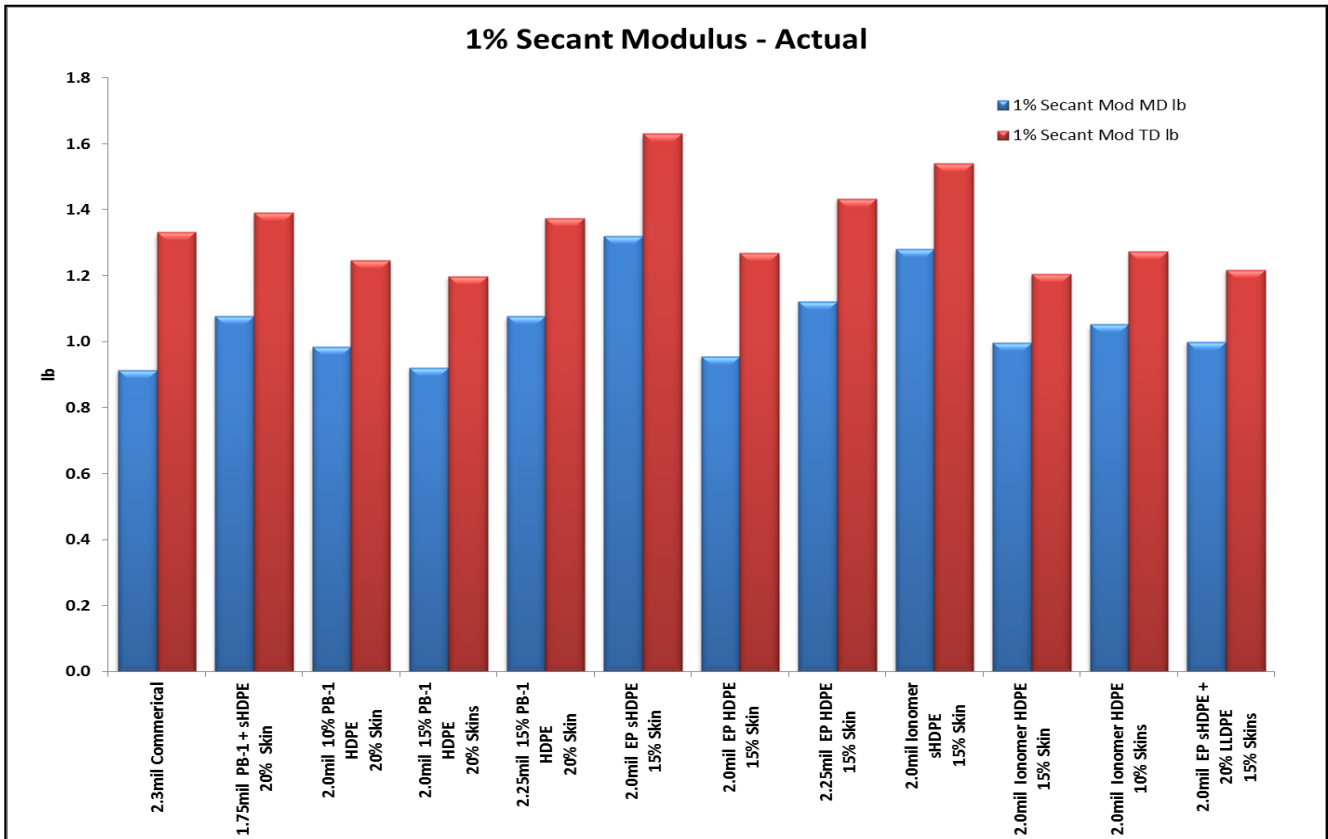


Figure 20: 1% Secant Modulus

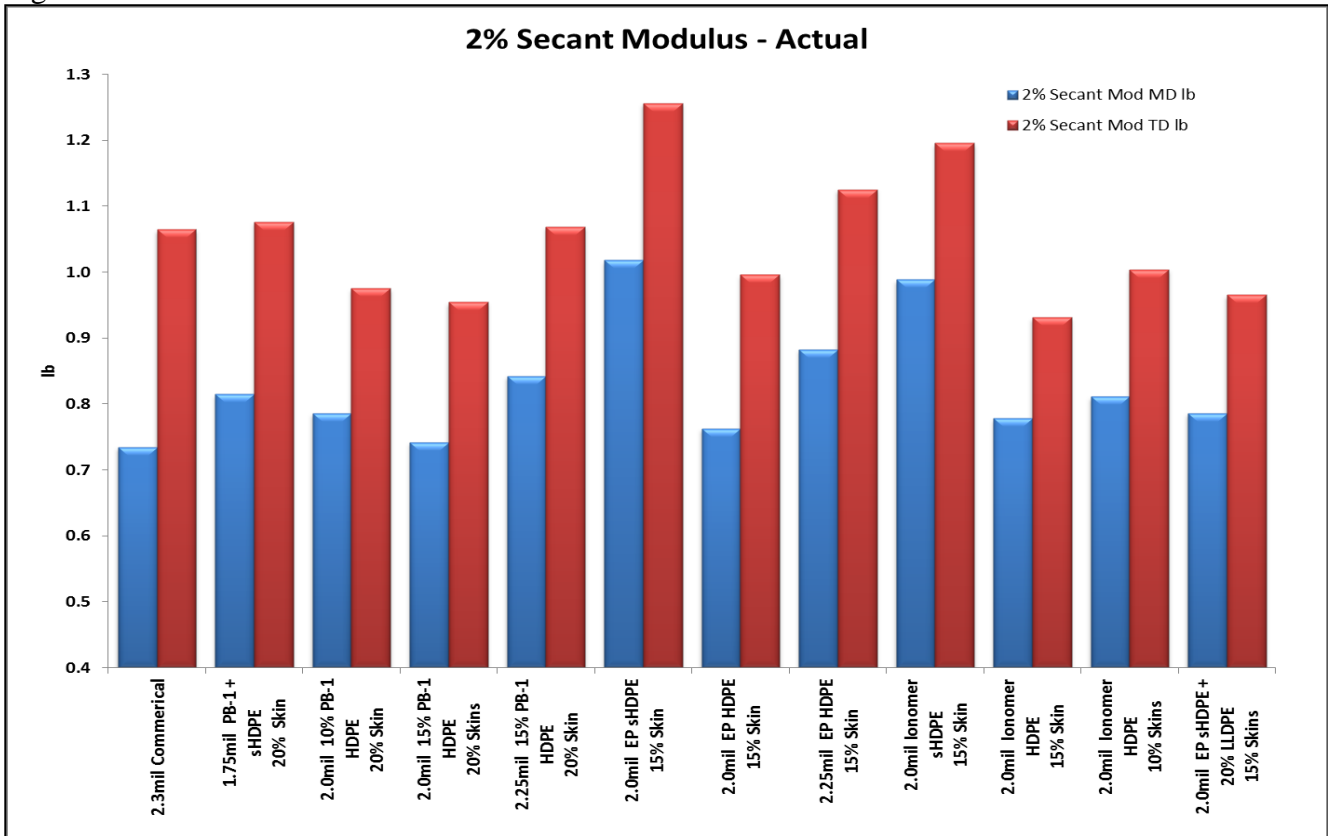


Figure 21: 2% Secant Modulus

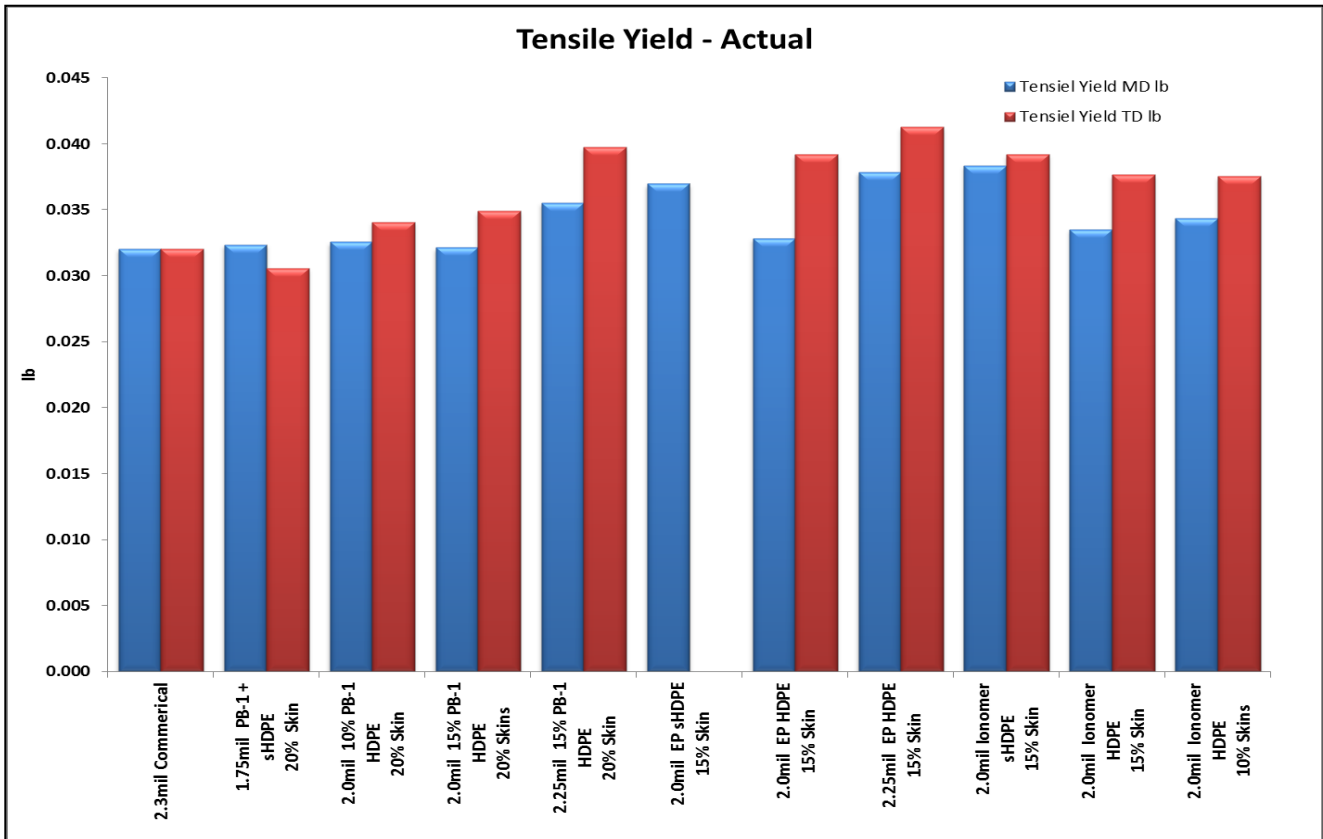


Figure 22: Tensile Yield

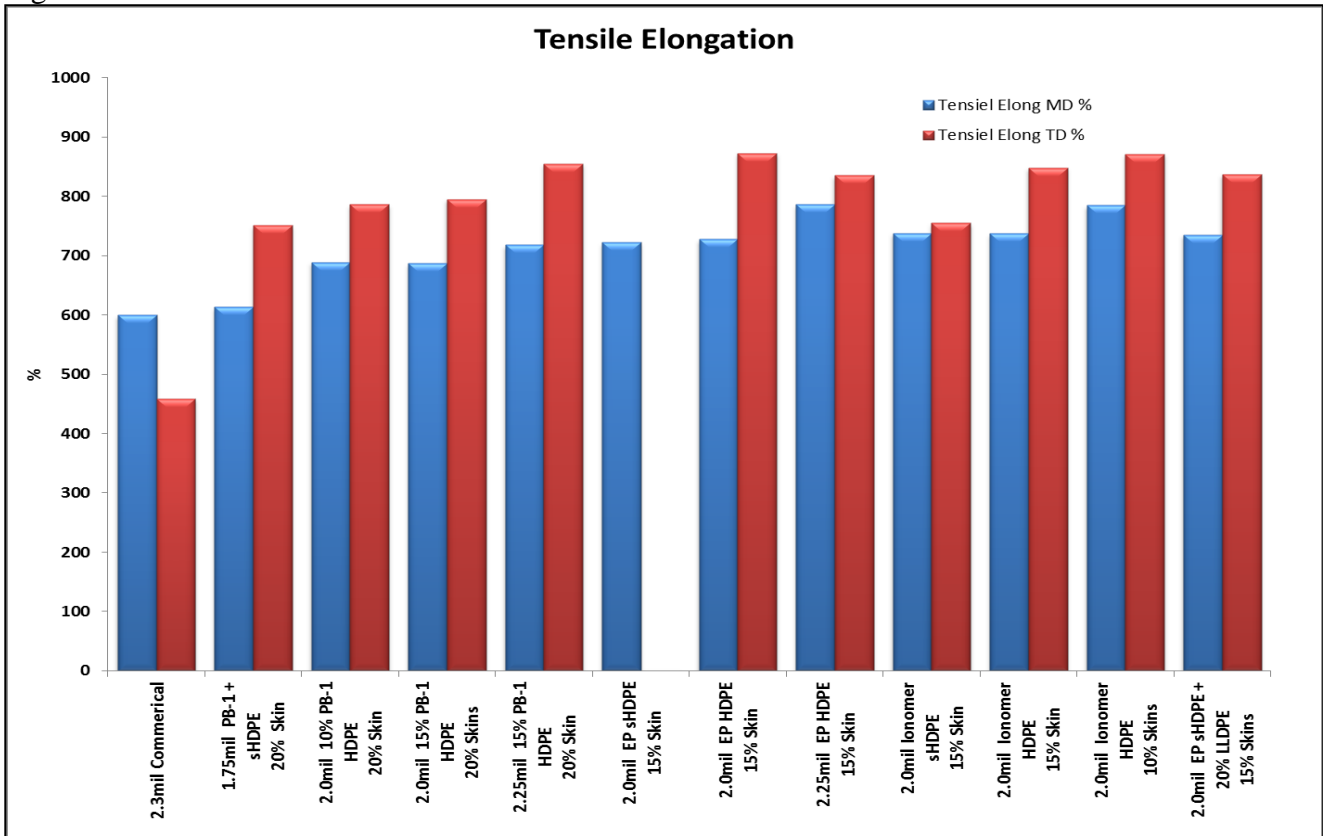


Figure 23: Tensile Elongation

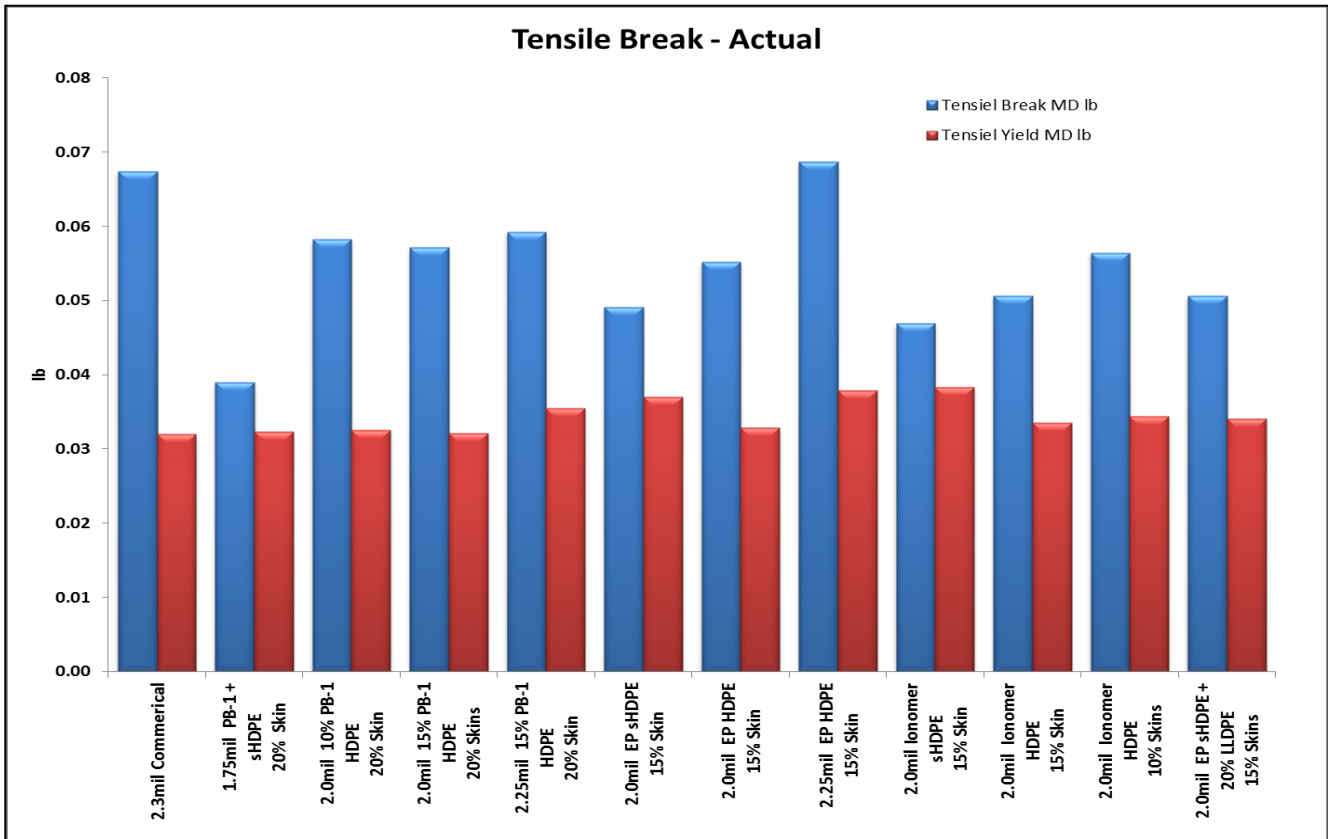


Figure 24: Tensile Break

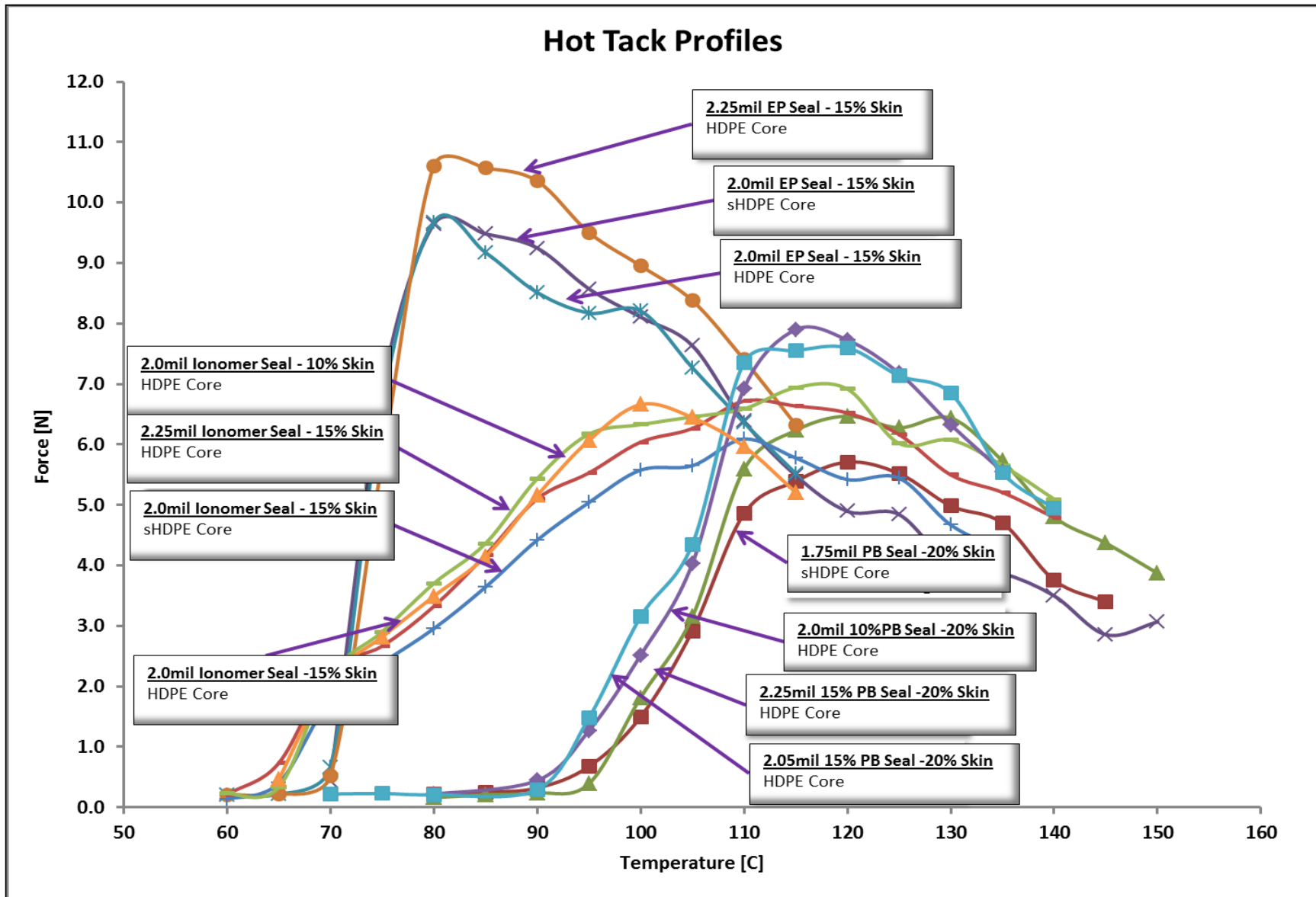


Figure 25: Hot Tack Strength

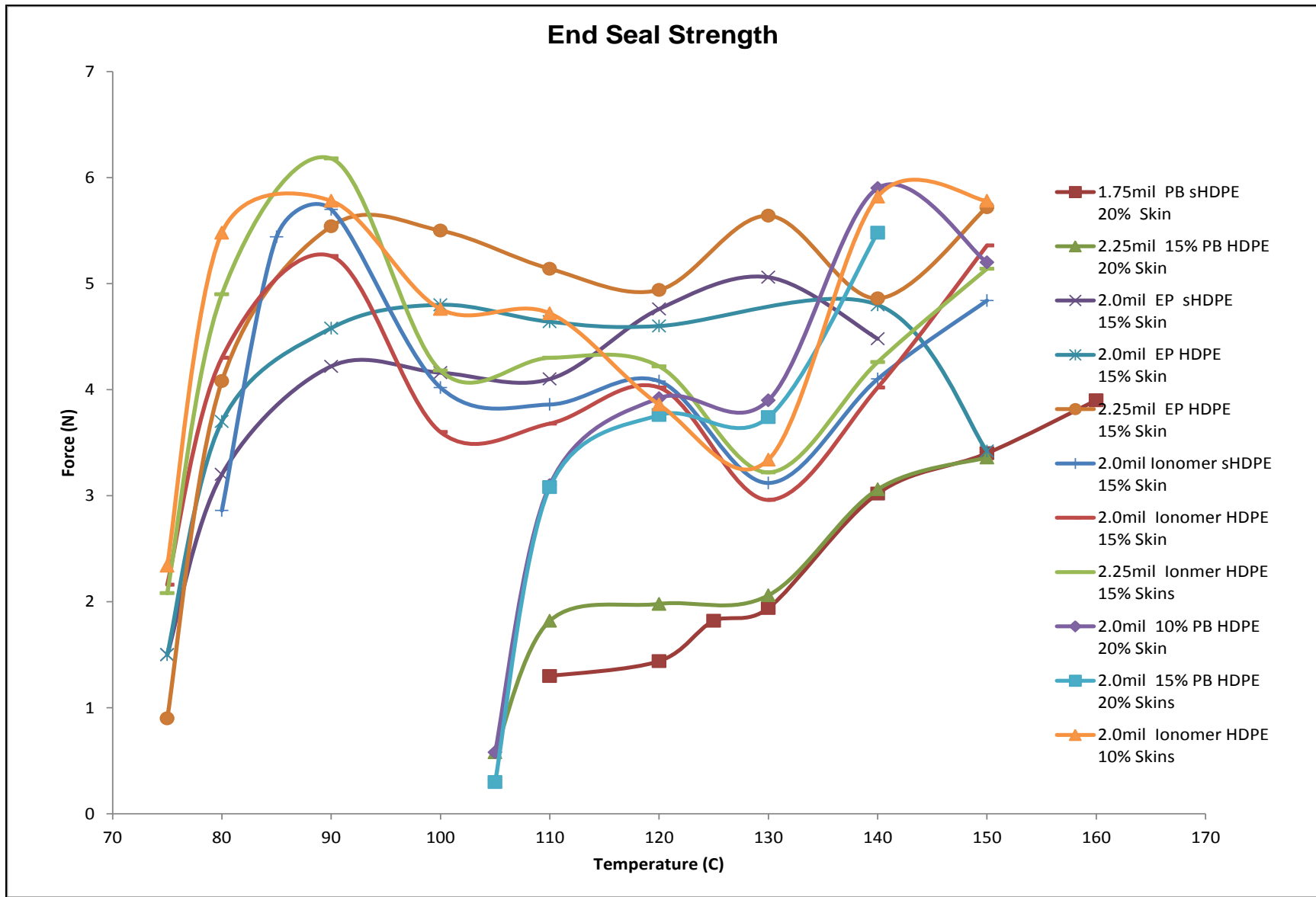


Figure 26: Film Heat Seal Strength - All after two weeks

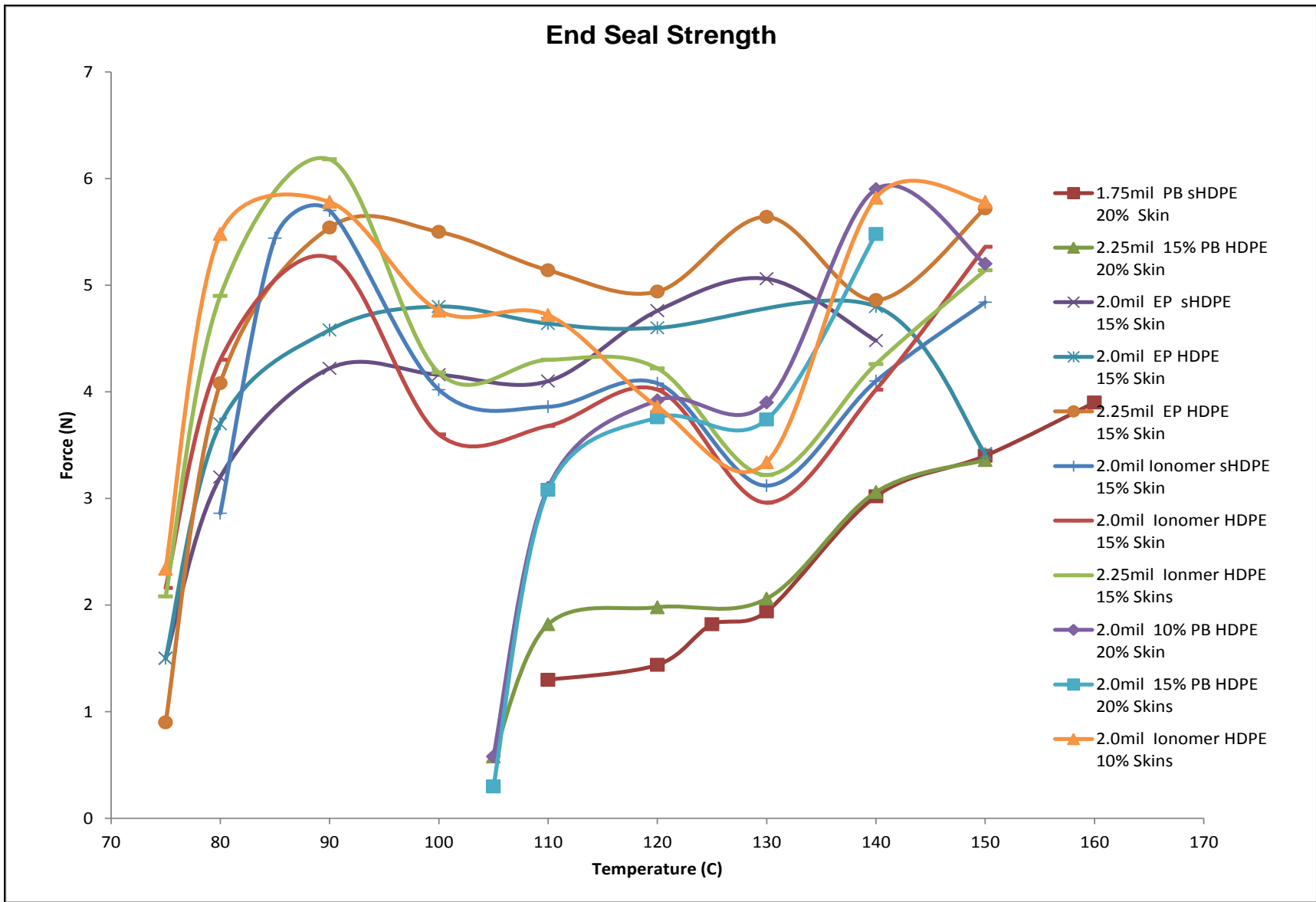


Figure 27: Pouch End Seal Strength - All after two weeks

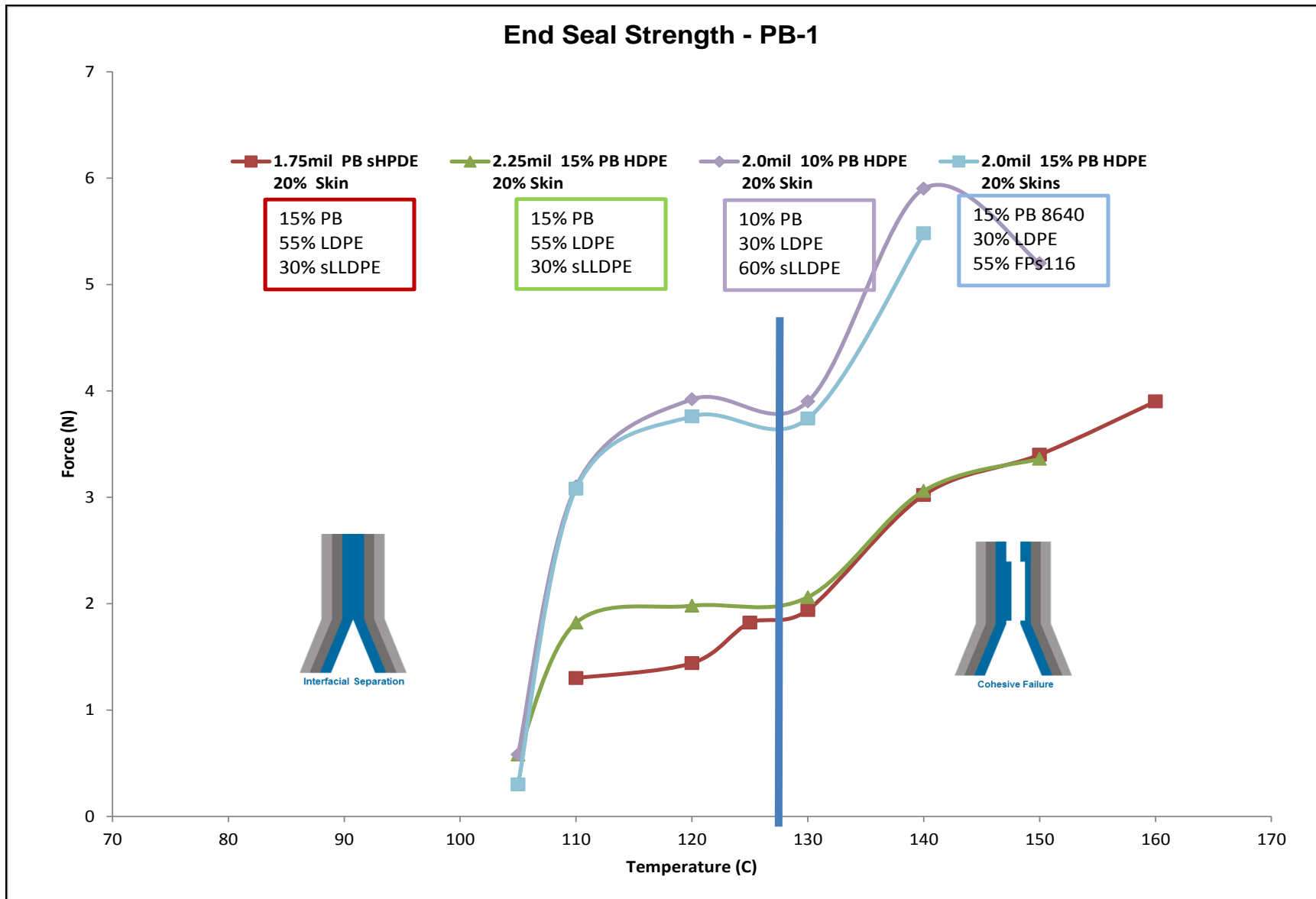


Figure28: Pouch End Seal Strength – PB-1

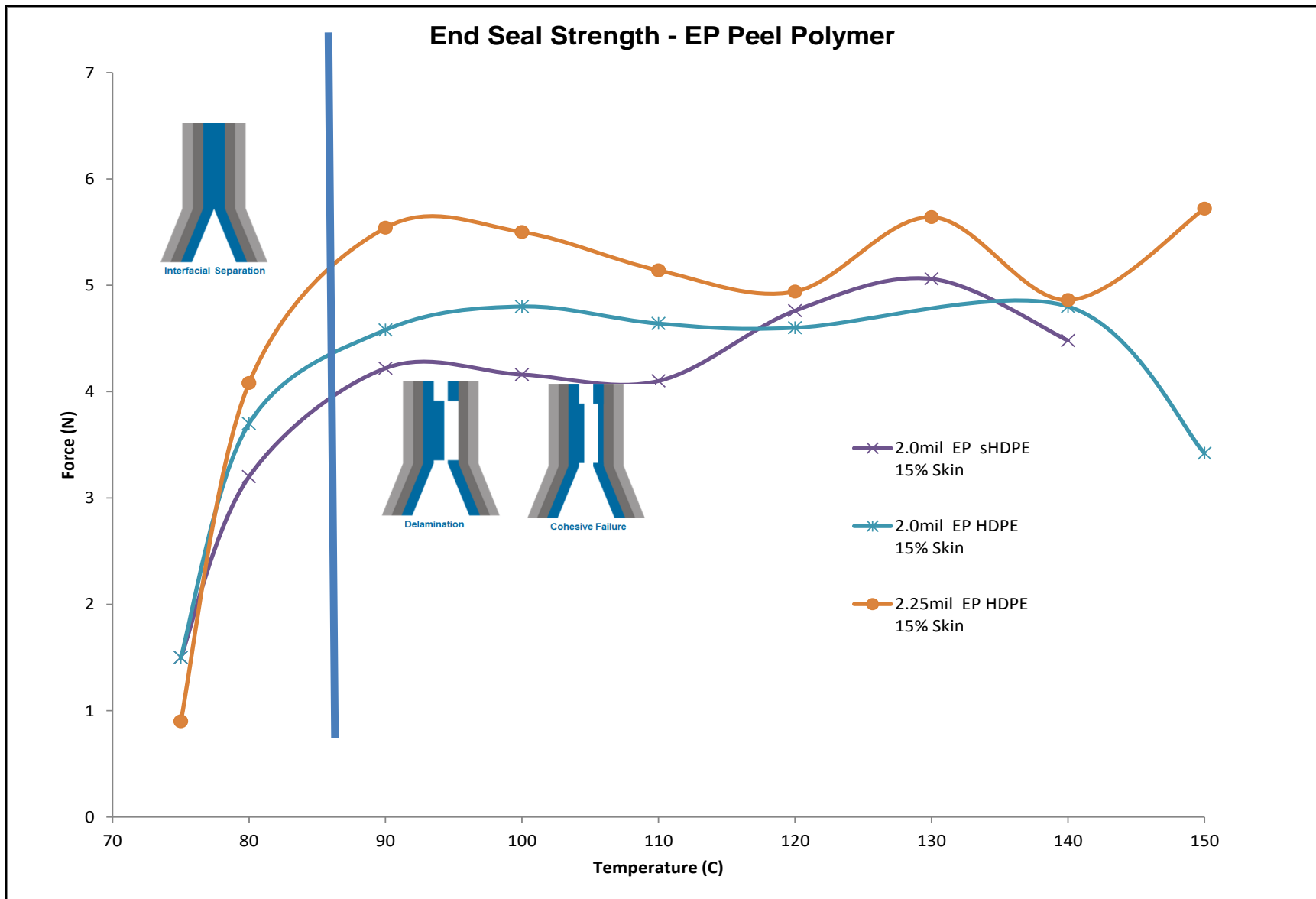


Figure 29: Pouch End Seal Strength – EP Peel Seal Polymer

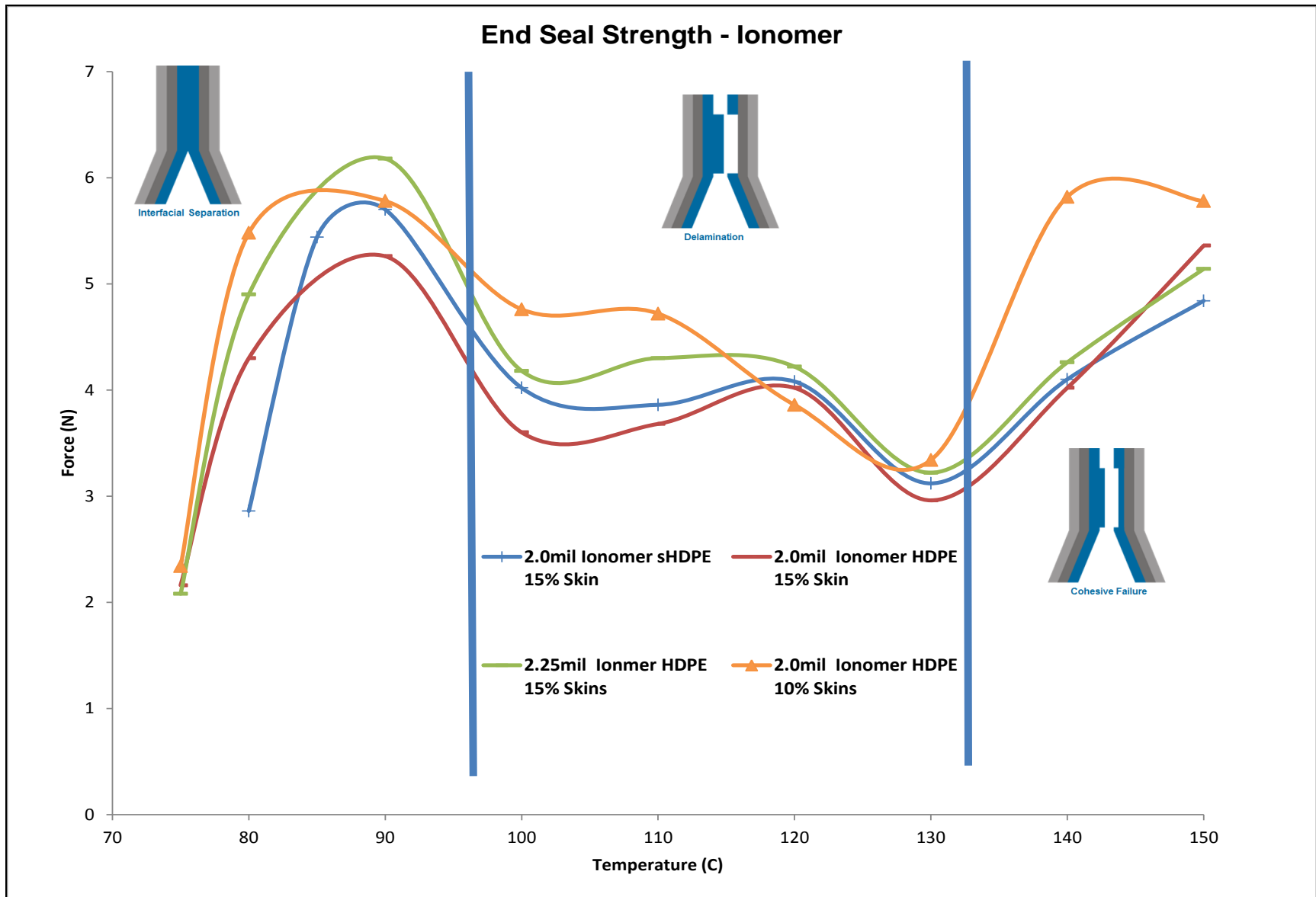


Figure 30: Pouch End Seal Strength - Ionomer

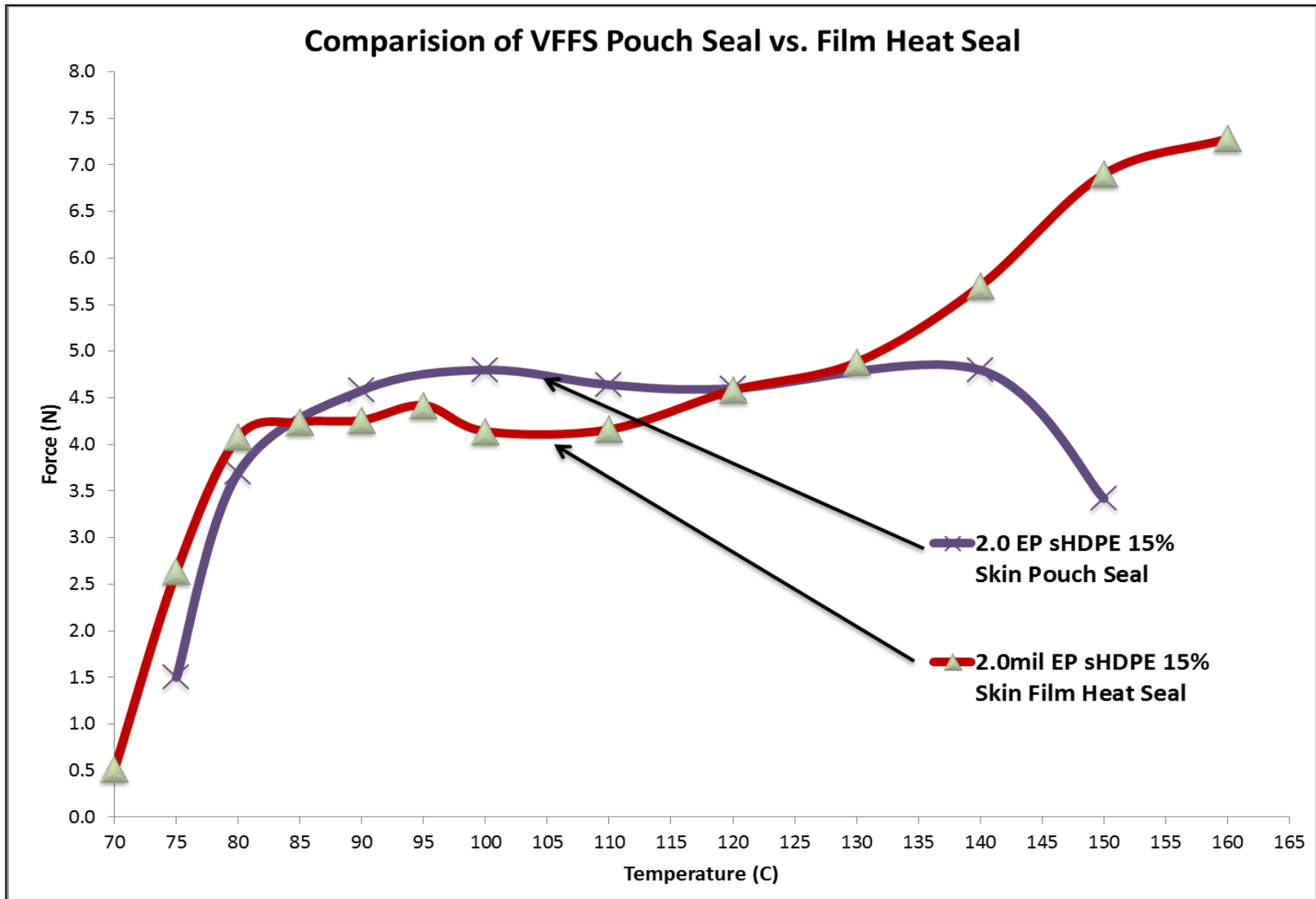


Figure 31: Pouch End Seal Vs Laboratory Heat Sealed Film

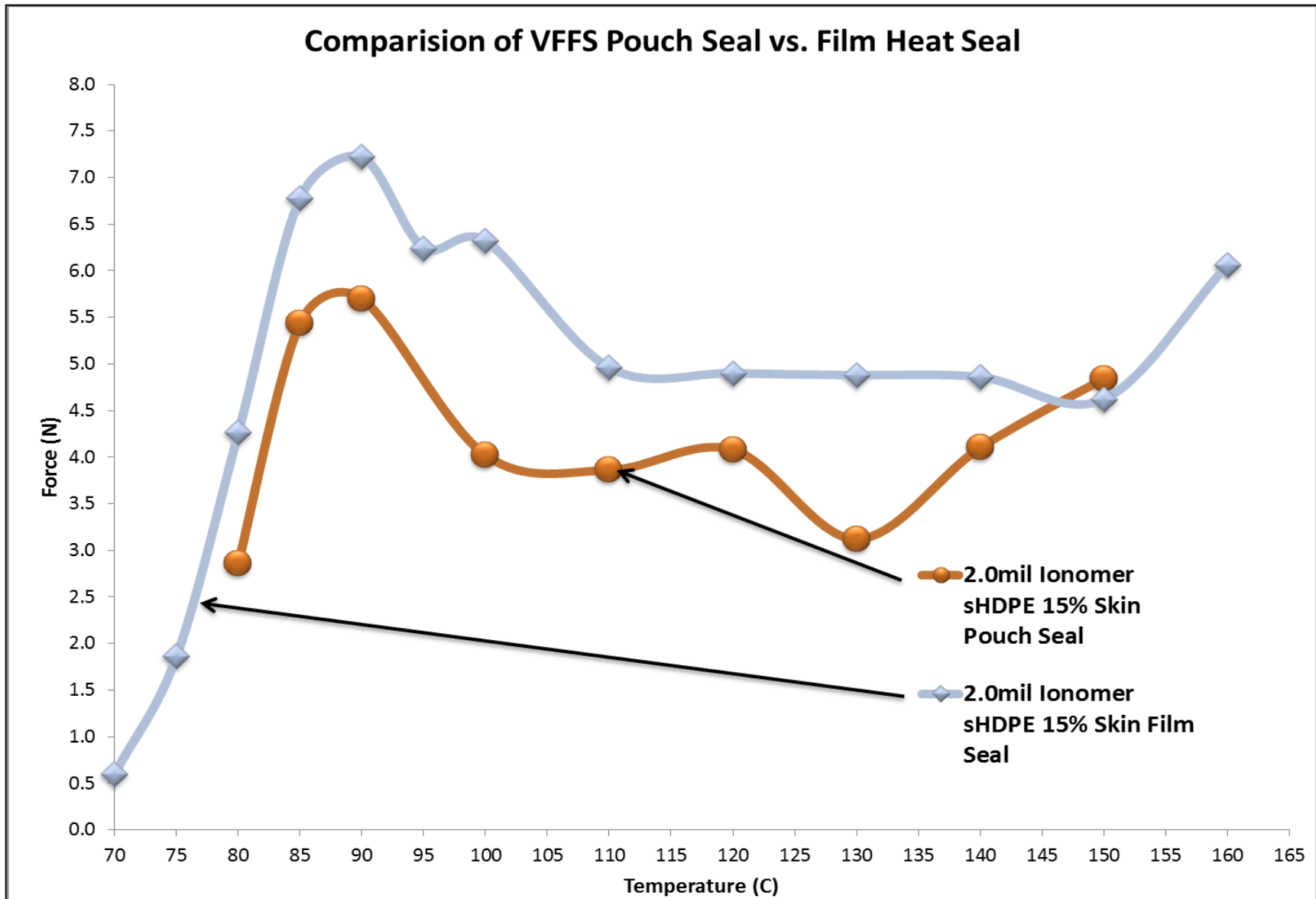


Figure 32: Pouch End Seal Vs Laboratory Heat Sealed Film

VFFS Pouch (Dry Filled) Compression Test

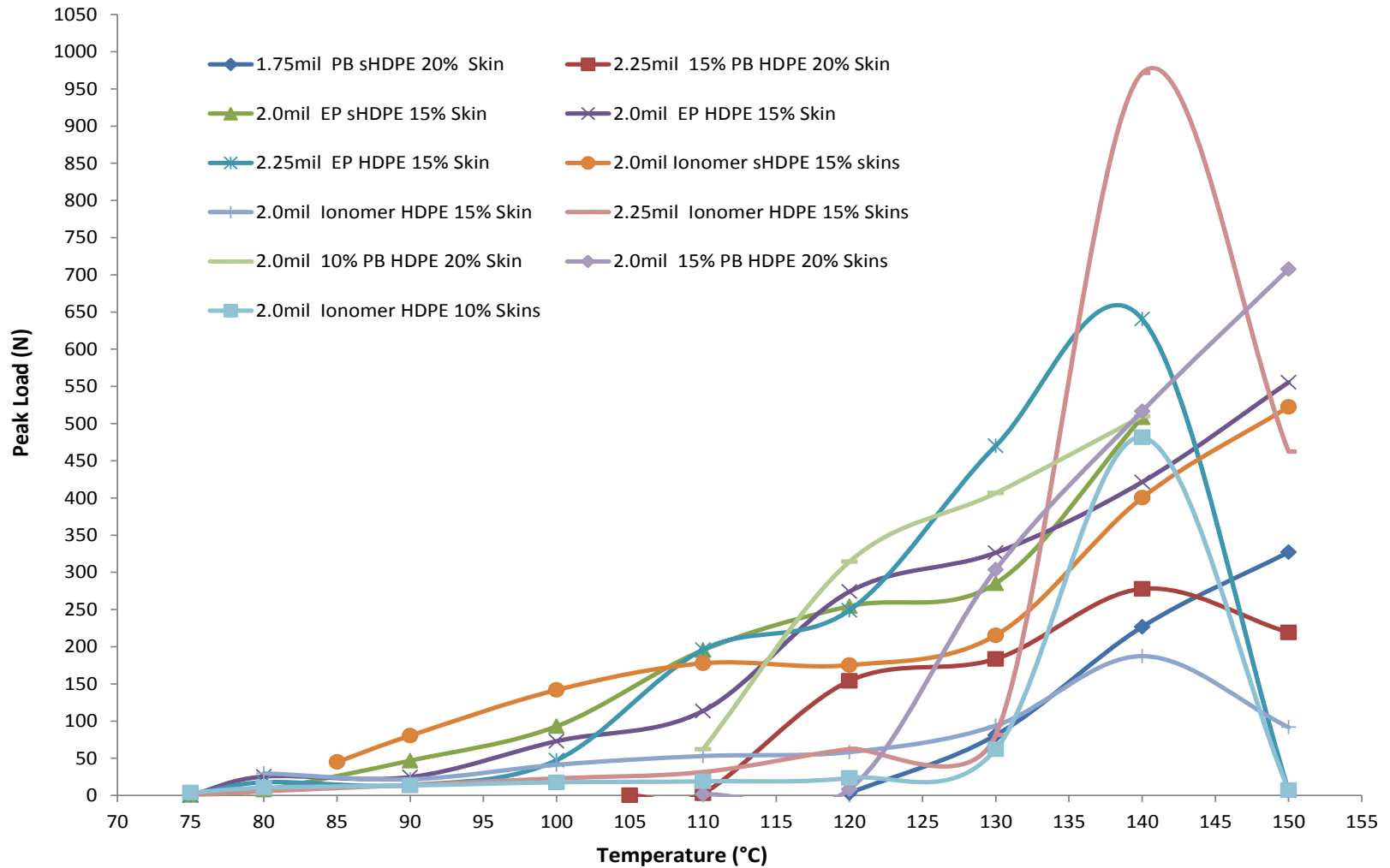


Figure 33: VFFS Pouch Compression Test

VFFS Pouch (Dry Filled) Compression Test - PB-1

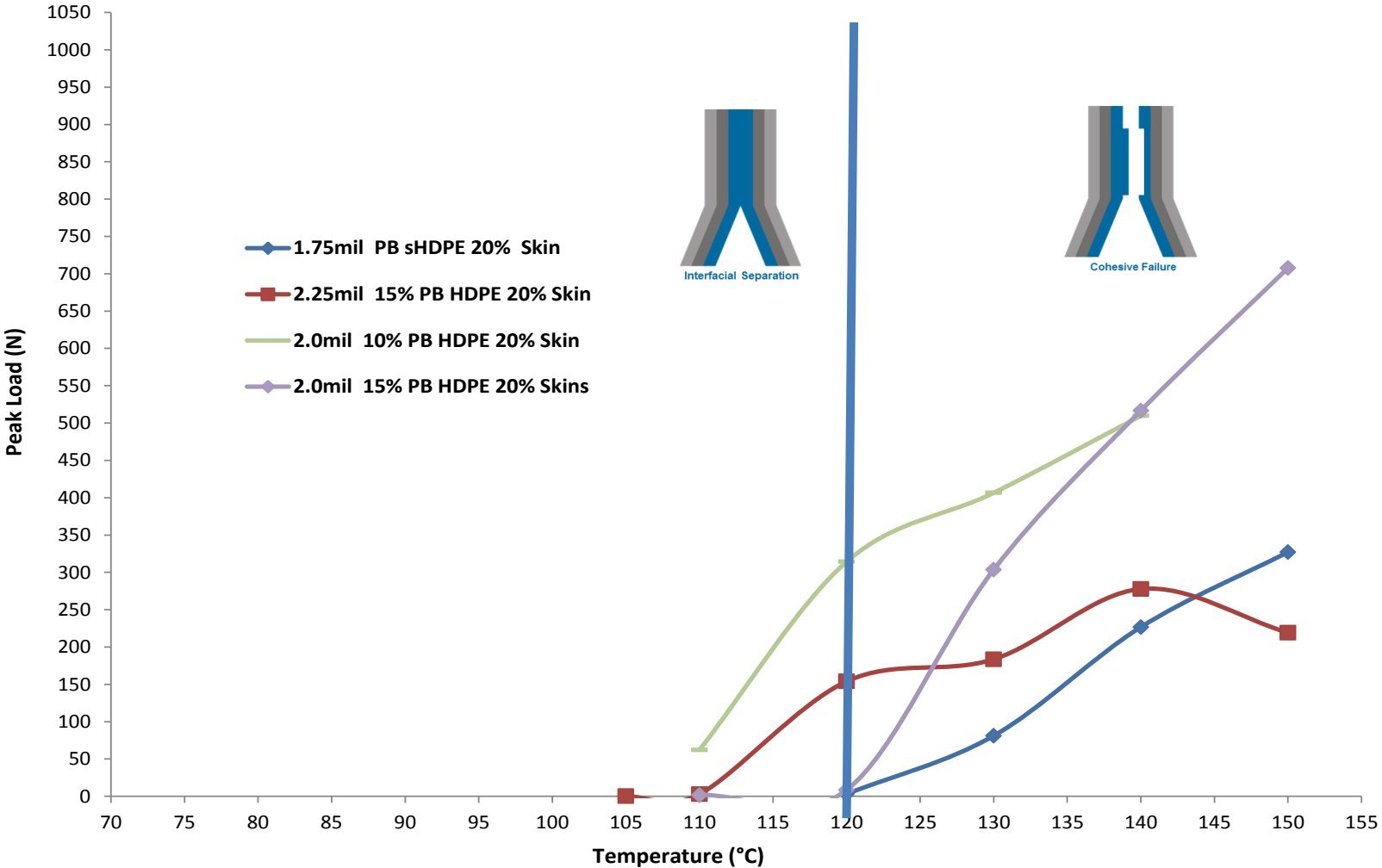


Figure 34: VFFS Pouch Compression Test – PB-1

VFFS Pouch (Dry Filled) Compression Test - EP Peel Polymer

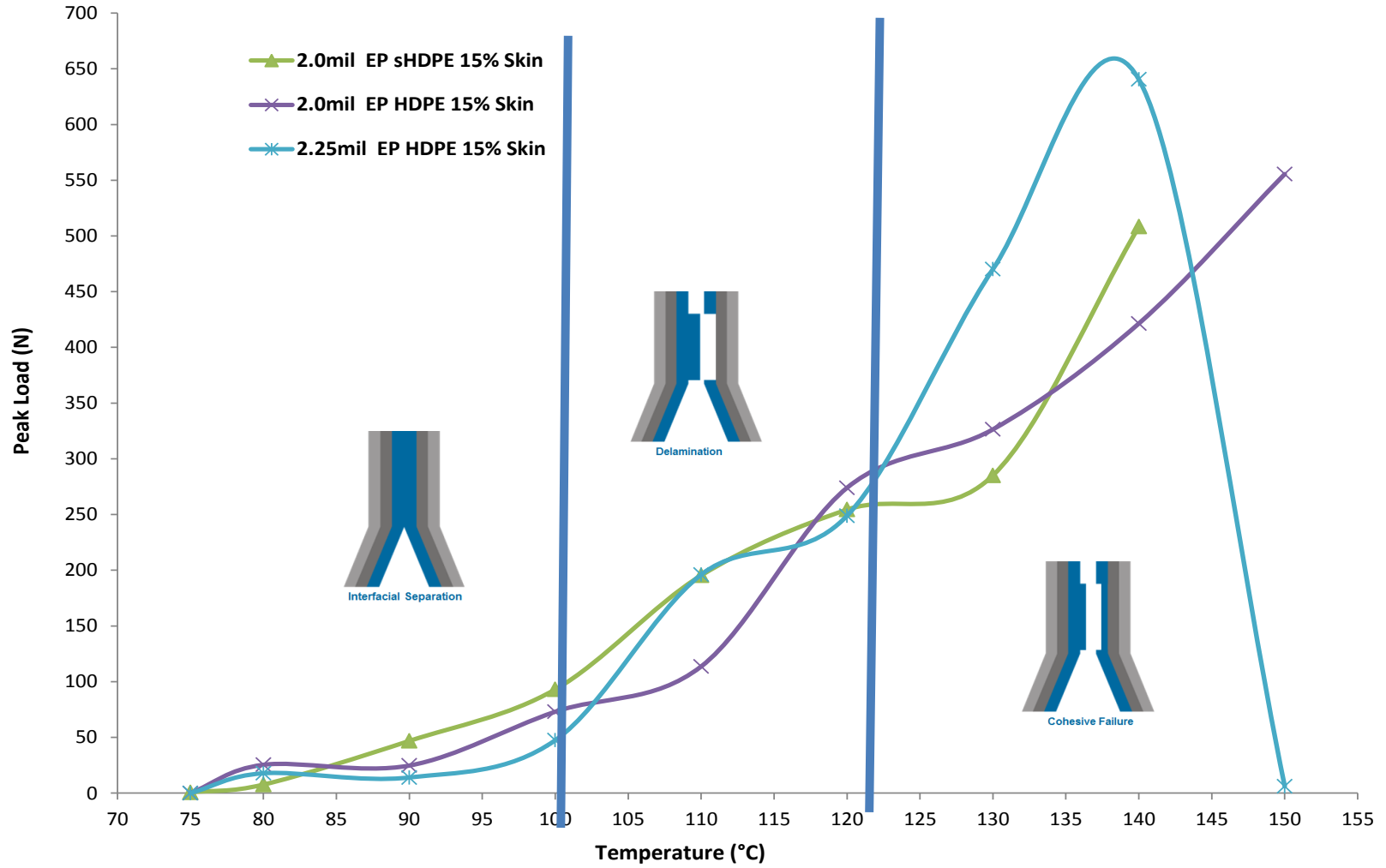


Figure 35: VFFS Pouch Compression Test - EP Peel Polymer

VFFS Pouch (Dry Filled) Compression Test - Ionomer

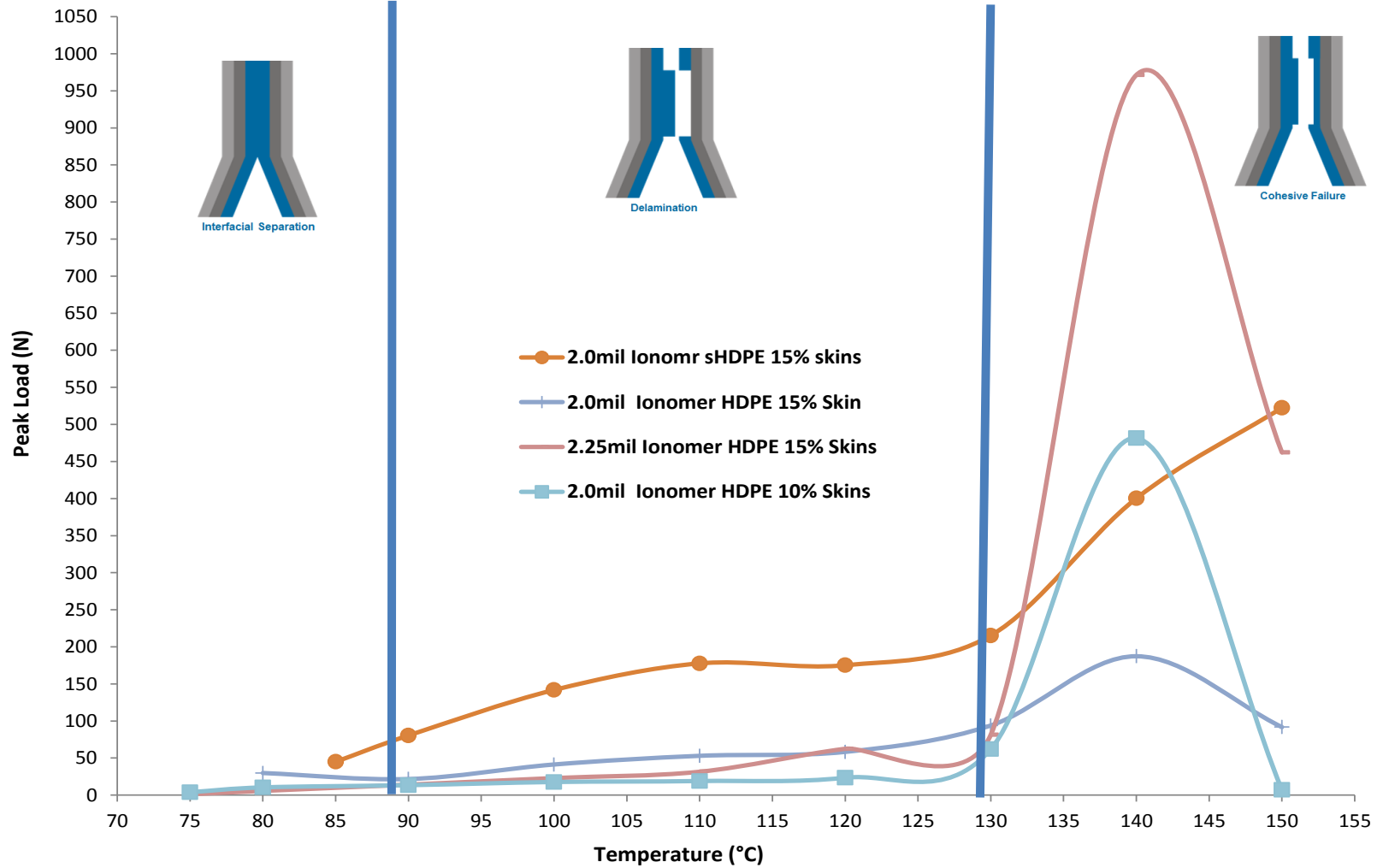


Figure 36: VFFS Pouch Compression Test - Ionomer

LASER IMAGEABLE POLYMERIC FILM

Patrick Thomas, PRThomas Technologies, LLC

Abstract

It has been found that polymeric film formulated with a photochromatic pigment offers a substantial advantage over prior art methods of laser imaging film. Polymeric film formulated with a photochromatic pigment which undergoes a white to black color change upon exposed to laser irradiation are durable and abrasion-resistant since the photochromatic pigment is within the body of the plastic film.

Introduction

Product labeling is becoming increasingly important in almost every branch of the industry. Markings such as production dates, expiration dates, bar codes, and serial numbers are applied using conventional methods of printing and labeling. Marking photochromatic film using lasers permits contactless and high-speed application of graphics.

Previously, two traditional methods of laser marking systems include the use of tapes or sprays [2]. Tape coated with a laser-imageable ink composition can be applied using adhesives. A laser-imageable spray coating can also be applied using a spray application system. Laser image markings applied using either of these coating processes are susceptible to smearing and fading upon sterilization.

Furthermore, the heat of reaction generated upon exposure to the laser irradiation can distort the marking and deform the film. Consequently, the legibility of the marking is adversely affected.

Thus, the industry needs a marking solution that allows for smaller production runs with customized labeling included on individual packages. Traditional printing methods such as flexography and rotogravure are relatively expensive and labor intensive considering the need for printing plates and frequent material changes.

Consequently, the application of laser marking provides an economical method of converting and allows the end user to reduce waste and printing material inventory. Moreover, this laser marking system provides a means of specific unit traceability of various products.

The purpose of this paper is to describe a method of producing a laser-imageable polymeric film formulated with photochromatic pigment that provides high-contrast

marking while retaining a smooth undistorted film surface [1].

Materials

Marking Layer

The marking layer can be fabricated using polyolefin material, photochromatic pigment, and an additive (See TABLE 1). The photochromatic pigment such as Datalase Pigment A is an ammonium octamolybdate (AOM). AOM forms a monochromatic grey/black marking when exposed to a CO₂ laser. The addition of an additive such as talc will help to prevent film distortion by diffusing the heat of reaction generated upon exposure of the pigment to the energy source.

Table 1 Material

Material	Source	Trade Name	Type	MI (g/10 min)	Density (g/cc)
A	LyondellBasell	Alathon L5885	HDPE	0.85	0.958
B	Ampacet	110313-B	White Conc	2-6	2.02
C	ExxonMobile	EXCEED 1012CA	ULDPE		0.912
D	Datalase, Ltd	Datalase Pigment A	Photochromatic Pigment		
E	Ampacet	100622	LDPE/SLIP		
F	Polyfil Corp	ABC-5000PB	LDPE/Talc		
G	LyondellBasell	Petrothene NA 345-013	LDPE	1.8	0.921
H	LyondellBasell	Polybutylene-1 PB 8640M	PB	1	0.908
I	LyondellBasell	Alathon L5045	HDPE	0.45	0.95
J	Ampacet	11853	White Conc/LLDPE	2.9	1.513
K	Nova Chemicals	SURPASS HPS-167-AB	HDPE	1.2	0.966
M	Polyfil Corp	PHC-0001HD	Nucleating agent		
N	Polyfil Corp	PAC-0154LL	LLDPE/PA	2	0.92
O	LyondellBasell	Alathon L6012	HDPE	12	0.96
P	PMC Biogenia	Kenamide W-40 Prill	Amide Wax		0.995

Method of Making Film

Film can be manufactured by such processes as blown film, cast film, lamination, or extrusion coating. The pigment and additive components of the marking layer can be mixed together in any conventional manner. Pigment and additive can be mixed with the polymer components of the marking layer by dry blending or compounding in an extruder. Masterbatching technology can also be employed.

Methods of Using Film

While it has been difficult to laser image films, the addition of a photochromatic pigment and additive facilitates the laser imaging process. Film formulated with the photochromatic pigment undergoes an irreversible color change when exposed to the laser beam. Since the lasers are controlled by computers, graphic images are programmable.

The shade and depth of color obtained are determined by the laser parameters, such as radiation time and output. Consequently, low energy density lead to light markings in the film, while high-energy densities lead to dark markings. Film formulated with AOM can be marked by a laser with a wavelength ranging from 300 to 10,000 nm.

The optical density of film markings preferably ranges from about 0.65 to about 0.90, based on a 0.0 (white) 1.25 (black) ODB scale. The text is clearly legible and is distinguished by a high degree of resolution.

Furthermore, upon sterilization, it has been shown the markings do not fade as is common with labels coated with ink formulations containing a photochromatic pigment and applied via pressure sensitive adhesives. Spray coated labels with inks containing photochromatic pigment also have a tendency to fade upon sterilization.

Advantages of Laser-imageable Film

This laser imaging system (film formulated with a photochromatic pigment and method of laser imaging) can replace conventional thermal transfer, inkjet, digital, or gravure-base printing processes. This laser imaging process reduces printing time, improves efficiency, reduces the costs associated with inks, and provides greater flexibility compared to current imaging methods. In addition, this marking system does not require formulating, cleaning, and provides for reduced scrap with no changeover required. Further, transitions between films are automatic and the lasers are capable of changing images instantly. Moreover, customers using this marking system method can customize packaging for specific lot traceability as required for many pharmaceutical products that require every unit to be controlled and traceable. These markings on packaging film are wipe-resistant, scratch-resistant, and stable during subsequent sterilization processes.

Preparation of Pigment Masterbatch

The pigment masterbatch was formulated with 64% O, 35% D, and 1% P (see Table 1). The mixture was compounded on a WP twin screw extruder at 100

g/minute. The 35% pigment MB was then incorporate into films 1-17 (see Table 2 and 3).

Preparation of Films

Films 1–8, with the compositions and construction shown in Table 2, were prepared on a conventional blown film line.

Table 2 Films 1-8 Structure/Formulation

Films	Layer 1	Layer 2
1	62 % A	88% A
	10% B	10% D
	28% C	2% E
2	62 % A	78% A
	10% B	20% D
	28% C	2% E
3	62 % A	68% A
	10% B	30% D
	28% C	2% E
4	62 % A	58% A
	10% B	40% D
	28% C	2% E
5	62 % A	48% A
	10% B	50% D
	28% C	2% E
6	62 % A	38% A
	10% B	20% D
	28% C	2% E
		40% F
7	62 % A	28% A
	10% B	30% D
	28% C	2% E
		40% F
8	62 % A	18% A
	10% B	40% D
	28% C	2% E
		40% F
Layer Ratio (%)	90	10
Thickness (mils)	3.15	0.35

1 cm x 1 cm square samples were prepared. Each sample was imagined using a Videojet 3320 laser with 127 mm lens to produce square block images. Such lasers generate high power light via excitation of the CO² within a sealed chamber. The light is focused to a small, intense beam that is used for writing or marking. The whole

process, from excitation to writing or marketing, is controlled by computer software supplied with the laser system.

Observations of imaged films are given below in table 4. Observation of "best" refers to film with minimal film distortion and very dark, legible images. An observation of "good" refers film with slight to minimal distortion and dark, legible images. An observation of "fair" refers to a film with slight distortion and light images. An observation of "poor" refer to film with some film distortion and very faint images.

After imaging, it was observed that films 4 and 5 with 40% and 50% pigment and no filler were legible. It was also observed that films 7 and 8 with 30% and 40% pigment and filler were legible. Films with 40% pigment and filler" want legibility to the film samples with 50% pigment and no filler. It appears that the filler reduces the film distortion due to the heat from the chemical reaction of the pigment.

Table 4 Observations of Imaged Films 1-8

Film	Poor	Fair	Good	Best
1	X			
2		X		
3		X		
4		X		
5			X	
6		X		
7			X	
8				X

Preparation of Films 9-17

Films 9-17 with compositions and construction shown in Table 3 below, were prepared on a conventional blown film line.

Optical Density Testing of Films 9-17

Label-sized samples of films 9-17 were prepared using a Videojet 3320 laser with 127 mm lens to produce a label with square blocks, text and single line graphics, but 2D data matrix code, and an EAN-13 barcode.

The black optical density (ODB) value for each sample was measured with SpectroEye spectrophotometer. The ODB values for each sample are given below in table 5, with a 0.0= white and a 1.25=equal black.

Table 5 Optical Density Values for Films 9-7

Film #	ODB
9	0.65
10	0.79
11	0.78
12	0.83
13	0.88
14	0.85
15	0.85
16	0.88
17	0.81

Conclusions

A polyethylene film formulated with a photochromatic pigment MB and additive provides a laser-imageable substrate capable of high resolution markings with an undistorted film surface.

The optimal marking layer formulation as determined by ODB measurements and film distortion comprises:

- 40% pigment MB
- 45% talc MB
- 9.5% HDPE
- 3% nucleating agent
- 1 % Process Aid
- 1.5 Slip/AB

References

1. Merial; Rick, Darley; Russell, and Thomas; Patrick, U.S Patent Application US 2013/0189617
2. Green; Williams, Phillips; Tristan, Jarvis; Anthony, Wyres; Christopher, Wilson; Trevor, U.S. Patent 8,173,253

Table 3 Films 9-17 Structure/Formulations

Film	Layer 1	Layer 2	Layer 3	Layer 4
9	57% G	48% C	88% K	40% F
	20% H	40% I	12% J	35% D
	23% C	12% K		19.5% K
				3% M
			1.5% E	
			1% N	
10	57% G	48% C	88% K	45% F
	20% H	40% I	12% J	35% D
	23% C	12% K		14.5% K
				3% M
			1.5% E	
			1% N	
11	57% G	48% C	88% K	50% F
	20% H	40% I	12% J	35% D
	23% C	12% K		9.5% K
				3% M
			1.5% E	
			1% N	
12	57% G	48% C	88% K	40% F
	20% H	40% I	12% J	45% D
	23% C	12% K		14.5% K
				3% M
			1.5% E	
			1% N	
13	57% G	48% C	88% K	45% F
	20% H	40% I	12% J	40% D
	23% C	12% K		9.5% K
				3% M
			1.5% E	
			1% N	
14	57% G	48% C	88% K	50% F
	20% H	40% I	12% J	40% D
	23% C	12% K		4.5% K
				3% M
			1.5% E	
			1% N	
15	57% G	48% C	88% K	40% D
	20% H	40% I	12% J	54.5% K
	23% C	12% K		3% M
				1.5% E
			1% N	
16	57% G	48% C	88% K	40% F
	20% H	40% I	12% J	40% D
	23% C	12% K		17.5% K
				3% M
			1.5% E	
			1% N	
17	57% G	48% C	88% K	40% D
	20% H	40% I	12% J	57.5% K
	23% C	12% K		1.5% E
				1% N
Layer Ratio (%)	25	45.3	19.7	10
Thickness (mils)	0.87	1.59	0.69	0.35

Figure 1 Laser-Imageable Polymer Film

