EVALUATION OF PACKAGING FILM MECHANICAL INTEGRITY USING A STANDARDIZED SCRATCH TEST

A Thesis

by

BRIAN ANTHONY HARE

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

August 2011

Major Subject: Materials Science and Engineering

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ABSTRACT

Evaluation of Packaging Film Mechanical Integrity Using a Standardized Scratch Test. (Ausgust 2011)

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Chair of Advisory Committee: Dr. Hung-Jue Sue

Polymeric packaging films see widespread use in the food packaging industry, and their mechanical integrity is paramount to maintaining product appearance, freshness, and overall food safety. Current testing methods, such as tensile or puncture tests, do not necessarily correlate well with field damages that are observed to be scratch-like. The standardized linearly increasing load scratch test was investigated as a new means of evaluating the mechanical integrity of packaging films.

Mechanical clamp and vacuum fixtures were considered for securing the films to a set of backing materials and tested under various testing rates and film orientation conditions. Film performance was evaluated according to their puncture load. Based on the above study, the vacuum fixture offers the most consistent and meaningful results by providing a more intimate contact between film and backing and minimizing extraneous motion of the film during testing. Additional testing was also carried out on a commercial film to confirm similarity between damage observed in the scratched films and that from the field. The scratch test gave good correlation between field performance and scratch test results on a set of commercial films.

Scratch-induced damages on multi-layer commercial packaging films were investigated using cross- and longitudinal-sectioning. Scratch test results showed clear distinction between the two tested systems on both the inside and outside surfaces. Microscopy was performed to investigate the feasibility of utilizing this methodology as a tool for packaging film structure evaluation by determining the effect each layer has on the resistance of scratch damages. It was shown that the film showing superior scratch test results also shows significantly better stress distribution through its layers during the scratch test, as well as better layer adhesion during severe deformation.

The scratch test showed good correlation between field test results and lab results for the tested film systems, and could be used to replace unnecessary or expensive testing methods. In addition, the scratch test showed good ability to provide more in-depth analysis of tested films by allowing for layer-by-layer analysis of damages and layer adhesion after testing, unlike current testing methods.

NOMENCLATURE

FEM	Finite Element Modeling
LDPE	Low Density Polyethylene
LLDPE	Linear Low Density Polyethylene
MRE	Meal, Ready to Eat
met	Metalized
oPET	Oriented Polyethylene Terepthalate
PMMA	Poly(methyl-methacrylate)
MD	Machine Direction
TD	Transverse Direction
AS	Air Side
FS	Food Side
VLSCM	Violet Laser Scanning Confocal Microscope

TABLE OF CONTENTS

	Page
ABSTRACT	iii
NOMENCLATURE	v
TABLE OF CONTENTS	vi
LIST OF FIGURES	vii
LIST OF TABLES	ix
1. INTRODUCTION	1
2. EXPERIMENTAL	6
2.1 Model Film Systems	6
2.2 Test Variables	7
2.3 Scratch Damage Analysis	10
3. RESULTS AND DISCUSSION	13
3.1 Effect of Scratch Testing Parameters on Scratch Performance	13
3.2 Effect of Film Orientation and Film Side on Scratch Performance	18
3.3 Observations of Scratch Induced Damages	20
3.4 Correlation with Commercial Films	22
3.5 Scratch Test Results of Multi-Layer Films	27
3.6 Observations of Multi-Layer Film Scratch Induced Damages	30
4. CONCLUSIONS	45
REFERENCES	47
VITA	52

LIST OF FIGURES

		Page
Figure 1	Scratch-like damages in production packaging	2
Figure 2	Structure schematic for the tested System 1 and System 2 films	7
Figure 3	(a) Clamp fixture and (b) vacuum fixture	9
Figure 4	An example of periodic stick-slip behavior observed on the surface of the System 1 film	11
Figure 5	Rate effect on the stick slip point	14
Figure 6	Rate effect on puncture point	15
Figure 7	Effect of variable vacuum pressure on control film	16
Figure 8	Effect of backing material on scratch induced puncture load	17
Figure 9	Effect of PMMA backing material on critical loads for onset of stick-slip and puncture	18
Figure 10	Film side effect for the stick slip point	19
Figure 11	Film side effect for puncture point	20
Figure 12	Damages observed on control film surface using the clamp fixture	21
Figure 13	Damages observed on control film surface using the vacuum fixture	22
Figure 14	Damages observed on commercial film surface using the clamp fixture	23
Figure 15	Damages observed on commercial film surface using the vacuum fixture	23
Figure 16	Scratch test results for System 1 and System 2 films tested on both the inside and outside surface	28

Page

Figure 17 Illustration of deformation undergone by test films when tested on (a) inside and (b) outside surfaces 29

Figure 18 Top down view of a scratch on the System 1 film on the inside surface 31

Figure 19 Top down view of a scratch on the inside surface of the System 1 film after the sealant layers have been removed... 31

Figure 20 Top down view of a scratch on the outside surface of the System 1 film.. 32

Figure 21 Comparison between undamaged reference images for System 1 33

Figure 22 Comparison between undamaged reference images for System 2 33

Figure 23 Comparison between scratch damaged sample images for System 1 35

Figure 24 Comparison between scratch damaged sample images for System 2 36

Figure 25 Delamination occurring between polyolefin layer and oPET layer in System 1 without scratch induced damages.. 37

Figure 26 Cross-sectional imaging showing the progression of scratch induced damages on the layered structure of System 1, on the left, and System 2, on the right 39

Figure 27 Cross-sectional imaging of scratch induced damages on the outside surface of System 1, on the left, and System 2, on the right.................. 41

Figure 28 Puncture caused by failure of the oPET layer in System 1 42

LIST OF TABLES

Page

Table 1 Variable Vacuum Pressure Effect on LDPE Based Commercial Film 24

Table 2 Rate Effect on LDPE Based Commercial Film....................................... 24

Table 3 Backing Effect on LDPE Based Commercial Film................................. 25

Table 4 Ranked oPET/LDPE Commercial Films 26

1. INTRODUCTION

Polymeric packaging films dominate the food packaging industry, and their strength and integrity are important factors in maintaining product life, appearance, and freshness. While other properties are important in packaging films, including glass transition temperature, melting temperature, heat-seal temperature, biodegradable properties, printing capabilities, and oxygen and moisture permeability, the mechanical properties of packaging films that are relevant to field performance are not as easy to quantify. Current test methods used to evaluate the mechanical integrity of packaging films include puncture, Trouser tear, tensile, J-Integral, essential work of fracture, and Gelbo flex [1-4]. It is not clear which of these tests provides the most useful information required in determining the relevant mechanical properties needed to evaluate the mechanical integrity of each film during packaging assembly, transportation, and handling. The tests also do not take into account all relevant properties which could affect field performance, such as the coefficient of friction of the packaging surface. For example, a higher coefficient of friction could cause an increase in the stress build-up on the surface of a package, potentially causing an earlier failure than a similar material with a lower coefficient of friction. It is understood that often times the above test methods do not correlate well with field performance. Thus, it is desirable to adopt a new testing method that may correlate better with performance observed in the field and provide meaningful information for ranking and developing better packaging films.

This thesis follows the style of Journal of Materials Science.

Observations of several commercial packages indicated that failure was not necessarily a simple puncture, with observed field damages similar to a scratch. These scratch-like damages can be seen in Fig. 1. It would therefore be desirable to replicate these damages in a controlled fashion in order to provide meaningful, quantifiable testing results for packaging film performance evaluation in a laboratory setting.

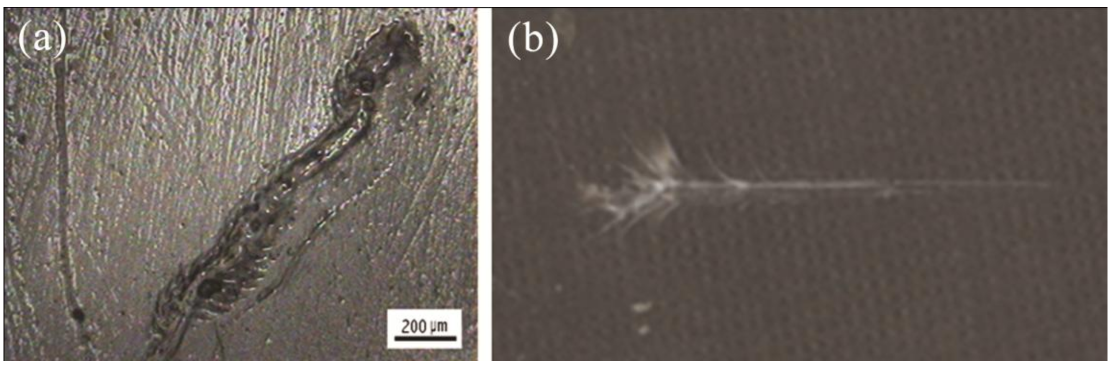

Fig. 1 Scratch-like damages in production packaging. (**a**) Microscopic damage and (**b**) macroscopic damage

Because of the complex requirements of food packaging films, multi-layer structures are typically utilized in order to fulfill different requirements. It is therefore important that each layer maintain its integrity and adhesion to the surrounding layers even under high imposed stress and strain conditions during filling, shipping, or handling. By understanding how specific layers of laminated films influence its resistance to scratch-induced damages, a better film structure can be engineered, allowing for better maintenance of product appearance, freshness, sterility, or other critical attributes. A key advantage of the scratch test over other testing methods is the potential ability to analyze damages associated with the test on a layer-by-layer basis through cross- and longitudinal-sectioning of scratch-damaged samples.

The ASTM polymer scratch test method was originally developed and demonstrated to be able to quantitatively evaluate the scratch performance of bulk polymer and coating samples [5-8]. This method utilizes a constant velocity linearly increasing normal load with a specialized tip traversed over a fixed length of the specimen. Significant research has been carried out in order to develop an objective method for standardized scratch testing [9-15], as well as research carried out to understand the effects of various parameters and material characteristics on the damage mechanisms associated with scratch. This includes research on the effect of additives [9,15-17], testing rate [18], and temperature [19]. Significant research has also been conducted on coating systems using various substrates, including determination of adhesion strength between coating and substrate [20-30] and wear and scratch resistance characterization [31-35]. In addition to experimental observations, numerical modeling has been performed on both bulk systems [36-38] and coating systems [39-48]. With this method, not only can damage type be assessed, but the damage transition zones can be seen and quantitatively pinpointed due to the correlation between normal load and scratch length. These findings have provided an excellent understanding of the scratch behavior and visibility of both bulk and coating systems, but little research has been done on scratch testing of polymeric films.

Due to the flexible nature of free standing films, applying the scratch test methodology requires the use of a backing material, which performs a similar function as the substrate in a coating system. Because of the free standing nature and thinness of the packaging films, often less than 100 micrometers thick, the effect of the backing material can have drastic influences on the scratch performance. Three-dimensional modeling using finite element methods (FEM) analysis has been performed to determine the stress states for coating systems with various thicknesses, and to correlate the stress states with damages observed during the scratch [39-42,44-48]. Based on these FEM modeling efforts, it is intuitively clear that the film thickness will have a notable effect on its scratch performance, having thicker films performing better.

Prior research has been performed attempting to apply the scratch testing methodology to films, and to correlate other test results to the scratch performance [49]. Based on a series of laminated films, it was shown that there tended to be a good correlation between scratch-induced failure loads and puncture test loads. This trend continued when tests were performed at low (-30°C) and elevated (82°C) temperatures. It was seen that the failure loads from the scratch test dropped significantly with an increase in temperature. High temperature performance of the tested films was confirmed to degrade through tensile tests, specifically a decrease in Young’s modulus and tensile strength. Additional damages were also observed with the scratch test, including layer delamination and foil layer failure prior to puncture. It was also shown that the scratch rate had a small but noticeable effect on the film tested, and was expected to exhibit similar trends for other packaging films. Testing also confirmed that scratch performance may vary with film orientation and the backing material utilized, such as aluminum or rubber.

Current research focuses on refining the testing parameters used during the scratch test so that reproducible, reliable, and meaningful results are obtained. The scratch test variables that will be tested include the fixture used to secure the film, scratch speed, backing material, film orientation, and film side. It is hoped that the present study will help develop an acceptable test methodology that can be reliably utilized for evaluation of packaging film mechanical integrity in a lab setting, but correlating well with their field performance. Current research also investigates the feasibility of utilizing the scratch test to investigate the effect a film structure has on the resistance of scratch-induced damages on a layer-by-layer basis. By combining scratch test results with the layer analysis of the damaged sample, a film can be more quickly and easily evaluated and retailored to meet the specific needs of the product.

2. EXPERIMENTAL

2.1 Model Film Systems

The model material used in the test method development experiments is a nominal 7.0 mil (177.8 μm) thickness low-density polyethylene (LDPE)/linear-low-density polyethylene (LLDPE) blend single-layer film provided by Cadillac Products Packaging Company (Troy, MI). This film was created as a high strength replacement for Meal, Ready to Eat (MRE) menu bags. Commercial films were also tested, including a 3.0 mil (76.2 μm) LDPE based film, and a set of three multi-layer oriented polyethylene terephthalate (oPET)/LDPE films with a total thickness of 97 μm, 88 μm, and 123 μm, respectively.

Two commercially available model film systems were used in these experiments. The first is a three-layer film with nominal thickness of 3.5 mil (88.9 μm), containing a metalized oPET film laminated to coextruded with two additional polyolefin films. The second is a four-layer film with a nominal thickness of 3.2 mil (81.3 μm), containing a metalized oPET film laminated to coextruded films containing both nylon and polyolefins. A schematic of their structures can be seen in Fig. 2. As shown, the interior surface of the film refers to the scratch tip being initially in contact with the polyolefin sealant layer, and the exterior surface of the film refers to the scratch tip being initially in contact with the oPET layer. The films are labeled as System 1 and System 2, respectively.

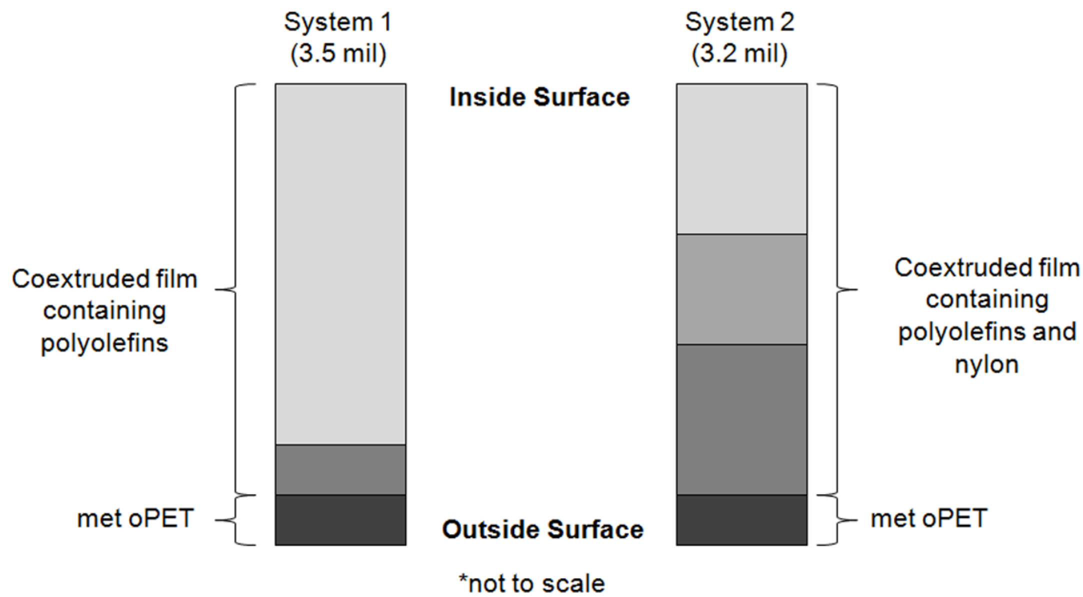

Fig. 2 Structure schematic for the tested System 1 and System 2 films

Scratch test specimens were cut to 6” in length, and 2.5” wide (15.24 cm x 6.35 cm) for vacuum fixture samples, and 1” wide (2.54 cm) for clamp fixture samples. Scratch testing was performed on a fourth generation scratch machine built by Surface Machine Systems. A minimum of four samples were used for each test.

2.2. Test Variables

The testing methodology for this project is based on the current progressive load scratch testing standard, ASTM D7027-05/ISO 19252:2008, but modified for use with free standing films [5,6]. A number of variables were investigated to determine an acceptable testing methodology for packaging films. These variables were also used to learn how free standing films behave when subjected to the progressive load scratch test.

The scratch test parameters investigated included the method with which the film is secured, the scratch speed, the amount of vacuum drawn on the specimen when using the vacuum fixture, and the backing material. The film variables investigated included film side and film orientation.

2.2.1 Scratch Test Parameters

The scratch tip used for these tests comes from the original ASTM standard, and is a 1mm diameter spherical stainless steel tip. Different tip geometries can also be used depending on field conditions and preference.

The loading applied to the film is dependent on the film and the backing material on which it is being tested. The criteria for selecting a loading level is to ensure that the puncture caused by the scratch test does not occur too soon or too late in the test, but will still occur in a repeatable fashion. Generally, it is desirable for the puncture to occur between 50-75% of the scratch length. In general, the loading range for the films tested was set at 1 – 30N.

Because free standing films are so flexible, the method with which to secure the film was also investigated. Two fixture types were used to secure the film during the scratch test: a mechanical clamp fixture, which holds the film around the edges of the scratch path, and a vacuum fixture, which draws a vacuum beneath the sample and uses ambient air pressure to secure the film to the backing. Images of these two fixtures can be seen in Fig. 3.

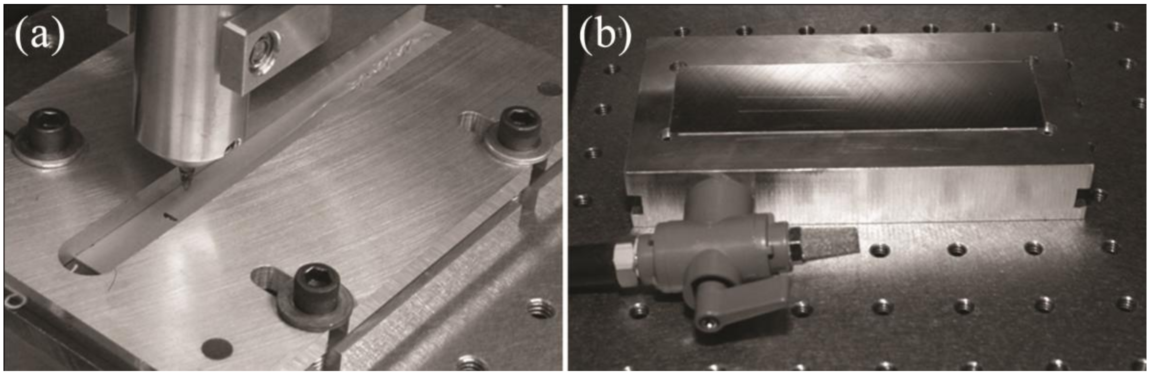

Fig. 3 (**a**) Clamp fixture and (**b**) vacuum fixture

Since mechanical properties of polymers can be highly rate dependent due to their viscoelastic nature, scratch speeds at 1 mm/s, 10 mm/s, and 100 mm/s were chosen to determine how the scratch behavior varies with rate of testing. These speeds were chosen to correlate with typical filling, transport, and handling speeds, respectively, experienced by food packaging films. The scratch length was set at a constant of 100 mm.

The amount of vacuum drawn on the film was also investigated, with vacuum pressures of 25 in. Hg, 15 in. Hg, and 5 in. Hg (84.65 kPa, 50.79 kPa, and 16.93 kPa). Finally, because packaging films are so thin, their performance can be significantly influenced by the backing material they are tested on. This is analogous to a coating's performance differing based on different substrate type [49,31,50]. In this regard, the backing material used can be selected to create a laboratory analog with which to mimic real life performance. For example, if a film is to experience field conditions that subject it to contact with metal or very hard surfaces, an aluminum backing can be used. Conversely, a neoprene rubber backing could be used to simulate a softer backing such as air. Additional backings could be used depending on the product requirements and the meaningfulness of the results acquired.

All tests were performed at room temperature.

2.2.2 Film Property Variables

To address the film orientation effect due to blown film processing, from which the molecular chains are likely to orient on the stretching or extrusion direction, two different film orientations were tested: the Machine Direction (MD), and the Transverse Direction (TD).

The different sides of the film were tested, and are labeled as Air Side (AS) and Food Side (FS). The sides were differentiated based on their intended use in field as MRE menu bags. The different sides for the films used during multi-layer film analysis are labeled as the interior and exterior surfaces, and are differentiated by their intended use in field service.

2.3 Scratch Damage Analysis

Similar to scratch testing of bulk polymers, stick-slip behavior can be observed on packaging films when tested against a relatively stiff backing material, such as PMMA or aluminum [43,51]. According to Jiang et al, while the scratch test is designed to move at a constant velocity, the actual tip velocity oscillates due to the surface contact between the tip and the tested material [11]. Stick-slip behavior is observed when the tip induces large deformation of the material, resulting in a build-up of material in front of the tip, which introduces additional resistance force. When this stored strain energy becomes greater than the resistance due to both friction and material deformation, the scratch tip moves over the material hindrance and resumes moving forward at full speed. This process repeats itself, resulting in a periodic stick-slip pattern, which can be readily observed through optical microscopy of the film surface (Fig. 4).

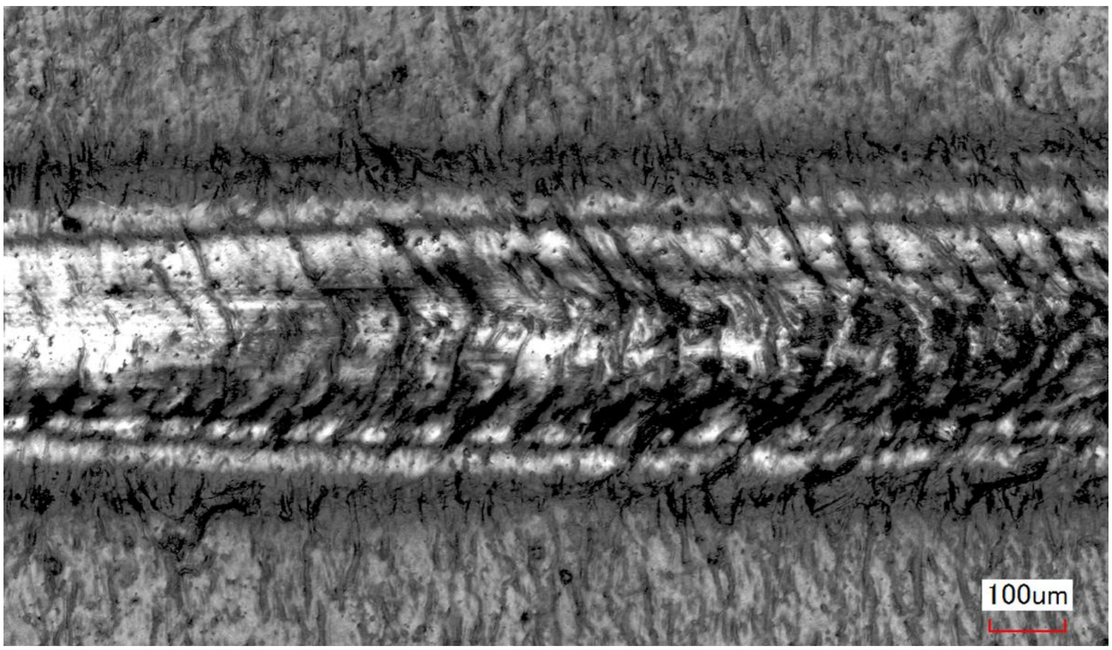

Fig. 4 An example of periodic stick-slip behavior observed on the surface of the System 1 film

The point at which the first puncture occurred was analyzed visually, and confirmed using an Olympus BX60 optical microscope. The point at which puncture occurred was measured using a millimeter scale ruler. The load at which puncture occurred was determined directly from the output of the scratch machine and based on the measurement of the distance from the start of the scratch test to where the puncture occurred.

Cross- and longitudinal-sectioning of scratch damaged samples was performed. Samples were sectioned by isolating the entirety of the scratch from the test sample, resulting in a sample approximately 10mm wide by 110mm long. The film was placed on a glass backing with the oPET layer in contact with the glass. A fresh razor blade was pressed straight down through the soft polyolefin layers towards the oPET layer perpendicular to the scratch direction at the location of interest, resulting in a cross-sectional view of the damaged film at the desired location when viewed looking head-on to the scratch path. Similarly, longitudinal-section cuts were made parallel to the scratch direction and in the center of the scratch path, thus allowing a view of the damage alongside the scratch path. Sectioned samples were placed in modeling clay for stabilization and viewed under a Keyence VK-9700 Violet Laser Scanning Confocal Microscope (VLSCM). To confirm that the sectioning method described above did not introduce artifacts, sectioned film samples were placed in epoxy and allowed to set for over 24 hours, followed by polishing using sandpaper followed by polishing cloths using a solution of 0.3μm alumina oxide to achieve a 0.4 μm average smoothness for VLSCM observation. Microscopy images of the damaged film samples prepared between the two methods were then compared.

3. RESULTS AND DISCUSSION

3.1 Effect of Scratch Testing Parameters on Scratch Performance

3.1.1 Fixture Analysis

Since the clamp fixture secures the sample around its edges, there is no intimate contact between the film and the backing except immediately beneath the scratch tip and around the edges where they were secured. Because of this, the film deforms and buckles uncontrollably over the length of the scratch test. The vacuum fixture draws a vacuum beneath the edges of the sample, allowing atmospheric air pressure to push the sample onto the fixture. This provides intimate contact between the film and the backing whether the tip is in contact with the film or not. This results in a controlled buckling of the film during the scratch.

3.1.2 Scratch Rate

From food filling to transport to handling, packaging films can experience a wide range of damages introduced at differing speeds. The critical loads for the formation of the stick slip point for all the films investigated are shown in Fig. 5, and those for the puncture point shown in Fig. 6. Tests were performed on the food side of the film using aluminum as the backing material. As seen in Fig. 5, the films do show some rate dependency on the critical loads for the onset formation of stick-slip damage, with an overall decrease in performance at slower test speeds. Due to the complex multi-axial stress state involved during scratch testing, no explanation is currently given for this behavior, as this section focuses on test method development. What can be definitively seen is that for this model system, there is a noticeable rate dependency on scratch test results. As can also be seen, the vacuum fixture produces consistent trends with lower standard deviations than the clamp fixture. The critical loads for puncture formation show similar trends, as seen in Fig. 6. Again, the vacuum fixture shows more consistent trends with lower standard deviations. For the tested film, it can be seen that the largest differences between the different film orientations can be observed at a slower testing speed.

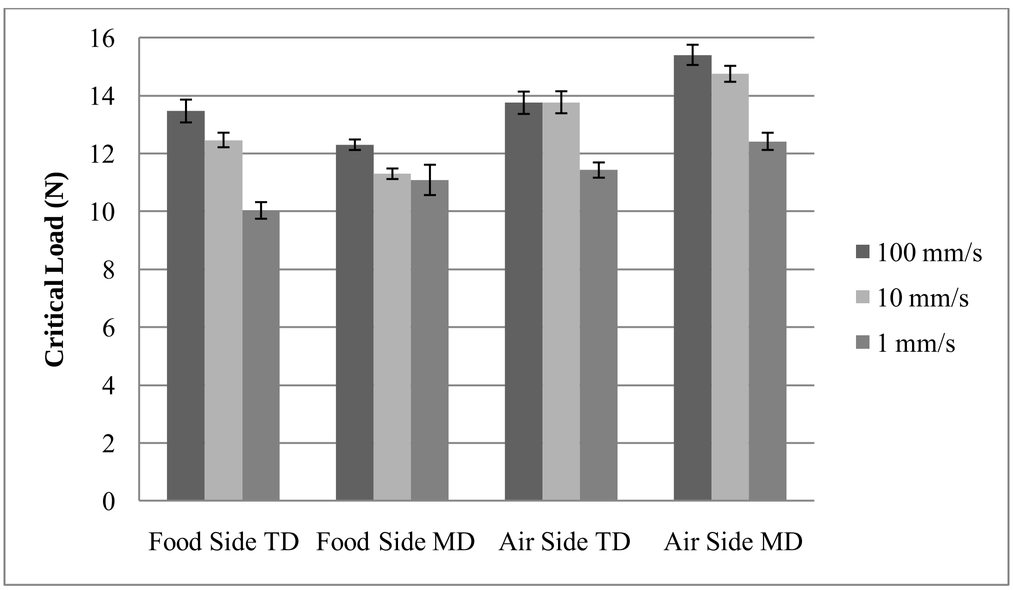

Fig. 5 Rate effect on the stick slip point

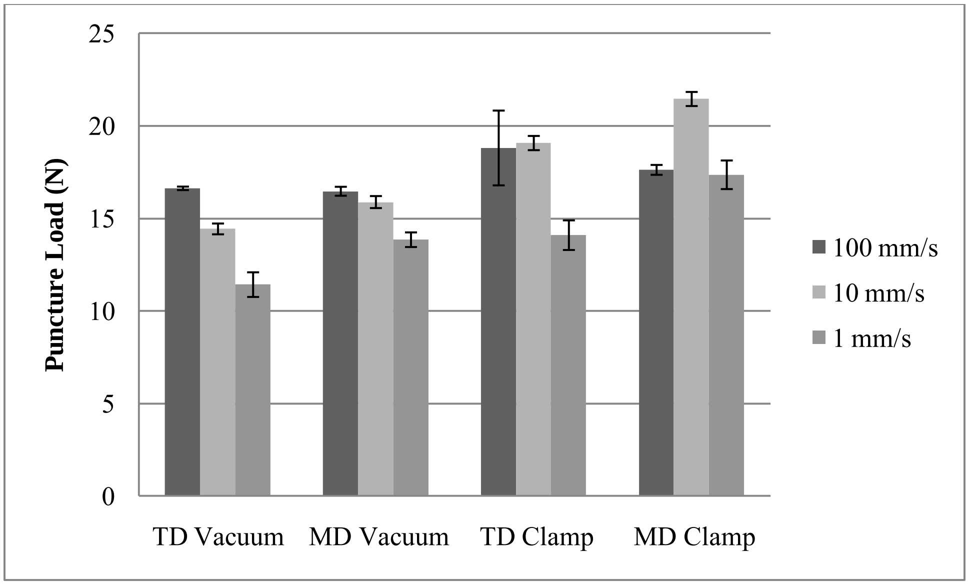

Fig. 6 Rate effect on puncture point

3.1.3 Vacuum Pressure

The amount of vacuum drawn was varied to 25, 15, and 5 in. Hg. This corresponds to a holding pressure of 12.3 psi, 7.4 psi, and 2.5 psi, respectively (84.65 kPa, 50.79 kPa, and 16.93 kPa). The test was performed on the air side of the control film on an aluminum backing at 100 mm/s. The results from the test for the MD orientation can be seen in Fig. 7. As seen in the figure, the critical load for stick-slip formation shows a minor decrease with a decrease in holding pressure, and there is a relatively minor decrease in the loads required to cause puncture despite a large range of holding pressure changes. Vacuum pressures below 5 in. Hg (2.5 psi, 16.93 kPa) were also tested, but the tests could not be completed as the film was pulled from the fixture during the test. This indicates that results should remain relatively consistent with the range of holding pressures used here, with the best performance seen by using the maximum holding pressure of 85 kPa.

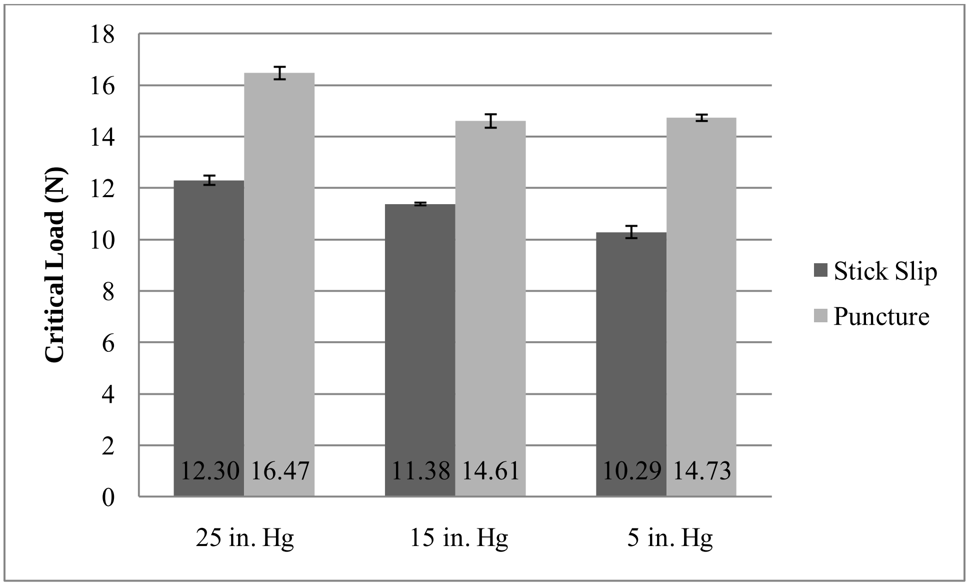

Fig. 7 Effect of variable vacuum pressure on control film

3.1.4 Backing Material

The backing on which the film was tested was changed from aluminum to rubber in order to provide two extreme scenarios for testing conditions. Tests were performed on the air side of the film at 100 mm/s with the vacuum fixture. Because the rubber readily deforms beneath the scratch tip, there is no large-scale stick-slip point as with a hard backing, thus the evaluation method to compare the two backings is based on the first instance of puncture. The results are shown in Fig. 8. As seen in the figure, the use of a rubber backing delays puncture, as the film is able to move around the tip allowing it to avoid the tear induced puncture seen with the aluminum backing. While there appears to be only a minor difference between values for MD and TD orientation, the rubber backing seems to neutralize any anisotropy effect due to more uniform deformation of the film around the scratch tip.

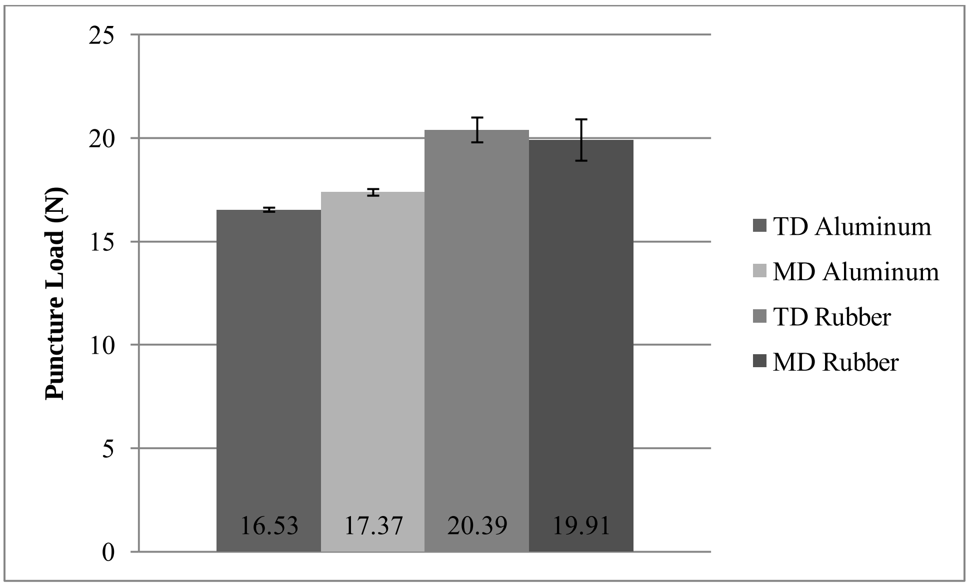

Fig. 8 Effect of backing material on scratch induced puncture load

The films were also tested using a poly(methyl methacrylate) (PMMA) backing. Tests were performed on the food side of the film using the vacuum fixture at a scratch rate of 100 mm/s. The critical loads for both the initial stick-slip point and the first instance of puncture are shown in Fig. 9. As seen, there is no significant difference in the critical loads for either metric. This finding indicates that any sufficiently rigid backing compared should provide similar results. This information correlates well with the work by Jiang *et al* who found that the scratch performance of a coating will not show change with a ratio of yield strengths between the film and the substrate above 0.5 [39].

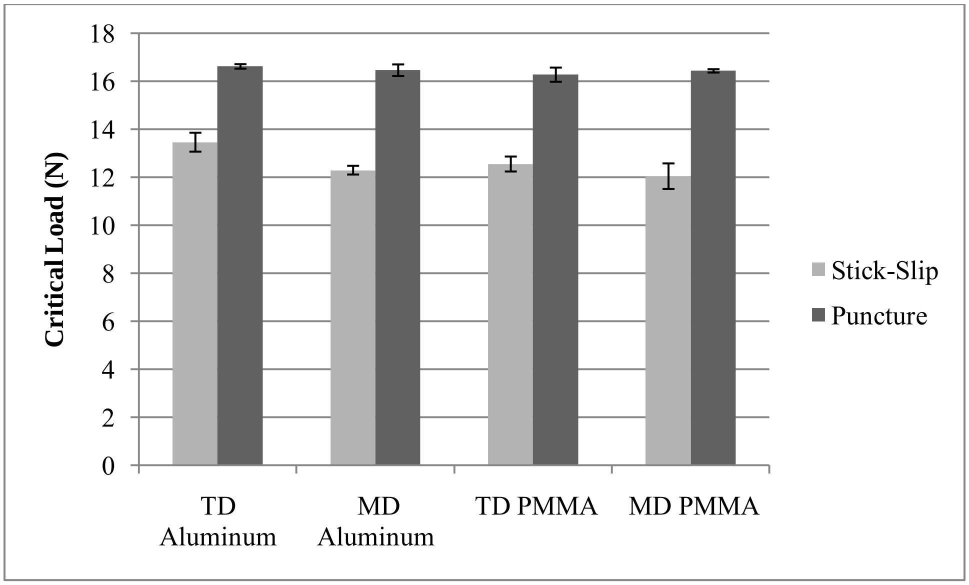

Fig. 9 Effect of PMMA backing material on critical loads for onset of stick-slip and puncture

3.2 Effect of Film Orientation and Film Side on Scratch Performance

As the vacuum fixture provided the most consistent and more meaningful results, it was used to further test the food and air sides of the film. For consistency and ease of analysis, tests were performed on an aluminum backing at 100 mm/s, 10 mm/s, and 1 mm/s. The critical loads for the stick-slip point are shown in Fig. 10, and those for the first instance of puncture in Fig. 11. As seen in the figures, the film side can have a dramatic impact on the scratch performance. This can be caused by different surface characteristics of the films, processing effects, or film structure in the case of multi-layered films. In this case, the control film has a different texture on the air side than the food side. Again, the same trend of decreasing performance with decreasing testing speed is evident, with good repeatability for the formation of both stick-slip and puncture.

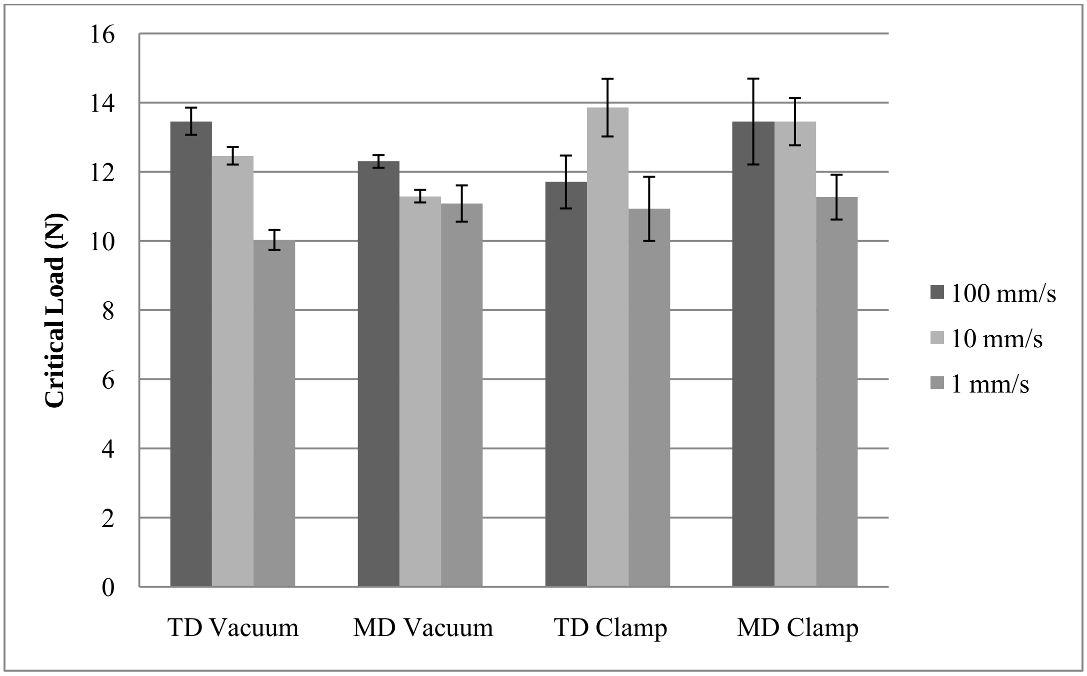

Fig. 10 Film side effect for the stick slip point

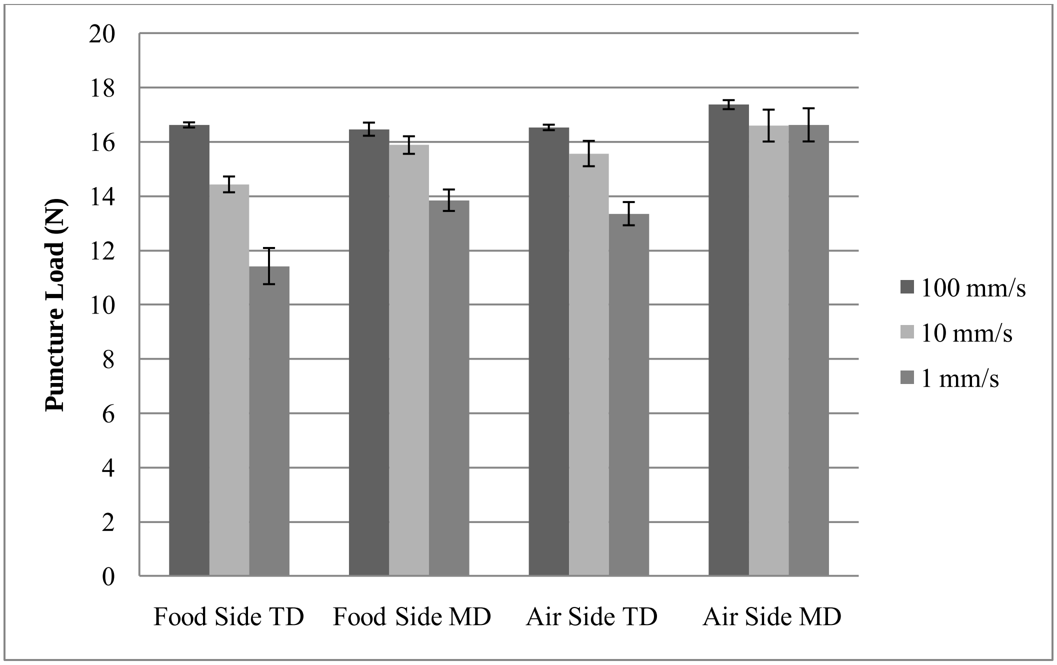

Fig. 11 Film side effect for puncture point

3.3 Observations of Scratch Induced Damages

Typical scratch induced damages along the length of the scratch are presented for the tested film in Fig. 12 and Fig**. 13** for the clamp and vacuum fixture, respectively. As seen in the figures, both the macro-scale and micro-scale damages for the film show numerous similarities to the damages seen in field (Fig. 1). For the control film, the damages between the two types of fixtures look similar. Damage begins with smooth deformation and transitions into stick-slip behavior, followed by a tear-induced puncture behind the scratch tip. The vacuum fixture shows much more uniform damages over the entire scratch length, attributed to the intimate contact between film and backing. The vacuum fixture introduces damage features more similar to those seen in the bulk material than those introduced by the clamp fixture, indicating good materialistic response of the film, absent of bucking induced damages caused by poor contact between film and backing. Because of the consistent trends in testing performance and the more uniform and meaningful scratch damages produced by the vacuum fixture, it is recommended that vacuum fixture be utilized for securing polymeric films for scratch testing.

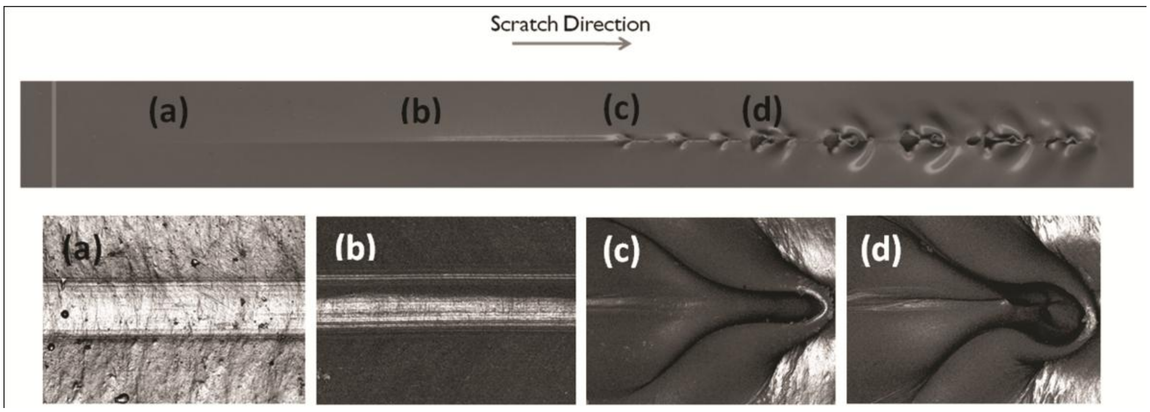

Fig. 12 Damages observed on control film surface using the clamp fixture. (**a**) Smooth deformation, (**b**), continued smooth deformation, (**c**) stick-slip point, and (**d**) puncture. Test performed on aluminum backing at 100 mm/s scratch rate, using 1mm SS spherical tip

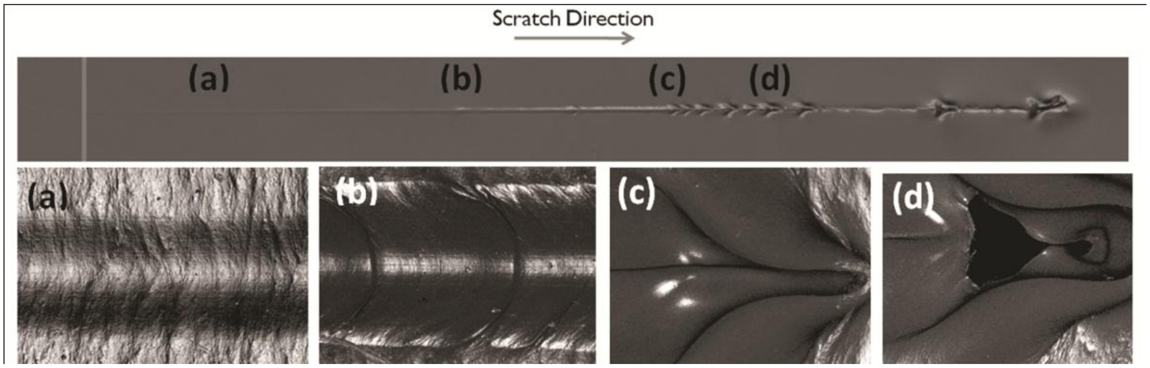

Fig. 13 Damages observed on control film surface using the vacuum fixture. (**a**) Smooth deformation, (**b**) small-scale stick-slip, (**c**) stick-slip point, and (**d**) puncture. Test performed on aluminum backing at 100 mm/s scratch rate, using 1mm SS spherical tip

3.4 Correlation with Commercial Films

A 3.0 mil (76.2 µm) nominal thickness film used by a large commercial food packaging corporation was also tested. Products in field were observed to have scratch-like damages on their packaging (Fig. 1). Damages observed by the clamp and vacuum fixtures were similar to the control film, and can be seen in Fig. 14 and Fig**. 15**. The damages observed in the commercial film are similar to what is seen in the control film, with uniform damages similar to that of the bulk when tested with the vacuum fixture.

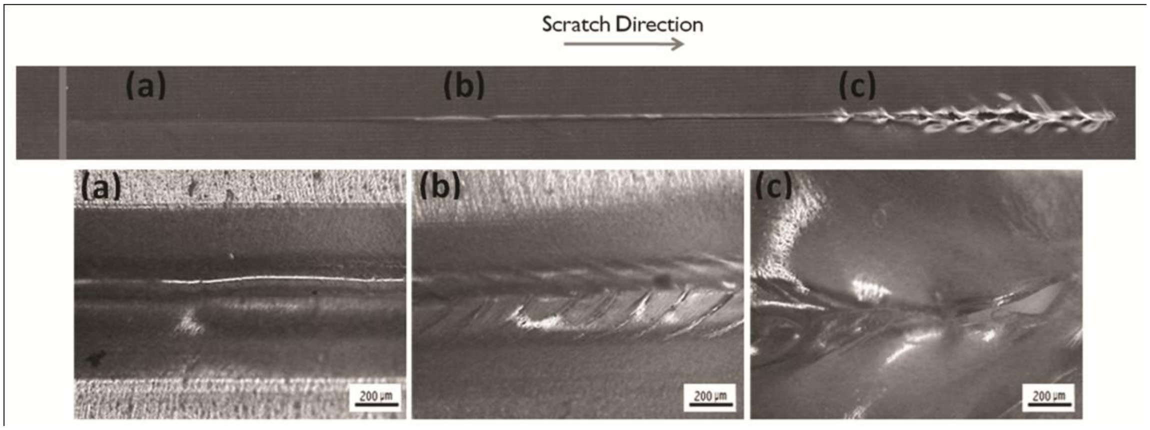

Fig. 14 Damages observed on commercial film surface using the clamp fixture. (**a**) Smooth deformation, (**b**), periodic stick-slip, and (**c**) puncture. Test performed on aluminum backing at 100 mm/s scratch rate

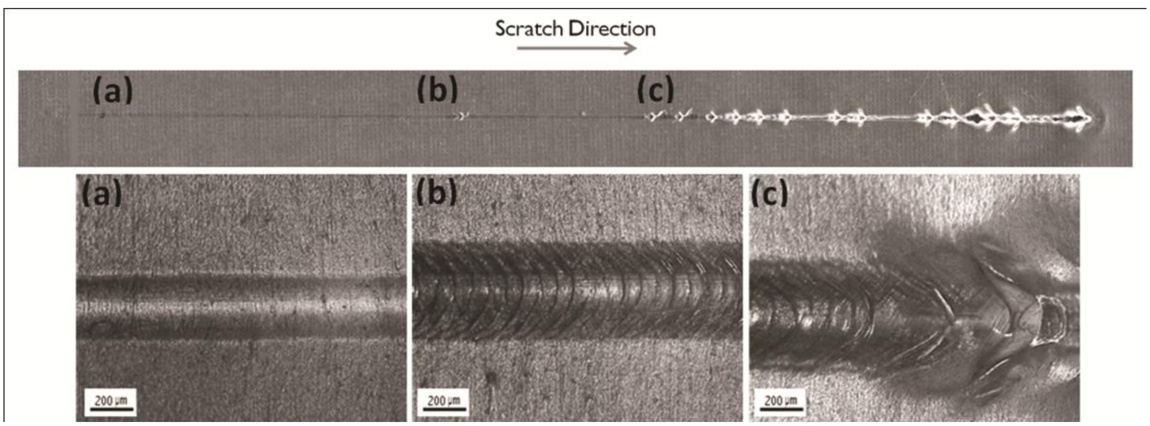

Fig. 15 Damages observed on commercial film surface using the vacuum fixture. (**a**) Smooth deformation, (**b**) stick-slip, and (**c**) puncture. Test performed on aluminum backing at 100 mm/s scratch rate, using 1mm SS spherical tip

When testing the effect of vacuum pressure, the control film shows similar minor decrease in performance as the control film did, and was consistent between the MD and TD orientations (Table 1). Interestingly, the commercial film showed no scratch rate dependency, with uniform onset puncture performance with the differing scratch rates.

While this is unlike the control film, it is evidence that different films can show differing behaviors depending on structure, as expected (Table 2).

Table 1 Variable Vacuum Pressure Effect on LDPE Based Commercial Film

Film	Orientation	kPa	Puncture (N)	Std. Dev. (N)
LDPE Commercial Film	TD	85	11.21	0.23
LDPE Commercial Film	TD	51	11.02	0.18
LDPE Commercial Film	TD	17	10.31	0.39
LDPE Commercial Film	MD	85	11.17	0.58
LDPE Commercial Film	MD	51	11.03	0.86
LDPE Commercial Film	MD	17	10.67	0.51

Table 2 Rate Effect on LDPE Based Commercial Film

Film	Orientation	Scratch Rate	Puncture (N)	Std. Dev (N)
LDPE Commercial Film	TD	100 mm/s	11.82	0.61
LDPE Commercial Film	TD	10 mm/s	12.19	0.36
LDPE Commercial Film	TD	1 mm/s	11.27	1.08
LDPE Commercial Film	MD	100 mm/s	12.12	0.71
LDPE Commercial Film	MD	10 mm/s	12.83	0.06
LDPE Commercial Film	MD	1 mm/s	12.70	0.27

The LDPE based commercial film was tested on an aluminum backing and a PMMA backing. The test was performed at 100 mm/s on the vacuum fixture. The results are summarized in Table 3. As seen, there is no significant difference in scratch load required to create a puncture between the aluminum and PMMA backings. These results correlate well with the control film in Fig. 9.

Table 3 Backing Effect on LDPE Based Commercial Film

Film	Orientation	Backing	Puncture (N)	Std. Dev (N)
LDPE Commercial Film	TD	Aluminum	11.82	0.61
LDPE Commercial Film	MD	Aluminum	12.12	0.71
LDPE Commercial Film	TD	PMMA	11.28	0.92
LDPE Commercial Film	MD	PMMA	11.45	0.70

Additionally, three commercial films intended for use in the same product as used by a major food packaging company were also tested. These films were ranked in field performance as good, better, and best. The “good” film is nominally 97 µm thick; the “better” film is nominally 88 µm thick; the “best” film is nominally 123 µm thick. The films were tested using the vacuum fixture at 100 mm/s using a PMMA backing. The results are shown in Table 4. As seen in the table, the critical load required for scratch induced puncture increases in both TD and MD orientations. While the thicknesses are different between the three films, the increase in the puncture loads is significantly more than the change in thickness would indicate. Despite the “better” film being thinner than the “good” film, it still performs better under the scratch test. This indicates that the scratch test could be used as a testing method to evaluate film performance, and to better correlate lab results to field results.

Table 4 Ranked oPET/LDPE Commercial Films

	Orientation	Puncture (N)	Std. Dev. (N)
"Good"	TD	9.87	0.72
"Better"	TD	11.07	1.13
"Best"	TD	17.1	1.86
"Good"	MD	10.57	0.46
"Better"	MD	10.85	0.46
"Best"	MD	16.98	0.58

As most packaging films contain multi-layer structures either through co-extrusion or lamination, their mechanical integrity depends not only on the material performance of each layer but also on how the multi-layer films are formed. In the case of the laminated multi-layer films, the choice of tie layers and their thicknesses can critically influence their performance. It is therefore important that the lab testing protocol be able to differentiate how the above factors can individually affect the packaging film performance. The proposed scratch testing methodology appears to have the potential to address the above concerns. It has been shown earlier that the scratch test can reveal the load from which delamination takes place in some foil laminated films [49] and on some coating systems [50]. It is therefore possible to analyze specific failures between layers or within a layer itself through three-dimensional imaging of transparent samples via laser confocal microscopy or through other microscopy tools for opaque samples. By understanding how and where the laminate structure fails, a packaging film can be more accurately analyzed and its structure formulated to better resist scratch-induced damages. Consequently, it can be logically seen that the proposed scratch test can serve as an effective tool for both evaluation and development of packaging films with greatly improved mechanical integrity.

3.5 Scratch Test Results of Multi-Layer Films

Films were subjected to the modified scratch test methodology described previously to determine mechanical integrity and scratch performance. The results can be seen in Fig. 16. Because these films provided were used in commercial applications, it is important to compare these laboratory results to field performance. Field performance data indicates that System 2 is the superior film, and that the majority of punctures were initiated from the exterior surface. This correlates well with the data generated by the scratch test, with System 2 showing significantly better performance on both the interior and exterior surfaces compared to System 1. Additionally, the lowest puncture loads are found when testing the exterior surface of the films.

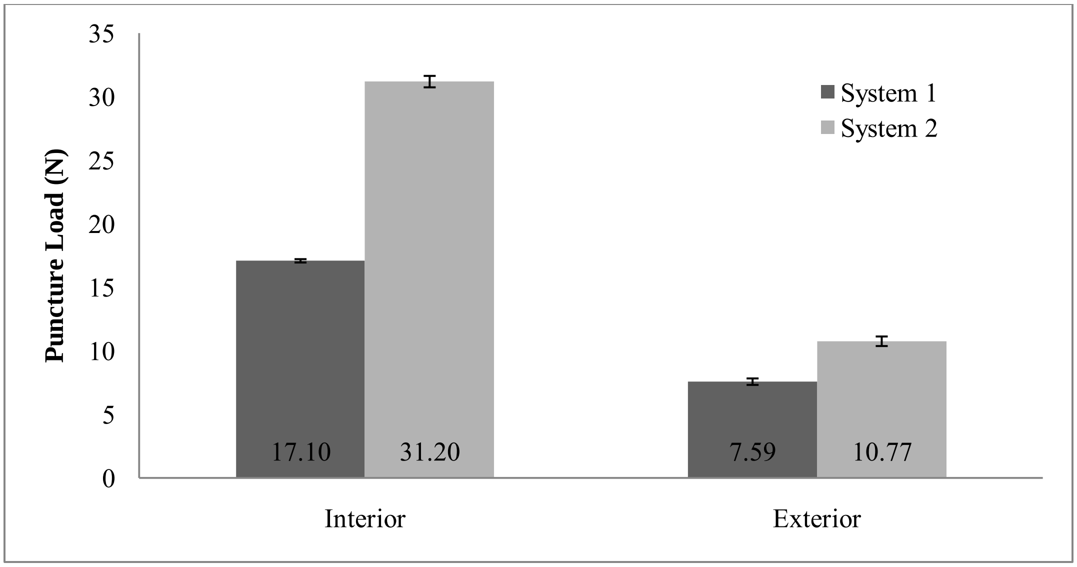

Fig. 16 Scratch test results for System 1 and System 2 films tested on both the inside and outside surface. Films were tested in the TD orientation at 1 mm/s on a PMMA backing

The difference in scratch performance between the two film sides can be attributed to the structure of the films. The scratch behavior of the films shows strong similarity to the scratch behavior of coating systems. Jiang, *et al* performed modeling of soft coatings on hard substrates and soft coatings on hard substrates and found that when a soft coating on a hard substrate undergoes a scratch, only the coating layer shows significant deformation, while the substrate remains undamaged. However, when a hard coating is tested on a soft substrate, the soft substrate deforms along with the hard coating layer, distributing the stresses over a larger area [50]. In relation to the tested film systems, the soft coating/substrate corresponds with the polyolefin sealant layers and the hard coating/substrate corresponds with the oPET layer. As shown in Fig. 17a, when tested on the exterior surface, the polyolefin sealant layers will show significant damage, while the oPET layer will remain undamaged. Puncture occurs after the scratch tip removes the sealant layers entirely and traverses across the oPET layer until failure. When tested on the outside surface, illustrated in Fig. 17b, the oPET layer deforms along with the polyolefin sealant layers beneath it. When the normal load increases to a critical point, the oPET layer can no longer conform to the deformation of the soft sealant layers, and subsequently breaks. Following this, the scratch tip pushes easily through the softer layers, generating a puncture.

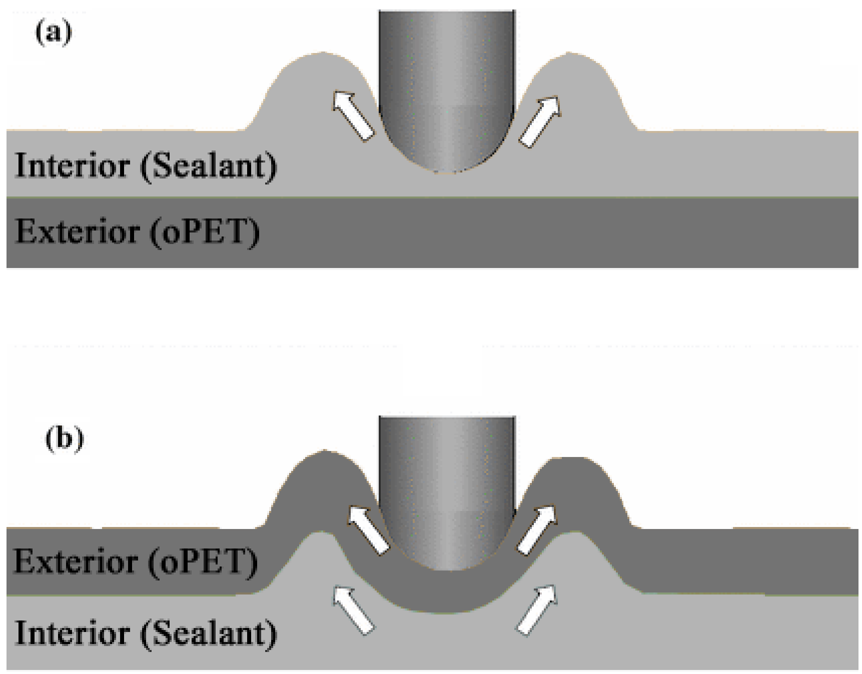

Fig. 17 Illustration of deformation undergone by test films when tested on (**a**) inside and (**b**) outside surfaces. Illustration adapted from [50]

3.6 Observations of Multi-Layer Film Scratch Induced Damages

3.6.1 Top View of Interior Film Surface of Multi-Layer Films

Both systems deform in a similar manner, and show similar damage features when viewed from the top of the scratch. Microscopy was performed to determine major damage feature and their transitions during the scratching process. The representative damage features are shown based on System 1, but are applicable to System 2, as well. Subtle scratch damage is readily visible at the start of the test, beginning with smooth deformation, seen in Fig. 18a. As the normal load increases, damage becomes more severe, transforming into periodic stick-slip behavior similar to bulk material, seen in Fig. 18b. Stick-slip behavior continues and becomes more severe as the load increases. Eventually, the polyolefin sealant layers are removed, fully exposing the oPET layer to the scratch tip, seen in Fig. 18c. Following the sealant removal point, the scratch tip continues traversing along the oPET layer, damaging it while pushing the sealant layers to the edge of the scratch path, shown in Fig. 19. The critical load is reached and the oPET layer fails, generating puncture.

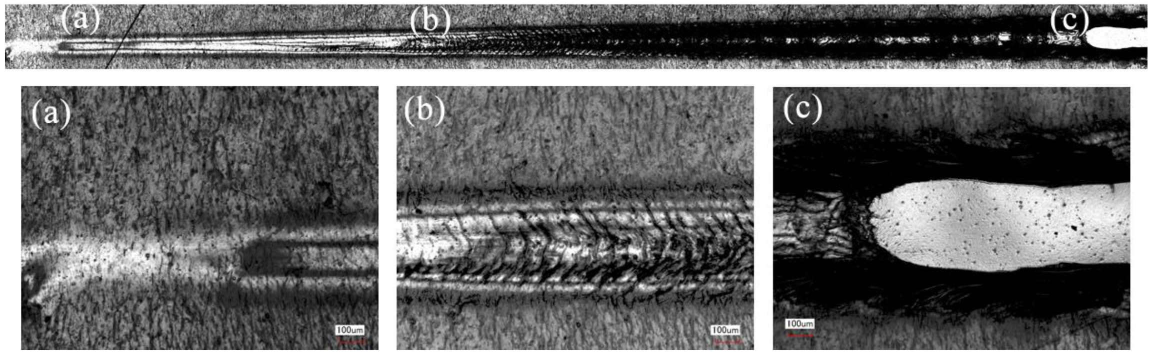

Fig. 18 Top down view of a scratch on the System 1 film on the inside surface. (**a**) Smooth deformation, (**b**) transition to periodic stick slip behavior, and (**c**) removal of sealant layers

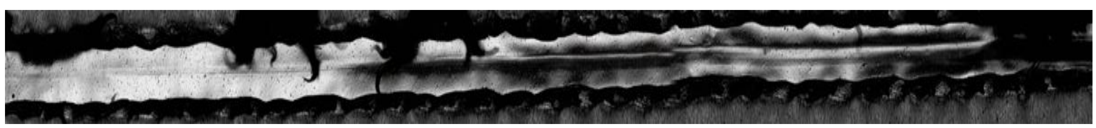

Fig. 19 Top down view of a scratch on the inside surface of the System 1 film after the sealant layers have been removed

3.6.2 Top View of Exterior Film Surface of Multi-Layer Films

As with the interior surfaces, both model systems show similar damages when viewed from a top down view of the interior film surfaces. Damage is slightly visible from the beginning of the test, seen in Fig. 20. However, because of the significantly lower loads involved and the sealant layers distributing the stress beneath the tip over a larger area, the damages at the start of the test are much less severe. As the normal load increases, smooth deformation becomes noticeably visible, seen in Fig. 20a. Smooth deformation eventually leads to large scale stick-slip behavior, Fig. 20b left, as the tip moves over the buildup of material in front of it. Once the critical load is reached, the sealant layers deform to a point that the oPET layer cannot, resulting in failure of the oPET layer. Following failure, the exposed sealant layers are easily removed by the scratch tip, resulting in puncture as seen in Fig. 20c right.

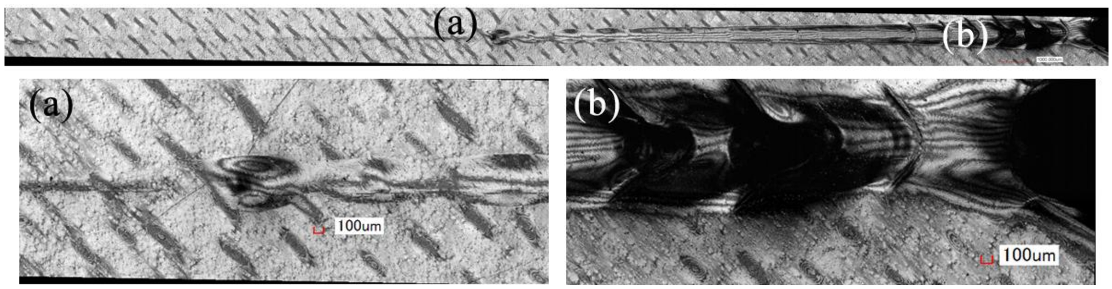

Fig. 20 Top down view of a scratch on the outside surface of the System 1 film. (**a**) Beginning of visible smooth deformation, and (**b**) stick slip and puncture

3.6.3 Cross- and Longitudinal-Section View of Multi-Layer Film Reference Samples

Cross- and longitudinal-sectioning of the scratch-damaged samples allows us to investigate, on a layer-by-layer basis, the damages associated with the scratch test and how each layer contributes to resisting scratch damage. The sectioning and imaging method used in this study utilizes freshly cut samples for imaging without sample embedding and polishing, as is customary for optical microscopy sample preparation. It is important to verify that any artifacts generated do not noticeably affect the analysis of the damages observed using the direct razor blade cutting. Artifacts can include, but are not limited to, damages such as compression of the sealant layers or delamination between layers. Comparisons between razor blade cutting and polished undamaged reference samples for Systems 1 and 2 can be seen in Fig. 21 and Fig**. 22**. No delaminations were observed due to razor blade cutting, or after polishing. As shown in both figures, the films show good layer integrity and demarcation.

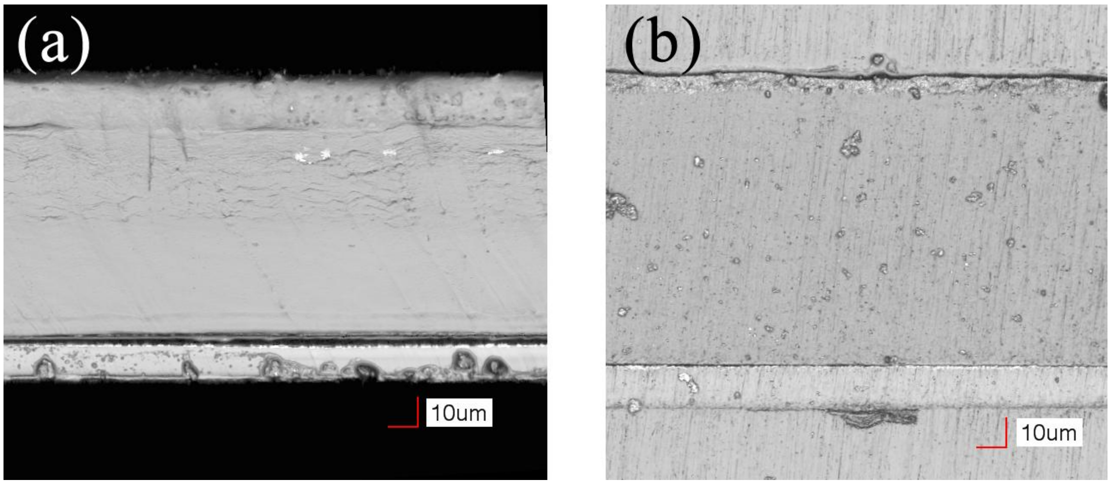

Fig. 21 Comparison between undamaged reference images for System 1. (**a**) Before and (**b**) after polishing

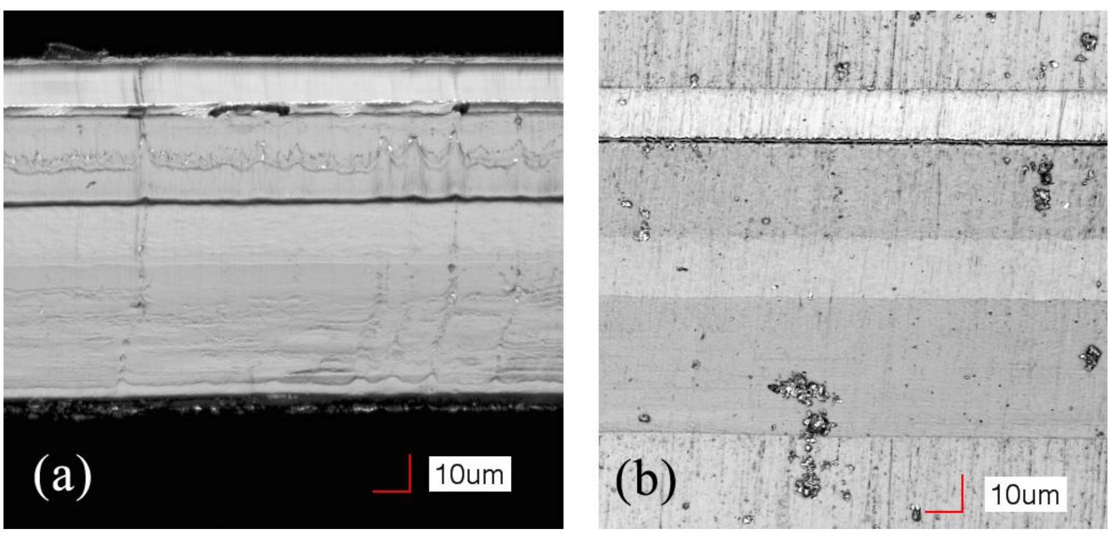

Fig. 22 Comparison between undamaged reference images for System 2. (**a**) Before and (**b**) after polishing

Verification of the sectioning and imaging method of scratch damaged regions was also completed. Cross-sections were taken from the late stick-slip region prior to sealant removal. The straight razor blade cutting and polished sample images for System 1 and 2 are shown in Fig. 23 and Fig**. 24**. Like the undamaged reference images, damaged samples show good layer demarcation, integrity, and adhesion, without observable delaminations or significant artifacts before or after razor blade cutting. The two images, while taken from the same location of the same sample, are not exactly the same as material is partially removed during polishing and possible partial relaxation of the deformed material during curing of the epoxy mount. Through these comparisons, it is seen that the sectioning and imaging method developed in this study allows us to effectively analyze scratch induced damages, layer integrity, and inter-layer adhesion of multi-layer packaging films without the need for additional polishing steps.

In all the undamaged samples observed, delaminations were rarely seen. System 2 was never observed to have delaminations between any layers. System 1 occasionally showed delaminations between the oPET layer and the sealant layers, an example of which is shown in Fig. 25. This indicates that, even before undergoing damage, the adhesion between these two layers is not ideal.

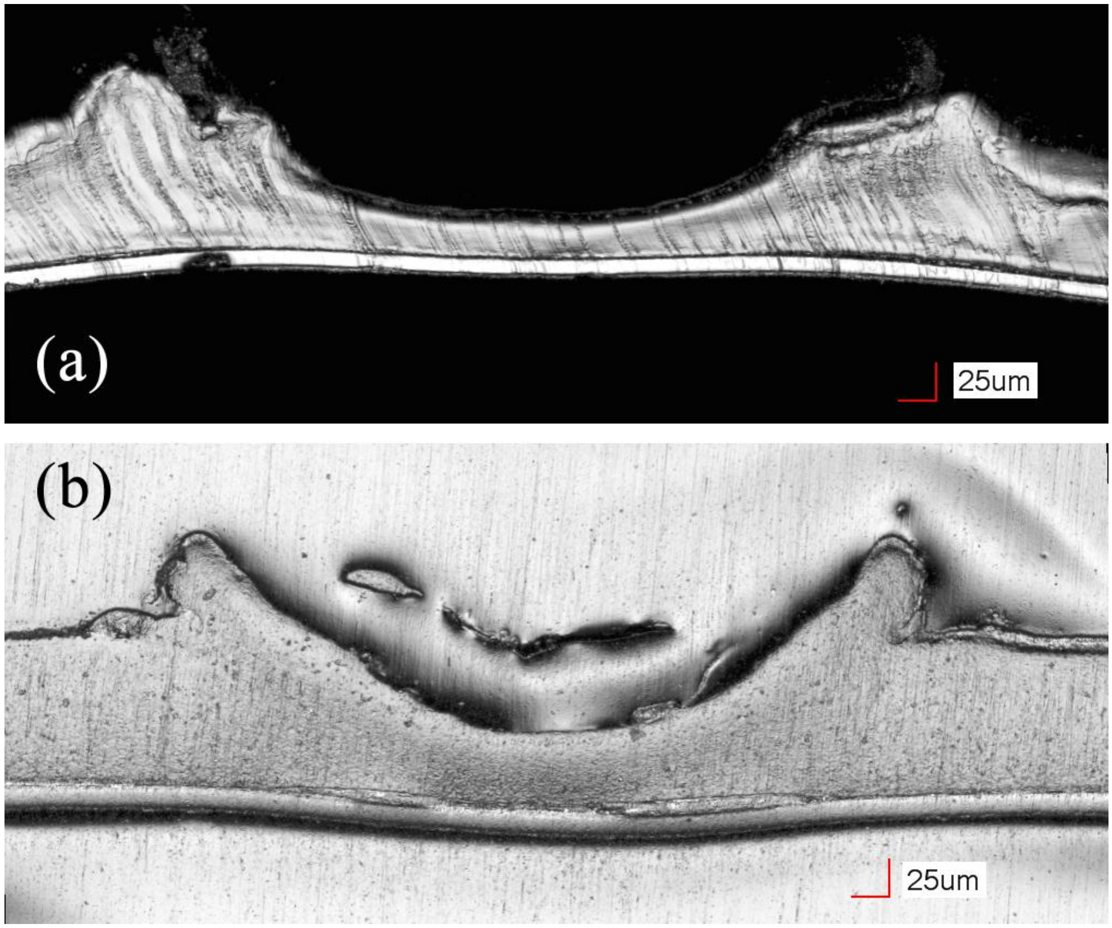

Fig. 23 Comparison between scratch damaged sample images for System 1. (**a**) Before and (**b**) after polishing

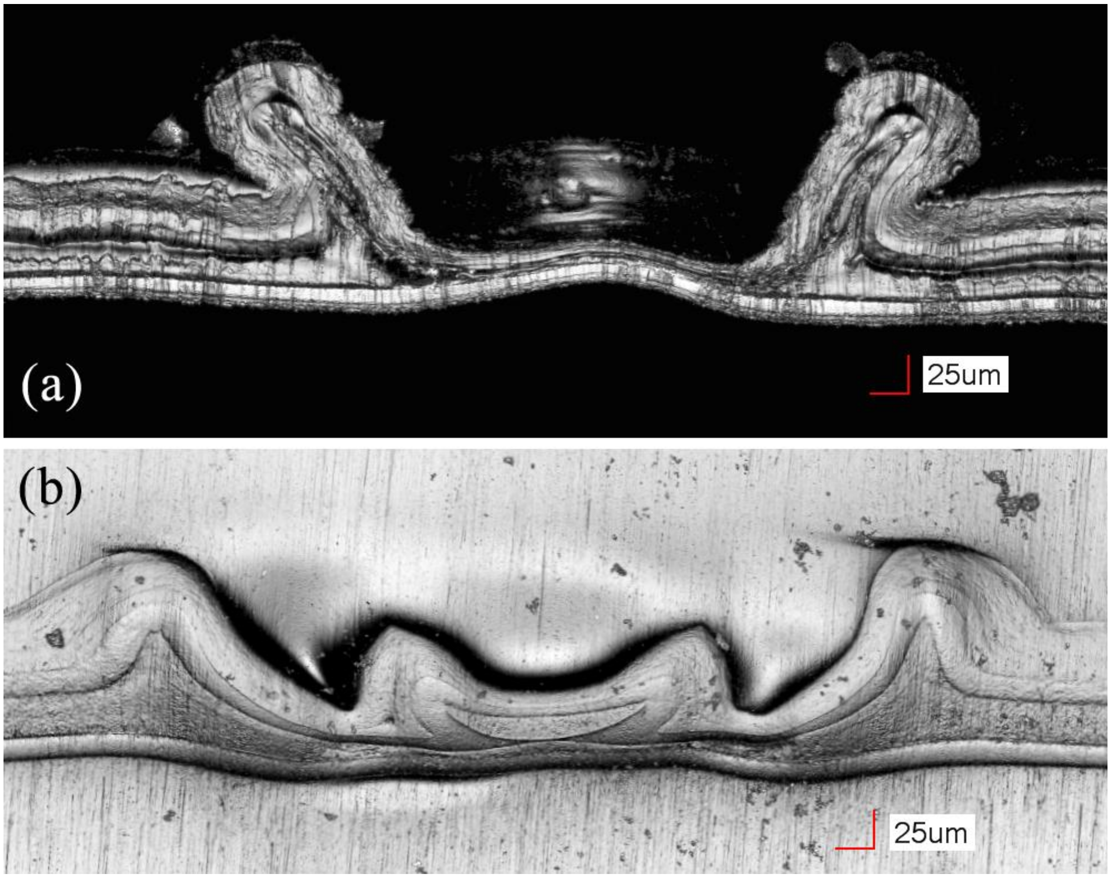

Fig. 24 Comparison between scratch damaged sample images for System 2. (**a**) Before and (**b**) after polishing

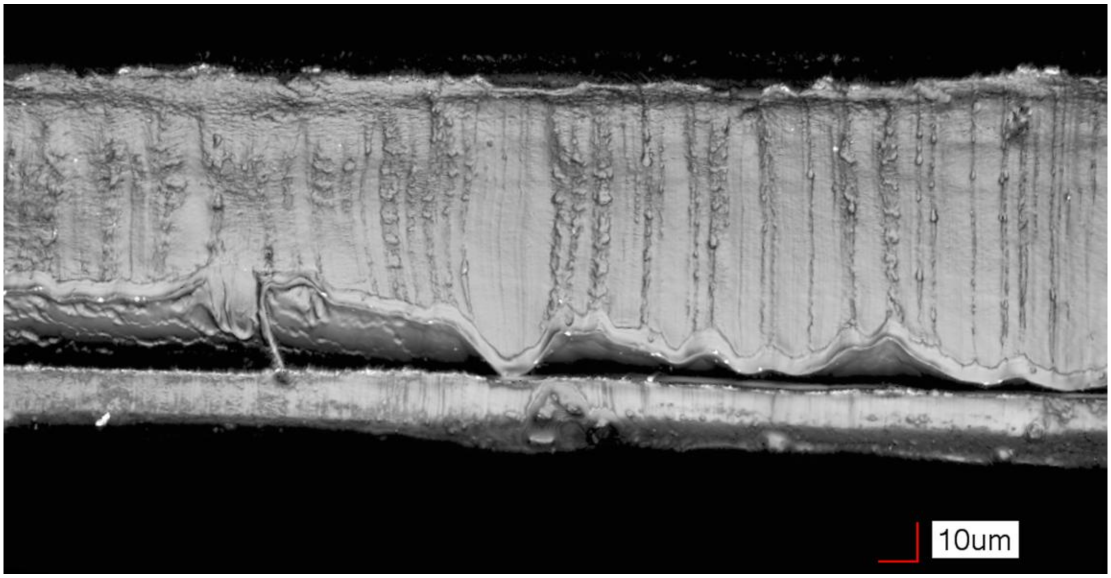

Fig. 25 Delamination occurring between polyolefin layer and oPET layer in System 1 without scratch induced damages

3.6.4 Cross- and Longitudinal-Section View of Multi-Layer Film Interior Surface Scratch

Cross-sectional imaging was performed on samples tested from the interior surfaces. The cross-sectional views of each film correspond with the damage zones described previously, smooth deformation, early stage stick-slip, late stage stick-slip, and sealant removal. The tests begin with smooth deformation, shown in Fig. 26a and b. As can be seen, System 1 shows compressive damage in the very top of the polyolefin sealant layer; while System 2 shows its layers deforming more uniformly. Damage continues to increase with an increasing normal load as seen in Fig. 26c and d. System 1 shows tearing in the top sealant layer, whereas System 2 continues to distribute the stresses throughout all of the layers. As damage continues into the late stick-slip stage

(Fig. 26e and f), the tearing of the top layer in System 1 continues. On the other hand, System 2 exhibits pile up around the edges of the sample, similar to bulk material behavior [11]. Throughout the test to this point, the second polyolefin layer of System 1 has shown no damage, with the entirety of damage concentrated in the top sealant layer. Contrary to this, System 2 distributes the scratch-induced damages throughout its layers, and layers remain adhered even under severe deformation.

A longitudinal-section of both systems was taken at the point of sealant removal. Fig. 26g and h shows the removal of the sealant layers, exposing the oPET directly to the scratch tip. It is important to note that the secondary polyolefin layer is removed quickly following the removal of the top sealant layer. This furthers the conclusion that this secondary layer does not aide in resisting scratch damages. System 2 shows a significant reduction in size of the final polyolefin layer, indicating it was compressed prior to final removal. Thus, the final polyolefin layer in System 2 contributed to resistance of scratch damages. The superior layer integrity and stress distribution of the System 2 structure contributes to its higher scratch resistance and higher normal load required to generate a puncture.

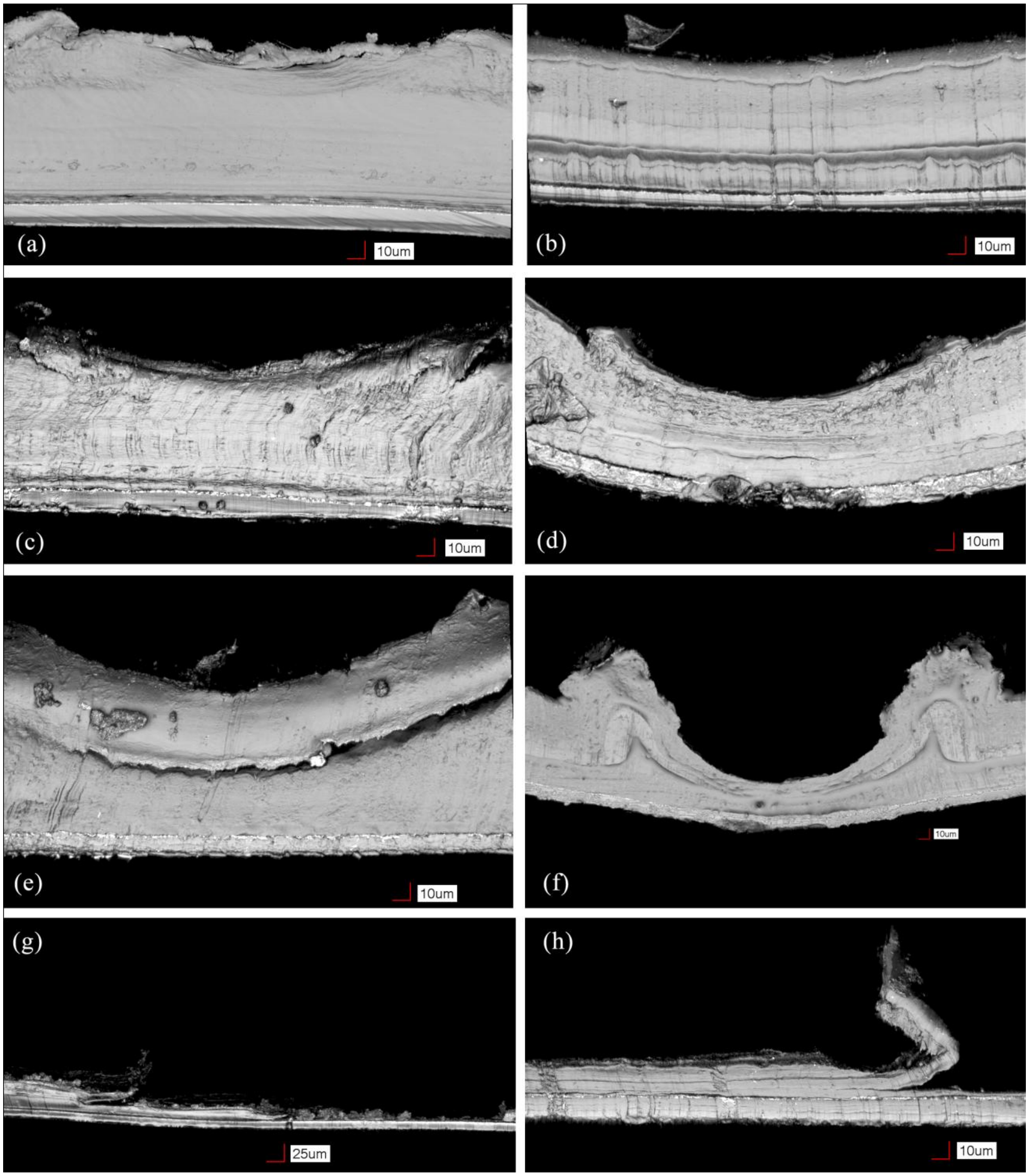

Fig. 26 Cross-sectional imaging showing the progression of scratch induced damages on the layered structure of System 1, on the left, and System 2, on the right. Damage begins with (**a**,**b**) smooth deformation, transitioning into (**c**,**d**) early periodic stick slip behavior, continuing into (**e**,**f**) later and more severe periodic stick slip behavior, followed by (**g**,**h**) sealant layer removal

3.6.5 Cross- and Longitudinal-Section View of Multi-Layer Film Exterior Surface Scratch

Cross-sectional imaging was performed on samples tested on the exterior surface. Because of the low load ranges and distribution of the stress induced by the scratch tip through the soft polyolefin sealant layers, the point at which a scratch could be seen in a cross-sectional image was not observed until approximately half-way through the test, where visible smooth deformation begins. Any visibility of the scratch in the top down view is likely attributed to an increase in surface roughness of the oPET layer, which is known to cause an increase in light scattering and therefore scratch visibility [16]. Additional images were taken during the stick-slip phase and a longitudinal-section was taken at the puncture point.

From the start of visible smooth deformation seen in Fig. 27a and b, System 1 again shows more damage to the polyolefin sealant layer, with in-layer tearing caused by its deformation beneath the scratch tip. System 2 does not show this, with all layers deforming concurrently during the scratch. The curvature of the scratch damaged region is likely caused by friction between the oPET surface and the scratch tip, causing the film to be pulled up slightly behind the scratch tip. When the stick-slip point is reached for the films, shown in Fig. 27c and d, delamination is observed on the edges of the scratch for System 1, whereas System 2 exhibits similar behavior as during smooth deformation. As the normal load continues to increase, System 1 shows more delamination between the polyolefin layer and the oPET layer, shown in Fig. 27e. However, System 2 continues to deform uniformly until puncture.

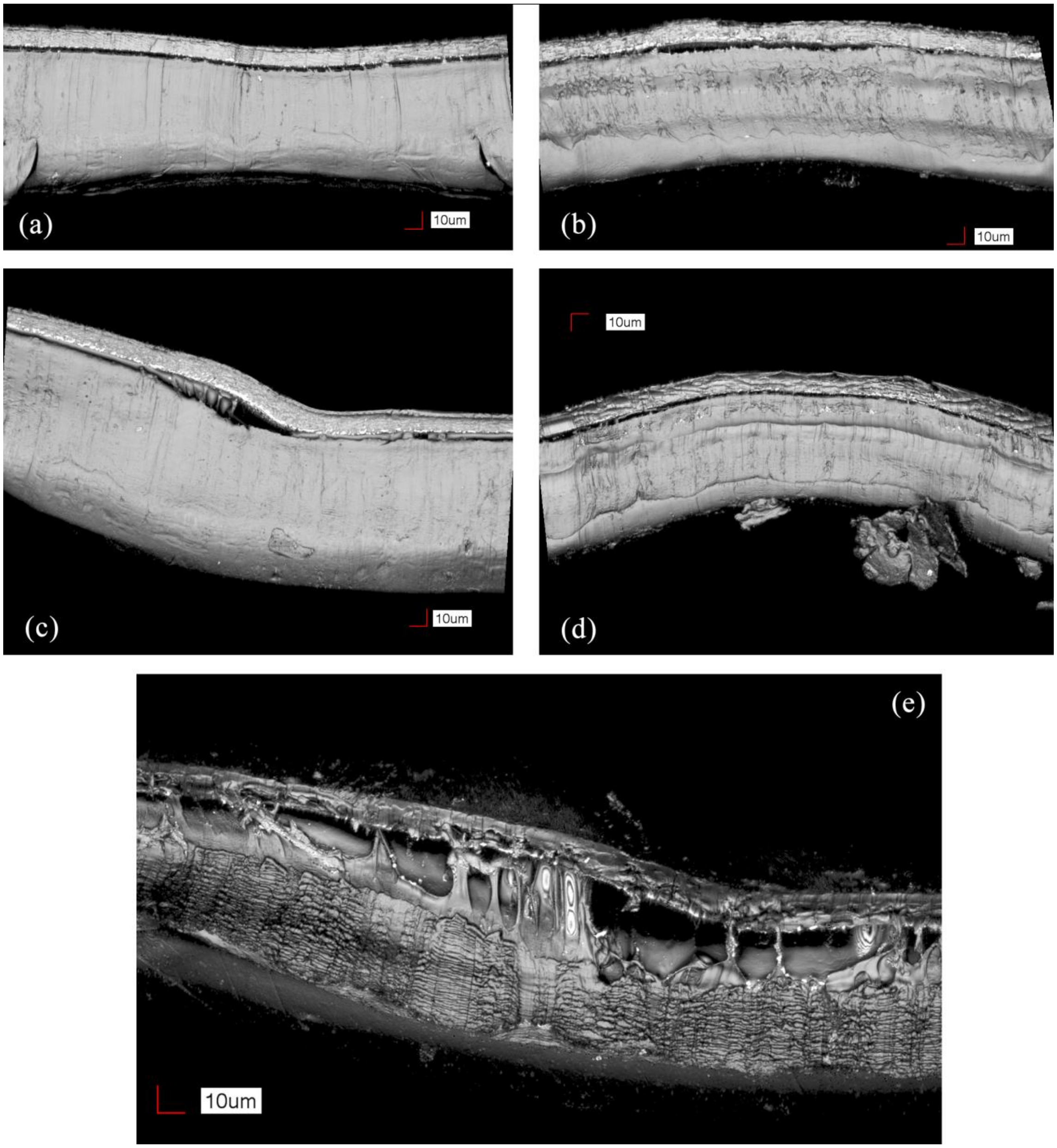

Fig. 27 Cross-sectional imaging of scratch induced damages on the outside surface of System 1, on the left, and System 2, on the right. Damage becomes visible within the layers approximately halfway through, and begins with (**a**,**b**) smooth deformation, followed by (**c**,**d**) early periodic stick-slip, System 2 continues this damage until failure. This continues into the (**e**) later stick-slip region for System 1

A longitudinal-section was taken of the puncture point, shown in Fig. 28. Since both systems fail in the same manner, only System 1 is shown to provide a clearer image. The oPET layer fails, and the remaining sealant layers are readily pushed away by the scratch tip. Puncture is observed on the right side of the image. As with scratch damages on the interior surface, the second polyolefin layer in System 1 does not show significant damage during the scratching process, with all visible damage observed in the sealant layer. In addition, the second polyolefin layer continues to show delamination between it and the oPET layer, again highlighting that the adhesive bonding between the two is not sufficient. System 2 shows excellent layer integrity and adhesion through the test, with no visible delamination occurring. These results correlate well with the scratch test, and the layer adhesion and integrity and stress distribution characteristics of System 2 structure contribute to its higher load required to generate a puncture.

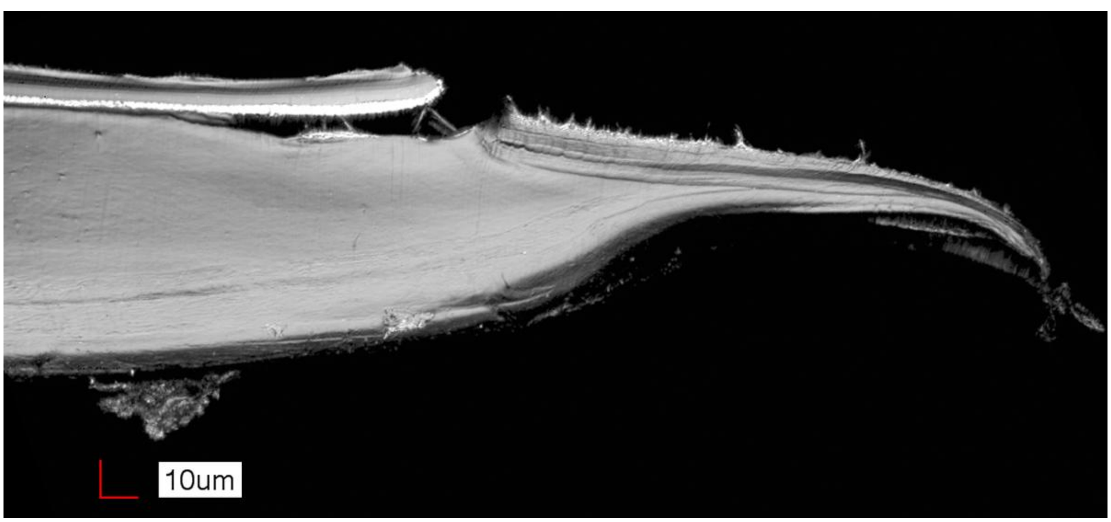

Fig. 28 Puncture caused by failure of the oPET layer in System 1. System 2 shows same failure mechanism

The present study has shown that the proposed scratch test is effective for quantitative evaluation of the mechanical integrity of polymeric packaging films. Damages produced through the scratch test methodology show correlation to damages observed on some commercial films. The scratch test provides a relevant alternative testing metric with which packaging films can be evaluated. In addition, the scratch test can allow for a more comprehensive evaluation of film performance by enabling analysis of the film structure on a layer-by-layer basis after scratch damages are introduced to the sample. Because the proposed scratch test provides meaningful and repeatable results, this methodology may give packaging film engineers with more in-depth analysis of packaging film structure and their corresponding mechanical performance.

While this study has highlighted the usefulness and applicability of the scratch test in the scenarios presented, there are limitations that must be mentioned. The scratch test may not correlate well with field performance results in all cases. Some film damages observed in field service may not be scratch-like, and may be dominated by tearing or puncture. The scratch test may better correlate with film field performance if the film is subjected to abrasive or scratch-like damages, such as during transportation and sliding of the packages. The scratch test is also unlikely to correlate with mechanical integrity of heat sealed or bonded joints between two films, where a T-peel test would be applicable.

Furthermore, the tip used in these experiments was a 1mm stainless steel spherical tip. However, a different tip geometry and material type may better correlate with the product field performance depending on the objects causing damage to the film. Similarly, a different backing material, such as neoprene rubber to simulate an air backing, can also result in different scratch performance. It is prudent that the scratch tests be performed under scenarios closely simulating realistic field conditions in order to achieve good correlation with field performance. While there are limitations, the scratch test methodology offers significant flexibility in testing conditions to address the needs of packaging film mechanical integrity evaluation and laminate structure design optimization.

4. CONCLUSIONS

While still in the early stages of development, the application of the progressive load scratch test for evaluating mechanical integrity of packaging films shows promising preliminary results. By using the vacuum fixture to secure the film, repeatable and meaningful results have been observed for both stick slip and puncture points along the scratch path. The use of the vacuum fixture coupled with a hard backing for the film yields damages that are similar to those seen in the bulk material behavior during the scratch test. For the tested control film, slower testing rates show the most significant differences between different film orientations. Tests on commercial films showed similar trends and behavior as the control film, and a set of field-ranked commercial films correlated well with the scratch test results.

The scratch test method has been shown to differentiate between good and bad multi-layer commercial food packaging films. Through cross-sectional and longitudinal-sectioning of the damaged films, the scratch test can be used as a tool to evaluate the integrity and adhesion of the films on a layer-by-layer basis, and how each layer contributes to the scratch resistance of the films. Two model films were tested and it was found that System 1 had a polyolefin layer that did not appear to contribute to the strength of the film, and was prone to delamination between it and the oPET layer. System 2 shows good layer integrity and adhesion throughout the test, which contributed to its higher normal load required to generate a puncture in the film. While still early in the development process of this methodology, meaningful correlations are observed. The scratch test offers a key advantage over other traditional testing methods in that it allows for investigation of inter-layer adhesion and the role of each layer plays in the mechanical integrity of the laminated films.

Future work will focus on refining and standardizing the testing methodology described here such that additional multi-layer commercial film systems can be effectively tested and compared to observed field results. Secondly, further testing of different backing materials, such as polypropylene or polyethylene, will be carried out in order to investigate their effect. Thirdly, the effect of different scratch tip geometries will be investigated. Finally, an investigation will be carried out in order to investigate the differences in scratch performance and layer integrity and adhesion between a set of model film systems formed through adhesive lamination and extrusion lamination.

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