Testing and Predicting Impact Puncture Resistance of Multilayer Flexible Packaging Films

Leopoldo A. Carbajal, Rong Jiao, Barry A. Morris, Diane M. Hahm, Randy R. Kendzierski DuPont, Wilmington, DE

Abstract

The resistance of films to high speed punctures, such as those that occur during filling operations on vertical-form-fill-seal packing lines, is difficult to measure and predict from laboratory tests. New test methods and computer modeling capability are described that correlate well with drop tests, promising to shorten development time for improving package performance. The creation of pinholes by snack crackers during filling operations is chosen as the initial focus of the study. High speed video is used to understand what happens to the cracker and film during a puncture event. From this study, a high-speed reverse-impact puncture test with a needle probe is developed that mimics the puncture event. The ultimate work to puncture the film is found to correlate with puncture resistance. Test results on seven coextruded barrier films show that the new test is able to quantitatively rank the performance of the films, give results that correlate with known structure variables, and provide input into model development. The reverse impact test results also agree with drop impact tests performed with bullets. An explicit nonlinear finite element model capable of predicting the impact puncture resistance of multilayer flexible packages is developed. The model explicitly considers each layer so that the contribution of the film structure can be determined. Initial results show excellent agreement between the model predictions and the multilayer barrier film test results. Future work will explore the role of material selection, layer placement and interlayer adhesion on puncture resistance, and ultimately lead to better performing packages.

Introduction

Pinholes created by puncture of flexible packaging films can allow unwanted communication between the product and the outside environment, leading to loss of product freshness. Impact events resulting in pinholes are particularly prevalent during filling operations on vertical-form-fill-seal (VFFS) packing lines. Common laboratory film tests such as Spencer impact resistance, dart impact resistance, slow puncture, Gelbo flex tests and Elmendorf tear resistance do not correlate well with packaging line failures associated with product impact during filling on VFFS lines.[1] A practical test often used in the industry to assess the impact puncture resistance of these flexible packages is to conduct a drop test using screws as projectiles. The output of the test is the number of pinholes present on the structure at the end of the test. Flexible packages with fewer pinholes are considered to have higher impact puncture resistance than those showing a higher number of pinholes. The simplicity of this test is a desirable characteristic for package designers and material developers (no advanced data acquisition systems or sensors are needed), but unfortunately, the discrete nature of the output (number of pinholes) and intrinsic variability of test parameters conspire against the resolution of its results and consequently on its ability to help guide the design process.

The goal of this work is to develop better laboratory test methods that correlate with puncture results from field studies, and have sufficient resolution to allow the package designer to quantify the effect of layer material properties, layer arrangement, layer thickness and interlayer adhesion on puncture resistance. Predictive capability, through the development of computer based modeling, is a second goal. In tandem, these tools will reveal key design insights and accelerate the development of new flexible packaging structures with enhanced puncture resistance. Progress against both of these goals is described in this paper.

Reverse Impact Puncture Test Development

Although several authors have reported methods for measuring the puncture resistance of polymeric materials [2-5], impact puncture of flexible food packages has some unique characteristics. Among these are the speed of the event,

the diameter of the punctured holes, and the relative low stiffness and strength of the food (this could mean that significant deformation of the impacting projectiles is taking place during the event).

To help guide the development of a more representative test method, preliminary food drop tests were conducted to understand the physics of the impact event. A woven wheat snack cracker was chosen as the initial focus of the work. Crackers are particularly prone to cause pinholes during filling due to their sharp edges and points. Figure 1a shows a typical penetration velocity chart obtained using a single cracker as the projectile (no added mass to the projectile). The graph shows that for this particular flexible film structure penetration starts to happen at 6.1 m/s. This velocity is much higher than the typical velocity at which puncture resistance tests are conducted [1, 4].

Also shown in Figure 1b is a micrograph of the impacted zone for a test velocity of 6.3 m/s that resulted in penetration. The failed zone is relatively small (the horizontal dimension of the failed zone is less than 0.5mm) when compared with the diameters of indenters typically used to characterize the puncture resistance of polymeric materials.

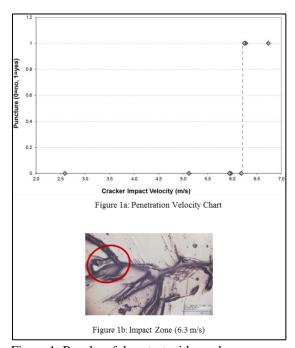


Figure 1: Results of drop test with cracker

High speed video of drop tests did not reveal crackers experiencing significant deformations during the impact event (or any signs of significant permanent deformation after the event). This can be seen in Figure 2, which shows a series of snapshots from the video sequence. Since the presented area of the projectile to the target does not change significantly during the food drop test, using an indenter made of steel for the puncture test is a reasonable choice.

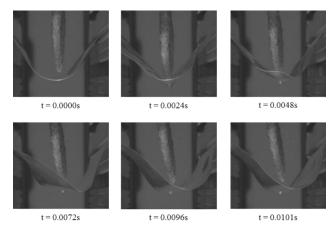


Figure 2: Drop test deformation sequence.

As a result of these findings, a reverse normal puncture (RNP) test was developed. Details of the equipment set-up can be found in Carbajal, *et al.* [6]. A photo of the instrument is shown in Figure 3. The film is mounted horizontally onto a hydraulic load frame capable of maintaining speeds of up to 12.7 m/s. Oblique angles were also tested but the results are similar to the horizontal configuration. The frame moves upward at constant speed (4.2 m/s for these tests) and impinges on the steel needle probe, with a shape profile given in Figure 4. The dimensions of the probe were chosen to mimic the size of the pinholes from the cracker drop test. Load transducers record the force as the film in punctured by the probe. The force vs. displacement is recorded, which is later integrated to produce work vs. displacement curves, as shown in Figure 5. Moving the film rather than the probe, as well as integrating the results to calculate the work, helps to reduce the noise of the experiment. The whole event is recorded on two high-speed video cameras, filming at a rate of 50,000 frames/s, as shown in Figure 3.

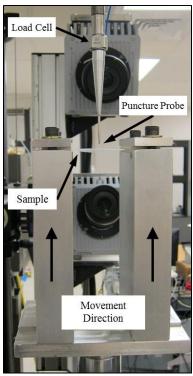


Figure 3: Reverse impact puncture test assembly

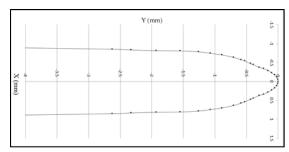


Figure 4: Puncture needle profile

Impact Puncture Test Results

A series of multilayer films were made in-house on a 9-layer Macro Engineering blown film line. The films had the 5-layer structure (HDPE - Tie Layer – Nylon - Tie Layer – Sealant) which is typically used in dry food packages. The layer compositions and thicknesses are listed in Table I, II and II. The thicknesses of the layers reported in Table III were measured using a microscope. The percentages may not add up to exactly 100% due to rounding errors.

Table I: Resins used in study

Resin	Supplier	Grade	Description	
HDPE1	Chevron Phillips	9659		
Tie Conc.	DuPont	Bynel® 41E710	Anhydride modified PE	
LLDPE1	Dow	Affinity 1880G	mPE, 0.902 g/cc	
Nylon1	BASF	Ultramid C	PA 6/6,6	
EVA1	DuPont	Elvax® 3174SHB	EVA (18% VA)	
Ionomer1	DuPont	Surlyn® 1650B	Zn-ionomer	
Ionomer2	DuPont	Surlyn® E185SB	Zn-ionomer	
SB1	DuPont	Elvax® CE9619-1	Slip & antiblock concentrate	
SB2	DuPont	Conpol™ 5B10S1	Slip & antiblock concentrate	

Table II. Flexible structures compositions

Short Name	Moisture Barrier	Tie Layer	Barrier	Tie Layer	Seal Layer Blend
1	HDPE1	LLDPE1 + 15% Tie Conc	Nylon 1	LLDPE1 + 15% Tie Conc	Typical combination of EVA1, Ionomer1 and SB1 used in commercial films
1A	Same as 1	Same as 1	Same as 1	Same as 1	Same as 1
1B	Same as 1	Same as 1	Same as 1	Same as 1	Same as 1
1C	Same as 1	LLDPE1	Same as 1	Same as 1	Same as 1
2	Same as 1	Same as 1	Same as 1	Same as 1	97% Ionomer2 +3%SB2
2A	Same as 1	Same as 1	Same as 1	Same as 1	Same as 2
2B	Same as 1	Same as 1	Same as 1	Same as 1	Same as 2

Table III. Layer thickness (as % of total thickness)

Short	Thickness of the	Moisture	Tie Layer	Barrier	Tie Layer	Seal Layer
Name	Structure (µm)	Barrier (%)	(%)	(%)	(%)	Blend (%)
1	55	73	2	6	2	17
1A	75	68	4	7	4	17
1B	83	64	3	18	2	12
1C	72	71	3	10	2	14
2	65	73	2	7	2	17
2A	74	70	3	11	2	14
2B	80	66	3	18	2	12

By integrating, numerically, the force signals over their corresponding displacement signals, the work history can be obtained. This is shown in Figure 5. The red line corresponds to the average work history and the black lines the actual range of the data. The ultimate work of the structures is used to characterize the puncture resistance of the films. A bar graph of the values obtained is shown in Figure 6. The test method is found to provide a rank order of the samples with good resolution (as indicated by the short error bars in Figure 6).

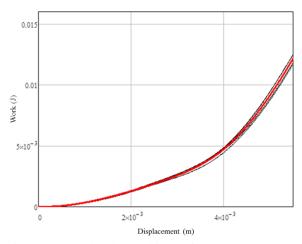


Figure 5: Calculated work history for structure 1.

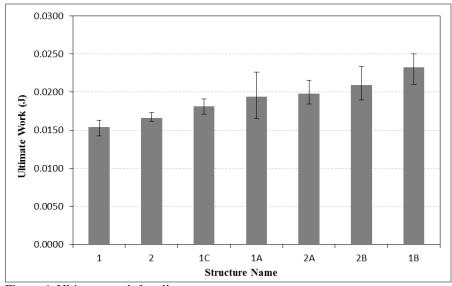


Figure 6: Ultimate work for all structures

The ranking in Figure 6 can be explained largely by the amount of nylon (given by % thickness provided in Table III) present in them. The explicit relationship between these two quantities can be seen in Figure 7. The sensitivity of the test to nylon layer thickness variations is evident from Figure 7. These results are encouraging since it is well known that increasing nylon layer thickness improves puncture resistance. Other laboratory tests do not always show this relationship.

To be able to judge the validity of the new test method, it is necessary to compare it against the results of a drop test. Since the cracker drop test did not reveal any significant deformation of the crackers, it was decided to use 0.223 Remington Full Metal Jacket bullets by Perfecta as the projectiles. The flying stability (better than that of screws) of the bullet was one of the reasons for the selection. The other reasons were the weight of the bullet (average weight equal to 3.56 grams) is similar to those of the crackers (average weight equal to 4.74 grams), and the dimensions of the bullet tip are in the range of those observed in the holes left during the dry food drop test.

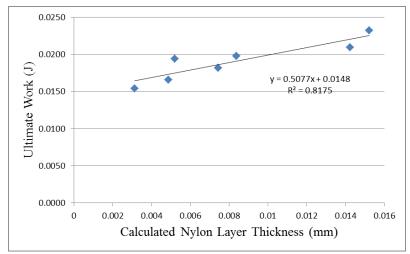


Figure 7: Ultimate work and nylon thickness.

Drop Test Results

Impact velocities of the bullets were measured using high speed video images, and only normal impacts were considered for the computation of the penetration velocities. The films were examined for pinholes following the impact event using a dye. By testing impacts at several velocities, the puncture velocity is determined, as illustrated in Figure 1a. Note that this approach is more quantitative than the typical screw drop test described in the Introduction. Here the velocity of penetration is measured directly whereas in a typical drop test the puncture resistance is inferred from the number of pinholes measured after the drop. Figure 8 shows a ranking based on the puncture velocities obtained during this test.

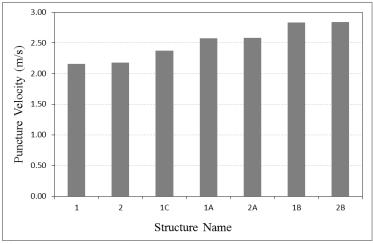


Figure 8: Drop test ranking

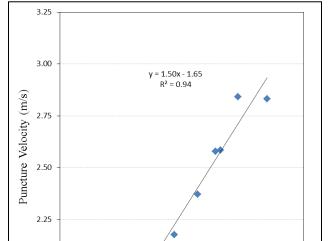
To further compare the results of the impact puncture test with those of the drop test, it is desirable to use quantities having the same dimensionality. For this purpose, the ultimate work is transformed, using Equation 1, into a quantity that has the same dimensions as the reported Puncture Velocity (Length/Time).

$$\psi = \sqrt{\frac{2*Work_{ultimate}}{Mass_{projectile}}} \tag{1}$$

where

Work_{ultimate} is the ultimate work obtained in the puncture test

Mass_{projectile} is the mass of the projectile of interest. In this case, the mass of the drop test projectiles were used.



2.50

 ψ (m/s)

2.75

3.00

A plot of puncture velocity vs. ψ in Figure 9 shows good agreement between the two rankings.

Figure 9: Ranking verification

2.25

Model Introduction

2.00

2.00

The development of the test method is an important first step for developing a predictive computer model of the impact resistance of multilayer films. It provided key insights into the time scales, deformation behavior and dimensions of the puncture event, which factored into the modeling approach. Impact events considered in this study usually have a short dynamic response time, large local deformations and failure of some or all the materials involved. The short duration of the event can be clearly appreciated in Figure 2. This sequence of images shows that the entire event duration (from impact until the wheat snack cracker is arrested) is less than 10 milliseconds. The nature of the permanent deformation and the failure of the structure can be observed in Figure 1b; the failed zone is relatively small (the horizontal dimension of the failed zone is less than 0.5mm).

3.25

The commercial software Abaqus/Explicit is used for the development of the models because of these characteristics. Explicit central-difference time integration is utilized. Details of the model development are provided in Carbojol, *et al.* [7]. Since the model is expected to provide insight about the contribution of individual material properties and/or geometric selections to the overall impact puncture resistance (IPR) of the multilayer structure, it is necessary to consider each material explicitly. Figure 10 shows the level of detail in the thickness direction for the two impact events considered in this paper. Three-dimensional finite element models were built for the different layers of the flexible structures. Since the projectiles are stiffer and stronger than the flexible structures, they are assumed to be perfectly rigid and are idealized using rigid shell elements (all their geometric shapes and dimension are preserved).

The procedure used to obtain material properties for each of the layers of interest consisted of a two-step process. The first part of the process entailed conducting tensile tests of each individual layer at two different strain rates: $0.001s^{-1}$ and $1.00s^{-1}$. Mathcad with the Kornucopia® toolbox was then used to process and convert all the raw force-displacement data into true stress and strain.

The second part of the procedure consisted of performing a reverse impact test for the individual material layers, using the same set-up described earlier for multilayer films. A numerical model of the puncture test was created. Using the material properties obtained in the tensile tests an initial force-displacement response is predicted, and

compared with the experimental responses. Following an iterative process, small modifications are made to the damage initiation criteria and the material properties until the predicted response matches the average experimental response. The refined properties are those corresponding to the best prediction.

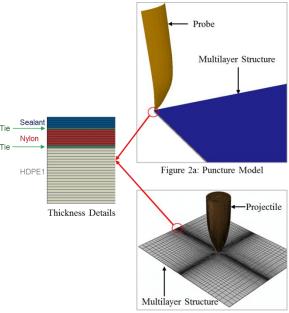


Figure 2b: Projectile Drop Model

Figure 10: Finite element details

This procedure was used to obtain each of the layer material properties needed to predict the IPR of the multilayer flexible packages in Table II. As an example, Figure 11 compares the experimental and predicted work-displacement responses for Nylon1.

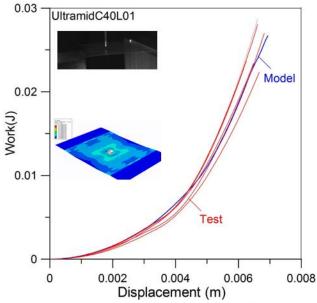


Figure 11: Experimental and predicted work-displacement responses for nylon1.

Multilayer Layer Model Predictions

Model predictions of the two experimental impact events (reverse normal puncture (RNP) and bullet drop tests) are considered to assess the ability of the models to predict the IPR of the multilayer structures. The first set of predictions corresponds to the RNP impact event. Figure 12 shows a sequence of images depicting typical deformed shapes observed during the simulation of the RNP event. These agree qualitatively with high speed video images from the actual experiment.

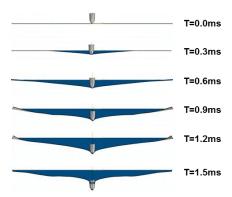


Figure 12: Typical predicted deformed shapes

Figure 13 compares the predicted and experimental values for ultimate work for all seven structures (Table II). Average material properties were used to predict the response of all structures.

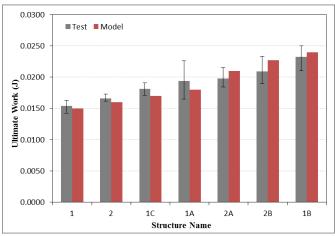


Figure 13: Experimental and predicted ultimate works

As it can be observed in Figure 13, the model and the test method rank the structures in exactly the same order. When judging the accuracy of the prediction, the largest registered error between predicted and the average experimental results is 8.5% (structure 2B), and the average error for all structures is 5.4%. These values are considered acceptable for this type of simulation. As stated earlier, the ranking in Figure 13 can be explained largely by the amount of nylon present in the structures. Figure 14 shows the explicit relationship between the nylon thickness and the predicted ultimate work of the structure.

The second set of predictions corresponds to the bullet drop impact event. Figure 15 shows the deformed predicted shapes as seen from the back face of structure 1B for a velocity that ultimately resulted in puncture. Figure 15a corresponds to an instant just before the puncture, and Figure 15b corresponds to the instant when the projectile has penetrated the structure.

Similar to the corresponding test, in this simulation the bullet is assigned an initial velocity and then the model predicts whether the structure can arrest the projectile. All predictions were performed using average material properties. Figure 16 compares the experimental and predicted V_{50} (the velocity at which 50% of the projectiles would puncture the structure) for six of the seven structures. All predictions needed to calculate the puncture velocity of structure 1 were not finished at the time of writing this paper.

The model and the test rank the structures in exactly the same order. When assessing the accuracy of these predictions, the largest registered error between predicted and the average experimental results is 4.5% (structure 2A), and the average error for all structures is 3.6%. These values are considered acceptable for this type of simulation.

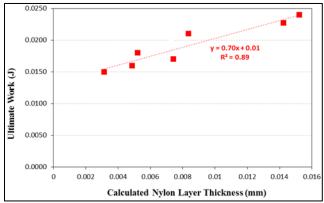


Figure 14: Predicted ultimate work and nylon thickness

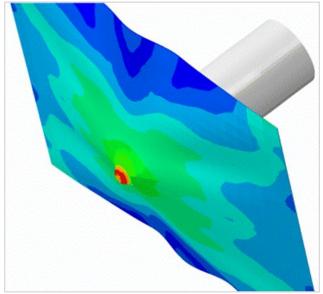


Figure 15a: Instant before penetration

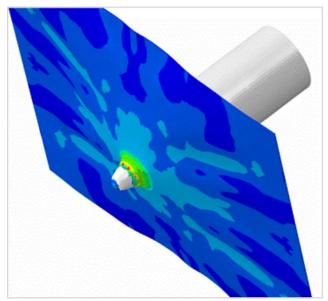


Figure 15b: Instant after penetration

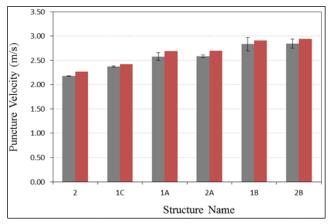


Figure 16: Experimental and predicted puncture velocities

Conclusions

A laboratory high-speed puncture test with a needle probe was developed that measures the ultimate work of puncturing the film. The test is based on studies of the fundamental dynamics of puncture of films by falling crackers (speed and deformation). Rankings of film structures tested with the method correlate with a projectile drop test. Nylon layer thickness was chosen as the first film structure parameter to study since increasing nylon layer thickness is often used to increase puncture resistance of films. Ultimate work shows good correlation with nylon thickness. The new method should prove useful in screening flexible packaging and help guide the development of improved structures.

A practical procedure has been developed for predicting the impact puncture resistance of multilayer flexible packages using numerical models and the mechanical properties of the materials involved. Model predictions for seven multilayer flexible packages and two types of events were conducted. The model predictions are in close agreement with the experimental data. Future publications will cover further validation of this capability, and its use to design higher impact resistance flexible packages.

References

- 1. Brian A. Hare, Hung-Jue Sue, Lora Ying Liang and Panos Kinigakis. Scratch Behavior of Extrusion and Adhesive Laminated Multilayer Food Packaging Films. Polymer Engineering and Science, 54, 71-77, 2014
- 2. Hu, K. H. and J.D. Breyer. Pinhole Resistance of Flexibles. Part I. Modern Packaging 44(12): 46-48, Dec. 1971. Part II. Modern Packaging 45(1): Jan. 1972.
- 3. Furno, F. H., R. S. Webb and N.P. Cook. High Speed Puncture Testing of Thermoplastics. J. Appl. Polymers Sci. 8:107-110, 1964.
- 4. Lynch, L. W. Evaluation of Plastic Films. Modern Packaging 38(2): 177-183, 226-227, 1964.
- 5. Hu, K.H. Measurements on the resistance of flexible packaging materials to puncture, abrasion, and flexure and the relationship of these measurements to the performance of packages subjected to conditions causing pinhole formation. US Army Natick Laboratory Technical Report, 1973.
- 6. Leopoldo A. Carbajal, Rong Jiao, Diane M. Hahm, Barry A. Morris, Randy R. Kendzirski, "Impact Puncture Resistance of Multilayer Flexible Food Packages". ANTEC, Orlando, FL, March 2015.
- 7. Leopoldo A. Carbajal, Rong Jiao, Diane M. Hahm, Barry A. Morris, Randy R. Kendzirski, "Predicting the Impact Puncture Response of Multilayer Flexible Food Packages Using Explicit Finite Element Models," ANTEC, Indianapolis, May 2016.