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Non-destructive testing of foil pouches. Determining a reliable method for checking for punctures

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Non-destructive testing of foil pouches. Determining a reliable method for checking for punctures

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ABSTRACT

Several technologies used for checking for punctures in foil pouches were reviewed. There were two that would be applicable, these were the Vacuum decay and Force decay methods. Vacuum decay is a method where the pouch is placed into a sealed chamber and a vacuum is applied. This vacuum will create a pressure differential from the inside of the pouch to the outside. After the vacuum is switched off the vacuum level is monitored and a change is measured. If a puncture exists there will be a change that is larger than one expected due to imperfections in the test chamber. This method will work unless the pouch has a very large puncture or it is not sealed. In this case there will not be a change in the vacuum decay measurement.

The force decay starts like the vacuum decay except that a transducer is used to measure the force exerted on a plate resting on the pouch. The inflation of the pouch due to pressure differential is the source of the force. The vacuum is applied until a predetermined force measurement is achieved then it is monitored for the rate of decay in the force reading. The accept/reject set point determines if the pouch is good or bad.

We tried to locate off the shelf test units capable of predicting a 50μm hole 100% of the time. Pouches were provided to various manufactures and they were asked to create specific holes and do the required testing. It was determined that the Force Decay method is the most appropriate test.

1. INTRODUCTION

In most packaging processes that involve foil as the final barrier to moisture, oxygen, and light there is a concern about the incidence of punctures in the foil. These punctures can be created as the foil is being rolled in thin sheets when particles are rolled into the foil, these are called micro voids. The most common defect is caused from the handling of the packages during the assembly of the foil with product. These defects are sometimes hard to see as the Human eye can only reliably see punctures that are larger than 100 µm. Punctures smaller that this can still pose significant concern about the efficacy of the product. There are several technologies available to check for this type of defect, some are destructive while others are not. Most were developed for the food industry and involve sampling a small portion of the total quantity produced. The cost of designing a system that can reliably inspect 100% of product from an automated line would be significant. The reliability of such a system and its capability would be in question if this is required for a medical product as this could add sterility to the other requirements for a barrier package. Because it is a medical product and could be considered a release test this test would need to meet the stringent requirements of the FDA. During the manufacture of a medical device there were several occurrences of foil pouches with punctures. These punctures were random in location and position in the batch. There was only one discovered in each batch and not all batches had occurrences. These punctures were found at different stages of the process. They appeared to have the same shape and size, see Figure 1. The packages, in general, ranged from pristine looking (Figure 1) to poorly handled (Figure 2). Most of the punctures had the same appearance where it is evident something impacted the package and scraped it from the outside (see figure 1). The average puncture size was ≈ 711 µm. After several investigations the source of the punctures was not evident. It was then decided that an inspection other than 100% human eye needed to be developed as punctures were found after 200% inspection. According to the University of Utah, a naked eye (an eye without any mechanical assistance that has 20/20 vision) can see objects about 0.1 mm or 100 µm

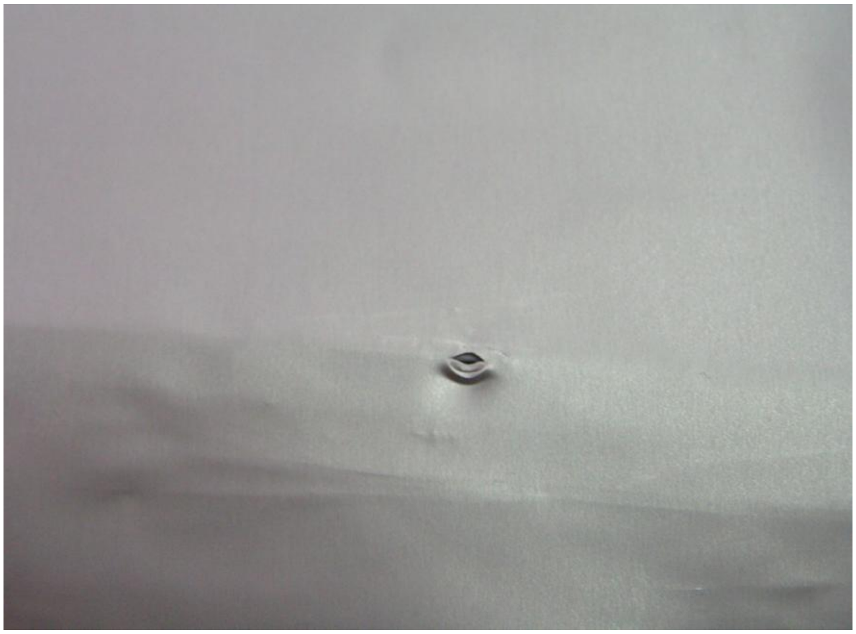

Figure 1 Magnified Actual Puncture

Figure shows the puncture at 50 times magnification

Figure 2 Actual Size of Hole

Figure shows the actual size of puncture in foil

2. PROBLEM STATEMENT

We set as a goal to find a technology that can be used to inspect foil pouches. The methods must be non-destructive and cannot alter the current manufacturing process. It must be capable of finding punctures smaller than 100 µm. The smallest puncture that can be found reliably needs to be determined and verified. We decided to proceed towards this goal as follows. We will look at the technologies available and determine which ones might meet the criteria. Find vendors that can supply the technologies and contact for possible testing. Perform a preliminary test (minimum of 30 data points) of the technology and collect data. Perform a statistical analysis of data and determine if additional testing is warranted. Secure more materials for testing and test station and perform larger test based on previous data set. Perform statistical analysis of data and make recommendations (yes/no). If yes then procure a station and install

3. TECHNOLGIES

In this section we list the most common methods of testing available.

3.1 BUBBLE LEAK TEST:

This test is outlined in the American Society for Testing and Materials (ASTM) Test Method F 2096 [1]. This test is performed by submerging the pouch in a liquid (usually water) and pulling a vacuum on the system. If a puncture exists then air will escape from the interior of the pouch through the puncture and form bubbles. This could be considered a destructive test because of the submersion in a liquid. See Figure 2 for a typical bubble leak test set up. Each pouch would have a known size precision hole attached to it. The pouch is the place in a clear glass “bowl” and weights are placed on top of it to keep it submerged while a vacuum is pulled, see Figure 3. The top is then placed on the bowl and a vacuum is pulled creating a pressure differential between the inside of the pouch and the interior of the “jar”. Pouches with 12.5 µm and 25.0 µm precision holes attached were tested and no bubbles were observed however pouches with 50 µm holes did produce bubbles. This test is not acceptable at this time based on problem statement of non destructive test.

Figure 3 Bubble Leak Test Station

Figure 4 Weights Holding Package Submerged

3.2 HELIUM LEAK TEST:

This test is out lined in ASTM Test Method F2228 with Helium substituted for CO_2 [2].This test involves replacing the normal atmosphere in the pouch with Helium The Test then pulls a vacuum on the pouch and sensors check for the helium. This test can find small holes but would cause a change in the process. To do this it would require a complete revalidation and more clinical trials to prove that there is zero impact on the product. This test is not acceptable at this time based on problem statement.

3.3 VACUUM DECAY LEAK TEST:

This test is outlined in ASTM Test Method F2338 [4]. This test requires the pouch to be placed under vacuum then monitoring the change in vacuum The change in vacuum is dependent on the size of hole and time allowed to monitor. On large holes this is a very fast test, as the holes become < 100 µm the test time grows exponentially as the size gets smaller. This test is acceptable based on our problem statement.

3.4 FORCE DECAY LEAK TEST:

This test is outlined in ASTM Test Method F2095-07 [3]This test has a plate with a pressure transducer attached above the pouch. When a vacuum is instituted the package inflates due to the pressure differential and applies a force to the plate. The vacuum pump is turned off and the force is monitored for a specified time, the final reading is then subtracted from the measurement at the start of the monitoring. This becomes the force decay value and based on it the pouch is accepted or rejected. This test is acceptable based on our problem statement.

3.5 THE BURST TEST:

This test is outlined in ASTM Test Method F1140-07 [5].This test requires the pouch to be inflated until the seals “burst” if there is a puncture it will tear at that point This test is not acceptable at this time based on problem statement.

4. Technology Choice:

There were two technologies that fit the problem statement and would fit all of the requirements. They were the Vacuum Decay and Force Decay tests. Vendors were identified and contacted for each of the technologies. They were given the nominal size of 50 µm puncture as a starting point. The 50 µm was chosen as it is used in the manufacture of the foil stock for micro-voids as a maximum size. A micro-void is created during the rolling of the aluminum when a piece of slag creates a hole in the material. The aluminum is then laminated on either side with a polypropylene or polyester layer. These layers of plastic provide a sterile barrier but not a moisture barrier. It was determined that a 50 µm void would not allow enough micrograms of water to permeate across the plastic to endanger the five year shelf life of moisture sensitive medical products. The equipment that makes the packages has Micro-void detectors that checks for these. This sensors is only capable of inspecting a single layer of foil the pouches that need inspecting would be double layer (top and bottom). Ten sample pouches were prepared for each vendor that agreed to the User Requirements.

The vendor for the Vacuum Decay type of test could not state if they could find a puncture of the size requested. The pouch was larger than any of their equipment could handle (9 inches by 13 inches). They were asking for $12,000 dollars to build and develop a R&D device to run the tests.

The vendor for the force decay performed tests on one of their lab units the results are in table 1. The fact that the initial testing shows 12.5 µm as a possibility and the speed with which they did the tests was encouraging. They offered to allow more extensive testing on the lab unit before any money was committed. They did say that this was not the ideal unit but should give a sense of the capabilities of the technology. They would supply a new concept when the testing was completed.

First Test			
Test Part ID	Decay Value	Result	Notes
	Mg/6 seconds		
1 (A-1)	24000	Reject	Cut Open Part
2 (C-3)	22	Reject	12.5 µm
3 (A-2	50	Reject	25 µm
4 (B-3)	259	Reject	50 µm
5 (C-2)	9	Pass	
6 (A-3)	2	Pass	
7 (B-2)	6	Pass	
8 (C-1)	7	Pass	
9 (B-1)	6	Pass	
Repeat Test			
1 (A-1)	24000	Gross E	Cut Open Part
2 (C-3)	19	Reject	12.5 µm
3 (A-2)	48	Reject	25 µm
4 (B-3)	272	Reject	50 µm
5 (C-2)	7	Pass	
6 (A-3)	1	Pass	
7 (B-2)	6	Pass	
8 (C-1)	6	Pass	
9 (B-1)	4	Pass	

Table 1 Testing Of Force Decay by Vendor

4.1 FEASIBILITY TESTING PERFORMED ON LAB UNIT:

Thirty packages were assembled with dummy product inside for the feasibility test. It was decided we would test 12.5 µm, 35 µm, and Zero hole packages. The precision holes were supplied from a company in Germany that used a laser to create the hole, size ± 10%, in the center of a 5 mm metal disk. These disks were then attached to a ring that had adhesive on one side and a 2.5mm hole in the center. The packages had large punctures created where the disks would be attached over this puncture and sealed around the edges. The punctures were made either by using a small needle or by cutting a square out with an Exacto knife. All of the packages were tested and results recorded. Then five packages from each size were randomly selected and five repeat tests of these were performed with a minimum of 3 minutes between tests to allow the package to equilibrate to the environment. This gave a total of 35 data points for each size hole. The results showed a good separation between the zero hole and 35 μm holes force decay values (see Figures 5 and 6). The repeat results indicated that the force decay values might be increasing with each repeat test (see Table 2).

		Force Decay in mg/6 sec.					
PKG ID	HOLE SIZE µm	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6
1	0	7	4	5	6	7	7
2	0	5					
3	0	3	1	4	4	4	4
4	0	1					
5	0	-1	-2	3	4	3	4
6	0	-1	2	4	5	5	5
7	0	3					
8	0	-1					
9	0	-1					
10	0	0	0	3	4	5	6
11	12.5	34					
12	12.5	22	24	27	28	27	27
13	12.5	60					
14	12.5	9	10	14	15	15	15
15	12.5	3					
16	12.5	2					
17	12.5	8	10	13	12	12	12
18	12.5	2	4	9	9	11	11
19	12.5	5					
20	12.5	3	9	11	11	11	11
21	35	114	135	142	143	143	146
22	35	138	152	156	156	153	156
23	35	134					
24	35	125					
25	35	125	148	153	155	153	157
26	35	111	154	161	161	166	170
27	35	117					
28	35	142	141	156	155	157	154
29	35	129					
30	35	129					

Table 2 Feasibility Run

Table 2 shows the results of the test values based on hole size and repeat tests

However the 12.5 µm holes overlapped the zero hole results indicating that the reject decay value would need to chosen so that in all probability “good” packages would be rejected, this would not meet six sigma standards for process capability.

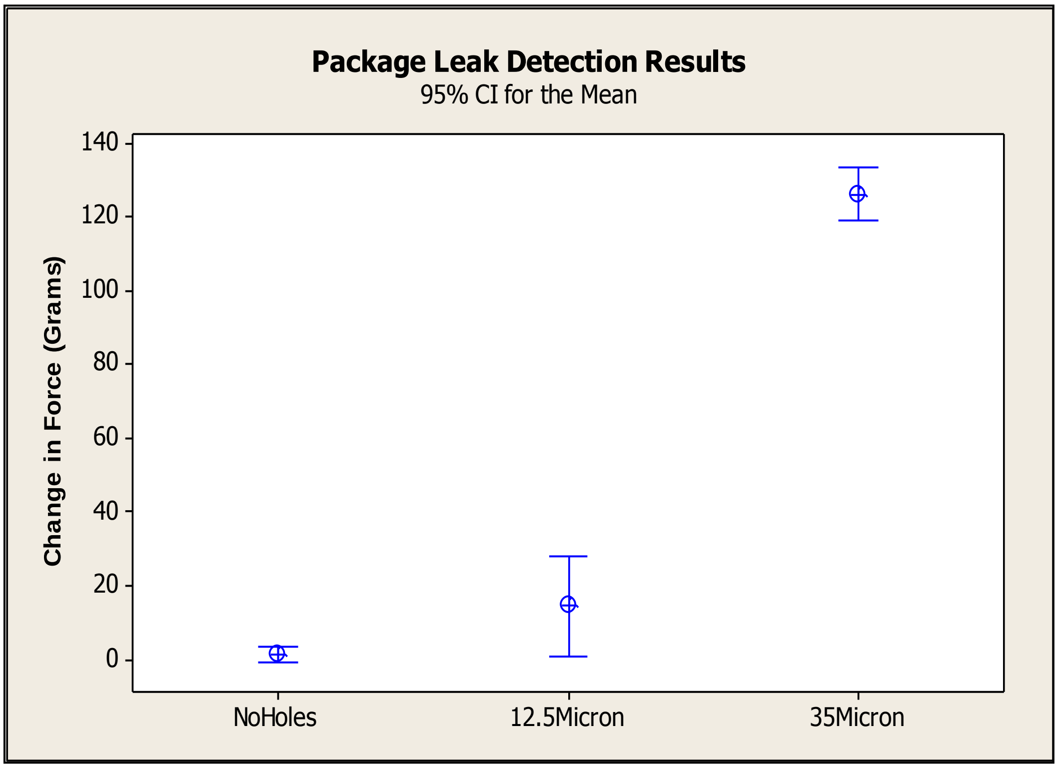

Figure 5 Package Leak Detection Results Feasibility Run

The data for the 35 µm holes was normal with a p-value = 0.008 using the non-parametric Anderson-Darling test. The other data sets are not normal with a p-value < 0.005using the same non-parametric test. This data was also not normal using the non-parametric Kolmogorov-Smirnov test.

This can be expected as the precision holes have a tolerance of ± 10% also the packages have variable headspace in them and this could affect the test. See Figure 7 to see how the test works.

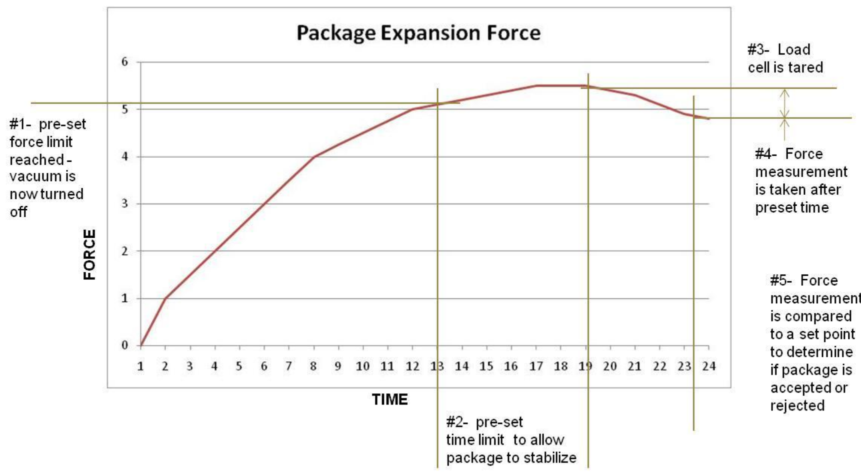

Figure 6 Graph Of Force Decay Test

5. PRODUCTION UNIT TESTING AT VENDORS

After the second feasibility the question of the variability of the force decay value was discussed. It was suggested that the variability of the head space in the pouches may be contributing to the variability. The vendor offered up a variation of the test unit where the cycle would change from pull vacuum until the pre-set force limit is met to pull vacuum for 2 seconds then raise package until force gage reads 0.5 kg then stop. Restart vacuum until force set point is reached and the rest of the test cycle remains the same. The vendor was commissioned to build a single unit for testing. After completion a Factory Acceptance Test (FAT) was performed. This FAT was executed in two phases. In Phase 1, packages with no hole, packages with 25 µm holes, and packages with 50 µm holes were evaluated using the Package Tester to determine force decay value for the respective package types. From these values a force decay threshold for rejecting was determined.

In Phase 2, the threshold decay value determined from Part 1 was used to demonstrate that seeded packages with 25 and 50 µm holes could be distinguished from packages with no holes. In Phase 2, packages with ~ 711 µm holes were also created with a 28 gage needle and introduced randomly and tested in order to mimic the defect size found in the current process.

5.1 TEST DESCRIPTION:

The foil pouch was placed into the nest in the test chamber by the operator. The cover was closed and locked by the operator. Operator then pressed start button. The chamber then pulled a small vacuum, allowing the package to inflate due to the pressure difference between inside of package and the chamber. The package was then moved up via a servo motor until the force meter read 0.5 kg. Following step 5.3.5, the chamber pulled a vacuum until the force gage read 15kg. Note: If the machine did not register the required force within 60 seconds, the test was marked as a failure. The vacuum was then turned off and a 12.5 second settling time occurred. At the end of the 12.5 seconds, the force gage was read (Force 1). If the reading after the settling time was below 13 kg, the package was marked as a failure and rejected. After step 5.3.7 there was a six second measurement time and the force gage was read again (Force 2). That reading (Force 2) was then subtracted from the measurement at the end of the settling time (Force 1); the difference was the force decay value. This value is shown negative because it is the amount the force decreased. This value can be compared to a pre-determined decay set point. A value between 0 and the set point is marked as a pass. Note: A large puncture may receive a value like this, but it would have failed in a previous step due to the fact that with a large puncture the package will not inflate to apply force on the gage (as described in 5.3.6).

5.2 DETERMINING THE DECAY SET POINT VALUE

Thirty packages with 25 µm holes and thirty packages with 50 µm holes were made. First, a primary hole was created in the foil pouch using a 28 gage needle (which was used for the development runs). The correct precision hole was selected. This is a round metal plate that has a laser cut hole in it to within ± 10% of indicated size. It was supplied from a vendor in Germany. Then an adhesive ring was set up to attach the plate with the hole in the center of the adhesive ring. The adhesive ring was then centered over the primary hole such that the precision hole is in the center of it, and the edges gently rubbed to get a good seal around the primary hole. Thirty packages without holes (Zero hole), thirty packages with 25 µm and thirty packages with 50 µm were tested in the package integrity tester. One 0 Hole package (#195) was removed because it failed to achieve a vacuum. This was due to the Plant vacuum not having enough capacity for all users. The descriptive statistics for the force decay values for the three groups are provided in Table 3: (Note the values are shown negative because this is a decay value and moves in the negative direction).

Statistic	0 Hole	25 µ hole	50 µ hole
n	29	30	30
mean	-0.03424	-0.260284	-0.743369
median	-0.03064	-0.152477	-0.520390
min	-0.05518	-1.052395	-2.620551
max	-0.02131	-0.047635	-0.427313
stdev	0.01117	0.217249	0.577085

Table 3 Descriptive Statistics For Force Decay Values

The sample size of thirty was selected to provide a 95% Confidence Interval of the mean pressure decay value of ± 1.08 for the 0-Hole packages and ± 3.80 for the 25 and 50 µm packages, and to provide a precise estimate of the standard deviation for each group. The non-parametric confidence intervals for each group are provided in table 4. The desired precision was achieved, even for the 0-hole group which finished with 29 samples.

Variable	CI for Mean	CI for Stdev
0 Hole	(-0.03849, -0.02999)	(0.008446, 0.016238)
25 µm Hole	(-0.3414, -0.1792)	(0.164919, 0.313323)
50 µm Hole	(-0.95886, -0.52788)	(0.438081, 0.832292)

Table 4 Confidence Intervals By Hole Size

The data for all three groups (0-hole, 25µ-hole and 50µ-hole) were not normally distributed using Anderson-Darling test (p-values: <0.005 for all three groups). The 0-hole packages had a high negative kurtosis value, indicating a distribution that is flatter than normal.

The 25μ-hole and 50μ-hole packages showed evidence of negative skewness. See the Individuals Chart for the force decay values for the three groups in Figure 7 below.

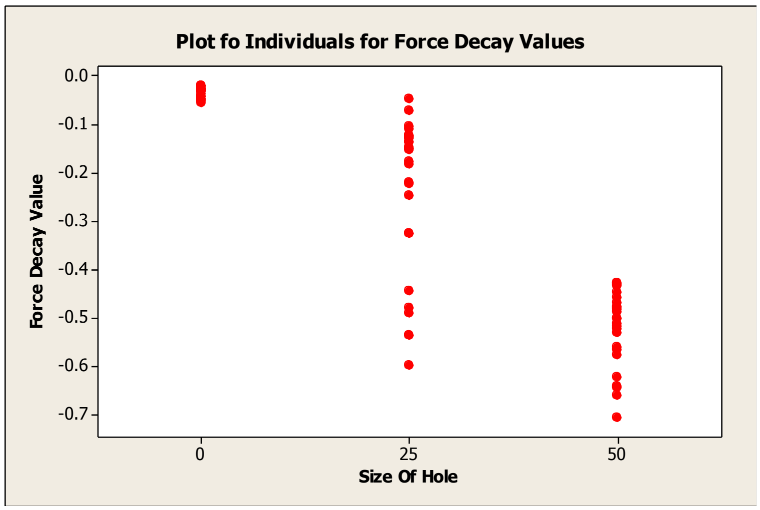

Figure 7 Individuals Plot Of Force Decay Values

There is one package (#56) in the 25μ-hole group with a low force decay value that is a statistical outlier. There are five packages (#8, #13, #61, #62, and #78) in the 50μ-hole group with low force decay values that were removed. After examining these packages it was determined that the decay values were due to packages not being prepared correctly and they were removed. The three distributions for force decay were compared in order to establish a possible force decay threshold value. The proposed decay threshold value was then established by taking the average value for the 0-hole packages and subtracting 5 standard deviations, resulting in a proposed threshold limit of -0.09. The five standard deviations was chosen to provide a factor of safety to prevent any good packages from being rejected. All 50μ-hole packages had actual force decay values at least

4 times greater than -0.09 and thus can clearly be distinguished from the 0-hole packages. The proposed value allows essentially no overlap with the predicted distribution of 50µ-hole decay values with the 0-hole (good packages) (see Figure 8).

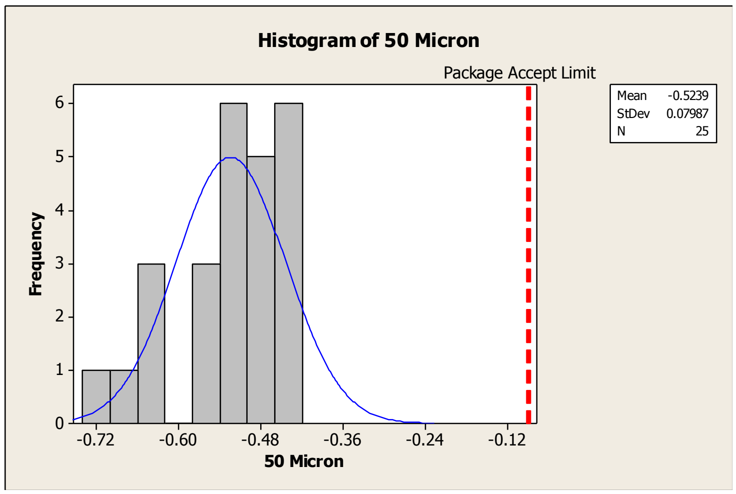

Figure 8: Histogram Of The 50 µm Decay Value

A similar graph comparing the distribution of force decay values for the 25µ-hole group to the proposed force decay threshold is shown in Figure 9. Two 25µ-hole packages (#51, #125) had force decay values that passed the proposed threshold value. Two other packages (#42 and #47) had decay values close to the proposed limit (-0.11 and -0.10, respectively). The graph also shows the negative skewness, potentially due to decay values associated with incorrectly-prepared packages. The area under the curve (to the right of) the -0.09 proposed threshold reflects the predicted false-accept rate for the 25µm-hole packages using this data. An analysis estimates this rate at about 21%. Although this rate is overestimated by assuming a normal distribution in the calculation it is unacceptable. When the single high decay value (-1.05, #56) is removed the distribution is still negatively skewed. Eliminating the skewed results by improving the variability of the 25µ-hole packages to make the data normal this would reduce this rate. We could also move the set point to the right closer to the 0-hole packages possibility having some "acceptable" packages rejected.

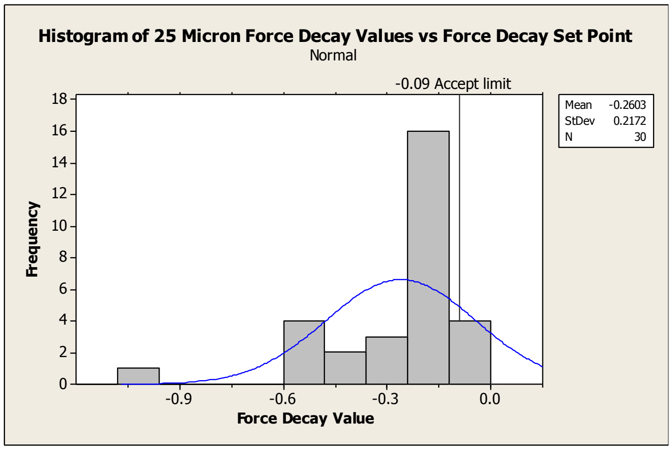

Figure 9 Histogram Of 25 µm Decay Values vs. Reject Value Set Point

This test was performed using plant vacuum. During the 0-hole run, one package (#195) failed to achieve the minimum force set point (15 kg) due to a failure of the plant vacuum. This could possibly cause the variability of the tests After experiencing this failure a venturi vacuum system was installed prior to running the next tests. The thirty 0-hole packages were tested again using the Piab system. An Individuals Plot for each condition is shown in Figure 10. The descriptive statistics for each condition are provided in Table 6. The consistent vacuum source reduced the standard deviation from 0.01117 to 0.004435. The data with the Venturi Vacuum passed the normality test (p-value: 0.559). In addition, the variance (standard deviation) was significantly less (p-value: 0.000).

Therefore it was determined this was the vacuum source that would be used for Part 2

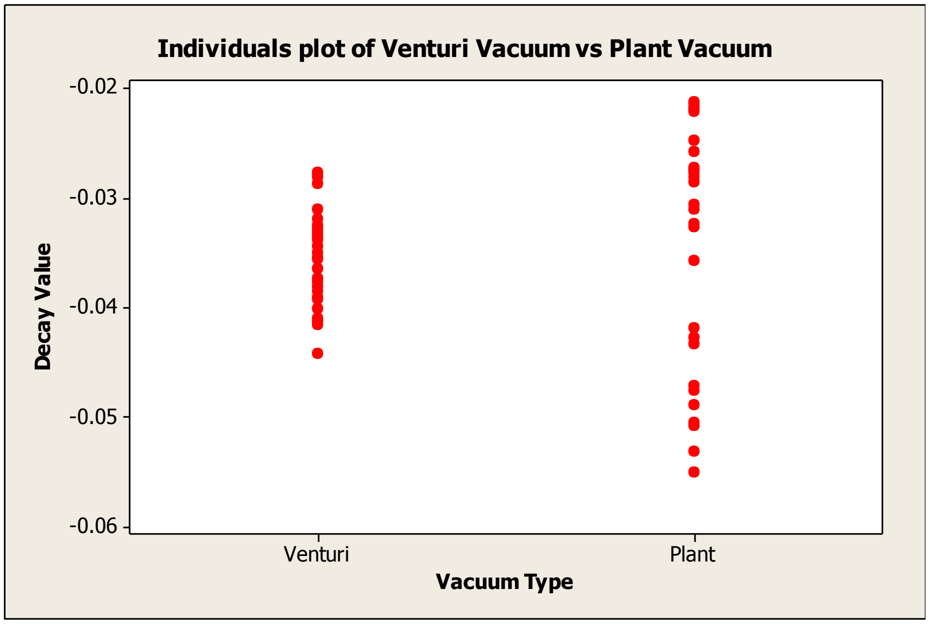

Figure 10 Plant Vacuum vs. Piab Source

Statistic	Piab System	Plant Vacuum
n	30	29
mean	-0.035554	-0.03424
median	-0.035230	-0.03064
min	-0.044295	-0.05518
max	-0.027809	-0.02131
stdev	0.004435	0.01117

Table 5 Statics For Plant Versus Piab

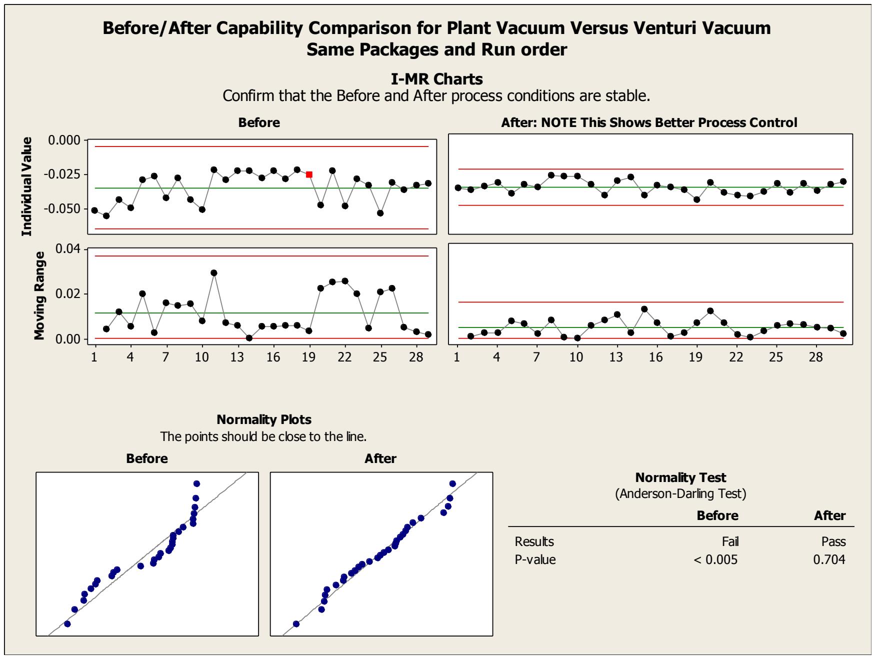

Figure 11 Before/After Comparison of Vacuum Sources

6. VERIFING THE DECAY SET POINT VALUE

A total of 100 samples were prepared in the following quantities: 48 packages with no holes (0-hole), 30 packages with 25 µm holes. 10 were prepared using the 28 gage needle for the primary hole. A second method using an Exacto knife was used to cut a hole and the material was physically removed. This method was used on twenty of the thirty 25 µ holes.

Ten packages with 50 µm holes (these were prepared using a 28 gage needle for the primary hole). Twelve packages with 711 µm (28 gage needle) holes, in order to represent the type and size of holes that had been observed.

The packages were randomly distributed and tested together using the pressure decay threshold value of -0.09 established in Part 1. Table 6 shows a summary of the results. The descriptive statistics are provided in Table 7.

Package type	Qty	Qty accepted	Qty rejected
Zero hole	48	48	0
25 µm	30	2	28
50 µm	30	0	30
711 µm	12	0	12

Table 6 List of packages tested

Statistic	0-	25µ-	50µ-
n	48	30	10
mean	-0.02897	-0.23029	-0.60115
median	-0.02826	-0.12509	-0.52951
min	-0.04270	-3.18392	-0.92215
max	-0.01855	-0.02751	-0.45268
stdev	0.00516	0.56008	0.15663

Table 7 Descriptive Statistics

All of the seeded packages with holes were found except two of the 25 µm holes. An investigation of these packages was performed. One package (#28) did not have a hole. Figure 12 shows the inside of the package, notice the oblong shape of the primary hole in the foil, this was created by the 28 gage needle and it appears as if the foil closed up over the hole on the disk.

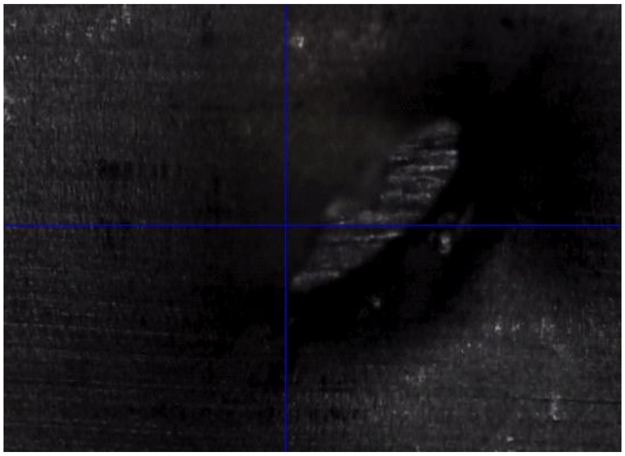

Figure 12 Package #28 Back Side 25 µm hole

The second (Package #114) had a hole (see figure 13) but was partially blocked (see figure 14).

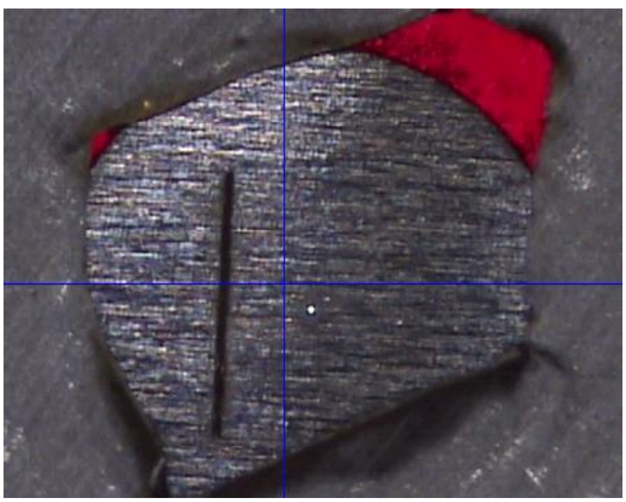

Figure 13 Package #114 Back side of 25 µm hole 50x Magnification

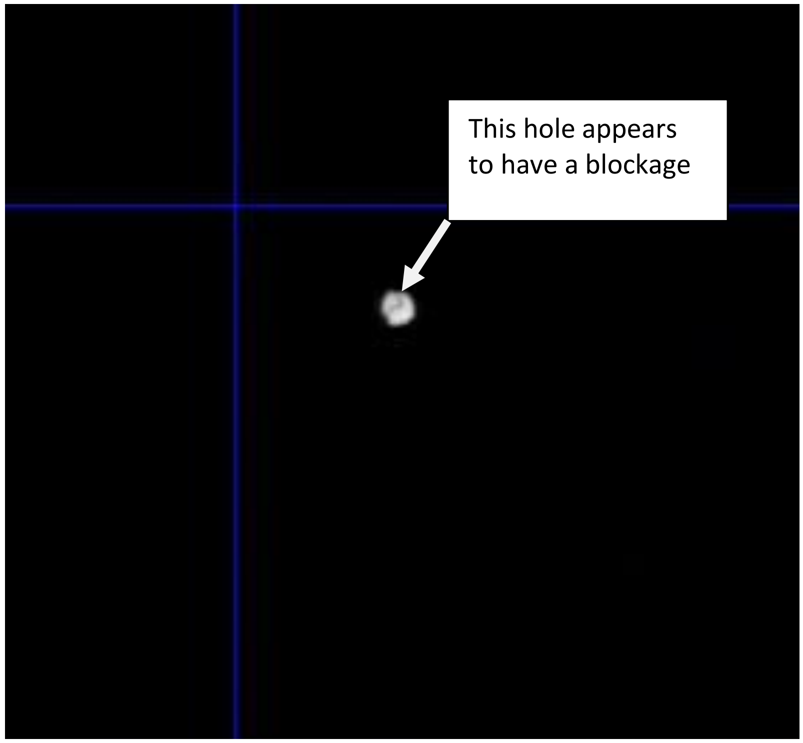

Figure 14 Package #114 at 100x Magnification

The individuals chart in Figure 15 shows the results. There was one 25μ-hole package (#33) that failed at the stabilization step so it was removed from the analysis in figure 15.

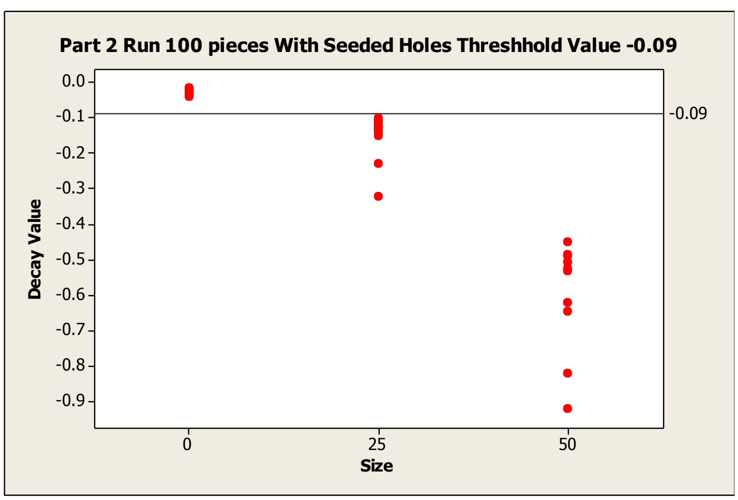

Figure 15 Individuals Chart of 100 Piece Seeded Run

The ten 50 µm holes was analyzed and the data was not normal (Anderson Darling Test P-value <.005).

In Figure 16 a histogram of the pressure decay values is shown for the 0-hole packages. The distribution is normal (Anderson Darling Test P-Value = .559).

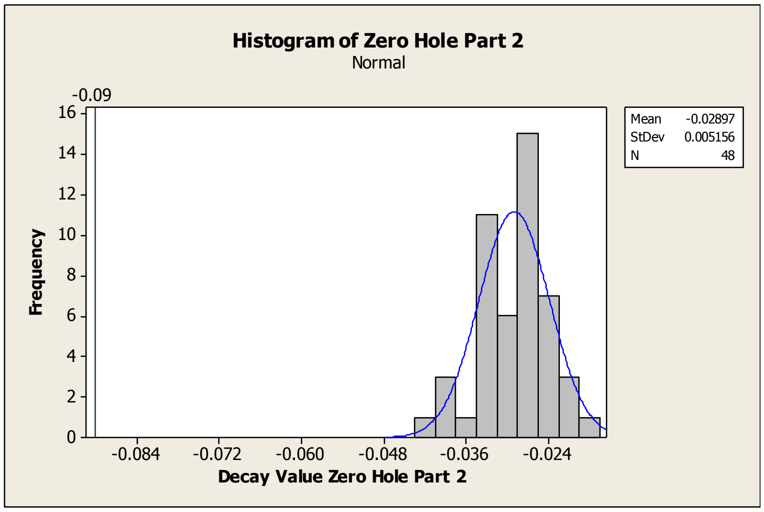

Figure 16 Histogram of the Zero Hole Packages

Figure 16 shows the variation in 0-hole decay values has been reduced from the results obtained in the first test. Given the reduction in the standard deviation of the pressure decay in the 0-hole packages, it looks like 0.09 threshold value is 12 standard deviations from the 0-hole average. This provides an opportunity to re-establish the pressure decay threshold value.

7. REVISITING THE DECAY SET POINT VALUE

After the unit was installed at the production facility the results of the previous testing was reviewed and it was decided that the accept set point value needed to be optimized. Samples for this study used precision holes supplied by Uson. These are laser drilled within ± 10% of designated hole size, both 25 µm and 50 µm. Packages (foil pouches) containing only empty trays were used for testing purposes. The representative packages from the manufacturing process and the packages modified with precision holes at 25µm and 50 µm was evaluated using the leak detector. Data from the representative samples was used to determine the threshold accept value, while data from the 25 µm and 50 µm were used to ensure that no overlap of the populations exists. Following determination of the threshold value, additional experiments were conducted to determine the number of repeat tests a package can withstand before results shift. It is intended to utilize this information to support ongoing verification of this method during routine production. The sample sizes used in this study were established (based on pressure decay data from prior studies) to provide the necessary precision for the estimates of the averages and standard deviations to establish the pressure decay threshold value.

The 90 piece run was performed including 30 packages without holes, 30 packages with 25µm holes and 30 packages with 50µm holes. The descriptive statistics are shown in Table 8 below.

Package Type	Count	Mean	Stdev	Minimum	Maximum
0 hole	30	-0.0115	0.0037	-0.0250	-0.0060
25µm holes	30	-0.1309	0.0263	-0.2200	-0.1000
50µm holes	30	-0.5728	0.4240	-2.2400	-0.1880

Table 8 Descriptive Statistics For The Secondary development

Each population was assessed for normality, and the data set for 0-hole was normal Using the Kolmogorov-Smirnov test P= 0.028, however the 25µm and 50µm hole packages was not normal with a P-Value of <0.05. **T**he separation of the 25 µm packages from the 0-hole packages was significant enough, a factor of 10, that further analysis of the 50µm data was not warranted. In order to calculate the threshold accept value for the integrity tester, the standard deviation of the zero hole packages (0.0037) was multiplied by 6 and subtracted from the mean of the zero hole packages. The six standard deviations was used to help minimize the possibility of acceptable packages being rejected causing a high loss rate or lower yield. This resulted in an accept value of -0.0337, which must be rounded to -0.03 to accommodate machine limitations. After rounding, the threshold value is 5 standard deviations from the mean. The 0-hole data set was analyzed to determine an appropriate distribution. The Gumbel Smallest Extreme Value type 1 distribution was used and is shown in figure 17. The density function is $F(x)= 1/\sigma \exp(-z-\exp(-z))$, where $z =(x-\mu)/\sigma$, μ is the location parameter and σ is the distribution scale

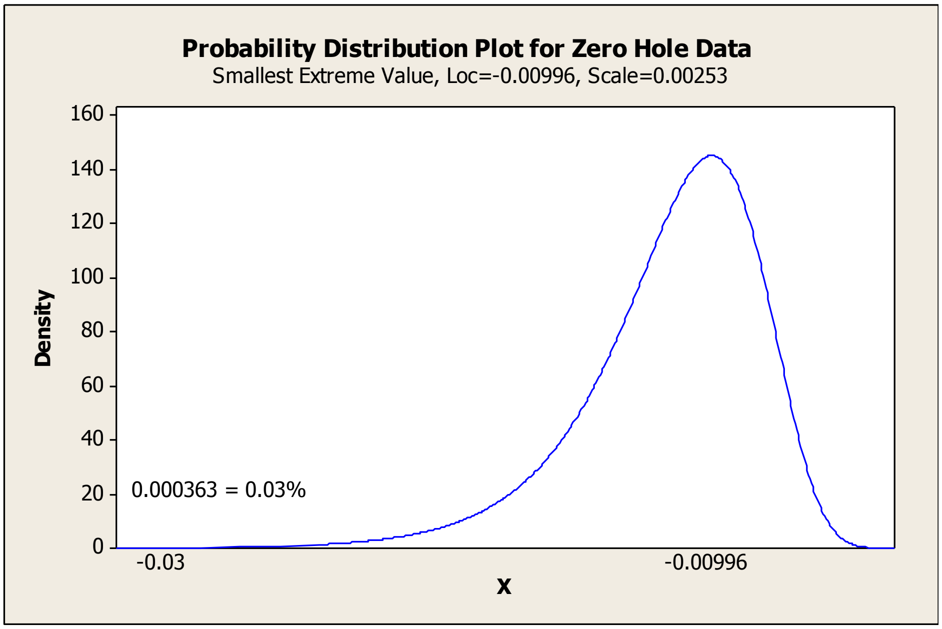

Figure 17 Distribution Plot of Zero Hole Secondary Development Run

the distribution plot shows that only 0.03% of the zero hole packages will fail at or below the -0.03 Decay set point. This threshold decay value was then compared to the data set generated from the 25µm hole packages. The proposed threshold value (-0.03) is 3.8 standard deviations from the mean of the 25µm hole packages. However, further analysis of the 25µm data set indicated that the standard deviation was affected by two decay values below -0.200 (packages #605 and #601. These two outliers cause the standard deviation to more than double. The cause of each of these outliers was the precision hole not attached correctly at the interface between the adhesive attachment ring and foil, leading to pressure loss between the foil and the ring in addition to through the hole.). Excluding these two points yields a standard deviation of 0.0115. The mean of the other 28 packages is -0.1247 and the distribution is normal with a Anderson-Darling test P-Value of 0.496. Using this data, the proposed threshold value (-

0.03) is 8 standard deviations from the mean decay value. A probability plot (excluding the two outliers) shows the probability of a 25µm hole passing is 1.11E^{-14} %. This is considered a more accurate reflection of the probability of a 25µm hole package passing the threshold decay value, as the excluded values were from the more negative side of the distribution.

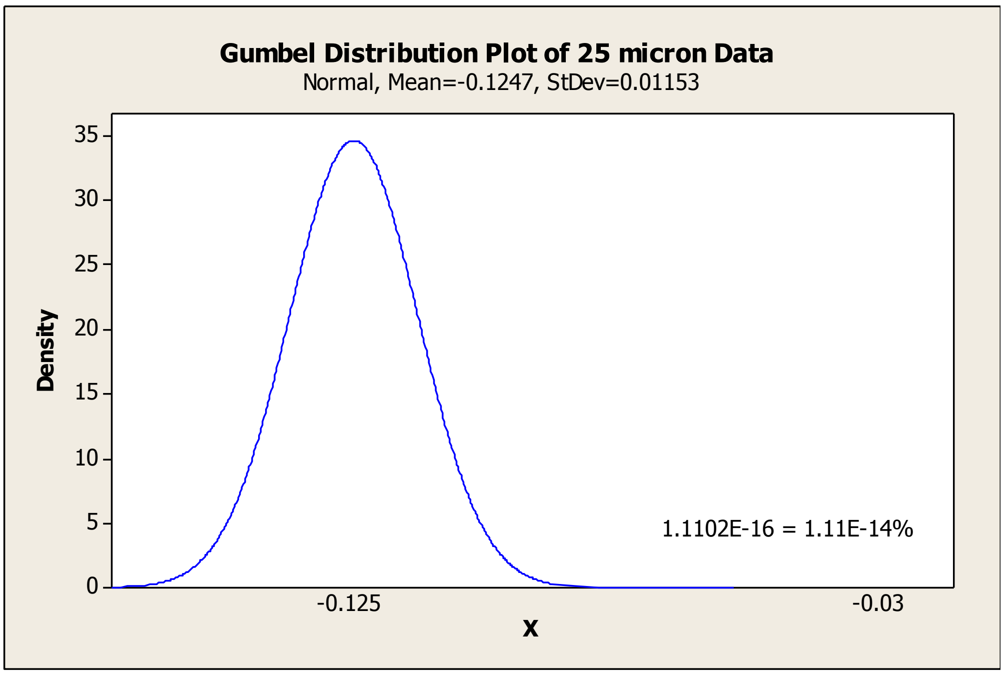

Figure 18 Gumbel Distribution Plot of 25 µm Holes

The threshold decay value of -0.03 provides sufficient protection against packages with 25µm holes. The two populations can be seen relative to the Accept decay value of -0.03 in Figure 19 below.

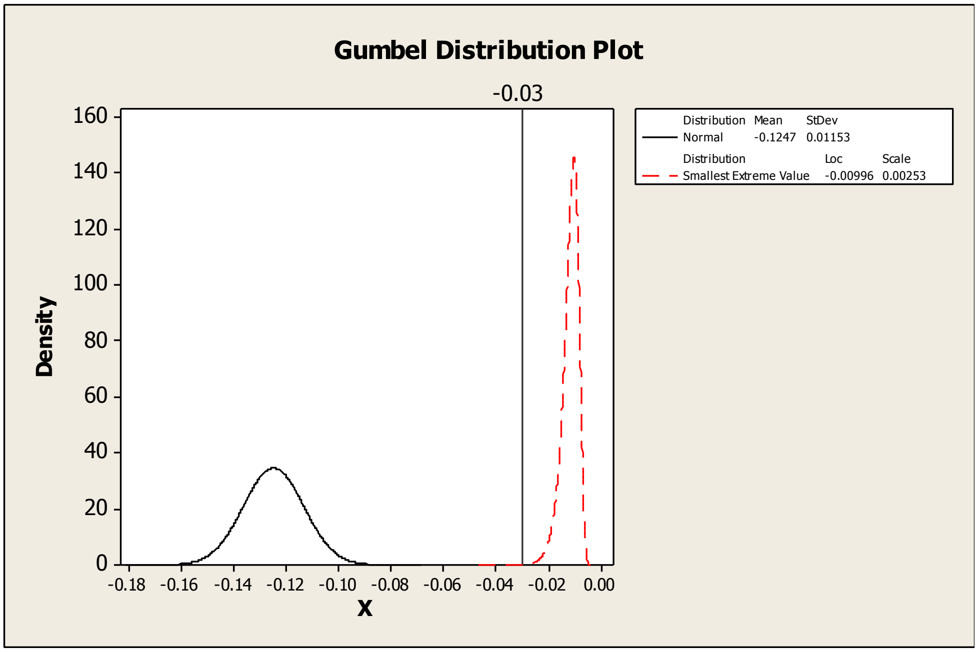

Figure 19 Comparison Plot of the two populations versus Accept decay value

8. Re-Testing OF Packages

Five packages each were chosen from each of the zero hole and 25µm holes groups and tested 50 consecutive times at 3 minute intervals The descriptive statistics are provided in Table 9 and Table10.

Pkg. #	Count	Mean	Stdev	Minimum	Maximum
808	50	-0.0121	0.0046	-0.0188	-0.0059
814	50	-0.0129	0.0038	-0.0189	-0.0068
810	50	-0.0087	0.0035	-0.0138	-0.0029
821	50	-0.0115	0.0035	-0.0175	-0.0070
805	50	-0.0082	0.0039	-0.0143	-0.0037

Table 9 Descriptive Statistics: Decay Values - Zero Hole

Pkg. #	Count	Mean	Stdev	Minimum	Maximum
334	50	-0.0981	0.0089	-0.1125	-0.0860
338	50	-0.1080	0.0010	-0.1293	-0.1010
129	50	-0.1150	0.0013	-0.1314	-0.1046
606	50	-0.1347	0.0017	-0.1626	-0.1228
604	50	-0.1457	0.0016	-0.1617	-0.1300

Table 10 Descriptive Statistics: Decay Values - 25µm Holes

The test for equal variances in the zero-hole and 25 µm data indicated statistically significant differences in variance (p=0.046) and (p-0.000), respectively, which prevented the data from being pooled. Individual/Moving Range Charts were used to evaluate overall trends for repeat testing of the 0-hole and 25µm packages. The charts are shown in Figures 20 and 21.

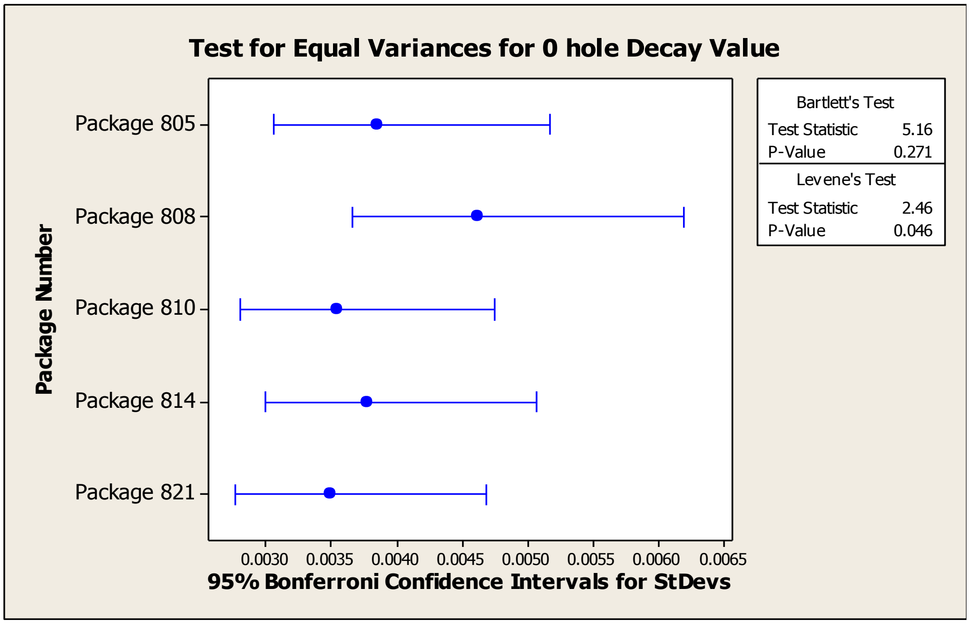

Figure 20 Test For Equal Variance of Repeat Testing of Zero Hole Packages

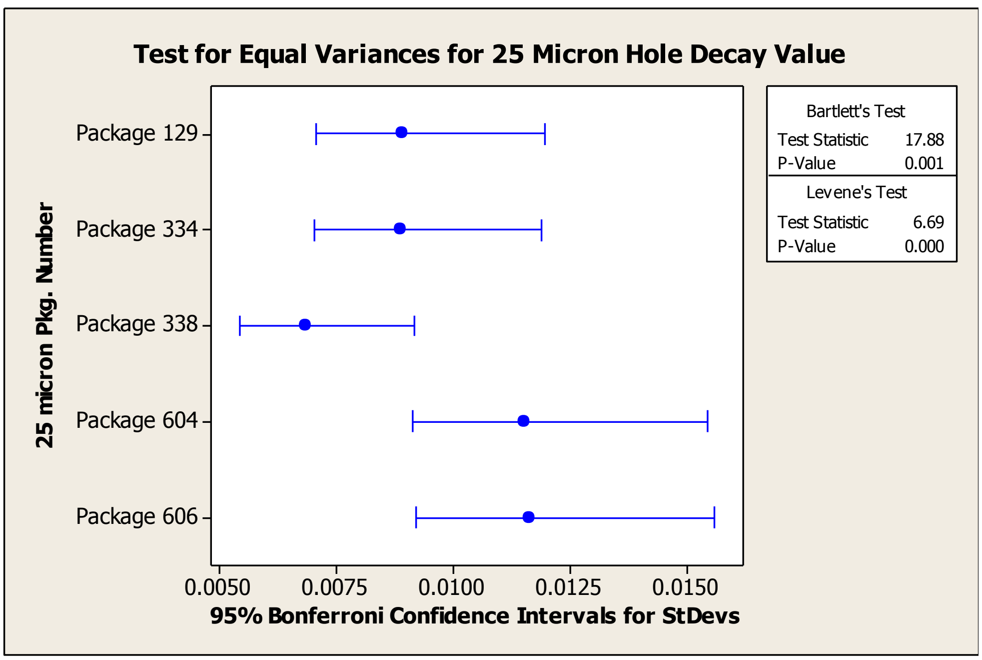

Figure 21 Test For Equal Variance of Repeat Testing of 25µm Hole Packages

The Individual Value charts for both groups show the decay value is relatively stable for 50 repeat cycles. The most variable zero hole data has a range control limit that is ½ of the control limit of the most variable 25 µm package. However, by looking at the data in stages, the range for package 338 is beginning to go out of control around observation 118 (repeat #18). This point corresponds with points below the control limits on the Individual Value chart. Likewise, package 606 begins to shift its range (though still in control) around observation 78 (repeat #28). The data indicates that the variation for two of the packages is beginning to increase after roughly 20 uses.

and below describe the original decay value and that after both 20 and 50 uses.

PACKAGE NUMBER	ORIGINAL DECAY	Mean of 1st 20 REPEATS	Mean - 50 REPEATS
805	-0.025	-0.009	-0.008
808	-0.010	-0.013	-0.012
810	-0.009	-0.009	-0.008
814	-0.008	-0.014	-0.013
821	-0.006	-0.013	-0.012

Table 11 Comparison of Original Decay Value to Mean of multiple test cycles 0 hole

PACKAGE NUMBER	ORIGINAL DECAY	Mean of 1st 20 REPEATS	Mean - 50 REPEATS
606	-0.144	-0.131	-0.130
129	-0.124	-0.117	-0.115
334	-0.100	-0.100	-0.098
338	-0.116	-0.108	-0.108
604	-0.138	-0.148	-0.149

`Table 12 Comparison of Original Decay to mean of Multiple Test Cycles 25µm holes

9. CONCLUSION

The original intent of this research had two parts, the first was to find a technology that could find a 100µm puncture in a foil package. The second was to determine the smallest puncture that could be found reliably. This needed be done without damaging the package so that a 100% inspection could be performed. The technology that was chosen, a Force Decay model, proved that it could find punctures less than 50 µm without fail so the first part was satisfied. The intent of the second part was to find the smallest puncture that could be found reliably. The reason for this is the stability of the package over time is affected by moisture permeating across a small void in the seal or puncture in the package. By knowing the minimum hole size this moisture permeation can be calculated and the stability over time determined. The other factor here was the desire not to reject packages that were acceptable as some of the products in the packages are very costly and 1% of rejections could cost $1,000,000 a year. The primary goal was to find 100% of the packages with a certain size hole without rejecting packages that do not have a hole. We found that this technology had the capability to find a 25µm puncture, however it needed to be shown that the accept value would not allow packages with punctures to be accepted. The data from the first phase of testing did not show a "goodness of fit" for all package groups tested using the Anderson-Darling test and Kolmogorov-Smirnov tests. After some investigation it was discovered that the plant vacuum was creating "noise" in the measurements as the usage went up and down due to the starting and stopping of equipment elsewhere in the plant. After switching the vacuum source to a venturi system the data on the zero size hole became normal using the Anderson-Darling test and showed a "goodness of fit". This allowed the accept value to decrease from -0.09 to -0.03

and increase the gap between the 25µm holes and the zero size holes therefore improving the reliability of the process.

We must ensure that 100% of the 25µm holes are identified without a high rate of “false” rejects. False rejects are when acceptable packages are rejected because of the accept value set point. The results of the testing indicate, using a Gumbel Distribution that the set point for acceptance at -0.03 would only allow 0.03% of acceptable packages to be rejected while allowing only 1.11×10^{-14}% of packages with a 25µm hole to pass. This meets the criteria for a reliable process. It is recommended that this testing method be considered for production. A gage R&R was performed and the method passed all of the criteria and was validated.

The process parameters were not looked at during this research due to time constraints but there are signs that this process could be improved so that a smaller hole might be reliably identified, the goal being 10µm. The findings from the research has been implemented and is currently be used and monitored.

10. REFERENCES

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5. ASTM F1140 - 07 “Standard Test Methods for Internal Pressurization Failure Resistance of Unrestrained Packages” (also called “The Burst Test”)

Minitab 16 was used for statistical analysis and graphs

11. VITA

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Accomplishments

- **Patents granted**:

US5623810, US5709067, US5732529, US5833055, US5987855, US6021625, US6047815, US6097427, US6135272, US6463719, US6804937, US6807796, US6820400, US6915623, US7467710, US7837455, US8128859.