Review of Metallized Film Adhesion Testing, Part 2: How Celplast Achieved Reproducible Results and Used This Test to Optimize Their Metallizing Process

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Introduction

There has been much discussion recently with respect to metal adhesion testing, and how to obtain reproducible results. As we have begun to investigate metal adhesion ourselves, we are particularly concerned with being able to identify any improvements being made in the metal adhesion of our metallized films in order to develop new products for demanding converting applications. Therefore, obtaining reproducible results became a top priority for our company.

Experimental Apparatus

Heat seals were carried out using a Wrapade Model K heat sealer, with 1" x 10" flat seal bars. Dwell time was maintained to within +/- 0.1 sec with a calibrated timer. Seal pressure was maintained at +/- 0.1 psi, with a calibrated pressure transducer. Upper and lower seal jaw temperatures were maintained to within +/- 1°F via heater coils controlled by an Omron PLC.

1" wide samples were cut out using a JDC 1-10 model shear sample cutter with protective shields.

All peel tests were carried out on a Thwing-Albert QC-1000 model tensile tester, with a calibrated 2000 g load cell and 1" wide rubber grips. Crosshead speed was set and periodically verified using a ruler and stopwatch. Raw & summary data was imported into an Excel spreadsheet via an RS-232 cable and the WinWedge software program.

All equipment conforms to the following ASTM test methods[2]:

- D882-02 – Standard Test Method for Tensile Properties of Thin Plastic Sheeting
- F904-98 – Standard Test Method for Comparison of Bond Strength or Ply Adhesion of Similar Laminates Made from Flexible Materials

Reducing Variability in the Test Method

Our initial experiments were carried out using the AIMCAL Metallizing Technical Reference Manual metal adhesion test method[1]. Due to the optimum heat seal pressure operating range of the Wrapade heat sealer, we were required to maintain a minimum 30 psi seal pressure during heat sealing, as opposed to the 15 psi recommended in the AIMCAL procedure. Also, we used a 610 Scotch tape backing on the metallized film and finished sample sizes of 1" x 3.5". We measured the peel force over a 1" x 1" seal area, testing several different metallized PET films.

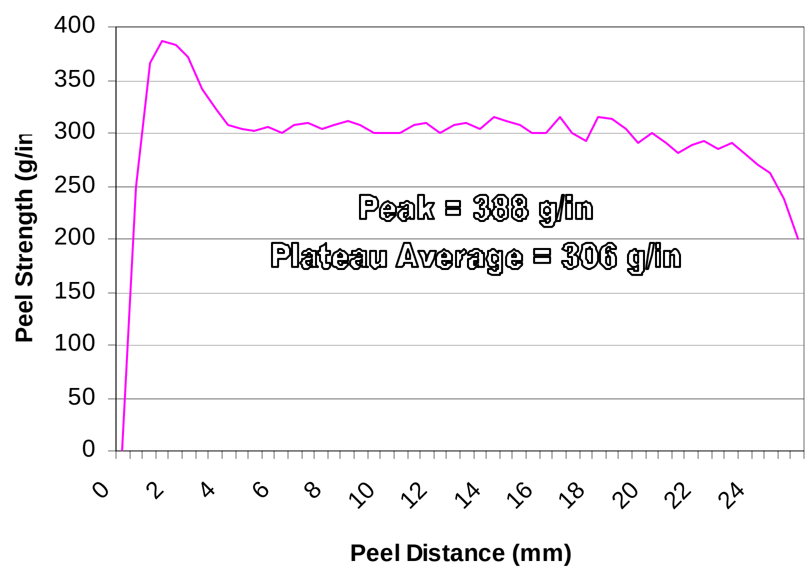
A few other details were optimized early on:

- Metal pull off the metallized film surface was more consistent when using the treated side of the EAA film.
- More consistent results for both the peak & average peel forces were obtained using a 180° T-peel, with the metallized film tail in the upper jaw of the peel tester, the EAA film in the lower jaw, and the heat sealed tail sticking straight out perpendicular to the peel direction. This minimized any peel force drift over the course of the test due to changing geometry of the peel angle.
- More reproducible results were obtained with the EAA film placed in the moving jaw (in the case of the Thwing-Albert QC-1000 unit, this is the lower jaw), as it led to fewer incidents of EAA film tear.
- We evaluated whether the conditioning time after heat sealing had a bearing on the peel test values. Conditioning was carried out in the Celplast lab, where humidity & temperature are always controlled (25 - 50% RH, 73-75°F). We found that for samples where peel tests were carried out 30 minutes or less after heat sealing, results tended to vary significantly. For peel tests carried out 45 – 180 minutes after heat sealing, there was no significant difference. Therefore, all peel testing was carried out out after a minimum 45 minute conditioning time.

Note that even though these details were optimized through our own testing, independently of AIMCAL, they coincide precisely with the techniques recommended in the AIMCAL metal adhesion test method.

We generated several data plots, of which a typical curve would be as follows:

Figure 1: Peel Force Curve for Typical met PET



We established early on that there was a closely linked correlation between peak & plateau average peel strength values. In particular, it appeared that the average value was consistently 18-20% lower than the peak force value. To determine whether there was a significant difference between the Peak and Average peel forces, we carried out an F-test treatment[3] based on 60 replicate samples obtained for a standard 2.3 OD met PET film, sealed at 40 psi, 240°F, 15 second dwell time, and peeled apart at a 9 in/min crosshead speed.

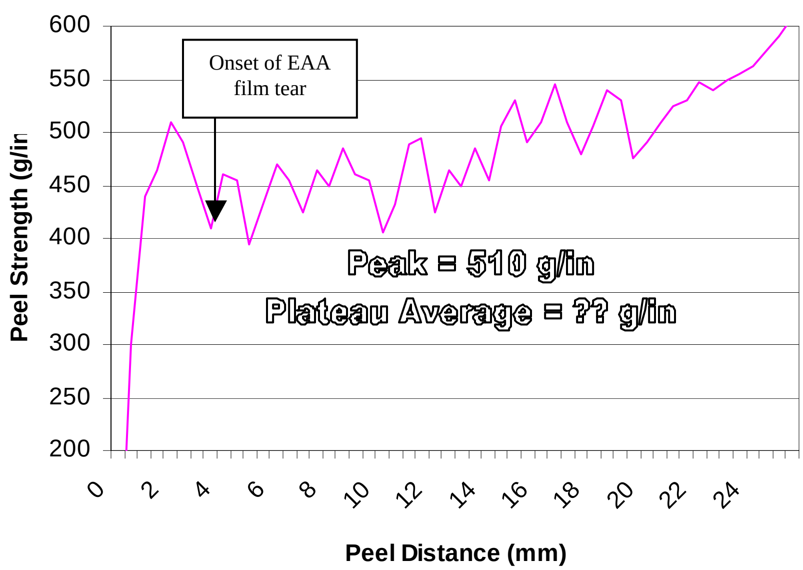
Table 1: Peak vs. Average Peel Forces & Variances (g/in)

	Peel Force - Peak	**Peel Force - Avg.**
Number of tests	60	60
Average	338	276
Std. Dev.	35	31
Variance	1225	961
F-test - observed	1.27471384	
F-test (59,59,0.05)	1.53	
Significant difference?	**No**	

In Table 1, we compare the observed F-test value (Variance Peak peel force / Variance Average peel force) with a calculated F-test value, based on 59 degrees of freedom for both the Peak & Average values, using a 95% level of confidence. If the observed F-test value is less than the calculated value, we can say there is no significant difference between them. In this case, we established that both values had an equivalent reproducibility, so we could use either the peak or average value going forward.

We also encountered some samples of met PET with even higher peel force values. In these cases, we would experience EAA film tear, which would lead to a highly inconsistent peel force curve. For these samples, we were able to measure the initial peak peel force, but not an average "plateau" peel force, since there was no discernable plateau. This situation is represented by a typical curve in Figure 2.

Figure 2: Peel Force Curve for met PET with Excellent Metal Adhesion



Therefore, going forward, in order to be able to measure & compare peel forces across a broad metal adhesion range, we focused exclusively on peak peel force measurements.

Now that we had established our dependent variable, we wanted to minimize the variability associated with the test method. To do so, we examined the four remaining independent variables that we believed would have the greatest potential effect on reproducibility:

- Heat seal pressure
- Heat seal temperature
- Heat seal dwell time
- Crosshead speed

Based on our experience so far, it was established that the reproducibility of the test was unlikely to get much better without having a large number of replicates included in any single test data point. Therefore, all future test results would be based on the average of peak peel results from 10 separate samples, as opposed to the 5 samples recommended by the AIMCAL test method.

At this point, we wanted to establish the reproducibility of the mid-point test method (40 psi, 240°F, 15 sec dwell, 9 in/min crosshead speed). By breaking up the 60 peak peel force sample results from Table 1 into 6 results made up of 10 samples each, we were able to calculate the variance of the peak peel force from each collection of 10 data points.

- Standard deviations for the 6 sample sets: 30, 44, 47, 29, 36, 22 g/in
- Standard deviation of the standard deviations: 9.54 g/in
- Variance of the standard deviations: $(9.54)^2 = 90.25$ (Mean of Squares)
- Degrees of freedom for variance calculation: 6 – 1 = 5

Now we can establish a Design of Experiments (DOE) for the four main factors under investigation, with the objective in mind of minimizing the standard deviation of the peak peel force (based on 10 samples) as our main dependent variable. Due to the large amount of testing involved, a 2^{4-1} fractional factorial design was developed. A high & low value was set for each independent variable, with an orthogonal design implemented. This will allow us to investigate the main effects of each variable, but not the interactive effects.

Table 2: Metal Adhesion of 2.3 OD met PET, 2^{4-1} DOE

Test #	Heat Seal Pressure (psi)	Heat Seal Temperature (°F)	Heat Seal Dwell Time (s)	QC-1000 Crosshead Speed (in/min)	Average Peak Peel (g/in)	Std. Dev. Of Peak Peel (g/in)
Mid	40	240	15	9	338	35
1	30	220	10	12	271	34
2	30	220	20	6	450	75
3	30	260	10	6	359	29
4	50	220	10	6	354	90
5	50	260	20	6	280	27
6	50	260	10	12	292	24
7	50	220	20	12	324	34
8	30	260	20	12	309	30

By producing an ANOVA table[3] on the main effects examined here, we were able to determine which effects had a significant impact on the variability of our peak peel force.

Table 3: ANOVA Table for Main Effects on Peak Peel Force Values (g/in)

	Effect	SS	df	MS	F
Heat Seal Pressure	7	196	1	196	2.17
Heat Seal Temperature	-123	60516	1	60516	670.54
Heat Seal Dwell Time	-11	484	1	484	5.36
Crosshead Speed	-99	39204	1	39204	434.39
Error			5	90.25	
F(1,5,0.05) = 6.61					

- "Effect" = ((sum of Std. Dev. of Peak Peels at high value of effect) - (sum of Std. Dev. of Peak Peels at low value of effect))/4
- "SS" = sum of squares = 2^{n-2} x $(\text{Effect})^2$ (where n = no. of factors)
- "df" = degrees of freedom for each test #
- "MS" = mean of squares = SS/df
- "F" = observed F-test value = MS_{Effect}/MS_{Error}

In order to determine the significance of each main effect in Table 3, we need to compare the observed F-test value to the calculated F-test value F(1,5,0.05). When we do so, it is apparent that only the heat seal temperature and the crosshead speed are significant factors, and both are negatively correlated to standard deviation. Therefore, as both are increased, we can expect the standard deviation to decrease.

We can verify that this is indeed the case by observing the variability of test #6 & 8 from Table 2, where both of these values are in the "high" position. The average standard deviation of these two tests is 27, compared to the mid-point standard deviation of 35. In terms of variance (the square of standard deviation), this is a 40% improvement in reproducibility of the test!

Since we had to ensure that our heat seal test would not distort metallized OPP samples we also wanted to test, the heat seal temperature was set to 250°F. Crosshead speed was set to 12 in/min, and heat seal pressure & dwell time remained at the "low" settings of 30 psi & 10 seconds, respectively.

Shortly after carrying out this DOE, we found that as we attempted to achieve better and better metal adhesion it was becoming more and more difficult to get consistent peel strength values. This was primarily due to the low tear resistance of the 1 mil EAA adhesive film. Therefore, we switched to a substrate with a similar treated EAA surface, but in a much more tear resistant structure: paper, extrusion laminated to foil, with a 1.0 mil EAA extrusion coating on the foil. Using this structure, we were able to maintain a high level of reproducibility up to peak peel force values of 1000+ g/in. In order to obtain a good seal using this bulkier structure, heat seal pressure was increased to 40 psi. Other settings remained the same.

Using these latest test conditions, the standard deviation for a test from a set of 10 samples with a peak peel force in the 300 – 600 g/in range is typically 25 – 35 g/in. Therefore, we can say with 95% confidence that when we measure the average peak peel force on two different samples, if the difference between these values is greater than 40 g/in the difference is greater than what can be explained by experimental noise, and the samples are fundamentally different in metal adhesion from each other.

Recent Test Results

With a more reproducible test method in place, we could investigate whether adjustments in our process would actually contribute to improved metal adhesion values. Evaluating different PET films under identical metallizing conditions, we can observe real differences between different treatment types as well as PET suppliers. In addition, by my making process modifications in our metallizing chambers, we have been able to achieve a wide range of peel force values. Some recent test results are shown in Table 4.

Table 4: Recent Metal Adhesion Test Results

Film Type	Metallizing Process Condition	Metal Adhesion Value (g/in)	Standard Deviation (g/in)
A	1	63	5
B	1	305	28
C	1	440	18
C	1(replicate)	450	20
D	1	480	31
D	2	580	36
D	3	860	39
D	4	680	37
D	4(replicate)	650	30

Conclusions

We have determined that there are several variables which can have an impact on metal adhesion test results. In particular, the reproducibility is affected by number of replicates tested per sample, heat seal temperature, type of heat seal substrate, orientation of your sample in the peel tester jaws, crosshead speed, and where on the peel force curve your measurement is taken. By optimizing each of these variables, reproducibility of this test can be greatly improved.

Acknowledgments

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