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Pull-Off Adhesion Testing of Coatings – Improve Your Technique

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ABSTRACT

Pull-off adhesion testing is widely used to assess the protective coating process. It is also used to determine if a coating is fit for service in new construction and for repairs to existing structures. The result is often critical to the acceptance or rejection of a coating process, as the adhesion value quoted by the paint manufacturer can be adversely affected by aspects of the coating process. Low adhesion values are indicative of premature failure of the coating and are often due to inadequate surface preparation of the substrate.

ASTM D4541 and BS EN ISO 4624 describe several different test apparatus; however, the basic approach of gluing a test dolly to the coated surface and then exerting a perpendicular force to the surface in an effort to remove both the dolly and the coating from the substrate is common to all these standards. A measure of the adhesion of the coating system is the force at which the coating fails and the type of failure obtained.

Trials have demonstrated that many aspects of the testing method, such as the mixing of the glue, the preparation of the coating surface and the face of the dolly and the temperature of the test, all affect the results.

This paper will investigate the effects of any deviation from the proscribed method in every aspect of the test. Each aspect is examined in turn, the results tabulated and the potential effect on a valid adhesion test result is discussed.

INTRODUCTION

The tensile pull-off method for adhesion testing, as outlined in ASTM D 4541 and similarly in BS EN ISO 4624, involves gluing a test dolly to the coated surface and then pulling the dolly by exerting a force perpendicular to the surface in an effort to remove the dolly with the coating from the substrate. The force at which this occurs and the type of failure obtained is recorded as a measure of the adhesion properties of the coating.

Several aspects of the test method were assessed, including the mix of the epoxy glue, different types of glue, surface preparation, the design of the dolly, temperature of the cure and the test, and cutting the coating or not. The difference between manual and automatic pull-off tester operation was also investigated. This paper evaluates the effects of any deviation from the required method in several aspects of the test. Each aspect is examined in turn, the results tabulated and the potential effect on a valid test discussed.

ADHESIVE MIXING

The test dolly should be glued to the surface using a suitable adhesive. Typically a two-pack epoxy adhesive is supplied with adhesion test units. The instructions for this type of adhesive state that the two components, resin and hardener, should be mixed in equal parts usually equal lengths of both parts. In order to achieve a more accurate mix, the amount of each component was measured by weight using an accurate electronic balance.

A test was carried out to understand the effects of mixing the adhesives incorrectly. 3 samples of glue were mixed, a 1:1 resin (a) to hardener (b) ratio, a 1:2 hardener to resin ratio and a 2:1 hardener to resin ratio. Unprepared dollies were stuck down on unprepared surfaces such that the only variable was the glue mix. The adhesive strength was not optimised. The dollies were then pulled using a manual Type V gauge.

Set	Glue Mix ratio (a:b)	Pull 1 (MPa)	Pull 2 (MPa)	Pull 3 (MPa)	Pull 4 (MPa)	Pull 5 (MPa)	Average Pull Value	% variation
1	1:1	6.0	6.4	7.6	7.6	4.5	6.67	-
2	1:2	5.8	4.0	3.9	3.6	3.9	3.93	-41
3	2:1	5.8	5.5	6.2	7.9	7.0	6.33	-5

Table 1 – Test Results for different ratios of two-pack epoxy glue

The average value is calculated excluding the maximum and minimum value in each set to avoid any skewing of the results due to any outliers. This approach was taken with all tests.

In Table 1 the results show an average failure value of 6.67 MPa when the glue is mixed correctly. If too much resin is used, then the failure force reduces by 5%, which is quite small, but is outside the ±1% stated accuracy of the gauge. However when the hardener quantity exceeded the resin, then a variation of -41% in failure strength was noted. It is unlikely that a user will mix the glue in such an erroneous way, indicating that an excess of resin to hardener would have little effect on the results, whereas too much hardener could still significantly affect any test results.

COMPARISON OF TWO ADHESIVE TYPES

ISO 4624 has guidance on the selection of suitable adhesives. The relevant section is reproduced as Figure 1 below.

Special attention is required in selecting suitable adhesives to be used in the test. To produce failure of the coating, it is essential that the cohesive and bonding properties of the adhesive are greater than those of the coating under test.

Preliminary screening of adhesives shall be carried out in order to determine their suitability for use. Suitable adhesives and, if applicable, their unmixed components shall cause little or no visible change in the coating under test when left in contact with the coating for a period equivalent to the curing time of the adhesive.

Adhesives which give the highest results, which means the most coating-substrate adhesive failure, are preferred.

In most cases, cyanoacrylate, two-component solventless epoxide and peroxide-catalysed polyester adhesives have been found suitable. In special tests under highly humid conditions, the curing time of the adhesive shall be as short as possible. The use of two-pack quick-drying epoxy adhesive is preferable in these situations.

NOTE Where failure is mainly associated with the adhesive, the use of another type of adhesive may enable more useful results to be obtained.

Figure 1 – Extract from ISO 4624

ASTM D 4541 states that the adhesive is for securing the fixture to the coating and that it does not affect any coating properties. Two component epoxies and acrylic adhesives have been found to be the most versatile.

Both specifications clearly state that there is no one single glue that can be specified for all coating pull tests; rather, glue suitable for the conditions of the test should be used. The correct glue is one that has bond strength greater than the adhesive strength of the coating being tested.

Different suppliers provide different adhesives with their adhesion test kits. Adhesives from the same supplier are often made in different parts of the world and the locally available version may differ from location to location. Indeed, in some cases, certain adhesives may be unavailable in given parts of the world.

The relative strength of both adhesives was compared. These adhesives are both commonly supplied in adhesion test kits. 10 dollies were stuck down to an uncoated, unprepared steel substrate, 5 using Adhesive A, and 5 using the Adhesive B. The glue was allowed to cure for 24 hours and the dollies pulled from the surface

Dolly	Glue	Test Value (MPa)	Failure Type
1	Adhesive A	5.8	Adhesive Dolly/Glue
2	Adhesive A	7.5	Adhesive Dolly/Glue
3	Adhesive A	8.7	Adhesive Dolly/Glue
4	Adhesive A	10.7	Adhesive Dolly/Glue
5	Adhesive A	8.3	Adhesive Dolly/Glue
6	Adhesive B	12.2	Adhesive/Cohesive 80:20 Dolly/Glue
7	Adhesive B	12.9	Adhesive/Cohesive 50:50 Dolly/Glue
8	Adhesive B	14.6	Adhesive/Cohesive 50:50 Dolly/Glue
9	Adhesive B	12.5	Adhesive/Cohesive 30:70 Dolly/Glue
10	Adhesive B	11.0	Adhesive/Cohesive 50:50 Dolly/Glue

Table 2 – Results for tests on a steel plate

As seen in Table 2, the values for the failure strength and the type of failure indicate that the Adhesive B has stronger adhesive properties than Adhesive A by approximately 4 MPa.

Dolly	Glue	Test Value (MPa)	Failure Type
1	Adhesive A	8.1	Adhesive Glue
2	Adhesive A	10.3	Adhesive/Cohesive 50:50 Glue/Coating
3	Adhesive A	10.9	Adhesive/Cohesive 50:50 Glue/Coating
4	Adhesive A	11.8	Adhesive Coating
5	Adhesive A	9.8	Adhesive/Cohesive 80:20 Glue/Coating
6	Adhesive B	8.2	Adhesive/Cohesive 80:20 Glue/Coating
7	Adhesive B	7.6	Adhesive/Cohesive 10:90 Glue/Coating
8	Adhesive B	12.0	Adhesive/Cohesive 95:5 Glue/Coating
9	Adhesive B	11.8	Adhesive/Cohesive 60:40 Glue/Coating
10	Adhesive B	11.0	Adhesive/Cohesive 50:50 Glue/Coating

Table 3 – Results for tests on a painted steel panel

Table 3 shows the results of a repeated test, this time on a coated surface. The average value for both adhesives is the same, 10.33 MPa. However with either adhesive, the crucial factor is that both have sufficient strength to carry out a successful adhesion test.

SURFACE PREPARATION

"To reduce the risk of glue failures, the surface of the coating can be lightly abraded to promote adhesion of the adhesive to the surface. If the surface is abraded, care must be taken to prevent damage to the coating or significant loss of coating thickness."

The preceding paragraph is taken from ASTM D 4541 and is not an instruction, rather a suggestion. A series of tests were carried out to examine the effect of preparing both the dolly and the surface.

4 sets of 5 dollies were glued to an uncoated surface with various combinations of preparation. The results for this test are shown in Table 4. Set 1 had both dolly and substrate prepared, Set 2 has only the surface prepared, Set 3 has only the dolly prepared and for Set 4 no preparation was carried out. After curing, the dollies were pulled from the surface and the failure value recorded.

Set	**Pull 1 (MPa)**	**Pull 2 (MPa)**	**Pull 3 (MPa)**	**Pull 4 (MPa)**	**Pull 5 (MPa)**	**Average Pull Value**	**% variation**
1	10.9	11.5	11.4	11.5	11.4	11.43	-
2	10.0	5.7	8.2	10.9	11.2	9.7	-15%
3	11.1	10.8	11.2	10.7	10.9	10.87	-4.9%
4	10.8	10.8	10.3	9.8	11.1	10.56	-7%

Table 4 – Results for the surface preparation trials

Using Set 1 as the "control" where both the dolly and the surface were prepared there are some marked differences to be seen when other combinations are utilised. If no preparation is done (Set 4), then there is a 7% reduction in the average failure value achieved. Preparing the dolly alone (Set 3) results in a negative variance of just less than 5% whereas preparing the coating surface only, (Set 2), results in a 15% reduction in failure value.

The average failure value for each set indicates that preparing both surfaces increases the strength of the bond between adhesive and coating, and adhesive and dolly. This greatly increases the chances of a successful test as the strength becomes greater than the specified coating strength. This leads to a further question: what level of surface preparation of the dolly should be undertaken for best results?

Tests were carried out using sanded and blasted dollies on a blasted metal surface and the results are listed in Table 5 below.

Dolly	Sanded (MPa)	Blasted (MPa)
1	8.7	10.0
2	10.3	14.2
3	11.3	14.4
4	14.8	11.9
5	10.8	13.4
Average Value	10.8	13.17

Table 5 – Test results for sanded and abrasive blasted dollies

These results clearly show that blasted dollies give a higher test value than a sanded dolly, in this case 22% higher.

In subsequent discussions with adhesive suppliers, it was recommended that both dolly and coated surface be abraded for their product to be most effective.

VARIATION IN LOADING FIXTURE (DOLLY) DESIGN

Any reference to the dimensions of the dolly, in any of the relevant standards, is only a recommendation that the length (height) of the dolly be at least half the diameter of the dolly. If this recommendation is taken literally, then most, if not all, commercially available dollies do not comply with this recommendation because dollies are shaped rather than cylindrical. However, taking the "spirit" of this recommendation, the thickness of the base should be a consideration.

Two designs of dolly were used, one having a base thickness of 4.0 mm (A) and the other a thickness of 2.6mm (B). The two styles of dolly are shown in Figure 2. Test results are given in Table 6.

Dolly Type A

Dolly Type B

Figure 2: Two dolly design types

Pull	A (MPa)	B (MPa)
1	10.4	9.4
2	10.7	8.2
3	11.3	9.1
4	9.8	8.8
5	10.3	8.5
Average value less outliers	10.46	8.8

Table 6 – Comparison of two dolly designs

Neither the dollies nor the un-coated surface received any surface preparation before the testing took place; hence any variation in readings can be attributed to the geometry of the dollies, as this is the only variable.

Results show a 16% higher pull strength is required to remove the thicker based dolly from the surface than the thinner based dolly. Video examination of tests carried out on glass showed that, in both cases, the dollies started to lift from the edges, but there was no discernible visible difference in the mechanical action of the pull test on each dolly.

TEMPERATURE OF ADHESIVE CURING AND PULL TESTING

Temperature and time of cure coupled with the temperature at the time of the adhesion test may have an effect on the results obtained. To investigate this, a series of tests were set up. Dollies were glued to a coated surface, both having been prepared as per the recommendations contained in ASTM D 4541. Various cure times and temperatures were used, and the tests were carried out at different temperatures. Table 7 summarises the conditions and results.

Set	Cure time (hours)	Cure temp (°C)	Test temp (°C)	Test value (MPa)
1	24	22	22	11.2
2	8	30	22	9.0
3	24	50	50	7.5
4	24	50	22	10.83
5	8	30	30	8.23
6	24	30	22	10.43

Table 7 – Results for tests at different temperatures

It must be noted that no combination of cure and test temperature gives a higher test value than the “control” conditions of set 1. In attempt to speed up the process, set 2 was cured at 30°C for 8 hours, a typical shift length, and pulled at room temperature with reduced results. These results suggest that under whichever conditions the cure occurs, the pull should be carried out at an ambient temperature of 22 ± 2°C.

TO CUT OR NOT TO CUT

ISO 4624 states "… carefully use the cutting device (5.4) to cut around the circumference of the dolly through to the substrate, unless otherwise specified or agreed" whereas ASTM D 4541 states "Scoring around the fixture violates the fundamental in situ test criterion that an unaltered coating be tested. If scoring around the test surface is employed, extreme care is required to prevent micro-cracking in the coating, since such cracks may cause reduced adhesion values. Scored samples constitute a different test, and this procedure should be clearly reported with the results."

The different approaches by the two leading standards institutions as to the cutting of a dolly underline the uncertainty of whether it is the best thing to do when carrying out an adhesion test.

10 dollies were stuck down on a coated surface, 5 were left uncut, and the other 5 were cut once the glue had cured. Results are presented in Table 8.

Dolly	Cut or un-cut	Test result (MPa)	Type of failure
1	Un-cut	10.3	Adhesive
2	Un-cut	8.5	Partial
3	Un-cut	10.5	Partial
4	Un-cut	10.5	Adhesive
5	Un-cut	10.6	Adhesive
6	Cut	9.7	Partial
7	Cut	9.6	Partial
8	Cut	9.0	Partial
9	Cut	10.6	Adhesive
10	Cut	9.2	Partial

Table 8 – Test results for cut and un-cut coating

"Partial" failure indicates a failure which is a combination of a partial cohesive failure of the coating and a partial adhesive failure between the glue and the coating.

Given the distribution of the partial type of failure, there was some thought that the cutting of the dolly once the glue had cured was affecting the bonding mechanism of the adhesive. Were micro-cracks being initiated by the act of cutting? In order to eliminate this possibility, 5 more dollies were stuck to the coated surface, but this time the cutting took place before the dollies were stuck down.

Dolly	Cut or un-cut	Test result (MPa)	Type of failure
1	Cut	9.8	Adhesive
2	Cut	10.1	Adhesive
3	Cut	9.5	Adhesive
4	Cut	9.9	Adhesive
5	Cut	10.5	Adhesive

Table 9 – Test results for a pre-cut coating

If we discount the highest and lowest value from all three groups of tested dollies, Tables 8 and 9, then take the average, the un-cut test shows an average of 10.43 MPa and the cut dollies 9.5MPa. This gives an 8.9% variation between cut and un-cut. If we now look in the same way at the pre-cut set, the average value is 9.93MPa, a variation of 4.3% from the un-cut test.

These results would indicate that cutting the dolly does have an impact on the test results, but this effect is minimised if the cutting takes place before the dolly is stuck down.

The cutting of the dollies prior to them being stuck down was carried out in a machine shop, with the test plate fixed and the cutting tool inserted into a drilling machine. These are most definitely not field conditions. Cutting the dollies prior to gluing, by hand, is difficult, if not impossible. The tool skates across the surface, similar to a needle across an LP record, and it is difficult to stop this "freehand". A guide was made using a steel disc 10mm thick with a hole slightly larger than the cutting tool drilled through it. Holding this guide down was difficult if not impossible, as the act of turning the cutting tool dragged/pushed the disc across the surface. This situation was rectified by using two G-clamps to hold the ring in place whilst the pre-cuts were made.
Table 10 lists the result obtained for 5 dollies where the coating has been cut after the dollies have been stuck down (Post) and 5 dollies where the coating was cut by hand as described above.

Dolly	Pre or post cut	Test results (MPa)	Type of failure
1	Post	8.5	60% Cohesive
2	Post	9.4	100% Cohesive
3	Post	9.8	100% Cohesive
4	Post	10.0	90% Cohesive
5	Post	10.3	100% Cohesive
6	Pre	11.9	100% Cohesive
7	Pre	10.9	100% Cohesive
8	Pre	9.8	100% Cohesive
9	Pre	10.4	100% Cohesive
10	Pre	11.9	100% Cohesive

Table 10 – Test results for pre and post cut films

Using the same approach of discarding the highest and lowest values before calculating the average, the average value of post-cutting the dollies was 9.73 MPa and pre-cutting the dollies 11.1 MPa. This would indicate that pre-cutting the dollies has less effect on the adhesion than cutting after the glue has cured.

One factor that must be considered when drawing this conclusion, is that if the guide ring needs 2 G clamps to hold it still, thus withstanding any lateral forces imparted by the act of dolly cutting, then these forces are being withstood by the dolly itself when cutting after the dolly has been stuck down. This lateral or shear force must be quite substantial, and must have an adverse effect on the adhesive strength of the test dolly set up, thus impacting negatively on the results.

MANUAL VS. AUTOMATIC ADHESION TESTERS

The Type V self-aligning adhesion tester is available as a manual, hand-operated, or automatic device. There are two known manufacturers of this apparatus. The automatic versions are similar in operation using a hydraulic pump to generate the pull force; however, the manual versions differ. One is operated by winding a handle, in a similar fashion to a fishing reel, the other by pumping a handle, similar to a car jack. The tests reported in this paper were carried out using the winding method.

A total of twenty dollies were glued to a coated surface using two-pack epoxy glue in the optimum manner as outlined in Tables 11 and 12.

10 were left uncut and 10 were cut. Half of each group were tested using a manual Type V adhesion tester (Figure 3) and half using an automatic model (Figure 4).

Figure 3 – Hand-operated adhesion tester (winding method)

Dolly	Manual Type V Adhesion Tester				Automatic Type V Adhesion Tester			
	Uncut MPa	Attributes	Cut MPa	Attributes	Uncut MPa	Attributes	Cut MPa	Attributes
1	10.8	100% A/B	9.2	90% A/B	10.68	100% A/B	9.71	100% A/B
2	10.6	100% A/B	7.5	100% A/B	10.44	100% A/B	10.12	100% A/B
3	10.6	100% A/B	9.4	100% A/B	10.91	100% A/B	10.00	100% A/B
4	10.5	100% A/B	9.9	100% A/B	10.80	100% A/B	9.84	100% A/B
5	10.1	100% A/B	10.2	100% A/B	10.33	100% A/B	9.50	100% A/B

Table 11 – Comparison of two adhesion testers, where an A/B failure is at the primer/substrate interface

It can be seen in Table 11 there is very little difference between the values obtained with a winding manual gauge, where the load is applied smoothly and evenly (1 turn per second, approximately 1 MPa/s) and an automatic gauge. The tests produce an average value of 10.56 MPa for the manual gauge versus 10.64 MPa for the automatic gauge on uncut dollies (9.50 MPa versus 9.85 MPa on cut dollies).

The main point derived from these tests is the significant difference (approximately 10% of the higher adhesion value) between the results for uncut and cut dollies, whichever gauge is used. 10.56 MPa for the uncut coating and 9.50 MPa for a coating that has been cut using the manual gauge. 10.64 MPa for the uncut coating against 9.85 MPa for a coating that had been cut using the automatic gauge.

This test result supports the results given in Tables 8, 9 and 10 where the tests were carried using a manual gauge only.

Figure 4 – Automatic Adhesion Tester

CONCLUSION

It is clear from this series of tests that the results can be significantly affected by minor variations in one or more of the test steps. From the choice of adhesive, through the preparation of the coating surface and the test dolly, to the decision to cut through the coating or not, the test needs to be precise and consistent. This allows results for the same coating under different conditions or different coatings under the same conditions to be compared with confidence.

Different adhesives have different operating constraints. It must be noted that the bond strength of the cured adhesive must be greater than the bond strength of the coating, either to the substrate (adhesive failure), to the coating beneath (also adhesive failure) or within a single layer (cohesive failure).

As with coating processes, the preparation and cleaning of the surface of the coating and the face of the dolly is crucial to optimising the adhesion of the dolly to the surface and therefore increasing the probability of a coating adhesion failure rather than a glue failure. It should be noted that glue failures are invalid adhesion tests unless the specified adhesion strength is exceeded. Such tests must be repeated until the coating fails or the minimum specification value for the coating adhesion is exceeded. ASTM D4541 states that when 50% or more of the dolly face is covered by adhesive then that result shall be disregarded.

The design of the dolly is significant in the adhesion values obtained but further work is required to determine why this is the case.

Care and best practice should be employed at all times when carrying out adhesion tests and if there are any concerns then any or all of the coating, adhesive and test gauge manufacturers should be consulted for advice.