Uniform Force Distribution in Adhesion Testing

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One of the key characteristics of any coating material is its bonding ability or adhesion to other materials. Without sufficient adhesion, a coating will fail. It is vital, therefore, to be able to measure adhesive strength objectively and accurately during research and quality control programmes.

Technological advances for coatings have progressed much further than for adhesion measurement equipment. It is well known that variations in measuring the pull-off strength of coatings can occur with different instruments and operating procedures. (See *PCE*, November 1996, pp. 24-32). Adhesion test methods that produce widely fluctuating test results have prompted some researchers to abandon objective adhesion testing and rely on other coating characteristics to gauge performance.

This article describes one manufacturer's research and development of a new test instrument that it says eliminates the variables involved in adhesion testing to obtain accurate, reproducible pull-off strength measurements.

The Problem

The challenge is to isolate unsystematic (i.e., random or unexplained) variations in adhesion measurements caused by test equipment from those caused by variations in coating material. The latter, of course, is the only relevant factor in coating analysis.

Factors that typically affect adhesion test results are thickness of the test panel, dolly size, type of test equipment, skills of the test operator, consistency and rate of load increase, and geometry of the surface.

These factors all contribute to uncertain test results, which with conventional testing technology are high by the industry's standards. The resulting problem is twofold: 1) test result variations are far higher than can be attributed to variations in the tested material, and 2) test results are much lower than the true mechanical strength of the coating material, but with no indication of how much lower.

Therefore, in light of the problem of reproducibility of results from adhesion testing, DFD Instruments in Norway conducted an analysis of mechanical adhesion testing.

New Pulling Device Developed

The study started with the invention of a pulling device, which by its design achieves and retains perfectly uniform force distribution across the test specimen, regardless of surface geometry or alignment of the test dolly.

Although any system that can distribute the force evenly can be used, an hydraulic system was chosen for the following reasons. First, hydraulic fluid is free-flowing; the sensitivity of the pulling device is limited only by the movement of the individual fluid molecules within the instrument. Second, fluid does not compress like gas in a pneumatic system, which can produce an unpleasant blast of air from the sudden release of pressure during testing. Third, an hydraulic system can produce a very high force. Fourth, there is no need for a supply of compressed gas to operate the system.

Surrounding the test dolly are a number of pistons in hydraulic cylinders, which are connected to a common hydraulic fluid system. When the fluid pressure changes, the pressure on each piston also changes but remains identical for all of them. Since the pressure on each piston is identical, it results in an even spread of force around the dolly, ensuring a uniform force distribution on the coating system under the dolly.

Test Panel Thickness and Dolly Size

It was discovered that thicker test panels gave higher pull-off values than thinner panels if the procedure for conventional pull-off adhesion equipment in accordance with ISO 4624 (Paints and Varnishes–Pull-off Test for Adhesion) was used, and that smaller dollies generally gave higher values than bigger dollies on a given panel.

Interestingly, these observations have been reported in other studies but with little or no explanation or investigation of the relationship between panel thickness and dolly size. The explanation, however, is very simple. All materials yield to a degree when subjected to pull or compression stresses, even if only a few nanometres. When the substrate bends (as a result of counter force during testing), the pull stress around the circumference of the dolly exceeds the strength of the

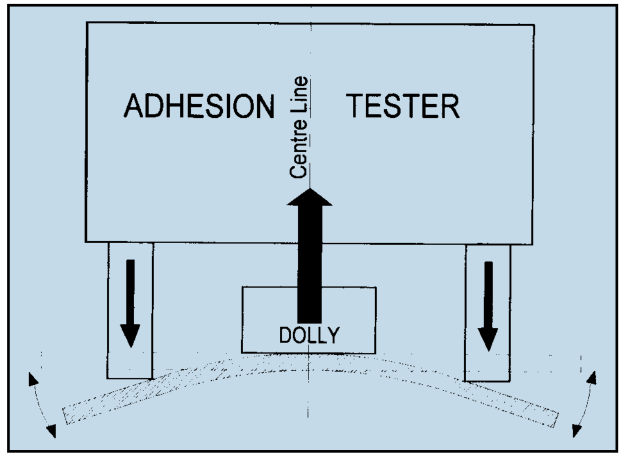

Fig. 1: Bend effect on test panel

coating. The coating toward the center of the dolly, meanwhile, is subjected to much lower pull stress, or it could even be compressed (Fig. 1). The result is a fracture at a recorded value far lower than the actual strength of the coating material.

The new instrument's sensitivity detects and reacts to variations on a nanometre scale. On this scale, detectable panel bending has been observed even on 12 mm panels at modest force.

Naturally, the smaller the dolly used, the less total force is required to pull it off and less bending takes place. Therefore, if a completely rigid test panel is used, dollies of different sizes should give the same results.

Reproducibility of Results

Having established the relationship between substrate flexing and dolly size, the reproducibility of the equipment could be tested. Due to the destructive nature of adhesion testing, it cannot be repeated on the same specimen. This adds uncertainty to the results because of variations in coating thickness, cure, etc., that must be minimised. Therefore, the following testing policies were adopted.

- Only coating materials known to be homogeneous were used. (For the purpose of this exercise, the material does not have to be a paint or coating. It could be glue, for instance.)
- The material had to produce a consistent fracture mode. Cohesive fracture (within the film) is, for this purpose, preferable to adhesive fracture (between the film and the substrate), since the latter may be caused by undesirable factors, such as surface contamination or inconsistent surface profile.

Table 1: Test Result Reproducibility

Test	Failure Stress in MPa	Failure Mode
1	91.48	100% cohesive
2	91.26	100% cohesive
3	92.06	100% cohesive
4	91.41	100% cohesive
5	91.04	100% cohesive

Average maximum strength: 91.45 MPa
Standard deviation: 0.38 MPa

• Any test with a deviating fracture mode (e.g., "glue failure") had to be eliminated from the sample, and no conclusions could be drawn from the result (even if the value appeared to be similar to the rest of the sample). Glue failure in this exercise represented a testing error.
• If several testing errors occurred, the whole exercise had to be repeated with a different coating and/or bonding agent.
• The substrate and/or dolly size had to be of such dimensions as to avoid any panel bending.

A single-pack (to avoid mixing variations and air bubbles) heat-curing adhesive was chosen as the material for testing. No additional bonding agent was required, thus eliminating potential glue failure. The test panel thickness was 12 mm. The dollies had a diameter of 8.2 mm. Five samples were tested. Other factors, such as curing time and temperature, were not important for this exercise, since they were identical for all the samples. The test results are shown in Table 1.

The results show very good agreement among the measured values on the five samples and that all samples failed cohesively. The standard deviation was low at 0.38 MPa, and the unexplained result variation (i.e., how far away the spread of results was from the mean value) also was very low at 0.42% (calculated as the standard deviation expressed as a percentage of the mean value). These low values demonstrate that using a uniform force distribution technique for the testing removed virtually all testing uncertainty and indicated that a high degree of reliance could be put on the failure stress measurement.

The results of pull-off adhesion tests with the new instrument on thermal-sprayed aluminium coatings are shown in Table 2. Again, very good reproducibility was obtained, particularly for the arc-sprayed coating where the unexplained result variation was only 0.9%.

Quantifying Disalignment

To quantify the disalignment (uneven pulling) in traditional test methods (i.e., ISO 4624), a special

Table 2: Pull-off Adhesion Measurements on Thermal-Sprayed Aluminium

	Arc-Sprayed Aluminium	Flame-Sprayed Aluminium
Sample Size	3	6
Fracture Mode	100% cohesive in the coating for all tests	100% interface to substrate for all tests
Average Value	27.64 MPa (4007 psi)	8.72 MPa (1260 psi)
Standard Deviation	0.25 MPa	0.55 MPa
Standard Deviation as a % of Average Value	0.9%	6.3%

Comment: Interfacial fracture to the substrate tends to produce a higher result variation than cohesive fracture, since it is caused by a combination of so many mechanical factors.

dolly was designed so the centre of the pull force could be moved away from the original centre, thus creating a measurable off-centre pull (Fig. 2).

The pull-off values in Table 3 and Figure 3 give the results obtained by gradually moving the force off-centre when the test was performed on an epoxy

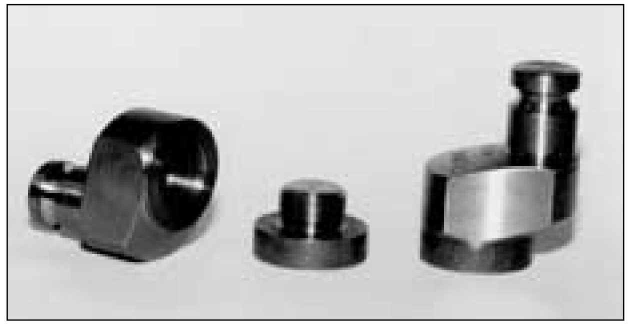

Fig. 2: Accessories used for measurable off-centre pull testing

vinyl and a thermal-sprayed aluminium coating. The results show the inconsistency and inaccuracy of the results obtained when the force on the dolly is not uniform.

DFD Instruments has found this to be borne out by repeated field tests involving other test equipment used in the normal manner compared with the reproducible results obtained from uniform force equipment.

Table 3: Results from Disalignment Test

	Epoxy/Vinyl	Thermal-Sprayed Aluminium
0% Displacement	15.15 MPa	18.72 MPa
25%	8.68 MPa	11.70 MPa
50%	7.35 MPa	10.79 MPa
75%	6.99 MPa	9.99 MPa
100%	6.60 MPa	8.87 MPa

Rate of Load Increase

The reproducibility and accuracy of the DFD test method also allowed objective testing of another assumption, namely that undue attention has been given to the speed of load increase, which is the speed with which the dolly is pulled off the panel during testing.

As expected, only a very small correlation was observed, with slightly increased pull-off values with increased speed, depending on the coatings tested. Since the measurable difference between the yield point and the break point of most coatings is very small anyway (using adhesion test methods as opposed to tensile strength/yield tests on free films), the issue of speed rate became almost solely academic compared with the variation caused by lack of force distribution. It was important, however, to check that it was not a factor in determining adhesion strength.

Is there an inconsistency in pull-off standards that dictate strict tolerances on the speed of load increase when, at the same time, they omit any tolerances on "perpendicularity" or, more correctly, force distribution?

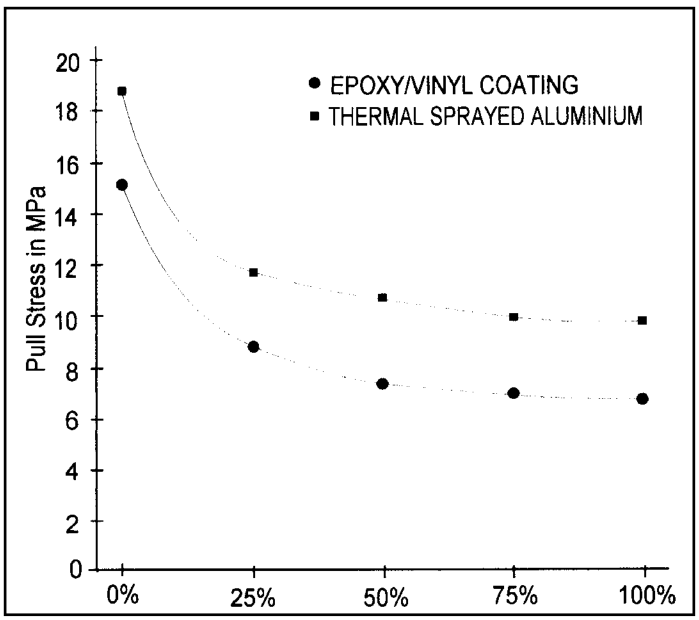

Fig. 3: Displacement of the centre of the pull force. 0% = in the centre 100% = at the edge of the dolly

Conclusions

Force distribution (or lack of it) is a significant factor in adhesion testing because it affects the accuracy and reproducibility of the results. The rate of load increase also is a factor in the test results, but for most paints and coatings its importance is not significant.

Traditional terminology like "perpendicular pull force" is meaningless in practical adhesion testing, because with conventional testing instruments, it cannot be guaranteed that force is evenly applied to the dolly so that it is pulled off in a perpendicular manner.

The fundamental objective of adhesion testing is to measure a material's ultimate strength expressed as pounds per square inch (psi), Newtons per square millimetre (N/mm^2), etc. It is critical, therefore, to avoid any stress concentrations in the specimen since these will cause premature rupture. To use perpendicularity as a measure of force distribution, therefore, is wrong for three reasons:

- It is not practical to quantify the tolerance within which perpendicularity would be allowed to deviate, hence any equipment purporting to pull vertically could be argued to be acceptable.
- Perpendicularity in the context of curved or spherical substrates is contradictory.
- Microscopic unevenness of the coating/adhesive thickness and local variations in surface profile cause the dolly to adhere at an angle (to the substrate) sufficient to spoil the test.

Because of a "domino effect," it only takes the rupture of a few molecules in the material to cause the whole specimen to fracture. Therefore, it is important that pull force is evenly distributed among all the molecules in the specimen. This can only be achieved with an instrument that senses variations and adjusts itself accordingly on the same nanometre level as the molecules.