Variability of Pipe Coating Pull-off Adhesion Measurements on Cylindrical Steel Pipelines

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Abstract

One of the most commonly utilized, and stringent, quality assurance tests for coatings or linings on steel pipe is the destructive pull-off adhesion test according to ASTM D4541. The specified value of adhesion is generally in accordance with AWWA C222 but for some projects adhesion values have been specified at very high levels which can be problematic from a testing or verification stand point. The rationale for high adhesion values (those greater than C222) appears to be a perception that very high adhesion values correlate to longer term corrosion protection. Usually, destructive pull-off tests are done by adhering metal "dollies" to the curved surface of the coated pipe using a fast reacting cyanoacrylate adhesive, and then mechanically pulling the dolly off in a radial direction. Tests are done not only on the very top of the pipe where it is possible to align the dollies well, but are also done around the sides of the pipe, where it is much more difficult to achieve ideal alignment of the dollies. Another limitation is that the adhesion of the glue to the coating may fail, giving a null result. Additionally, when the actual adhesion of coating is very high, the device for pulling the dollies from the pipe has a limit on the force that it can exert, and in necessity will terminate the test without giving a datum.

The paper will discuss testing that was completed to examine the consistency of adhesion testing to ASTM D4541 on steel pipe coating with polyurethane applied in accordance with AWWA C222. Pipe diameter, flat surfaces, glue type (cyanoacrylate and epoxy), dolly diameter, polyurethane formulations scoring around dollies, and type of equipment were all considered in the testing. Possibilities for improving testing reliability and accuracy are discussed.

Introduction

Steel pipe used to transport water is often buried underground where it cannot be easily checked. One of the principal threats, in the long term, is corrosion of the pipe, Polyurethane coatings are applied to the exterior of these pipes in order to provide long term protection from soil corrosion.. Simple adhesion testing, of a coating to the pipe, is commonly done during application in order to determine whether the coating and its application are competent and will provide the necessary protection.

The adhesion test usually comprises a few tensile butt ('pull-off') tests done to a sampling of a pipe lot with dollies of 20 mm diameter, glued to the pipe with quick-drying cyanoacrylate adhesive. Occasionally project specifications require very high adhesion values, on the assumption that very high values correlate to longer term corrosion protection. Tests are done, not only on the very top of the pipe, where the geometry of the test can be reasonably maintained, but are also done around the sides of the pipe in order to sample several locations on each section of pipe. The objective of this testing is to qualify each section of pipe in a lot for field use, and the decision is based on the results obtained from a few adhesion tests, on the assumption that they correlate with the longevity of the pipe, in service.

The work reported here examined the effect of several experimental variables in determining the pull-off adhesion values. Necessarily, adhesion results must be reliable so that they may be used to correlate with corrosion prevention in service. Any experimental variable that diminishes adhesion test values obtained, or introduces an extraneous cause of failure, devalues the data. The variables in this study included the choice of adhesive used between pull-off dolly and coating, the diameter of the dolly, the scoring procedure and the diameter of the pipe. Two polyurethane coatings were used in the study to ensure that trends were not unique to a particular coating. Overall, the testing reported here included more than 1000 adhesion pulls.

Background

Strength of adhesion is inevitably important in a coating's ability to resist the service environment and fulfill its protective function. It seems obvious that adhesion must play a role, especially in corrosion protection, but the correlation is unclear [Dickie 1986, Marsh 2001]. There is no study that shows how much adhesion is necessary before a coating successfully resists corrosion. If a polymer degrades in service, then its adhesion must diminish and eventually fail [Devashayam 2006, Jorgensen 2006, and Leidheiser 1986]. Often the ingress of water across the interface between coating and substrate is the culprit for diminished adhesion. Unfortunately, there are few investigations with quantitative data showing clearly how coating adhesion deteriorates, principally because adhesion is very difficult to measure unequivocally [Chen 2011, Gerberich 2006].

All measurements of adhesive, or cohesive, failure are prone to uncertainties in techniques, mixed modes of failure and large sensitivity to imperfections in the materials. There is a substantial literature of the mechanics of material failure in various geometries [Chung 2005, Hutchinson 1992, and Thouless 1991] that, for their predictions, require the interfacial adhesion and the cohesive strength of the materials being well characterized, so that the path of the failure can be predicted. Unfortunately, adhering coatings are formed at the interface, from liquids or melts that undergo complicated processes, so that is impossible to measure their surface properties without interfering with those processes. Nevertheless, quantifying adhesion is a necessary part of connecting composition to performance.

Coating adhesion must be determined after the coating has formed. Steel water pipeline coatings must be tough and sufficiently thick to be a barrier to water and to resist handling and damage during placement, so some methods of coating adhesion measurement cannot be used e.g. peel testing, cross hatch testing. Coating adhesion to these steel pipes is measured by pulling off, in a direction radial to the pipe, a metal dolly that has been glued to the coating (ASTM D 4541). This is also known as a tensile butt test. There are several important experimental variables that must be controlled before a reliable value of adhesion is obtained; these are the subject of this investigation.

Experimental Procedures

The adhesion of the glue between dolly and coating must be strong, so that the failure is between coating and substrate and it must be rigid so that deformation occurs only in the coating. The pulling direction must be perfectly perpendicular to the plane of the coating and the substrate so the stress is perfectly even across the area of the dolly-coating joint; otherwise a peeling failure will be initiated in the joint which will lead to artificially low values of stress. Typically, the glue and the coating are scored through to the metal substrate, around the circumference of the dolly. This defines the area of the coating and glue being stressed. Provided that the glue has not affected the chemistry of the coating, and that there is a perfect joint between them, without bubbles or dirt, and that the scoring process has not damaged the glue nor the coating, the dolly can be pulled to give the failure stress of the coating adhering to the steel pipe.

The adhesion testing was completed according to ASTM D4541 on steel pipe with polyurethane coatings, applied in accordance with AWWA C222. The dollies were pulled with a Defelsko PosiTest™ AT-A (automatic). New dollies were purchased, from the manufacturer of the adhesion equipment for this investigation and their surface roughened with 80 grit emery paper. Two sizes of dollies were used: 20 mm and 14 mm diameter. Pipeline coatings are conventionally tested with the 20 mm size, but the adhesion pulling equipment has an upper limit to the load that it can exert on the dolly and so 'terminates' the test before the adhesive joint fails. The smaller dolly was included because it reaches a larger value of pull-off stress.

Two 2-component polyurethane coatings, A and B, were applied to pipe sections manufactured in the Portland, Oregon, plant of Northwest Pipe Company using the factory's plural component spray equipment, according to the coating manufacturers mixing and application instructions. They were applied to whole, cylindrical pipe sections and flat plate that had been abrasively cleaned to SSPC SP5 white metal blast. AWWA C222 requires a minimum surface cleanliness of SSPC SP 10 near white blast. The surface preparation, blasting profile and coating application were checked and values recorded by quality control technicians from Northwest Pipe. The measured surface profile exceeded the AWWA C222 2.5 mil (64µm) requirement. Pipe diameters of 20 (0.51 m), 36 (0.914 m), 48 (1.22 m) and 84 inches (2.13 m) were coated. Flat panels of steel pipe stock had been welded to a section of the 84 inch pipe so that they were cleaned and coated by the same process as the cylindrical pipe, see figure 1.


(a)


(b)

Figure 1. Pipe with smaller diameter section to accommodate flat panels for the cleaning and coating processes (a) prior to blasting, (b) after blasting.

Prior to blast cleaning and coating, sections of each pipe was partially slit into convenient sections for subsequent adhesion measurements so after coating; the sections could be freed using a plasma torch with minimum damage. After blasting, while still part of the pipe, the slits were covered with masking tape prior to coating. The sections were stored at room temperature. Aligning and gluing dollies was much more reliable under laboratory conditions on sections of pipe (figures 2), than it would be on complete, cylindrical pipe, in factory settings.

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Figure 2(a). Internal view of precut pipe (1.22 m) after coating, but before final separation of the sections.

Figure 2(b). Separated, coated sections of pipe with dollies prior to adhesion testing.

In this investigation, a parallel study was done using a 2 component epoxy adhesive and a more conventional, cyanoacrylate adhesive. The two adhesives were 3M Scotch-Weld™ epoxy adhesive DP-460NS and Scotch-Weld™ Instant Adhesive CA100. After setting in place, the epoxy adhesive was warmed with hot air and allowed to cure for at least 24 hours before the dolly was pulled. The cyanoacrylate adhesive was not warmed, but the adhesion testing was usually delayed 24 hours, for convenience, to be done concurrently with those using the epoxy.

Before adhesion was measured, scoring through the coating to the metal surface was done around the dollies using the tool supplied by the manufacturer of the equipment and dollies.

Although it is not practical to perform many adhesion tests in the shop, because of time constraints, and the need to minimize repairs to the coating, each dataset here, i.e. combination of the variables, used 30 dolly adhesion tests in order to fulfill the common statistical criterion of having a large dataset for analysis. Imperfections in the adhesive, coating, substrate preparation or equipment will lead to results that are unusually low. Outliers were eliminated using the ‘Thompson tau’ technique [Thompson 1935].

Results and Discussion

In the shop, or field, dollies are adhered to coatings on pipes using quick drying (usually cyanoacrylate) adhesives because the adhesion test can be performed very quickly thereafter and in variable weather and temperature conditions. In other, laboratory testing epoxy adhesives may be used because they are very strong and adhere to a variety of surfaces. However, epoxy glue requires mixing and a significant curing time or heating. On curved surfaces, dollies glued with the epoxy were taped in place so that they did not move while the epoxy was still fluid. The cyanoacrylate cures quickly enough that it needed to be held in place only briefly.

The investigation used 30 samples in each set and for the most part, results were found to be from a normal distribution, or close enough that it was impossible to reliably decide an alternative form of distribution. Distributions of results were analyzed after eliminating the outliers after inspection using Thompson's tau analysis. Most of the outliers were on the low end of the data where it is easy to imagine reasons how the test was adversely affected by some imperfection. The very few cases where the outliers were on the high side (but not terminations) deserve further thought in a more basic investigation, as they may be the closest to actual adhesion levels of the coating, but their treatment not be discussed further here.

The initial comparison of the glues was done on flat panels and on high curvature, 0.51 m (20 inch) diameter pipe sections. Each set of circumstances was examined with 30 adhesion tests. The right-most table column gives comments and records how many useful data points were in each set of 30. For example, if there were 28 useful points, it means that 2 were outliers. Table 1(a), coating A, and (b), for coating B, show the results.

Table 1(a) Coating A: effect on adhesion failure stress (average and standard deviation) of dolly size and glue type for flat panels and small diameter pipe.

Substrate Diameter, m	*Dolly Diameter, mm*	*Glue Type*	*Average stress, MPa*	*Standard Deviation, MPa*	*Comment*
0.51	14	Cyanoacrylate	13.1	3.59	30 useful data
0.51	20	Cyanoacrylate	16.9	1.25	29 useful data
0.51	14	Epoxy	18.1	1.84	27 useful data
0.51	20	Epoxy	18.9	1.37	26 useful data
Flat	14	Cyanoacrylate	22.6	2.45	28 useful data
Flat	20	Cyanoacrylate	18.0	4.92	4 useful data, all others 'terminated'
Flat	14	Epoxy	27.5	0.72	25 useful data
Flat	20	Epoxy	-	-	All 'terminated'

N.B. 1000 pound/square inch = 6.895 MPa

Table 1(b) Coating B: effect on adhesion failure stress (average and standard deviation) of dolly size and glue type for flat panels and small diameter pipe.

Substrate Diameter, m	*Dolly Diameter, mm*	*Glue Type*	*Average stress, MPa*	*Standard Deviation, MPa*	*Comment*
0.51	14	Cyanoacrylate	-	-	All failed glue-coating
0.51	20	Cyanoacrylate	-	-	All failed glue-coating
0.51	14	Epoxy	21.3	2.26	28 useful data
0.51	20	Epoxy	-	-	All 'terminated'
Flat	14	Cyanoacrylate	-	-	All failed glue-coating
Flat	20	Cyanoacrylate	-	-	All 'terminated'
Flat	14	Epoxy	-	-	All failed glue-coating
Flat	20	Epoxy	-	-	All 'terminated'

N.B. 1000 pound/square inch = 6.895 MPa

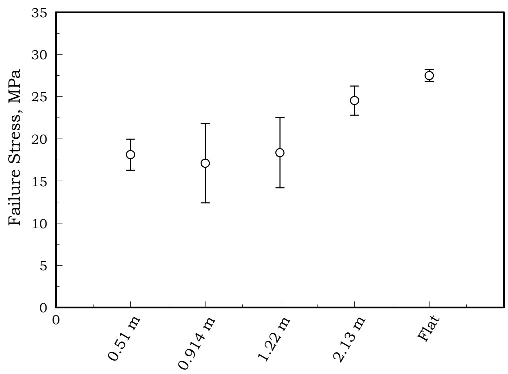
Several deductions can be made. Clearly, both coatings adhere very well. In many cases, with the wider, 20 mm dollies, the adhesion testing equipment could not exert enough force to cause a failure and the test ‘terminated’. All one can say in this case is that the adhesion must be greater than 20.7 MPa (3000 psi), which was the upper limit of the device. More useful results are gained with the 14 mm dollies because the upper limit of the device corresponds to 41.4 MPa (6000 psi). Nevertheless, much of this study was done with both 20 mm and 14 mm dollies, because the 20 mm are more frequently used. .

Coating B adheres very well to the steel pipe, but it has many failures between the glue and the coating and thus the test does not give a result. Coating B, in this series, adhered more strongly to the steel than the adhesives could adhere to this coating. The only data from this series was on the curved, 0.51 m (20 inch) pipe, where results were obtained using the 14 inch dollies and the epoxy adhesive.

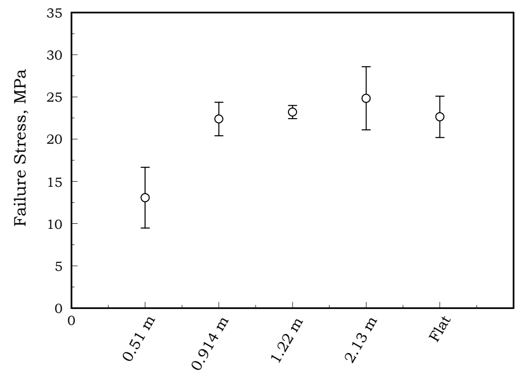
Fortunately, coating A gave more results. In all cases the value recorded for the failure stress was higher on the flat surface, than it was on the curved 20 inch pipe. The F-test and the T-test showed that these data (curved vs. flat substrates) were not from the same distribution (with > 95% confidence). While an adhesion test on a 20 inch diameter pipe gives a result, it cannot be claimed to be a representative of the adhesion of a coating to a flat surface. It is no surprise that the curvature substantially reduces the quality of the measurement. The glue thickness varies due to the gap width changing between the curved pipe and the flat bottom of the dolly. There are also opportunities for bubbles, for the dollies to slide, glue retraction and other problems in placing the dolly so that the adhesive joint is not optimally perpendicular to the coating (pipe) surface and symmetrical with respect to the dolly adhesive. Scoring of the dolly can also impact and apply torsion to the dolly or cause cracking of the glue.

The use of epoxy adhesive enabled a higher value of adhesion to be achieved, with a tendency here, and in other tests (below), to be a more reliable adhesive in the sense that it produces more useful data points that are not outliers or glue-coating failures. Epoxy adhesives are unfortunately more temperature dependent and normally take at least 24 hours to cure prior to testing.

Adhesive pull-off tests are done in the field on pipes of varying diameter. Here, datasets were gathered on sections cut from the various pipe diameters, as described above. Coating B, as before, produced too many glue-to-coating failures and test terminations for analysis. However, coating A, using the smaller diameter dolly, gave useful samples data, for both glues. As one would expect, the trend (figures 3) is clear for both glues, that adhesion measured on the larger diameter pipes is more characteristic of the adhesion on the flat panels. Measuring adhesion on flat panels reduces the possibilities for misalignment and incomplete glue lines and so is the best representative of the capability of the substrate, the coating and the glue. In addition, these tests were done by gluing the dollies onto sections of the pipes that had previously been cut out, so the dollies were be applied under optimum conditions, which would not be true in the field when the test has to be done at a place on the pipe that is hard to access and where it is difficult to ensure the alignment of the dolly and the integrity of the glue line. Comparing pull-off adhesion values obtained on 0.51 m (20 inch) diameter pipe to a value of adhesion required in a specification cannot be reliable.



(a)



(b)

Figure 3. Average failure stress in adhesion testing using 14mm dollies for coating A on pipes of various diameter, using (a) epoxy adhesive, (b) cyanoacrylate adhesive. The bars represent one standard deviation.

Comparative data using 20 mm dollies was not useful due to the number of terminations and other outliers. Even though there were 30 attempts using 20 mm dollies on each of the pipe diameters, it very telling that the number of glue failures or test terminations were so high that insufficient data is available to obtain a valid trend line.

The value of scoring around the adhesion dolly before pulling off was investigated. Scoring is done to clean excess glue from the circumference of the dolly and to scribe the coating, in order to define the area over which the dolly loads the coating during the test. The smaller dolly, 14 mm diameter, was used on coating system A, using both the epoxy and the cyanoacrylate glues. The scoring was done down to the metal, all around the dolly on both the 0.51 m diameter pipe and on flat panels. These dollies and pulls-off were done specifically for this part of the study, so the laboratory conditions and techniques were consistent for the 8 samples sets here. The results are given in Table 2.

Table 2. The effect of scoring on adhesive failure stress for both epoxy and cyanoacrylate glues on flat panels and small diameter pipe sections (coating A).

Substrate Diameter, m	*Glue Type*	*Average stress, MPa*	*Standard Deviation, MPa*	
Flat	Epoxy	26.1	0.96	Scored
Flat	Epoxy	27.3	0.94	Un-scored
0.51	Epoxy	16.1	3.3	Scored
0.51	Epoxy	17.1	3.1	Un-scored
Flat	Cyanoacrylate	25.1	0.81	Scored
Flat	Cyanoacrylate	25.4	1.22	Un-scored
0.51	Cyanoacrylate	14.1	3.6	Scored
0.51	Cyanoacrylate	13.9	3.0	Un-scored

N.B. 1000 pound/square inch = 6.895 MPa

The table is arranged so circumstances that are the same, except for scoring, are together. There is very little difference here in the average values or the standard deviations between scored and un-scored tests. Considerable care was taken when scribing on the curved surface that the scribing tool did not disturb the dolly that was adhering, even when scribing through the coating below the crest of the curve. This may not be possible in shop or field testing.

The values in Table 2 are consistent with the data in figures 3, although they were obtained separately. Again, values measured on flat panels are higher, and the epoxy glue gives somewhat higher values overall.

If the assumption is that high values of failure stress measured on pipes correlate with long term protection against corrosion, it is instructive to compare the values of adhesion obtained here with other studies. It is impossible to correlate adhesion with conventional surface tension studies via the Young-Dupré equation because surface tension of adhering materials can only be measured on drops in air, not against the substrate. It is clear that the arrangement of polymer molecules at the interface with a very polar metal/oxide/hydroxide will be very different to an interface with air. The actual interfacial energy of adhesion between the coating and the substrate must be larger. Coating adhesion must be measured; it cannot be estimated from surface tension data. Few methods can be used to isolate the adhesive interface and measure its strength. Values obtained for the interfacial work of adhesion, often using a peeling technique, are typically 2 - 20 J/m^2 [Croll 1980, Packham 1983, Tran 2011].

In contrast, strongly adhering polyurethane pipeline coatings might give a failure stress (tensile butt test) of 27 MPa (4000 psi) in good circumstances, and be 760 µm (30 mil) thick with a modulus of 1 GPa. The resultant, apparent, work of adhesion using conventional analysis [Chung 2005, Kendall 1971] would be 140 J/m^2, which is several times larger than values expected from more specialized techniques. It is well recognized that a large part of the energy or force expended in the tensile butt (pull-off) test is used in deforming the coating, and does not test the adhesive interface [Gerberich 2006, Gent 1971]. There is no rigorous theoretical framework for analyzing this apparently simple test [Chung 2005]. Further, the high loading rates characteristic of the equipment used to measure adhesion here, will amplify the stiffness and failure stress measured because the experiment is very fast compared to normal viscoelastic processes in polymers. This also means that testing equipment that uses manual application of load will give results that are sensitive to the nature, practices and fatigue of the operator, and will give lower readings than the much more rapid, and consistent, automatic equipment.

The pull – off test does not measure adhesion; it measures an overall failure of the coating system wherein adhesion plays only a very minor role. Since the standard deviation in a conventional, tensile butt joint measurement may be considerable, depending on the circumstances, there is no chance of identifying the small adhesion component within the overall experimental result, and this method would not be valid to quantify adhesion for correlation to corrosion protection.

There are other cautions in estimating likely performance over extended periods. In extended field service, stresses will arise much more slowly than the pull-off testing rate but failure will also occur at much lower stresses due to the influence of the long term viscoelastic response of the polymer to extended periods of stress. In addition, failure inevitably results from the accumulated damage from many occurrences of sub-critical stress, i.e. a fatigue failure. Adhesive fatigue failure, as such, is not discussed at all in coatings technology, but is explored [Ashcroft 2010, Gerberich 2006] in other industries, e.g. fiber reinforced composites and dental repair.

Taking all these considerations into account, failure stress from the tensile butt test cannot correlate to the long term corrosion protection offered by a coating. There is no doubt that coatings must adhere well, in order to resist the advance of water and other aggressive species across the surface of the metal pipe, but there is no indication that very high values of tensile butt failure stress provide improved corrosion protection. However, the coated pipe must resist corrosion after it has been placed and joined. After manufacture, coatings on pipes must survive impacts and accidents during storage, transportation and placement. Since the adhesion pull-off test gives a result that combines the cohesive toughness properties as well as the adhesive properties, then it is suitable test for quality control in evaluating whether a coating will survive its treatment and thus perform its ultimate task.

Summary

Pull-off adhesion testing is intrinsically very sensitive to minor variations in experimental procedures and circumstances. Overall, the testing reported here included over a thousand individual adhesion measurements. Standard deviations found here were as high as 20% of the value of the mean.

A pull-off test result is dominated by the force necessary to deform the coating, in transferring the load to the interface. Polyurethane coatings are deliberately designed to be tough. Hence interfacial adhesion contributes a small fraction of the test result. Methods that focus better on adhesion are done with different joint geometries, e.g. peeling, and at very slow testing speeds in order to reduce, as much as possible, the contribution from the deformation within the coating. Clearly, such testing methods are not practical for checking, in the shop or field, whether a thick, tough polyurethane coating has been applied well. However, a pull-off 'adhesion' test can be done very rapidly and, if done well, assesses the quality of the coating film as well as whether there is competent adhesion.

At the simplest level, it is evident from the variations encountered here, and the standard deviations found, that a handful of tests done on a length of pipe will not provide a characteristic value for adhesion failure, and certainly not one that could be used to estimate the long term corrosion protection, even if there were such connection. However, the use of pull-off tests according to ASTM D4541, used as quality control, is appropriate as long as it is recognized that large standard deviations will occur. Surface preparation such as grit blasting and ensuring a suitable angular surface profile and surface cleanliness are essential to providing much more accurate and repeatable results.

Due to the rate-dependent, viscoelastic nature of polymers the rapid loading rate of an automatic device will give a larger value for the test result than a manually applied load. This sensitivity to loading rate means that manually operated testers give results that are sensitive to the control and practices of each operator. The use of automatic testers removes the "human" variable in providing a constant force. However, their rate of loading should be standardized to ensure that the different automatic devices available perform as closely as possible.

There is no doubt that a pull-off test performed on a small diameter pipe will give a value of failure stress that is much less than the characteristic value of the coating measured on a flat surface. Such a value should not be compared to a value required in a specification that does not make an allowance for that situation. If there is a need for more accurate measurements of adhesion, it may be possible to measure performance on flat panels of the same steel incorporated into the cleaning, blasting and coating processes. This would provide the most accuracy available using the ASTM D4541 test.

The epoxy dolly glue produced higher values of coating failure stress. Unfortunately, it requires care in mixing and more time for curing. Thus the cyanoacrylate type is more practical for rapid shop or field testing, but one should be aware that its use provides an underestimate of performance. Using a dolly of 14 mm diameter was much more useful than one of 20 mm. It enabled coatings to be better evaluated without so many "terminations" by standard portable equipment. In addition, the glue line thickness between a small diameter dolly and the coating will have less variation on a curved surface than it would under a larger diameter dolly. In cases where the 14 mm dolly pull-off values are higher than the termination value for the 20 mm dolly, we know that the 20 mm dolly cannot possibly give us a true representation of the distribution of results, because any high values are not accessible and the resultant average value would not be meaningful.

There was no difference between scoring around the dolly and not-scoring on either flat panels or small diameter pipes, regardless of the glue used. Further investigation is needed, but one could argue that scoring is necessary to define the area of the coating that is subjected to the tensile butt test, but that it is probably not necessary to score below the crest of the pipe and risk upsetting the alignment of the dolly and damaging its adhesion.

There is no evidence that ever higher adhesion pull-off values correlate to longer service life for a coated pipe. Further, employing pull-off adhesion as the primary factor in gauging coating service life is not justified due to the intrinsic variability in the method. The adhesion test has low accuracy and repeatability even on flat, and especially on curved, surfaces.

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