ADHESION TESTING OF EPOXY COATING

by

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ABSTRACT

The hot water and knife adhesion tests developed in this study proved to be a valuable tool for quality control and for in-depth studies of coating adhesion. Hot water and knife adhesion tests were very useful in discriminating and identifying good from bad quality coatings. The tests were relatively easy to perform and did not require special or sophisticated equipment. Most of the subjectivity involved in other adhesion tests was eliminated or reduced through the use of a calibrated knife. Nevertheless, it was shown that the subjectivity of the tests had little or no effect in the detection of coatings with poor adhesion. Test parameters such as knife force calibration procedures, adhesion test method, test operator, type of knife and blade, and test evaluator had little effect on the test results. Sample source was the most influential factor in determining adhesion strength. The quality of coating application by different coaters can vary greatly and affects adhesion of the coating. An interesting finding was the good agreement observed between results from hot water-adhesion tests and those from the TxDOT peel test. Considering that the TxDOT peel test is simple and quick to perform, the test is very useful for adhesion evaluation, especially if a calibrated knife is not available. Another important finding was the poor correlation observed between knife adhesion tests and bend tests. Bend tests were not reliable indicators of coating adhesion and were more a measure of the coating flexibility. Therefore, the use of bend tests as the only method of testing epoxy coating adhesion (as proposed in some ECR standards) is discouraged.

PREFACE

This report is one of a series of reports on a project to evaluate the integrity and performance of epoxy-coated reinforcing bars used in transportation structures in the state of Texas. The report describes an investigation of tests to evaluate the adhesion strength of epoxy coating. Strong adhesion is considered an important property of the epoxy coating for satisfactory corrosion protection of steel reinforcement. However, reliable and practical tests to evaluate coating adhesion are not available. TxDOT specifies the Bend Test and the Peel Test (Tex-739-I) to evaluate coating adhesion. The Bend Test is not appropriate for adhesion evaluation and the Peel Test is very subjective. The objective of this study was to develop a simple, quick, and reliable test method that could be performed at the coating plant or elsewhere during the construction process.

SUMMARY

The importance of coating quality and adhesion was discussed. Quality control measures, industry efforts to improve quality (CRSI Certification Program), and industry standards and specifications were reviewed and discussed. The nature and factors affecting coating adhesion, mechanisms of adhesion loss, available tests to evaluate coating adhesion, and prior research on coating adhesion evaluation were analyzed. An experimental evaluation of hot water immersion and knife adhesion testing was conducted at three different stages to determine the feasibility of these tests for coating adhesion evaluation. The objective was to develop a reliable and practical adhesion test that could be performed quickly, repetitively, and economically at the coating plant and from which test results could be objectively interpreted. ECR samples from different coating applicators, with varying bar diameters, and both straight and bent samples were tested. Other test variables included the temperature of the hot water bath, time of immersion, elapsed time between hot water immersion and adhesion test, different adhesion test operators, and different adhesion test procedures. Test results were discussed and analyzed. Different adhesion rating systems were devised and evaluated.

IMPLEMENTATION

A test procedure to evaluate the adhesive strength of the epoxy coating was developed and is recommended for quality control. The test is simple, quick, and reliable, and can be performed at the coating plant or elsewhere during the construction process. The recommended test procedure is described in Appendix A of this report. The approach for the evaluation of coating adhesion included in this report should serve as an aid to engineers involved in the specification, quality control, and inspection of epoxy-coated reinforcement for concrete bridge and other transportation structures.

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CHAPTER 1

Introduction

1.1 GENERAL

The quality of epoxy coating has been shown to be a key factor affecting the corrosion performance of fusion-bonded epoxy-coated rebars in chloride-contaminated concrete. One measure of quality is adhesion of the coating to the steel substrate. Some have argued that the epoxy film relies on adhesion to the steel substrate to protect the steel surface against corrosion. A well adhered coating acts as an effective physical barrier that slows the arrival of corroding substances to the coating/steel interface. However, the role played by coating adhesion in the corrosion protection of steel reinforcement is not very well understood. It has been claimed that inadequate coating adhesion, along with the presence of discontinuities in the coating, may lead to film undercutting and early breakdown of the coating protection system.[1, 2, 3, 4] Poor adhesion may also reveal a poor coating application process. Yet adhesion of epoxy-coatings is not satisfactorily addressed in current specifications on ECR. One of the main problems has been the lack of an adequate test to measure adhesion. Quality of coating adhesion is determined by bending tests according to most specifications. However, bending tests are more indicative of the coating flexibility than of the coating adhesion. Specimens that passed the bend test have experienced adhesion loss and undercutting at bent regions in past studies.[5, 6]

In the early 1990's, a hot water immersion test was developed and used in several European countries for evaluation of coating quality.[2, 7, 8] In these tests, an attempt was made to address quality by evaluating the amount of coating damage after the test. Corrosive action of hot water accelerates formation of rust spots at coating imperfections and defects. The earlier tests were not intended to evaluate epoxy coating adhesion. More recently, the Ontario Ministry of Transportation (MTO) suggested a knife adhesion test of epoxy coating after immersion in hot water.[3, 9] Epoxy coatings tend to lose adhesion in moist environments and hot water accelerates this phenomenon. Because of high variability of test results, the test was not incorporated in standard MTO specifications for quality assurance. Coaters in Ontario use the knift test for quality control at their plants. Texas DOT specifications include a “peel test” for estimating coating adhesion.[10] This test is used for epoxy-coated elements that are too small to perform a bend test. Such elements include rebar couplers, plates, mechanical splices, etc. The test is performed by peeling the coating with a utility knife. The test has the disadvantage of being highly subjective and without sufficient background to support quantitative interpretation of test results.

1.2 RESEARCH OBJECTIVES

The main objectives of this research are the following:

- To develop a hot water test that can be performed quickly and economically.
- To develop a reliable adhesion test that can be performed repetitively at the coating plant and from which test results can be objectively interpreted.
- To determine the feasibility of incorporating hot water and adhesion tests in standard specifications for quality control of epoxy coated rebars.
- To understand the relationship between coating adhesion and corrosion protection.

The feasibility of hot water immersion and adhesion tests as a means for quality control of ECR was investigated by testing bar samples from different coaters, with varying bar diameters, and both straight and bent samples. Other variables that were evaluated include the temperature of the hot water bath, time of immersion, elapsed time between hot water immersion and adhesion test, different adhesion test operators, and different adhesion test procedures. Test results are discussed and analyzed. Different adhesion rating systems were devised and evaluated. The intent was to produce a test that could be easily and practically implemented without special or sophisticated equipment. With further research and refinement, developed tests may be incorporated in ECR specifications as an aid for quality assessment.

1.3 LITERATURE REVIEW ON COATING ADHESION

1.3.1 Nature of Epoxy Coating Adhesion to Steel [11, 12]

The Condensed Chemical Dictionary defines adhesion as the "phenomenon of the sticking of two surfaces together due to molecular attraction for each other." The American College Dictionary states the definition as "the molecular force exerted across the surface of contact between unlike liquids and solids which resists their separation." In both definitions, a molecular force or interaction is the fundamental feature of adhesion. Adhesion of epoxy compounds to metals is provided mainly by a) chemical or adsorption adhesion, and b) mechanical interlocking. Each of these components of coating adhesion is described below:

Chemical or Adsorption Adhesion

High polarity exists in the epoxy resin chain and the cured epoxy polymer due to the presence of aliphatic hydroxyl and ether groups. The presence of metal oxides in the treated steel surface causes a very strong electromagnetic attraction between both materials. The strength of coating adhesion to steel is directly proportional to the hydroxyl group content of the epoxy compound. The formation of chemical bonds between active hydrogen in the steel surface and epoxide groups in the coating contributes to coating adhesion.

Mechanical Interlocking

A roughened surface, pretreatment of the steel surface, or the presence of porous oxides on the surface allow prepolymeric epoxy resin and curing agents to penetrate into the crevices and pores provided by the pretreatment. Upon polymerization, the coating becomes mechanically embedded in the metal surface or the surface oxide structure. The cavities and pores formed during surface preparation provide a larger surface area for electrochemical reactions, further increasing the adhesive strength of the coating.

1.3.2 Factors Affecting Coating Adhesion [12]

Epoxy Coating Formulation

The formulation of the epoxy coating affects the chemistry of polymer chain formation and molecular weight. The ultimate form of the polymer chain, its length, shape, and configuration determines the properties and physical characteristics of the coating, such as flexibility, hardness, and adhesion. The viscosity of the epoxy during the transition from the wet to the cured state is particularly important. Adhesion develops as the coating is "wet-in" or absorbed into the substrate in a mechanism where the molecules of both materials are brought together in intimate contact. As the coating cures, its viscosity changes and increases, and its mobility or flow decreases. If the epoxy is not properly formulated, flow of the coating into the substrate microstructure may be hampered, adversely affecting adhesion and producing a number of voids and holidays in the film.[11]

Coating Process

Deficiencies in the following stages during the coating process may result in inadequate adhesion of epoxy coating to steel:[12]

a) Failure to provide an adequate surface profile (optimum number and depth of peaks and valleys) lessens the mechanical interlocking between the epoxy resin and the steel surface. Improper cleaning of the steel surface will result in mill scale and other surface contaminants (rust, loosely adhering deposits, oil, grease, chlorides, and other foreign matter) that can impair coating adhesion.

b) Improper heating of the bar causes incomplete reaction or degradation of the epoxy. On the one hand, if curing is incomplete as a result of underheating, the epoxy will not flow properly over the bar surface and fill in the cavities as needed for good mechanical interlock. On the other hand, overheating the steel degrades the epoxy and reduces the electrochemical bond by producing new oxide layers in the steel surface that will not react with hydroxyl groups in the epoxy.

c) Fast quenching of the coating can reduce the gel state time of the epoxy. A shorter gel time results in reduced time for the epoxy to flow and produce adequate mechanical bond. In addition,

fast cooling may produce high internal stresses at the interface due to differences in coefficients of thermal expansion between the epoxy and the steel.

Moisture

The presence of moisture around the epoxy can be detrimental because water is one of the most destructive agents of metal/polymer adhesion. In the field, moisture around the reinforcement may come from water penetrating the concrete. Sources of water are rain, deicing, sea water, and ambient humidity. Moisture is also available as part of the pore solution in concrete. Depending on the length of exposure, adhesion failure tends to change from the resin to the epoxy/steel interfacial region. If exposure time is sufficiently prolonged, the presence of water in the interfacial region is believed to produce large reductions in adhesion strength. Water may cause loss of adhesion by breaking the hydrogen bonds at the epoxy/steel interface or by hydrating the metal oxide layer.[2] The mechanism of wet adhesion loss will be discussed further in a subsequent section.

Temperature

If a polymerized epoxy is exposed to temperatures close to the transition temperature, the coating will become soft and fluid, and will be susceptible to deterioration. If moisture is present, there can be permanent loss of adhesion. The presence of high temperature alone causes momentary loss of adhesion, but immediately after the epoxy cools, adhesion can be regained. Therefore, high temperatures will generally produce loss of adhesion when moisture is present. Conversely, moisture alone produces loss of adhesion over time, but high temperatures help to accelerate the disbondment process. This principle is the basis for the hot water tests.

Coating Damage

Discontinuities in the coating are an indirect contributing factor to adhesion loss. Deleterious agents, such as water, chlorides, or diluted chemical substances can enter the steel/epoxy interface through coating discontinuities as small as pinholes and produce loss of bond. Corrosion cells forming at sites of coating damage can produce adhesion loss by cathodic disbondment and/or corrosion progression under the film.

1.3.3 Adhesion Loss Mechanisms and Relevance

Most corrosion failure mechanisms of epoxy coating in concrete discussed in the literature involve the progressive loss of coating adhesion to the steel substrate. Adhesion is usually lost as a result of one or more of the following mechanisms: a) Wet adhesion loss, b) cathodic disbondment, c) anodic undercutting, and d) bar fabrication. A description of each mechanism is described below:

Wet Adhesion Loss.

It has been theorized and observed that coatings lose adhesion when subject to moist environments.[2, 13, 14] The mechanism under which this phenomenon occurs is still unclear. Water can reach the epoxy/steel interface in two ways: 1) Diffusion through the epoxy because of coating permeability to water, and/or 2) transport across the interface itself because of discontinuities in the coating. In process (1), moisture permeates the coating in a complex and only partially understood manner. Propelling forces consist of osmotic and electroendosmotic pressures with transport aided by thermally induced molecular movements and vibrations within the polymer.[15] Although not completely understood, the following theories regarding the mechanism by which water promotes loss of adhesion have been proposed:[12]

a) Displacement of epoxy by water: Electrochemical adhesion in epoxy/steel interfaces depends on strong hydrogen bonds. Since water molecules are very strong hydrogen bonding agents, they will break the bond between epoxy and metal, and produce new hydrogen bonds with the hydrated oxide surface of the metal.

b) Oxide layer deterioration by hydration: Water hydrates the oxide layer above the steel surface. Since metal oxide hydrates have poor adherence to their base metals, mechanical adhesion is reduced considerably by the presence of a weak layer of hydrates at the interface.

Wet adhesion loss is often recoverable upon drying, but can become permanent in the presence of stress, through substrate deformations, or by build-up of underfilm corrosion products.[2]

Cathodic disbondment.

The anodic reaction that occurs at a coating defect is usually coupled to a nearby cathodic reaction beneath the coating. Oxygen and water migrate through the coating and support the cathodic reaction $O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$. This is possible because epoxy coatings can be permeated by oxygen, water, and ions.[16] Cathodically generated alkalinity can react with the organic polymer to disbond the coating at a defect at the interface between coating and metal. Such reaction is termed saponification.[17] It has also been theorized that cathodic disbondment may proceed by dissolution of the oxide film by hydroxides rather than by alkaline degradation of the coating itself. This is based on the good stability of epoxy coatings in alkaline environments.[14] Cathodic disbondment may also occur at microscopic or smaller flaws in the coating to produce blisters, which do not require a physically obvious defect for initiation.[17]

Anodic undercutting.

This mechanism is also known as oxide lifting. Briefly, corrosion products that are generated by the anodic reaction are deposited under the epoxy film during subsequent periods of wetting and drying, result in lifting or debonding of the coating from the substrate.[17]

Bar fabrication.

During bending, shearing stresses generated at the coating/steel interface weaken the adhesion of the epoxy film by mechanical action. Regions that are particularly vulnerable are the base of transverse ribs at the outer bend, because the coating stretches at these regions. If the coating is of good quality and properly applied, adhesion will only be weakened, but not lost after bending. It is usually the combination of bar bending with one or all of the above mechanisms that produces extensive adhesion loss in bent areas embedded in chloride-contaminated concrete.

Generally, more than one of the above adhesion loss mechanisms occur during corrosion of epoxy-coated bars, although it is unclear which one precedes the others. If concrete is of poor quality, the coating will still be adhered to the steel surface when the chlorides arrive, and the prevalent mechanisms will be a combination of cathodic disbondment, anodic undercutting, and water displacement. If concrete is of good quality, chloride penetration will be delayed, but adhesion may be lost by water displacement before chlorides arrive at the bar surface.

Regardless of which adhesion loss mechanism predominates, it is expected that a higher degree of initial coating adhesion before exposure will prevent or significantly delay the loss of adhesion during service, and therefore, decrease the extent of underfilm corrosion.

Pencil hardness measurements in a study by Clear for C-SHRP showed that, except for the effects caused by steel corrosion, the epoxy coating did not undergo physical deterioration after accelerated corrosion tests or exposure to chlorides during service in field concrete. These findings, coupled with the variable, and often poor, dry knife adhesion test results, led to the conclusion that loss of adhesion and underfilm corrosion originated at the coating /steel interface.[4]

1.3.4 Tests for Evaluation of Coating Adhesion

Peel or Knife Tests

Knife adhesion tests have been used because of their simplicity. The test procedure involves the application of a shearing force through the interface between coating and substrate with a sharp knife and successive prying of the disbonded coating. Pre-cuts (usually an X or V cut) through the coating are made to define the test section and eliminate the effect of cohesive forces by the surrounding coating. During the application of the knife force, the coating will lift from the substrate until the adhesion strength is larger than the applied shear stress. At that point, the knife will not advance further under the coating or will cut through the epoxy coating itself. The use of a hand-held knife has practical advantages and disadvantages. The main advantage is the portability of the knife, which enables testing of bars at any location or position; job sites, bar storage areas, coating applicator plant, or laboratory. Disadvantages include susceptibility of the test to operator error and variability, and the subjectivity of adhesion ratings.

Peel or knife tests are frequently performed after a preceding test has been performed on the bar, such as solution immersion, hot water immersion, cathodic disbondment, bend test, outdoor exposure, UV exposure, or accelerated corrosion inside concrete. These tests are intended to simulate the service environment to which the bars will be exposed in an accelerated way, and the subsequent knife adhesion test is intended to give a measure of the coating adhesion during the service life of the bar. The chemical component of adhesion is usually affected after the accelerated tests and the subsequent knife force breaks the remaining mechanical component of adhesion. If knife tests are performed without any previous accelerated test and bars have not been exposed to the environment, the knife force has to overcome the combined chemical and mechanical adhesion. In this case, the knife test would give an indication of the coating adhesion as produced by the coating applicator.

A variation of peel adhesion test was conducted by McDonald *et al.*[6] After making the two cuts through the coating, the coating was lifted and grasped with tweezers and then peeled back. The test was termed knife-peel adhesion test, perhaps because a knife was used to pre-cut the epoxy. The authors referred to the ASTM G1 specification as the background for the test, but after reviewing the standard, no mention is made of any knife adhesion test.

Presently, there is a lack of uniformity in different specifications and research studies regarding test procedure and adhesion evaluation criteria. Knife adhesion tests have been performed at ambient temperature, after hot water solution immersion, after cathodic disbondment, and after bending of the bar. Other variables that have not been uniform or defined include angle of X or V pre-cuts, knife force application, knife angle, and type of knife blade. An evaluation of knife adhesion test variables is presented in the following chapters.

TxDOT Peel Test

The Materials and Test Division of the Texas Department of Transportation developed an adhesion test procedure for steel elements that are too short for the bend test. Such elements include mechanical couplers, dowel bars, steel chairs and supports, steel plates, and others. The test is performed in accordance with test method Tex-739-I: [10]

> Perform the Peel Test by cutting or prying with the edge of a stout knife, applied with a considerable pressure in a manner tending to remove a portion of the coating. Testing should not be carried out at edges or corners (points of lowest coating adhesion) to determine adhesion. Adhesion will be considered inadequate if the coating can be removed in the form of a layer or skin so as to expose the base metal in advance of the knife edge. Removal of small particles of coating by paring or whittling will not be cause for failure.

As with most knife adhesion tests, the TxDOT Peel Test is highly subjective. Lorenzo discussed some of the difficulties of this test method.[12] The correct placement of the knife at the beginning of the force

application, the amount of force to be applied, and the acceptance criterion all depend on the operator's interpretation of the norm. Since no cuts are made through the epoxy to define the test area, the stiffness of the surrounding coating will tend to mask test results. An experimental evaluation of the Peel Test is later.

Hot Water Immersion

The German and Swiss guidelines for epoxy-coated reinforcement have placed emphasis on hot water testing as a quality and performance indicator. Hot water testing has a historical basis within ASTM, per recommended practices C870-86, C868-85, and D870-92. The buried pipeline industry has also used hot water testing. The Ontario Ministry of Transportation developed some draft specifications for hot water testing of epoxy-coated bar samples.[2, 9]

The procedure involves immersion of samples in hot water at a specified temperature for a given time. Different documents specify different water temperature and time of immersion. High osmotic pressures result in formation of blisters and cause vapor to migrate rapidly to the coating/steel interface at areas of marginal coating adhesion. As such, the procedure is an indicator of adhesion loss. Failure in water immersion may be caused by a number of factors, including deficiency in the coating itself, contamination of the substrate, or inadequate surface preparation. The test is particularly relevant to service performance because adhesion is considered a fundamental property for corrosion protection.[2]

Swiss and German guidelines specify a water temperature of about 10°C below the glass transition temperature of the epoxy coating.[18, 19] For typical coatings, temperatures range from of 75°C to 80°C. The Ontario draft specified a temperature of about 73 ± 2°C. It is recognized that as long as the temperature is below that range, the elevated temperature serves only to accelerate the water permeability of the coating and to speed but not alter the degradation process. Test immersion time was 7 to 10 days for German and Swiss specifications, and 48 ± 2 hours for the Ontario draft. Interestingly, German and Swiss specifications do not include adhesion testing following hot water immersion and base the acceptance criteria on the development of blisters or coating damage after immersion. A knife adhesion test is part of the Ontario draft specification.

Clear *et al.* incorporated electrochemical impedance spectroscopy (EIS) for the evaluation of samples following hot water immersion testing in a NCHRP study. EIS is particularly useful in providing mechanistic information and performance indications such as significance of defects and electrolyte take-up by the coating.[2] Direct tensile adhesion testing using a special test setup following hot water immersion was also evaluated in the NCHRP study.

McDonald *et al.* conducted knife-peel adhesion tests after immersing bar samples in four different solutions with and without chloride using a lower temperature (55°C) for a longer period (28 days).[6] Their rationale was that organic coating materials and steel have significantly different coefficients of thermal expansion and heat deflection temperatures. The 55°C was judged to provide a more reasonable in-service field temperature, yet provided a more aggressive condition than ASTM and AASHTO immersion tests at ambient temperatures. Two of the solutions contained sodium and potassium hydroxide and were intended to simulate the concrete pore solution environment. A summary of test parameters used in different specifications and research studies are included in Table 1.1.

An experimental evaluation of hot water testing was performed as part of the present research study and is presented in the following chapters.

Table 1.1 Test parameters for hot water test used in different specifications and research studies.

Study or Specification	Water Temperature	Time of Immersion	Evaluation
Swiss and German	10°C below glass transition temp.	7 to 10 days	Visual examination
MTO (Canada)[9]	73°C ± 2°C	48 ± 2	Knife adhesion test
NCHRP Study[2]	80°C	Variable	• EIS • Direct tensile adhesion testing
FHWA Study[6]	55°C*	28 days	• Knife-peel adhesion test • Visual examination

* Four different solutions with and without chloride were used

Cathodic Disbondment Tests

Cathodic disbondment tests consist of applying a cathodic potential to specimens immersed in an electrolyte solution for a specified period of time. The test subjects the coating to electrical stress in the highly conductive electrolyte. The coating is artificially perforated before starting the test. The test specimen is connected to the negative terminal of a source of direct current and an anode is connected to the positive terminal. At the end of the test, the extent of loosened or disbonded coating at the hole in the immersed area is compared with the extent of loosened or disbonded coating at a new test hole in the coating made in an area that was not immersed. A knife adhesion test is usually performed to determine the extent of disbondment.[20, 21]

The principle of the test is as follows: Water, ions, and oxygen are present at the steel surface by either permeating through the coating or moving along the coating/steel interface via a defect, and an

electrochemical cell with anode and cathode is established. When cathodic polarization is applied to a corroding metallic surface, the surplus or excess of electrons provided reduces the rate of the anodic reaction and increases the rate of the cathodic reaction

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$$

which increases the rate of oxygen reduction and OH^- production. The hydroxide ions will locally increase the pH at the coating/metal interface to as much as 14 or more. At very high pH levels, the polar bonds between the metal and the coating are significantly reduced.[6, 17]

Cathodic disbondment tests have been used in the pipeline industry to assess coating quality and to prequalify epoxy materials. There are numerous test procedures available for conducting cathodic disbondment tests, such as those described in AASHTO M284,[22] ASTM A775,[23] ASTM A934,[24] ASTM G8,[20] ASTM G42,[21] MTO,[25] and those performed by Schieβl and Reuter,[7] Sagüés and Powers,[26] and in the FHWA-RD-74-18[27] and FHWA-RD-94-103[6] studies. Different test methods will differ in their length of exposure time, applied potential, coating defects, temperature, and test solution. A reinforcing bar or a section of steel plate is used in the various procedures. For instance, the British Standard for cathodic disbondment is usually performed as a powder qualification test on plate samples.[28] In the FHWA-RD-94-103 study, cathodic disbondment tests were made particularly severe by testing bent bars instead of straight bars.[6] Table 1.2 summarizes parameters used in different tests procedures.

ASTM standards warn that although the ability to resist disbondment is a desired quality on a comparative basis, disbondment per se in the test is not necessarily an adverse indication. Although loosened coating and cathodic holidays may not result in corrosion, the accelerated condition for disbondment provided by the test gives a measure of resistance of coatings to this type of mechanism. According to ASTM, commonly used dielectric coatings will disbond to some degree and the test thus provides a means of comparing one coating with another. Adhesion strength may be more important for some coatings than others, and two different coating systems with the same measured disbondment may not have lost equivalent corrosion protection.[20, 21]

Table 1.2(a) Parameters for cathodic disbondment test from different standards and research studies [Adapted from Ref. 7].

Test Method	Time of Expos.	Temp	Potent (mV vs. SCE)	Intentional coating damage	Sample shape	Electrolyte solution	Accept Crit.
ASTM G8	30 days	23°C	-1500	6-mm (¼ in) drilled hole	straight	3% NaCl* pH 7	No
ASTM G42	30 days	60°C	-1500	6-mm (¼ in) drilled hole	straight	3% NaCl* pH 7	No
ASTM A775	7 days	24°C	-1500	3-mm drilled hole	straight	3% NaCl pH 7	No
ASTM 934 (Test A) ***	24 hr	65°C	-3000	3-mm drilled hole	straight	3% NaCl pH 7	Yes
ASTM 934 (Test B)[IV]	7 days	23°C	-1500	3-mm drilled hole	straight	3% NaCl pH 7	Yes
AASHTO M284	30 days	23°C	-2000	none**	straight	7% NaCl	Yes

* The standard specifies a 1% by weight of each NaCl, NaSO4, and NaCO3 solution (pH 11.2) but the pipeline industry generally uses the 3% NaCl solution.

** If no holidays develop in 30 days, a 6-mm diameter hole is drilled into the coating of both the anode and cathode. The test is continued for 24 hr, in which time no undercutting shall occur.

***Used for coating application requirements and pre-qualification requirements

[IV] Used for pre-qualification requirements only

Table 1.2(b): Parameters for cathodic disbondment test from different standards and research studies [Adapted from Ref. 7].

Test Method	Time of Expos.	Temp	Potent (mV vs. SCE)	Intentional coating damage	Sample shape	Electrolyte solution	Accept Crit.
MTO	7 days	23°C	-1500	3-mm drilled hole	straight	3% NaCl	Yes
Schieβl and Reuter[7]	30 days	23°C	-1000	3, 2.5 x 10 mm cuts	straight	3.5% NaCl* pH 7	N/A
Schieβl and Reuter[7]	30 days	23°C	-1000	3, 2.5 x 10 mm cuts	straight	0.3N KOH + 0.05N NaOH pH 13.3	N/A
FHWA-RD-94-103[6]	28 days	23°C	-1000 vs. rest potent.	6-mm (¼ in drilled hole)	bent	0.3N KOH + 0.05N NaOH pH 13.3	N/A

* The standard specifies a 1% by weight of each NaCl, NaSO4, and NaCO3 solution (pH 11.2) but the pipeline industry generally uses the 3% NaCl solution.

** If no holidays develop in 30 days, a 6-mm diameter hole is drilled into the coating of both the anode and cathode. The test is continued for 24 hr, in which time no undercutting shall occur.

***Used for coating application requirements and pre-qualification requirements

[IV] Used for pre-qualification requirements only

Salt Spray Tests

Coated samples are placed inside a chamber and subjected to salt spray comprised of a selected percentage of sodium chloride by mass dissolved in distilled water. A typical salt fog chamber contains an

air saturator tower, a salt solution reservoir, atomizing nozzles, sample panel supports, and heat controls to maintain the conditions of test at the desired temperature, and a relative humidity of 95% to 98% (Figure 1.1).[11] At the end of the exposure, a knife adhesion test is usually performed to determine the extent of disbondment and underfilm corrosion. This test is described in several standards such as ASTM A775[23], ASTM A934[24], and MTO Draft 93-10-01.[29] Different test procedures vary in concentration of salt solution, temperature, time of exposure, sample preparation, and acceptance criteria. For instance, both ASTM A775[23] and A934[24] specify 35°C ± 2°C salt spray of 5% NaCl solution for 800 ± 20 hours. Bars contain three intentional 3-mm diameter defects drilled through the coating evenly spaced along one side of the bar with the holes centered between deformations. The acceptance criterion is that the average coating disbondment radius should not exceed 3 mm from the edge of the intentional defect at 9 test sites.

Figure 1.1 Salt fog cabinet.[11]

In reality, the test is not an adhesion test but a corrosion test where adhesion loss occurs as a result of underfilm corrosion. Therefore, the mechanism of adhesion loss in a salt spray test is different from that in a cathodic disbondment test. While the latter test measures the amount of coating delamination that occurs because of cathodic reactions, the hot, wet conditions and high chloride concentration produced by the salt spray test will assay the adhesive strength of the coating and its ability to resist film undercutting in a hot, wet chloride environment. The ability of the coating to resist corrosion propagation initiating from damaged, bare areas is of utmost importance. Corrosion must be limited to the damaged areas by preventing film undercutting. The salt spray test is claimed to be an excellent procedure for testing these

characteristics. The test can be used as a screening test for material selection and/or as a quality control test to check coating at the plant.

Pull-Off Adhesion Tests

Coating is pulled off a steel substrate using a special adhesion test device. This procedure was developed and used in a NCHRP study.[2] In this study, the test procedure consisted of gluing the concave side of an aluminum pull-stub to the surface of the epoxy-coated bar using a two-part structural epoxy adhesive. To improve adhesion between the coating and the aluminum pull-stub, the coating surface was roughened with a rasp and subsequently cleaned with ethanol. Careful attention was given to properly aligning the pull-stub being placed on the bar using a specially designed alignment device. The adhesive was allowed to cure for 24 hours before testing. The coating was then scored around the pull-stub to expose bare metal to isolate the coating test area and eliminate the influence of cohesive forces that could be exerted by the surrounding coating. The bar/pull-stub assembly was mounted on a specially designed loading frame. The test set-up is schematically shown in Figure 1.2.

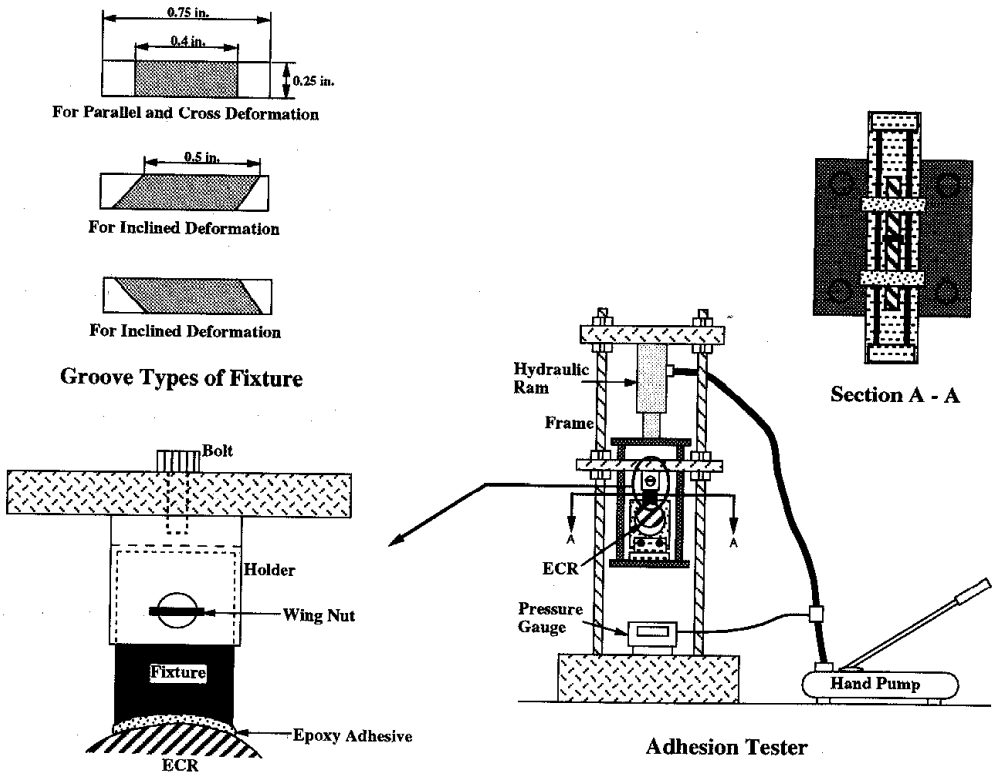


Figure 1.2 Schematic of pull-off adhesion testing system used in the NCHRP 10-37 study.[2]

This procedure is possibly the least subjective and most accurate way of determining coating adhesion, because the maximum amount of pull-off force applied with the hydraulic ram can be measured and divided by the test area to give units of stress (psi or Pascals) for the nominal coating adhesion strength.

This procedure is mainly suitable for research studies but may not be as practical as knife adhesion tests for field or plant applications. It should be pointed out that several pull-stubs with varying geometry are needed to fit bars with different diameters and corrugation patterns.

Bend Tests

In reality, bend tests do not assess the adhesive strength of the coating and it is a misconception to use a bend test for that purpose, as stated in previous ASTM and current AASHTO specifications. More accurately, bend tests provide an indication of the flexibility of the coating. Since epoxy-coated reinforcement in actual concrete structures has to be bent, it is important that the coating is flexibility enough to bend without developing tears, cracks, or to disbond. Epoxy coating must be formulated for adequate flexibility without sacrificing its corrosion protection properties.

1.3.5 Coating Adhesion Evaluation in Standards for Epoxy Coated Rebars[30]

Evaluation of coating adhesion according to typical ASTM and AASHTO standard specifications for epoxy-coated reinforcing steel bars is presented in the following paragraphs. The standards are divided in two parts: Post-fabricated bars and prefabricated bars. Post-fabricated bars refers to bars that are bent after application of the coating and prefabricated bars refers to bars that are bent before application of the coating.

Post-fabricated bars

AASHTO M284-91[22] specifies the use of the bend test to evaluate adhesion of the epoxy coating. A 120° bend around the appropriate mandrel with a slow bend rate is required. In contrast, ASTM A775-97[23] has incorporated the requirement of conducting a 7-day, ambient-temperature cathodic disbondment test on a regular basis to measure coating adhesion. There is no specified acceptance criteria. ASTM recommends the coating applicator use the test data as part of the plant' statistical process control.

A very important difference between the standards is that ASTM A775-97 no longer defines the bend test as an adhesion test (as in AASHTO M284-91) but as a coating flexibility test. The bend test does not always determine how well the coating is adhered to the steel surface nor does it indicate if such coating adhesion will be maintained in a chloride-contaminated concrete environment. The bend test is useful for quality assurance since failure of the test would indicate significantly problems with the coating process. The requirements for the bend test are more stringent in the ASTM standard (bend of 180° after rebound at a much faster rate).

Prefabricated bars

Prefabrication of rebar prior to surface preparation and coating application was developed recently as a solution to the coating adhesion lost and damaged by bending. A standard for prefabricated bars and

issued ASTM A934 / A934M in 1995 (later revised in 1996). For evaluation of coating adhesion, ASTM A934-96 requires a 24-hour, 65°C (150°F) cathodic disbondment test with a 6-mm maximum coating disbondment radius as on acceptance criterion of the bar lot.

1.3.6 Experience and Research on Coating Adhesion Evaluation

Experience by the Ontario Ministry of Transportation[3, 31]

In 1993, the Ontario Ministry of Transportation (MTO) asked coating applicators to significantly improve the quality of their product for the Ministry to continue specifying epoxy-coated bars. At the same time, the Ministry agreed to work with industry to develop test procedures and acceptance criteria. As a result of that work, three tests were investigated to measure coating adhesion: A hot water bath, cathodic disbondment, and salt spray exposure. The hot water test was found useful in discriminating and identifying bars with poor coating adhesion. However, knife adhesion ratings showed poor correlation when round-robin tests performed by different operators were compared. As a result, the hot water test was not incorporated into the 1994 specifications, and only cathodic disbondment and salt spray testing were introduced.

Experience in Europe

Test results by the German Institute for Building Technology (IFBT) showed that high powder quality and adhesion of the coating film to the steel surface were the most important parameters for corrosion protection. Consequently, German and other European standards for epoxy-coated bars placed great emphasis on these parameters. Investigations in Germany showed that immersion of coated bars in 90°C demineralized water was an excellent test for quality of adhesion and, to a certain extent, the permeability of the coating film. This test is accepted in Germany and Switzerland as a quality criterion in the pipeline industry, and is one of the main quality control tests in both the German and Swiss guidelines for epoxy-coated bars. In addition to the hot water test, the cathodic disbondment test has been used in Europe to evaluate the quality of adhesion and the quality of application at the coating plant.[7]

Research by the US Federal Highway Administration[6]

A five-year research project (FHWA-RD-94-103) commissioned by the FHWA was conducted by WJE to investigate the corrosion resistance of a variety of coated and uncoated rebars. The main objective was to derive a corrosion resistant reinforcing bar that will endure a 75 to 100-year design life for concrete structures. An additional objective was to develop appropriate new short-term test procedures that can be incorporated into the ECR standard specifications. In Task 1, 22 bendable and 11 nonbendable organic coatings were tested for coating adhesion following 28-day immersion tests in four solutions at 55°C. The four selected solutions were considered representative of the environments that coated bars may experience in service. Adhesion was also tested after cathodic disbondment tests on bent bars. For all

bars, holes were intentionally dulled through the coating. The main findings of Task 1 are summarized below:

In relation to hot solution immersion tests:

- Straight bars tested in hot deionized water can more easily pass the adhesion test than when tested in the other three hot solutions (NaCl, OH^-, and OH^- + NaCl). See Figure 1.3.

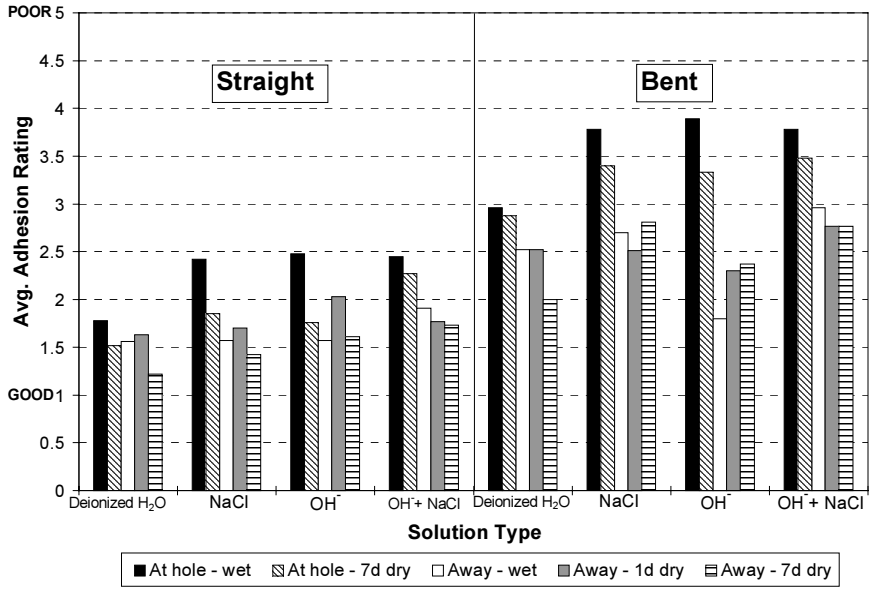


Figure 1.3 Average overall adhesion ratings for all coatings under all test conditions [FHWA-RD-94-103].[6]

- The use of bent bars in hot NaCl and OH^- + NaCl solutions produced the greatest number of poor adhesion ratings, indicating that these solutions are more detrimental to adhesion (Figure 1.3).

- For all types of solutions and conditions tested, bent bars experienced higher loss of adhesion (marginal to poor average adhesion) than straight bars after hot solution immersion (Figure 1.3). None of the bent bars with either bendable or nonbendable coating achieved perfect adhesion ratings in all four solutions following the immersion tests.

- The adhesion at the hole immediately after removal from the solution provided the worst adhesion. Adhesion was best away from the hole after 7 days of drying. However, the improvement of adhesion observed due to drying was regarded as minimal (Figure 1.4).

- Nonbendable coatings applied to straight bars exhibited the best overall performance, with more than 90% of nonbendable coatings on straight bars showing excellent adhesion ratings (Figure 1.5).

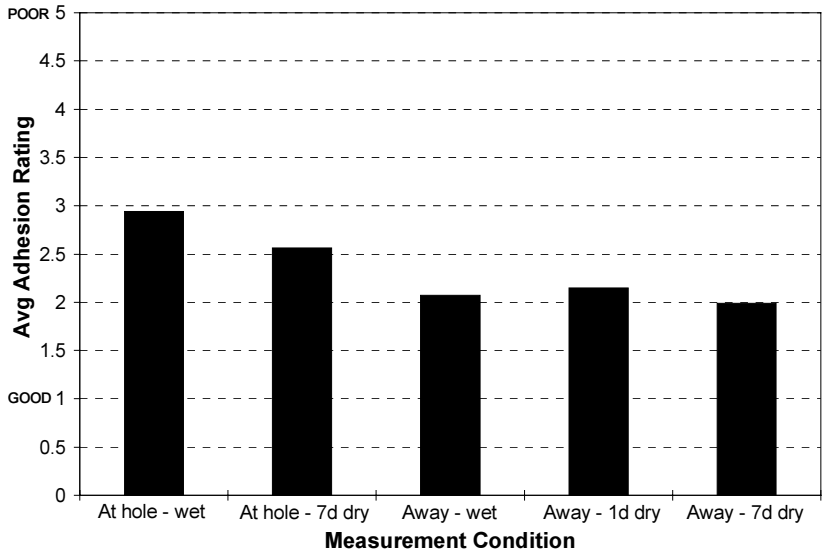


Figure 1.4 Average overall adhesion ratings for bent and straight bars at all test locations for all four solutions [FHWA-RD-94-103].[6]

- When nonbendable coatings were applied to prebent bars, the overall adhesion performance was worse than when applied to straight bars (Figure 1.5). This implies that it may be more difficult to coat and/or clean a prebent bar than a straight bar when applying nonbendable coatings.
- The poorest overall adhesion performance was achieved with bent bars using bendable coatings. Only 5% to 20% of the 20 or 21 bendable coatings on bent bars had excellent adhesion ratings (Figure 1.5).

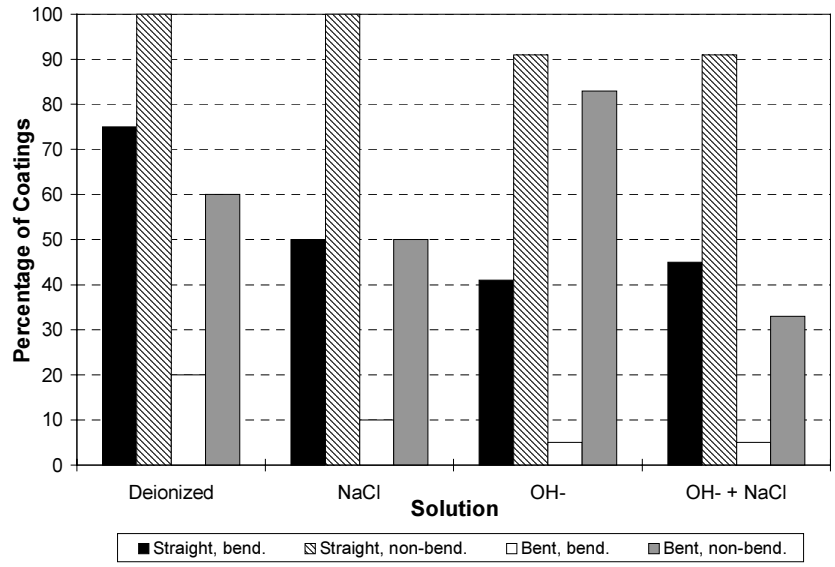


Figure 1.5 Percentage of coatings with average adhesion rating of 1 to 1.5 in different solutions [FHWA-RD-94-103].[6]

- Nonbendable organic coatings provided better adhesion than the average organic coating systems that are considered bendable, as suggested by the better average adhesion achieved when nonbendable

coatings were applied to straight bars compared to that of bendable coatings applied on straight bars (Figure 1.5). A direct comparison was made on straight bars to test the coating in a nonstretched condition for both cases.

In relation to cathodic disbondment tests:

- Cathodic disbondment testing on bent coated bars, which was particularly severe in this study, showed that nonbendable coatings performed significantly better than bendable coatings (Figures 1.6 and 1.7).

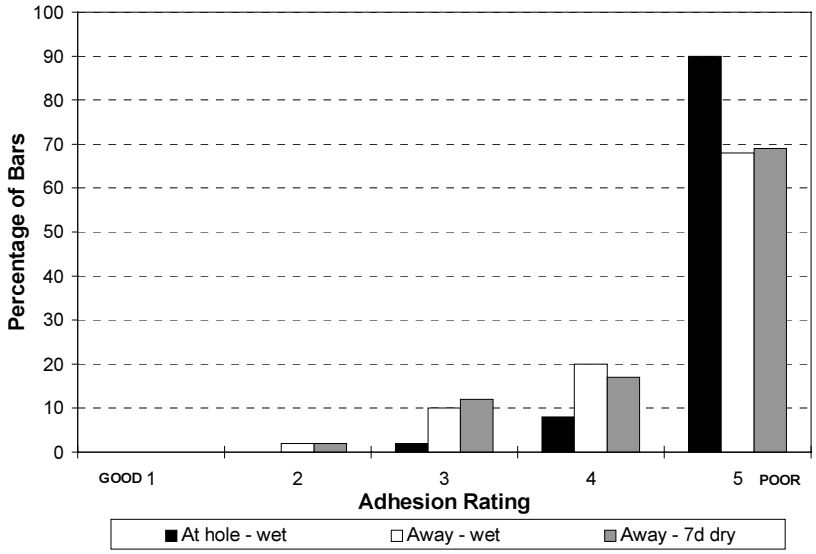


Figure 1.6 Average overall adhesion ratings for bendable coating at all three test locations [FHWA-RD-94-103].[6]

- Ninety-two percent of prebent specimens achieved excellent to good adhesion when tested away from the hole, either wet or after 7 days of air drying. In contrast, only one specimen achieved good adhesion when tested wet at the hole (Figure 1.7). The increased adhesion away from the hole showed that adhesion loss is created by conditions at the hole.

- Excellent adhesion was achieved on 92% of prebent bars when tested away from the hole after 7 days of air drying (Figure 1.7).

- With the exception of galvanized prebent bars, all prebent bars had good to excellent adhesion away from the hole under either wet or dry test conditions.

- Bendable coatings exhibited poor adhesion on 98% of the specimens when tested wet at the hole, 88% when away from the hole wet, and 86% when away from the hole after 7 days of air drying. The data revealed that moderate to severe coating disbondment resulted from bending effects that overshadowed the electrical disbonding effects of the test.

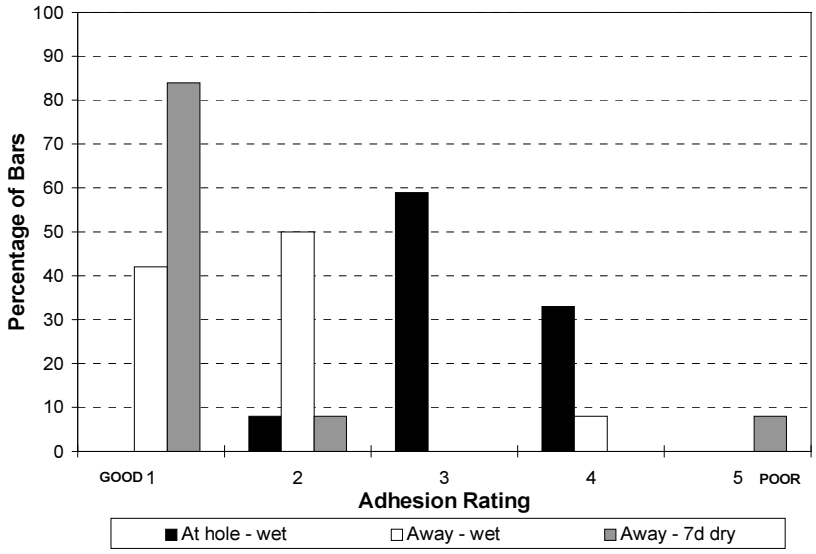


Figure 1.7 Average overall adhesion ratings for nonbendable coating at all three test locations [FHWA-RD-94-103].[6]

In relation to adhesion performance in defect-free coatings:

- The reduction in adhesion for those particular coating systems that exhibited overall excellent adhesion generally occurs only at the hole in the coating. The adhesion away from the hole in defect-free coated areas, or areas with fewer than 2 holidays per foot for prebent bars, is not reduced or affected by either hot solution immersion or cathodic disbondment tests.

In relation to the testing procedures:

- Knife-peel adhesion testing performed after hot solution immersion and cathodic disbondment tests proved to be a very useful method to prescreen the overall quality of 22 bendable and 11 nonbendable organic coatings on steel reinforcing bars.

National Cooperative Highway Research Program[2]

Research project 10-37 was sponsored by the National Cooperative Highway Research Program (NCHRP) and conducted by Kenneth C. Clear, Inc. (Virginia), with Florida Atlantic University as a subcontractor. Research results from hot water immersion and adhesion tests, and their correlation with electrochemical impedance spectroscopy tests are described here.

Coated bar specimens were specially prepared and placed into immersion test cells, which were filled with the desired solution (distilled water or 3.5% NaCl solution) to a level just below the top of the bar specimen, as shown in Figure 1.8. Multiple cell specimens were then placed inside a hot water bath (Figure 1.9). Bath temperature was 80°C and time of immersion was 14 days. Electrochemical impedance scans were taken at intervals of 1, 3, 7, and 14 days. The bar specimens were examined daily for blister

formation and rusting. At the end of immersion, adhesion testing was conducted using the special adhesion pull-off test device and procedure discussed earlier. Three test locations along the bar length were selected randomly per specimen. Adhesion tests were conducted after 1, 14, and 21 days of air drying.

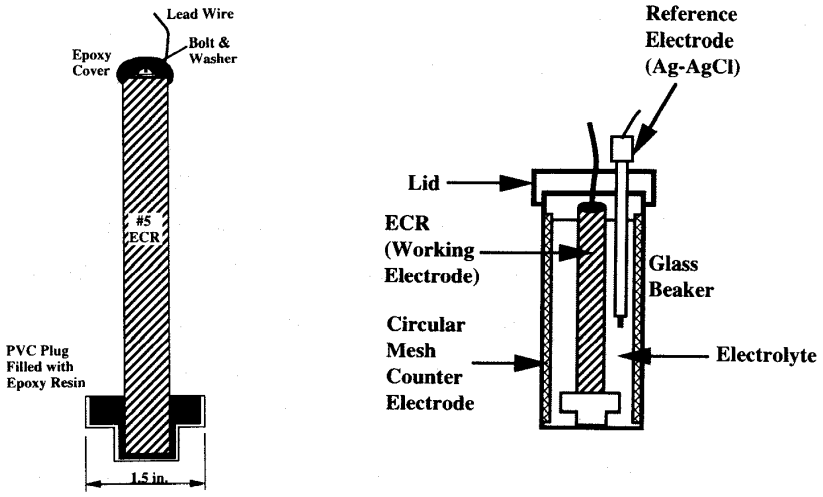


Figure 1.8 Schematic of epoxy-coated bar specimen and of immersion test cell for hot water test of NCHRP 10-37 study.[2]

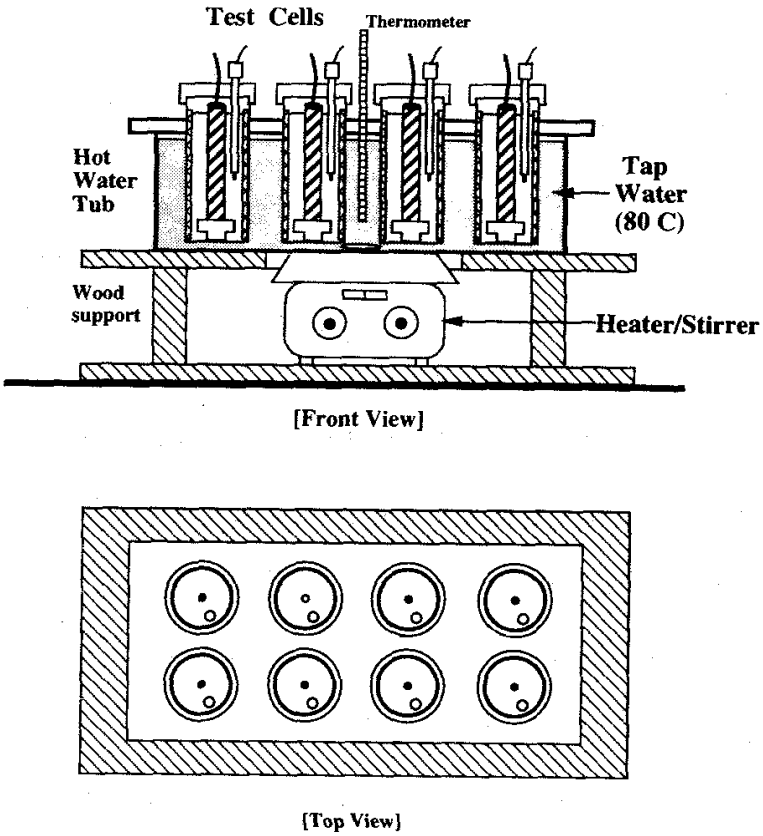


Figure 1.9 Schematic of hot water test apparatus for NCHRP 10-37 study.[2]

Macrocell concrete slabs were prepared with coated bars from the same sources. In the upper mat, two coated bar specimens were embedded in chloride-contaminated concrete, while three uncoated bars were placed in the bottom chloride-free concrete. Slabs were cyclically ponded with tap water for 10 months. At the end of exposure, coated bars were removed, visually inspected, and tested for adhesion. The main research findings are as follows:

- The amount of underfilm corrosion and the post-immersion (drying) period affected coating adhesion strength. In general, adhesion strength increased with extended drying time (Figure 1.10).

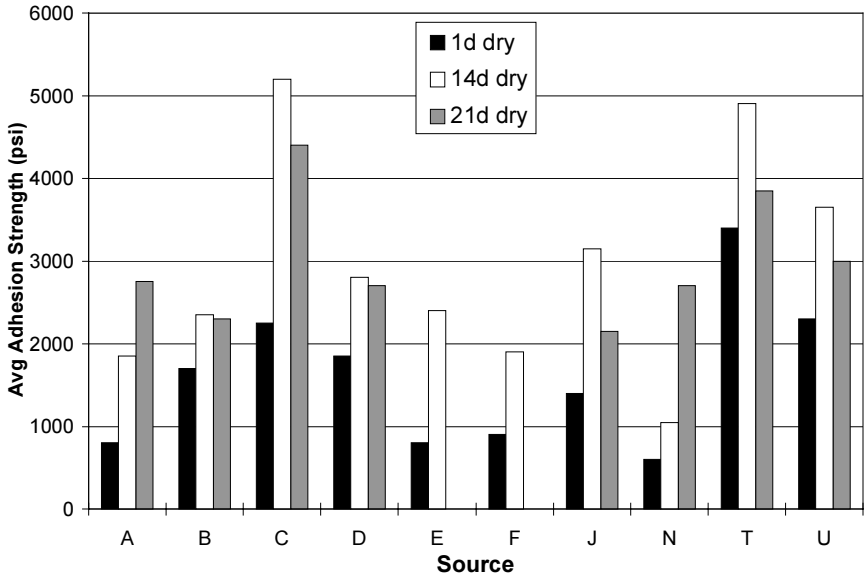


Figure 1.10 Average adhesion strength of coated bars from various sources after 14 days of immersion in 80°C distilled water [NCHRP 10-37].[2]

- Specimens from most sources that showed high impedance during exposure also exhibited relatively high adhesion after hot water immersion and concrete exposure. However, there were specimens that exhibited low impedance and marginal to low adhesion after concrete exposure (due to corrosion) but high adhesion after hot water immersion. Therefore, the development of conductive pathways and presence of coating defects (low impedance) did not preclude good adhesion in distilled water.

- As a corollary of the above, while good wet adhesion is a desired property, it is not sufficient, in and of itself, to ensure good performance when excessive defects are present.

- When hot 3.5% NaCl solution was used for the specimen cells, adhesion test results correlated much better with electrochemical impedance spectroscopy scans as compared with distilled water. Therefore, hot water testing should be performed in an aqueous chloride environment because chloride ions promote more underfilm corrosion compared to distilled water.

- Hot water tests may not correlate well with epoxy-coated bar performance in more aggressive environments.
- Acceptance criterion of no blistering in 7-10 days of exposure to hot water as specified by the German procedure did not seem adequate to predict poor performance.
- Distilled water was more effective than aqueous 3.5% NaCl in promoting wet adhesion loss to specimens with no discernible initial defects.
- Adhesion strength for defect-free specimens in distilled water did not correlate well, in general, with impedance results. Some specimens were highly susceptible to coating breakdown in the presence of defects that developed during the hot water exposure, but the occurrence of such defects did not compromise adhesion.
- The most significant change in impedance response occurred within 24 hours after immersion in hot distilled water or aqueous 3.5% NaCl solution. Therefore, the hot water test can provide useful information in one day.
- Unless a correlation between adhesion strength and long-term performance can be established, adhesion testing should not be included in a quality control protocol. EIS using single frequency measurements can provide a more reliable discrimination between "good" and "bad" epoxy coatings.

University of Texas at Austin

Durability studies on epoxy-coated bars were conducted as part of the TxDOT-sponsored research project 1265. The experiments included immersion in 3.5% NaCl solution,[32] macrocell[33] and beam[34] studies, and electrochemical impedance spectroscopy.[35] Relevant findings pertaining to the role of coating adhesion in the corrosion performance of the bar specimens are discussed here.

The corrosion of epoxy coated bars observed in the durability studies reveals that adhesion of the epoxy coating is inevitably lost after a prolonged period of exposure to water and chlorides in concrete (whether bars were bent or straight). Corrosion experiments and field inspections by others have also provided evidence of various degrees of coating disbondment after chloride exposure in concrete.[1, 36, 37, 38, 39, 40] Kahhaleh suggested that adhesion loss could be beneficial because corrosion would spread along the bar and would not concentrate at certain spots and cause severe localized damage.[5] Longer term exposure showed that this hypothesis may not necessarily be true. Although bar corrosion was less concentrated and severe in coated bars than on uncoated bars, several pits of moderate depth were observed in coated stirrups (beam study).[41]

The degree of adhesion loss after chloride exposure seemed to be affected by differences in coating integrity. Straight bars from beam B1 were in excellent condition with no visible damage before chloride exposure. The bar condition was preserved without signs of corrosion or extensive adhesion loss after 4.3 years of chloride exposure (Figure 1.11). Longitudinal bars in the remaining autopsied specimens had intentional damage, patched or unpatched, and exhibited adhesion loss within the wetted region with varying degrees of underfilm corrosion (Figure 1.12). Since bars for all beams came from the same lot, it is reasonable to assume that all bars had similar coating adhesion before chloride exposure. Clearly, coating integrity was fundamental in the preservation of adhesion and its protective capabilities. In addition, it was found that adhesion loss always occurred around areas of damaged coating and was least affected at locations farthest from damaged coating. Similar observations have been made by Sagüés.[1] Visible holidays and coating defects were present on areas that experienced coating disbondment in coated bar segments extracted from four bridge decks in California.[36] This evidence suggests that the agents causing coating disbondment migrated to the coating-substrate interface through coating defects rather than through the bulk of the coating.

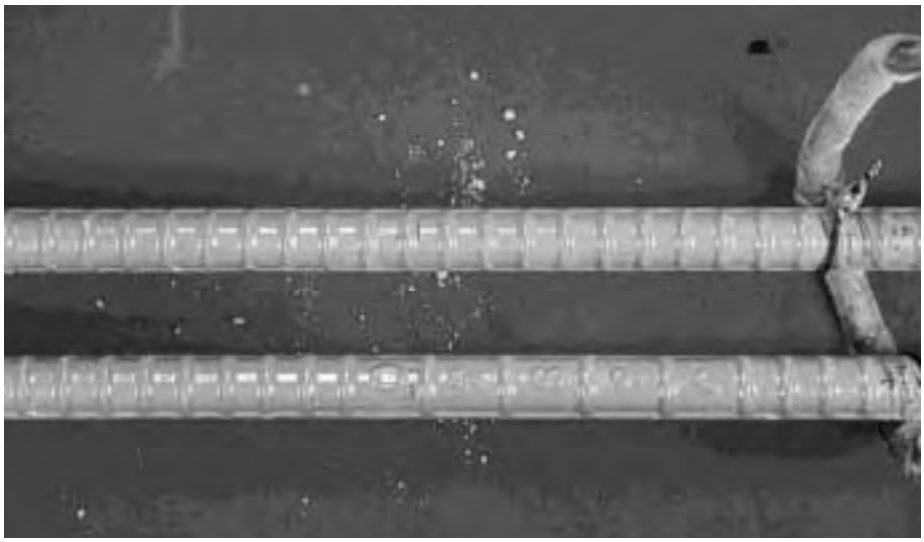


Figure 1.11 Longitudinal coated bars of beam B1 remained in good condition after 4.3 years of chloride exposure (undamaged coating before exposure).[34]

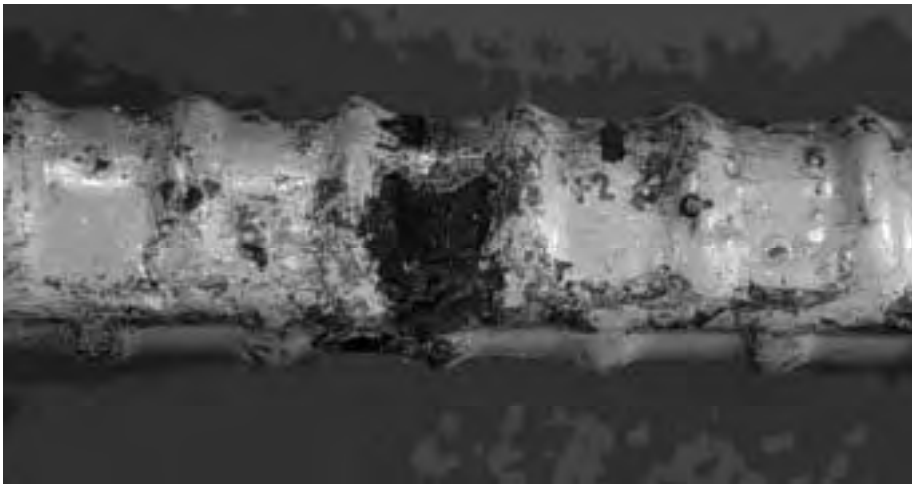


Figure 1.12 Build up of rust products at damaged spot on bar from beam B10.[34]

Fabrication (bending) of bars weakened coating adhesion. In durability studies, all macrocell specimens showed loss of adhesion at bend portions and adjacent straight legs after 2 and 4.5 years of chloride exposure, regardless of the level of corrosion activity (Figure 1.13). Likewise, coated stirrups in beam specimens showed widespread adhesion loss after one and 4.3 years (Figure 1.14). On most beams, adhesion loss was slightly more extensive on fabricated bars than on straight bars. Underfilm corrosion was noticeably more extensive on fabricated bars than on straight bars. Weakening of adhesion caused by bar fabrication seemed to be proportional to the observed adhesion loss and underfilm corrosion after chloride exposure. After fabrication, adhesion was weakened at bends in stirrups but was likely preserved along the straight portions. After chloride exposure, adhesion loss and undercutting progressed from weakened (bend) portions to initially well adhered (straight) portions.

EIS and polarization resistance tests on bent and straight coated bar samples performed by Chen showed similar results regarding adhesion loss and corrosion.[35] Adhesion strength before immersion was similar for both straight and bent samples. After immersion, bent samples experienced more extensive adhesion loss than straight samples did. Extent of adhesion loss was strongly dependent on the coating type and source. There was not a clear correlation between adhesion strength after immersion and extent of corrosion. Several bent samples experienced adhesion loss but no signs of corrosion after immersion in chloride solution. The coating surface in those samples had no visible damage, pinholes, or discontinuities. Even very thin coating at rib bases provided protection as long as the coating had no defects. Chen stated that "adhesion loss can be the result, and not necessarily the cause, of epoxy-coated bar degradation."[35]

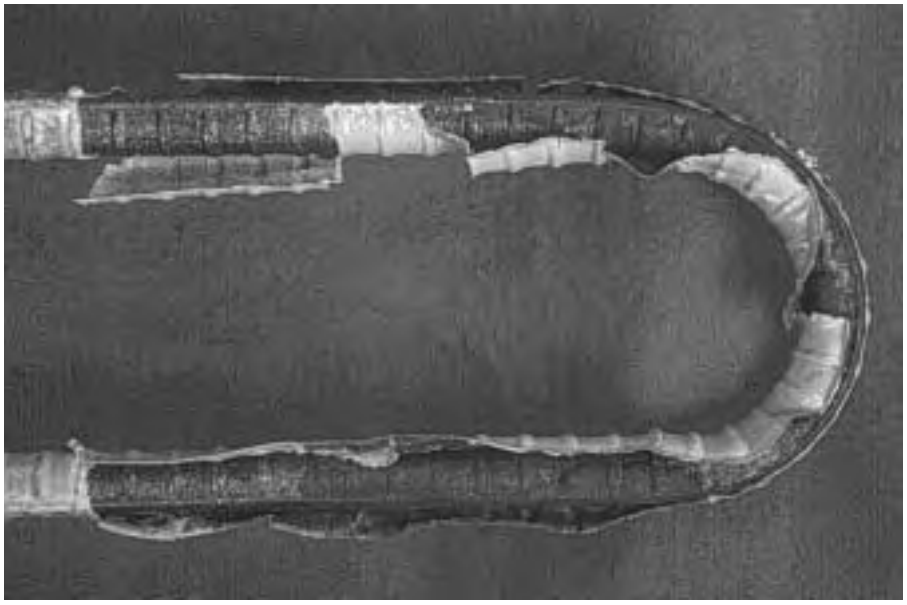


Figure 1.13 Coating debonding on a 13 mm (#4) bar from the macrocell study after 4.5 years of exposure.[33]

Figure 1.14 Coating extensively debonded on stirrups from beam specimens.[34]

Although coating adhesion was not measured before exposure, some hypothesis regarding the role of adhesion can be drawn from the exposure studies conducted in this study. The effect of adhesion on corrosion performance may be similar to that of flexural concrete cracks. Weakening of adhesion by bar fabrication will accelerate loss of adhesion and underfilm corrosion (similar to the presence of flexural cracks). Adhesion loss and underfilm corrosion will be significantly slowed if there is good adhesion before exposure (similar to the absence of flexural cracks). Nevertheless, in the long term, adhesion loss and underfilm corrosion will progress in bars with initial good adhesion (provided that the coating is damaged) to levels closer to that of bars with initial weak adhesion. The longer the exposure, the more similar the amount of corrosion will be between bars with initially poor or good adhesion.

Miscellaneous

Experiments conducted at the University of Western Ontario showed that the mechanism of adhesion loss appeared to be water permeating the epoxy coating. Water displaced the coating from the steel substrate.[3] Nevertheless, electrochemical tests indicated that the effect of adhesion loss on corrosion behavior was directly related to the presence of defects in the coating. If defects were absent, adhesion loss did not change the short-term corrosion behavior. However, if defects were present, corrosion rate was directly related to the adhesion of the coating, i.e. poor coating adhesion resulted in high corrosion rates. The main factors improving coating adhesion identified in that study were an increase in the surface roughness and a decrease in the presence of contaminants.[3]

In an attempt to clarify the role of holes in the coating versus coating adhesion, a numerical model was developed at UMIST University.[42] The "Cottis Model" revealed that in the presence of holes in the coating in a low permeable concrete, the bar corrosion rate was governed primarily by the coating adhesion, and not by the relative size of the defects in the coating (Figure 1.15). However, there was no explanation of the validity of the model for coated bar specimens, particularly in a real concrete environment.

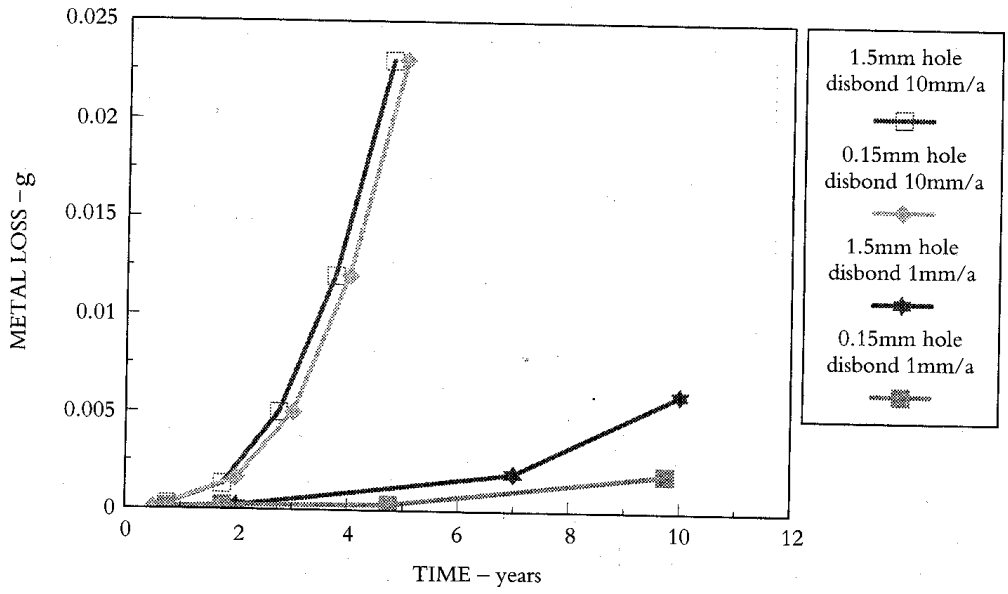


Figure 1.15 Cumulative corrosion with time for epoxy-coated steel in low permeability concrete according to "Cottis Model" [UMIST].[42]

1.4 ORGANIZATION OF STUDY

The adhesion study was divided into three phases. In the first phase, hot water tests were conducted following the Swiss and MTO procedures. The only modification that was introduced consisted of using an alternate adhesion rating system to evaluate test results by the MTO procedure. For the second phase, some modifications to the MTO hot water-adhesion test parameters were introduced and evaluated and the repeatability of the test was studied. In addition, a procedure to control the knife force was implemented. In the third phase, additional refinements in the test parameters and procedure were studied and evaluated. A self-calibrated knife was developed to measure and control the amount of force applied. Results from different adhesion test procedures were correlated. A short-term salt-solution immersion test was conducted in an attempt to understand the role of coating adhesion in corrosion protection. A proposed test procedure for hot water-adhesion is included in Appendix A.

CHAPTER 2

First Phase: Adhesion Study Following Existing Guidelines

2.1 HOT WATER TEST - SWISS SPECIFICATION[18, 19]

The test consists of immersing bent and straight epoxy coated bars in a hot water bath at a water temperature of 10°C below the glass transition temperature of the epoxy coating (typically about 80°C) for seven days. The bar ends and damaged areas of about 25 mm^2 are patched. A maximum of one damaged spot (of up to 25 mm^2) per meter before immersion is admissible. The bars are evaluated by visual inspection. Assessment of coating damage is based on the classification shown in Table 2.1. Acceptance criteria are as follows:

- In previously undamaged coating, a maximum of 6 spots of damage type S2 per meter is allowed.
- A maximum of 5 damaged spots of type S2 may appear in bent bars.
- In the patched areas, deterioration such as the formation of blisters and damage visible to the unaided eye (damage type S3) is permissible.

Table 2.1 Classification of damage of epoxy-coated reinforcement.

Damage Type	Description
S0	No damage (evaluated with visual equipment)
S1	Microscopic damage that can be recognized only with visual equipment (magnifying glass, microscope)
S2	Damage that can be recognized by the eye without visual equipment
S3	Clear visual damage (0.1 - 1.0 mm)
S4	Clear visual damage of larger dimensions (> 1 mm)
S5	Surface failure of corrosion protective system (over 5 mm)

In all cases the assessment is done with respect to the effective size of damage on the coating, without any consideration of corrosion products that may be deposited underneath the coating.

Regarding the first two criteria (damage types S0 and S1), microscopic examination of a bar is very tedious, time-consuming, and difficult. There is no guidance for recognizing and identifying microscopic coating defects. These characteristics do not constitute a practical test. Also, it is desirable to examine the steel surface underneath the coating at rusted spots to give a better indication of the extent of coating degradation. Often, undercutting corrosion beneath the coating is more widespread than the corrosion observed on the coating surface.

Previous work on this project explored the feasibility of the hot water immersion test using Swiss specifications.[5, 32, 43] The test was conducted on #4 and #8 bent, epoxy coated rebars, with both repaired and unrepaired damaged areas. The results showed that deterioration appeared at pinholes and cracks in areas deemed undamaged by visual inspection. Such damage was especially noted along the sides of the lugs. It was observed that the test was very effective in identifying pinholes in the coating on bent bars. The main conclusion was that the test was feasible for indicating the quality of coating application.

For the present study, specimens from one coating applicator (*A*), one type of epoxy (*a*), two steel mills (*H* and *N*), two bar sizes (#10 and #4), and both straight and bent samples were used. A total of 8 group combinations and 31 samples (at least 3 samples per group) were considered. Specimens were immersed in their "as-received" condition without repairing coating damage. Bar ends were sealed with silicone. Length of specimens was 12.5 cm. The glass transition temperature of epoxy coating "*a*" was 87°C, which led to a water temperature of 77°C for the test. Although not specified, samples were allowed to dry for 24 hours after immersion before visual examination.

Some of the major findings include: Black rust deposits appeared on previously damaged areas (coating damaged before water immersion) or on pinholes detected before the test. Coating defects and pinholes undetected before the test became visible as black dots or spots, dark-brown spots, or black or brown rusted cracks. Brown rust appeared much less frequently than black rust. About 90% of rusted areas appeared on or adjacent to bar deformations (longitudinal and transverse ribs). There were instances where large and small damaged areas did not experience any change in appearance nor did they exhibit rust formation.

On one hand, the hot water test following Swiss specifications seemed helpful in revealing coating defects such as pinholes, cracks, tears, thin coating, incipient damage, and other types of damage that were not evident to the unaided eye. On the other hand, the fact that no corrosion appeared at several locations with large and visible areas of damage raises questions about the reliability of the test. If large damaged areas withstand such test conditions, much smaller and less visible damaged areas could also sustain the test without corrosion attack. It may be possible for a bar with defective coating to pass the test.

For these reasons, the hot water test did not seem to be reliable for locating all possible defects and discontinuities in the coating. Clear *et al.* also found the classification and acceptance criteria of the Swiss procedure to be inadequate.[2] In addition, seven days of immersion and a very cumbersome microscopic examination process are not practical for a test intended to be completed quickly. With these factors in mind, no further tests using the Swiss specification were conducted in subsequent phases.

2.2 MTO HOT WATER-ADHESION TEST

MTO Draft Specification[9]

The Canadian draft specification requires submerging bar samples in a tap water bath at a temperature of 73 ± 2°C for a period of 48 ± 2 hr (Figure 2.1). Spacing of at least 25 mm between bar samples is required. The bars are removed from the water bath and stored in air at 23 ± 3°C for 24 ± 2 hr. Subsequently, samples are prepared for adhesion testing as follows*:* With the specimen securely clamped on a vise, an X-cut is made through the coating at six locations between bar deformations, as shown in Figure 2.2. Three test sites are located on each side of the bar. The cuts must extend through the coating so that the metal is visible. The cuts are made with a utility knife having a new, sharp blade for each specimen. The length of each cut should not be less than 10 mm or the distance between adjacent deformations. The two cuts forming the "X" should intersect a an angle as close to 90° as possible. The "X" cut defines 4 flaps of coating and each one is tested. All test sites should be located at least 25 mm from the bar ends.

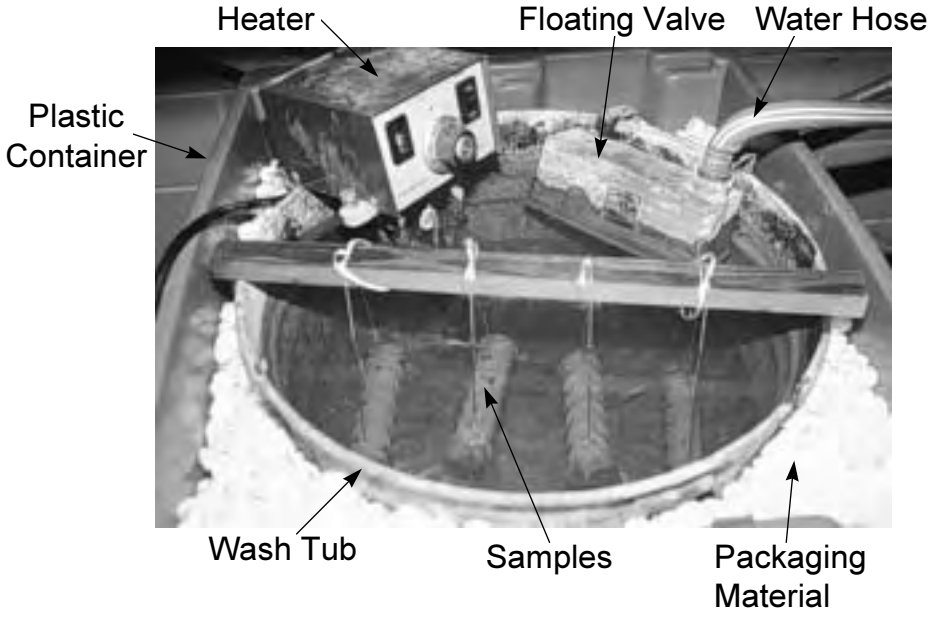


Figure 2.1 Hot water bath.

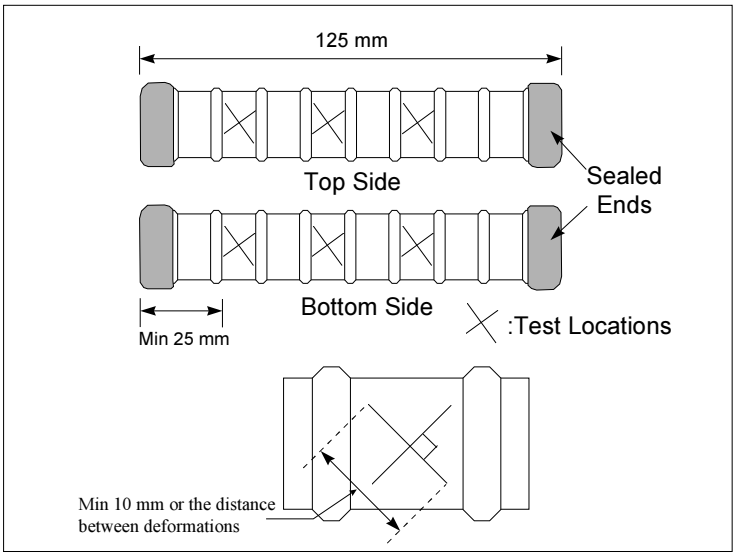


Figure 2.2 Test locations on rebar.

Adhesion testing on each "X" is performed as follows: The knife is positioned vertically on the bar so that the tip of the blade makes contact with the intersection of the two cuts and the plane of the blade bisects the two cuts (the plane of the blade is perpendicular to a line bisecting the flap to be tested, as illustrated in Figure 2.3). The blade is then rotated so that it makes a shallow (approximately 30°) angle with the bar while the tip of the blade remains in contact with the bar (Figure 2.3). The blade is inserted under the coating and a constant pressure of approximately 3 kg is applied until the coating resists the insertion (Figure 2.4). The pressure is maintained for at least 5 seconds. The knife blade should not cut through the coating. Any disbonded coating is removed by levering action of the blade. The procedure is repeated in all four flaps. An adhesion rating is assigned in accordance with Table 2.2.

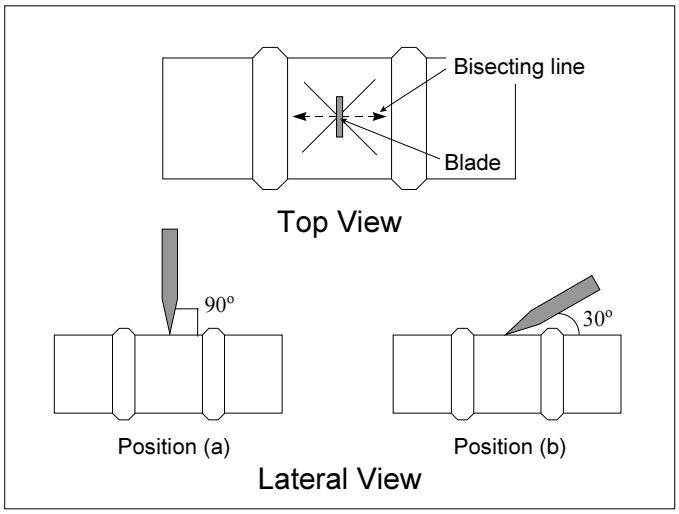


Figure 2.3 Position of knife and direction of force application.

Figure 2.4 Adhesion testing of epoxy-coated bar specimen.

Table 2.2 Adhesion rating of epoxy coating in Hot Water Test (MTO test procedure).

Adhesion Rating	Description
1	Unable to insert blade tip under the coating at all four sections
3	Blade tip can be inserted under the coating. Levering action removes small chips of coating but cannot remove the entire coating at any section.
5	Blade tip slides easily under the coating and the entire coating can be removed at one or more sections.

Refinements in Procedure

An interesting problem relates to the measurement and application of a constant knife force during the test. For this first phase, knife force was estimated very crudely. The procedure consisted of first pushing the knife against a digital scale until the desired amount of force was reached. The pressure on the scale was maintained for about 30 seconds so the operator would get a feel for the amount of force to be applied. The operator then tried to emulate that force during the test. Once the operator felt the desired force was reached, the knife pressure was maintained for at least 35 seconds (instead of the specified 5 seconds). This procedure is basically a calibration of the force applied by hand. Initially, the operator needed to calibrate his arm before testing every site. As the operator became more experienced, arm calibration was done after every 3 test sites. Estimating the knife force was subjective and very susceptible to human error and was the first step towards developing a more reliable method for force calibration in subsequent phases.

Another problem involved positioning the knife at an angle of approximately 30° with respect to the bar surface. It is difficult for the operator to concentrate on controlling arm force and keeping the proper angle of the knife during the test. Obviously, human error is to be expected. Besides, the reference plane against which the angle of the knife should be estimated was not clearly defined. Possible reference planes could be: a) a horizontal plane parallel to the longitudinal bar axis, or b) a plane tangent to the bar perimeter at every point the blade tip makes contact with the bar as the knife moves (Figure 2.5). For this study, the angle of the knife was measured with respect to the tangential reference plane. In this manner, the knife would be positioned at an approximately constant angle with respect to the bar surface as the knife moves. This procedure should produce an approximately constant force at the coating-steel interface.

A utility knife with snap-off blades was used for adhesion testing. With such a knife, new blades could be easily used for every test and the test was economical. However, the knife blade design was not very suitable for adhesion testing on epoxy-coated bars. The problem was that the sharp edge of the blade was parallel to the knife axis. The blade was not symmetrical and did not have a very pointed sharp tip. During the test, the blade had to be held at an oblique angle with respect to the path that the blade had to follow (the line bisecting the flap of coating) so that the blade tip could be inserted under the coating. For the operator, this was an awkward and uncomfortable position.

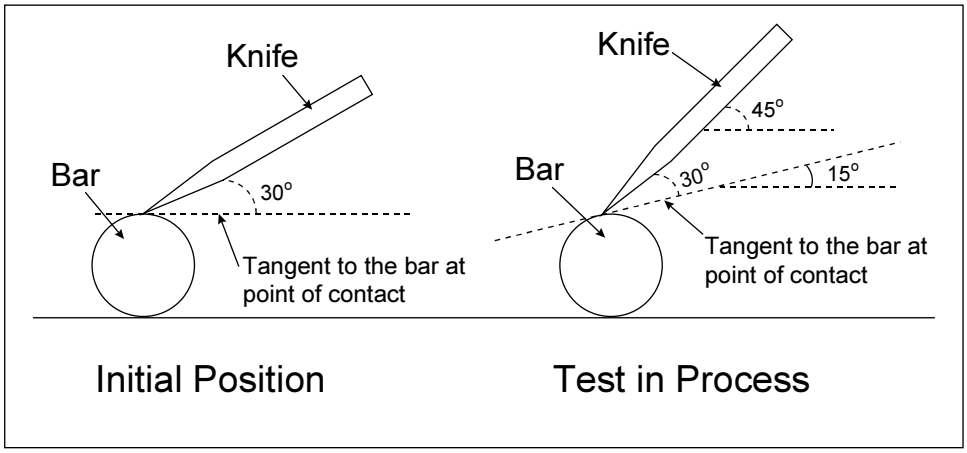


Figure 2.5 Angle of knife during adhesion testing.

Study Variables

Bars procured for this study had the following characteristics:

- Bars from three Texas DOT-approved coating plants (*A, B*, and *C*)
- Bars coated with two epoxy coating powders (*a* and *b*) pre-qualified by TxDOT
- Bars from four steel mills (H, N, I, and S)
- Bent and straight bars

- Bars of three sizes (#10, #9, #4)
- Bars with two rib deformation (parallel, diagonal)

Bars from coating plants *A* and *B* were coated with epoxy material "*a"* and bars from coating plant *C* were coated with epoxy material *"b"*. Coating plant *A* acquired bars from two steel mills (H and N) and coating plants *B* from BI and *C* from SM. Coating plants *A* and *B* supplied #10 and #4 bars, and plant *C* supplied #9 and #4 bars. This yielded a total of 16 groups, each one representing a bent or straight bar, of given diameter, from a specific steel mill, coated with a particular epoxy at a certain coating plant. Determination of study variables had several limitations because all three coaters did not use the same epoxy coatings, did not purchase bars from the same source, and did not provide bars of the same diameter. The primary variables were coating applicator, bent or straight bars, bar size, and steel source.

Shipped bars were 10 feet long and had a 180° hook at each end (Figure 2.6). Bars from coater *C* were individually wrapped in soft styrofoam sleeves to protect the coating from damage during shipment. Bars from coaters *A* and *B* were not as carefully handled during shipment. Bars from coater *A* had the most damage. Samples 12.5 cm long were cut from both the straight and bent portions of the bars. A total of 51 bar samples were cut, including at least two samples from each group. Samples were selected from locations with the least damage. Bar ends were sealed with silicone. Specimens were immersed in their "as-received" condition without repairing coating damage.

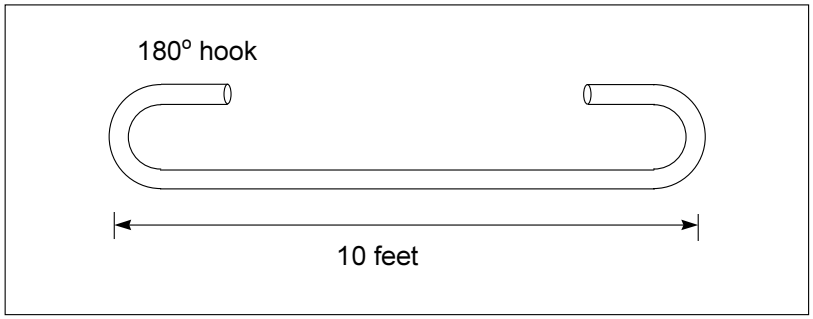


Figure 2.6 Epoxy-coated bar dimensions as-received from coaters.

MTO test procedures specify adhesion testing 24 hours after immersion. This requirement was followed at most test sites. However, adhesion tests were performed at selected test sites in 24 samples after varying post-immersion periods, ranging from 40 hours to 2 months after immersion.

Test Results and discussion

When the experiment was performed, the initial MTO rating system was different from that shown in Table 2.2. The earlier rating was based on measurement of the debonded coating area. A simplified system was developed for evaluation of test results and is described in Table 2.3. In this system, a "flap" rating was assigned to each individual flap and, depending on the combination of flap ratings, an adhesion

rating was assigned to each test site. This alternative rating system has the advantage of not requiring any measurement. The final MTO rating system, which is considerably simpler than the one proposed in their first draft, became available during the course of the experiment.

Table 2.3 Alternative adhesion rating system.

Flap rating	Description
A	Unable to insert blade tip under the coating
B	Blade tip can be inserted under the coating. Levering action removes small chips of coating but cannot remove the entire coating
C	Blade tip slides easily under the coating and the entire coating can be removed

Adhesion Rating	Description - Σ Flap ratings
1	A at all 4 flaps (Good adhesion)
2	B at one flap, A at remaining flaps
3	B at 2 or 3 flaps, A at remaining flaps
4	B at all 4 flaps
5	C at one flap, A or B at remaining flaps
6	C at 2 or 3 flaps, A or B at remaining flaps
7	C at all 4 flaps (Poor adhesion)

Figure 2.7 shows average adhesion test results of specimens from all three coaters. A rating of 1 indicates good coating adhesion and a rating of 7 indicates poor coating adhesion. Specimens from coater *A* exhibited generally poor coating adhesion. In most cases, the coating could be easily peeled with almost no force. Frequently, the coating started to debond while making the "X" cuts. Straight bar specimens from coaters *B* and *C* showed better coating adhesion, with few test sites having adhesion rating of 7 and several test sites with ratings of 1 and 2. On average, coating adhesion for straight bar specimens from coaters *B* and *C* was fair. A photograph showing typical adhesion results on several specimens from all coaters is included in Figure 2.8.

Figure 2.7 also shows average adhesion test results of straight and bent samples for all coaters. Clearly, bent bars from all three coaters exhibited poor coating adhesion. Poor adhesion of bent bar specimens was expected. During bending, the epoxy coating is stretched and loses some adhesion to the bar surface. Bent samples start with marginal adhesion compared to straight specimens. After hot water immersion, the already marginal coating adhesion of bent samples was even worse. It seems that the test conditions may be too severe for bent bars. In several bent samples, coating on the outside of the bend was easier to peel than on the inside of the bend, even when adhesion ratings were the same on both the inside and outside

of the bent. A possible reason is that during bending, coating on the outside stretches and coating on the inside compresses. Even if coating adhesion is poor, the compressed coating on the inside may offer some resistance to the knife blade.

If bent bars are not considered, the difference in coating adhesion between the three coaters becomes even more pronounced. Straight samples from coater *A* showed poor coating adhesion while straight samples from coaters *B* and *C* showed very good to excellent performance, as evidenced in Figure 2.7.

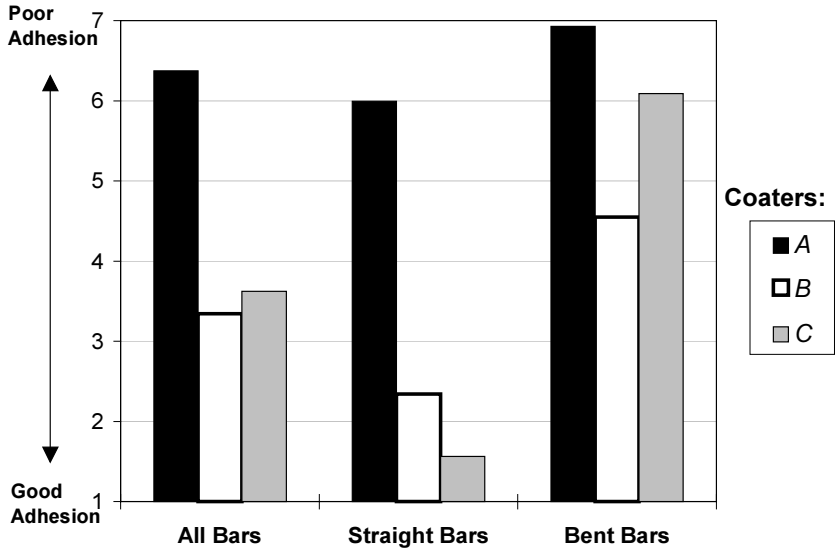


Figure 2.7 Average adhesion ratings of specimens grouped by coating plant and type of specimen (bent or straight).

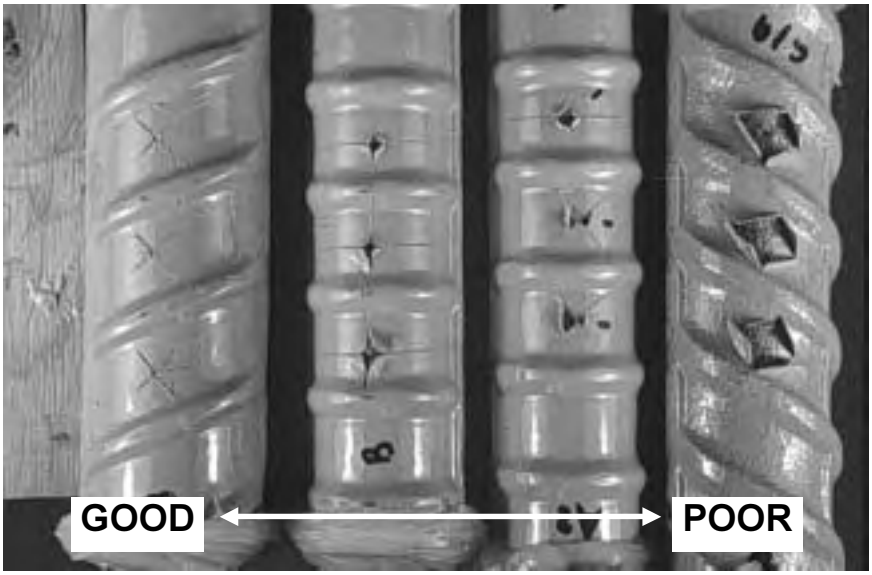


Figure 2.8 Typical adhesion test results of several specimens from all coaters.

In Figure 2.9, average adhesion ratings are categorized by bar size and coater. There is not a clear relationship between bar size and coating adhesion. Bars of smaller diameter from coating plants *A* and *C* performed better than bars of larger diameter. However, in the case of bars from plant *B*, larger bars showed better coating adhesion than smaller bars. Considering specimens from all coaters, smaller bars, with average rating of 4.7, tended to have slightly better coating adhesion than larger bars, with average rating of 5.4 (Figure 2.10).

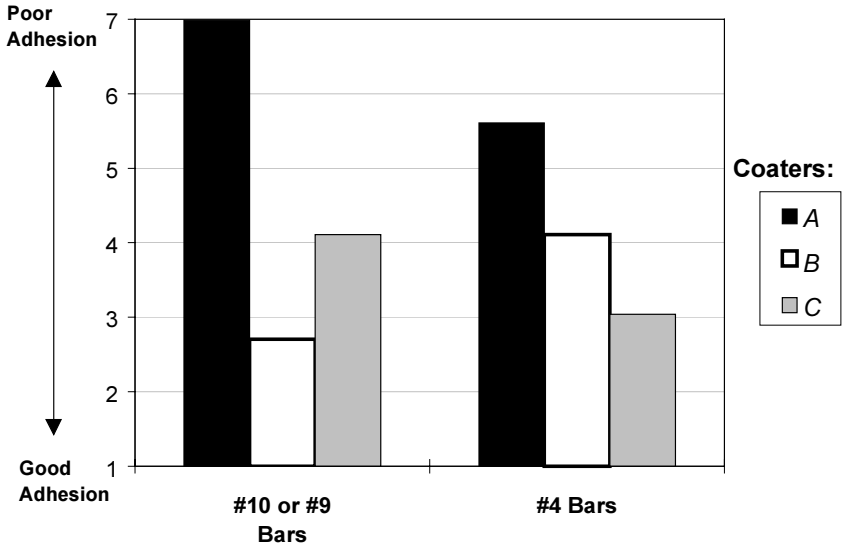


Figure 2.9 Average adhesion rating of specimens grouped by coater and bar size.

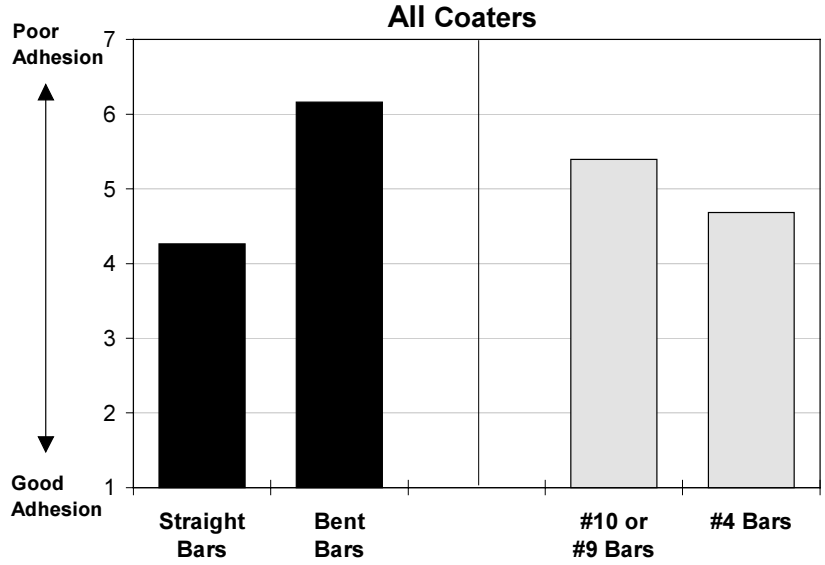


Figure 2.10 Average adhesion rating of specimens grouped by bar type and bar size.

Adhesion rating and coating thickness of individual specimens are plotted in Figure 2.11. The data points are widely scattered and there is no clear trend between coating adhesion and coating thickness. Coating

thickness variability, and adhesion rating of each specimen are plotted in Figure 2.12. Again, no clear relationship between these two coating characteristics was found.

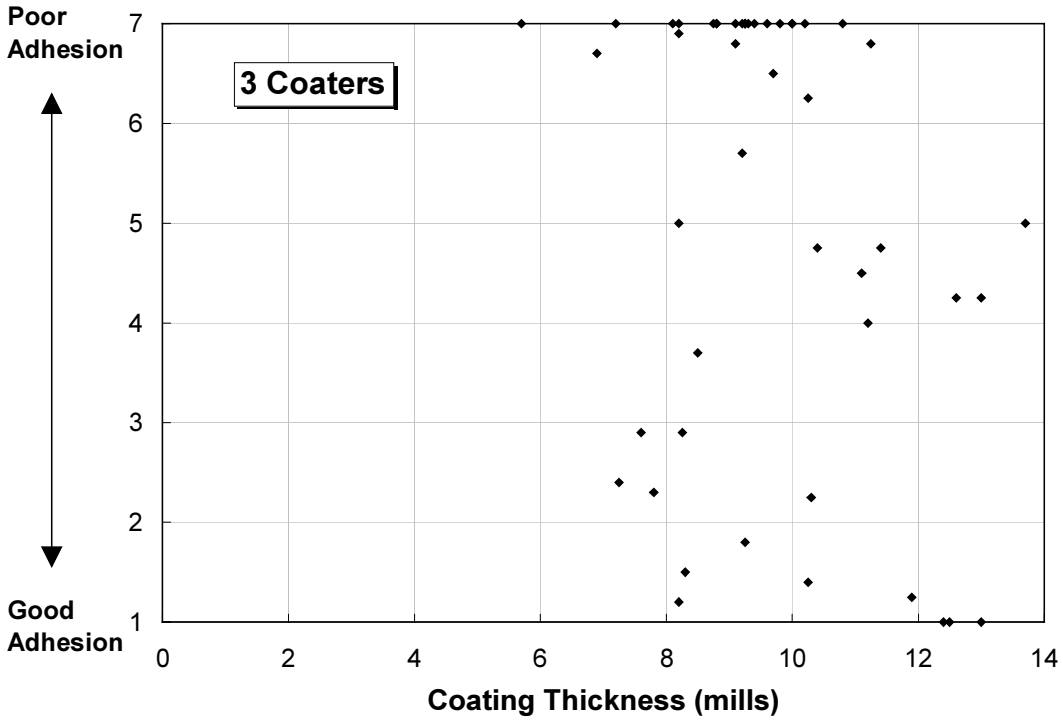


Figure 2.11 Adhesion rating vs. coating thickness of all specimens.

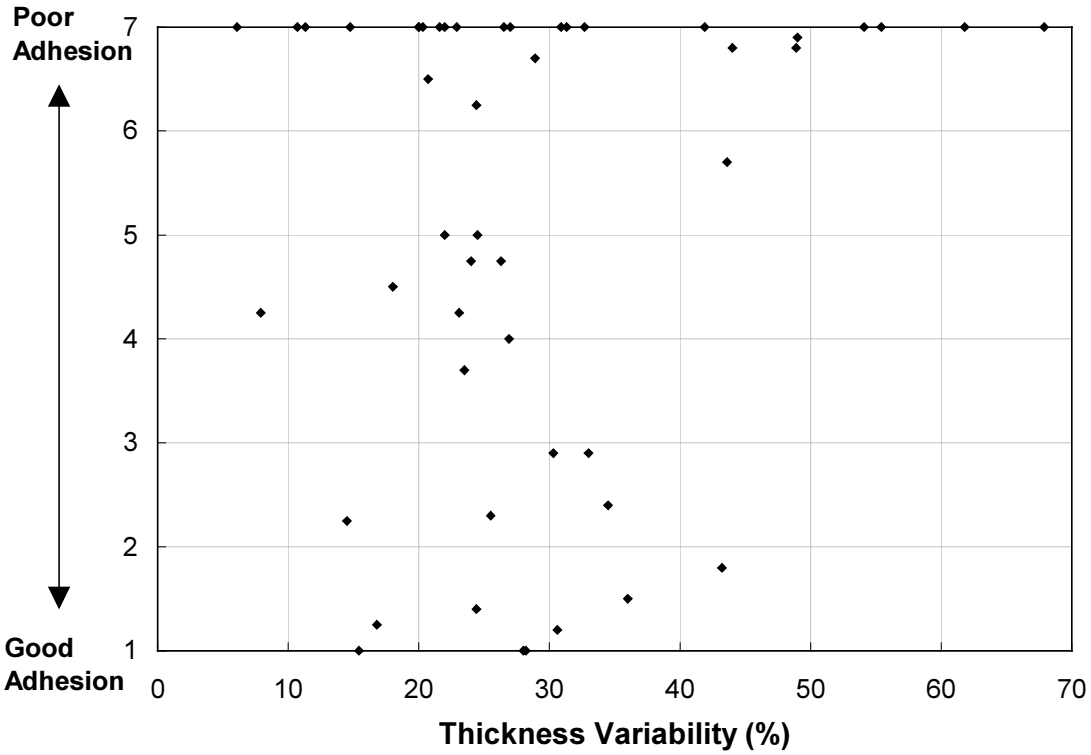


Figure 2.12 Adhesion rating vs. variability of coating thickness.

As mentioned previously, bars were immersed in hot water in their as-received condition. About one third of the specimens had some degree of coating damage and remaining specimens were undamaged. Both Swiss and MTO test procedures specify the use of bars free from holidays and bare areas. Therefore, it was of interest to observe the performance of bars that did not meet specifications. Some field studies reported in the literature showed that bars with damaged coating prior to exposure underwent worse adhesion loss than undamaged bars.[36] Loss of adhesion was observed at an intentional hole in the coating after hot solution immersion tests conducted by the FHWA.[6] Adhesion ratings of each individual specimen for coaters *A*, *B*, and *C* are plotted in Figures 2.13, 2.14, and 2.15. Data points are plotted in sequence and the horizontal axis only shows the sequence number of each sample. Black points represent specimens with coating damage and white points represent specimens with no damage in the coating. Figure 2.13 shows that most specimens from coater *A* were damaged and exhibited poor adhesion. One half of undamaged specimens showed poor adhesion and the other half showed better adhesion, with ratings between 2.4 and 3.7. Graphs for coaters *B* and *C* (Figures 2.14 and 2.15) showed more widespread scatter of data and no clear relationship between coating damage and coating adhesion. There were specimens with no coating damage and poor coating adhesion and specimens with damaged coating and excellent adhesion.

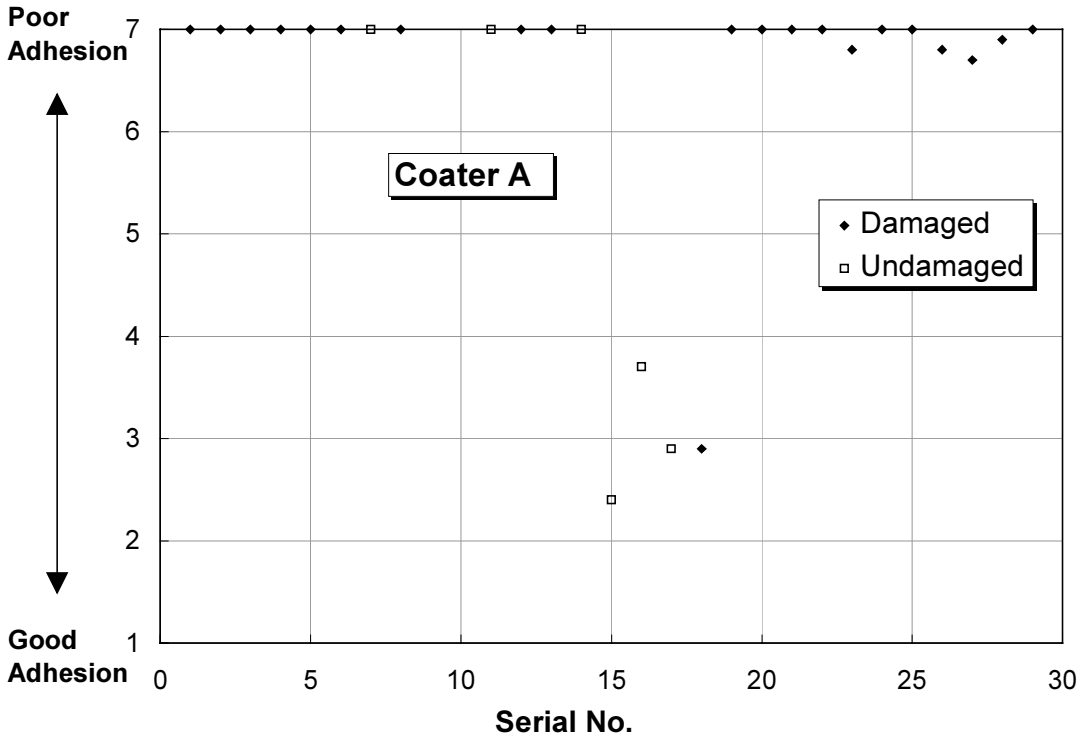


Figure 2.13 Effect of coating damage on coating adhesion (Coater *A*).

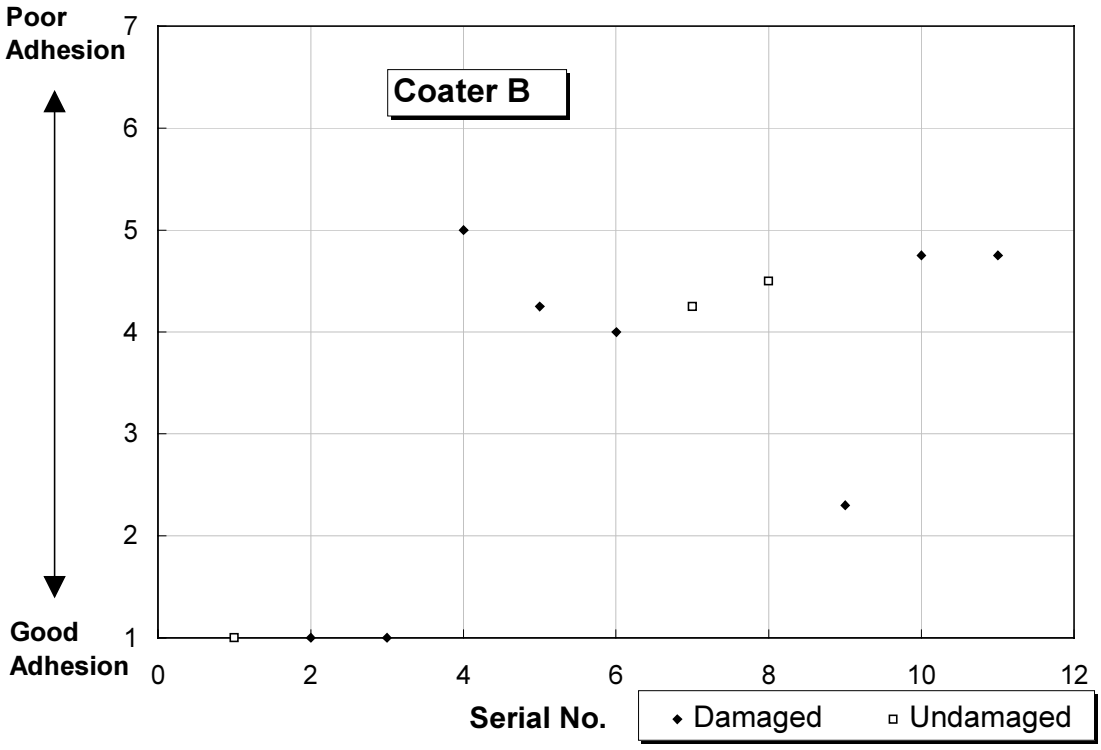


Figure 2.14 Effect of coating damage on coating adhesion (Coater *B*).

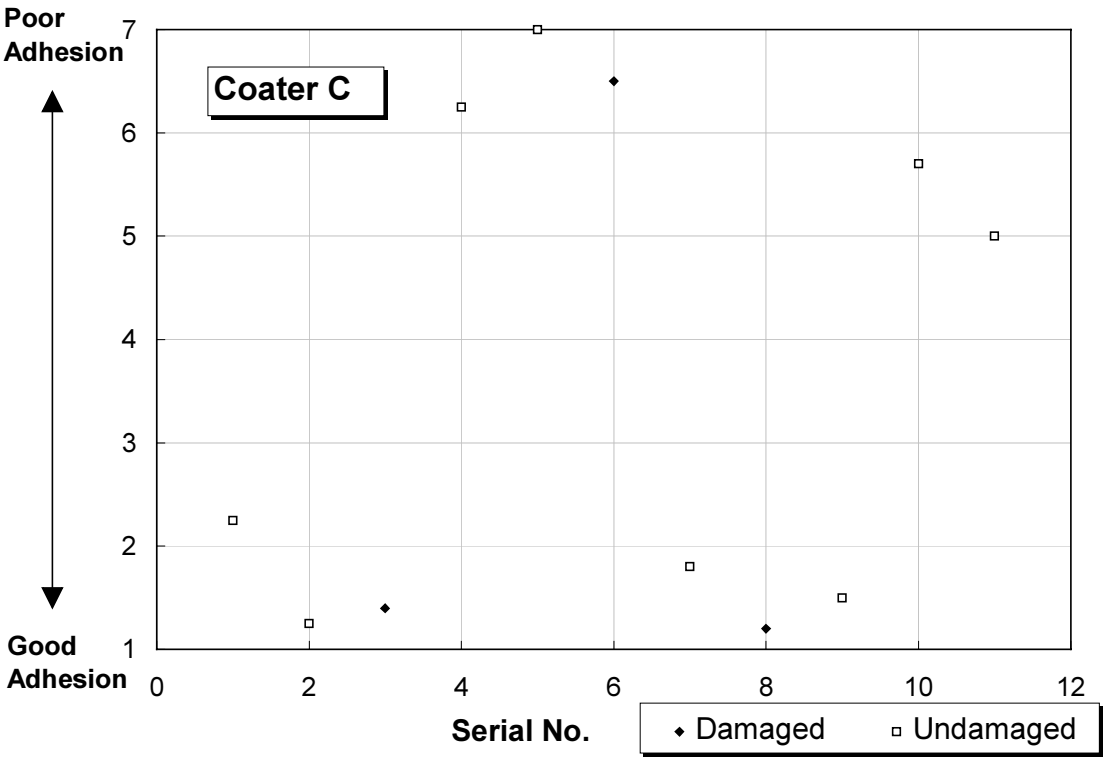


Figure 2.15 Effect of coating damage on coating adhesion (Coater *C*).

Tables 2.4, 2.5, and 2.6 contain adhesion ratings of specimens that were tested at different times after immersion in hot water. For each of those specimens, adhesion tests were performed typically 24 hours after immersion and either 40, 72, 90, 120 hours, or 2 months after immersion. In most cases, coating adhesion was either unchanged or slightly better when the test was performed at times longer than 24 hours after immersion. Examination of data from Tables 2.4, 2.5, and 2.6 reveals that most adhesion ratings were very similar and only in a few cases there was a drastic change (for the better or worse) in coating adhesion with respect to 24, 72, and 90 hours post-immersion times. Variability of adhesion ratings of tests conducted at varying post-immersion times was not significant and was similar to the variability of readings for tests conducted at a uniform post-immersion time of 24 hours.

Table 2.4 Adhesion ratings of tests conducted at varying post-immersion times.

Specimen	24 hours		72 hours		90 hours	
	NR	Avg. Adh. Rating	NR	Avg. Adh. Rating	NR	Avg. Adh. Rating
C23	4	7	2	6.5		
C24	4	7	2	7		
C25	4	7	2	7		
C26	4	7	2	6.5		
C27	4	7	3	6.3		
C28	5	7	2	6.5		
C29	6	7	2	7		
C32	3	1	2	1		
C33	2	6	1	4	1	4
C34	2	4.5	2	4		
C35	2	4			2	4
C36	2	3.5	2	5		
C37			2	5	2	4
C38	2	1.5			4	2.75
C39	2	4.5	2	5		
C40	2	4			2	5.5

NR: Number of readings.

Table 2.5 Adhesion ratings of tests conducted at 24, 40 and 120 hours post-immersion times.

Specimen	24 hours		40 hours		120 hours	
	NR	Avg. Adh. Rating	NR	Avg. Adh. Rating	NR	Avg. Adh. Rating
C41					4	2.25
C42	2	1.5			2	1
C43	2	1.5			3	1.3
C47			2	2	4	1.75
C48			2	1	4	1.25
C49			2	2.5	4	1

NR: Number of readings.

Table 2.6 Adhesion ratings of tests conducted at 24 hours and 2 months post-immersion times.

Specimen	24 hours		2 months	
	NR	Avg. Adh. Rating	NR	Avg. Adh. Rating
C6	6	7	2	7
C12	6	7	6	7

NR: Number of readings.

The knife force applied by an operator was not always constant. For instance, in samples with the best coating adhesion (ratings of 1 or 2), the actual applied force may have exceeded 4 kilograms. It is likely that the operator tended to push the knife strongly when the coating offered resistance to debonding. Despite the subjectivity of the procedure for estimation of knife force, the test seemed useful and produced some meaningful results. It should be emphasized, however, that only one test operator was involved. It may be expected that with more than one operator involved, applied knife force may vary significantly.

A common problem during the test was that of the coating ripping off as a result of: 1) high knife force, 2) slippage of knife, or 3) blade cutting through the coating. It was difficult to adequately interpret the results from these cases in terms of coating adhesion. Generally, only the area of coating lifted just before the coating tore was considered to have debonded. In cases where the knife slipped without tearing additional coating, the test force was re-applied at the position where the knife slipped. Any additional debonding was included in the test result. Such assessment was not always easy and required careful judgment. An interesting finding was that sometimes the blade could be inserted and advanced beneath the coating only for a short distance after maintaining the 3 kg force for 35 seconds; however, subsequent levering action of the blade would remove a larger portion of the coating. Another interesting

phenomenon was that at some flaps where adhesion was rated as "C" (poor), the coating would initially offer some resistance to the advancement of the blade, but after 20 to 30 seconds of maintaining the knife pressure, the coating would eventually yield and start peeling. This finding justified the procedure followed in this study for maintaining the knife pressure for at least 35 seconds. If this had not been done, some adhesion ratings may have been quite different.

The alternative adhesion rating system is compared to the MTO rating system in Figure 2.16. The average adhesion rating of each representative group of specimens was calculated using both rating systems and plotted on the graph. Since each rating system has a different range, the values had to be normalized so they could be plotted on the same graph. Normalization was done by first dividing the readings of the alternative system by 7 (the largest value of that system) to produce a range from 0.14 to 1.0. Subsequently, the values of the MTO system were converted to the normalized system by interpolation. A normalized rating of 1.0 represents poor adhesion and a normalized rating of 0.14 indicates good adhesion. It can be seen that curves representing each system follow very similar trends. The largest difference between the two ratings was 0.18 and the average difference was 0.06. Consistently, the MTO rating system gave equal or worse adhesion ratings than the alternative system. This indicates that the MTO system tends to be more stringent in certain cases. As opposed to the system in the first MTO draft, the newer rating system was devised to be very simple and easy to use but, because of its simplicity, it would be expected to err on the safe side.

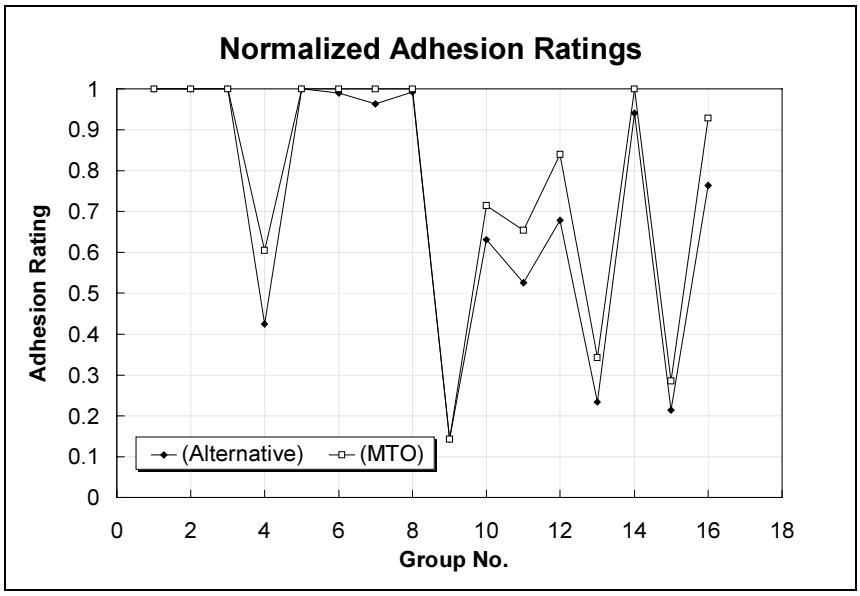


Figure 2.16 Comparison of adhesion rating systems.

2.3 CONCLUSIONS FROM FIRST PHASE

The main finding in the First Phase was that hot water-adhesion tests were useful in discriminating and differentiating good from bad coatings. Adhesion test results best correlated with sample source (coater). Bar diameter did not influence test results. In all cases, straight bars performed better than bent bars. For the range studied, coating thickness and thickness variability did not correlate with adhesion performance. Adhesion test results did not have any correlation with original coating condition. Test results were not significantly affected by changes in post-immersion time. A rating to evaluate adhesion test results was devised based on ease of use and practicality. An important issue to address in the next phases was defining a limiting adhesion rating as acceptance criterion for quality assurance. The tests were relatively easy to perform and did not require special or sophisticated equipment.

CHAPTER 3

Second Phase: Hot Water-Adhesion Test Modifications

In the Second Phase, additional test variables were studied and the repeatability of the test was improved by eliminating or reducing factors that make the test subjective. Four major areas were addressed: 1) repeatability, 2) effect of immersion time in hot water, 3) evaluation of knife blades and force calibration, and 4) effect of test operator.

Another important issue relates to the subjectivity of the procedure used in the First Phase for estimating and controlling the amount of force applied to the knife. It was important to develop a more reliable and objective procedure so that different operators apply approximately the same pressure to the knife.

3.1 SPECIAL DEVICE TO CONTROL KNIFE FORCE

Adhesion tests reported in Section 3.2 were conducted using a special device to control the applied knife force. A device was manufactured for this study and consisted of a wooden assembly in which a long, thin, flexible plywood strip was mounted so that it could deflect in the horizontal direction. The ends of the strip were free to rotate (simply supported). The bar specimen was then fixed to the plywood strip with a hose clamp at each end. A short chamfer was fixed at the mid-length of the plywood strip to hold the bar in place. During testing, the operator applied sufficient knife pressure to the epoxy to deflect the plywood strip laterally until it touched a limit, which was indicated using a nail. The device was calibrated so that the desired of knife force was reached when the plywood strip touched the nail. A sketch of this special device is shown in Figure 3.1.

The procedure for calibrating the special device was as follows: The device was turned so that the plywood strip (with the specimen mounted to it) deflected downwards. A known weight was positioned above the specimen and the amount of deflection was measured. The nail head protruded to the desired point. The calibration process was performed for each specimen because clamping the bars stiffened the plywood strip and it was not possible to clamp each bar identically.

The weight selected for calibration was based on the following considerations: X cuts at each test site were oriented as illustrated in Figure 3.1. The knife was positioned approximately 45° with respect to the bar axis in a horizontal plane, and at approximately 30° with respect to a tangential plane at the point of contact with the bar. When the knife was pushed against the bar, one of the three orthogonal components of the knife force produced lateral deflection of the plywood strip. The horizontal component is F_{knife} cos30° cos45°. For F_{knife}= 3.5 kg, the horizontal component is 2.14 kg.

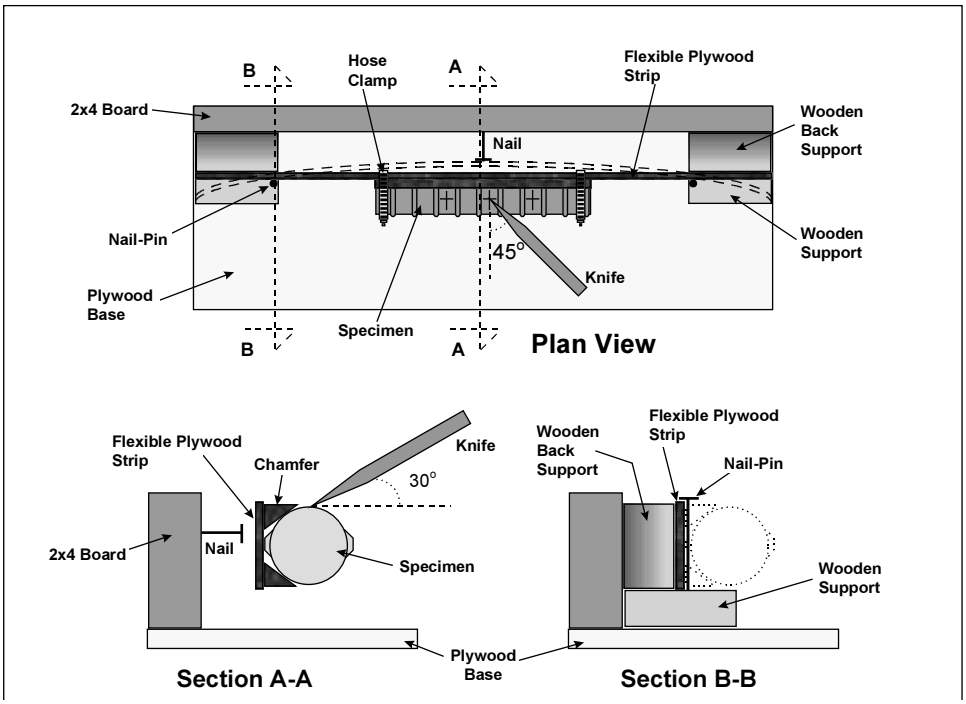


Figure 3.1 Special device to calibrate knife force.

3.2 TEST REPEATABILITY

One crucial question is how well an individual adhesion test represents the general properties of the coating application for a specific production run. The variability of coating adhesion throughout the length of a rebar is unknown. If adhesion test results from different portions of a rebar are available, variability may be due to the coating process itself, to inherent test errors, or to a combination of both.

Study Variables and Test Results

Main variables included bar source (coating plant) and location (Figure 3.2) along bar from which the sample was obtained. Hot water and adhesion tests were conducted on straight epoxy coated bar samples from coating plants *B* (#10) and *C* (#9). For each coater, 8 specimens were cut from 2 rebars (eight feet long) at several locations along the rebar, typically 3 specimens from both bar ends and one specimen from the middle portion (Specimens 1, 2, 3, and 4 in Figure 3.2). Six tests were conducted (three on each side) for each sample. Comparison of test results from different bar locations gave an indication of the variability of coating adhesion along an individual rebar. Test parameters included temperature of 75°C for water bath, 48 hours of immersion, 24 hours of post-immersion, and 3.5 kg of knife force.

A summary of adhesion ratings and average values for all specimens is included in Table 3.1. Statistical values based on all individual adhesion ratings are summarized in Table 3.2.

Specimens 1 and 2 were located at one end of the bar, specimen 3 was located at the middle portion of the bar, and specimen 4 was located at the other end of the bar (Figure 3.2). From Tables 3.1 and 3.2, the data show that on average, bars from coater *B* had better coating adhesion than bars from coater *C*. Also, all specimens from coater *B* performed better than those from coater *C*.

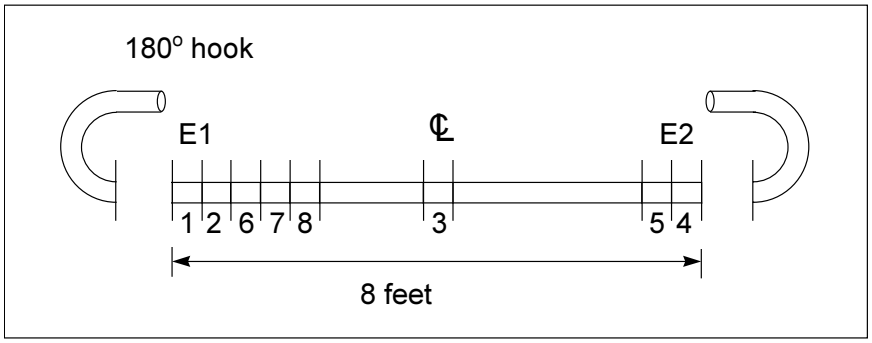


Figure 3.2 Location of specimens obtained from epoxy-coated bar.

The observations indicate that coating adhesion tends to be relatively uniform for specimens cut from the same bar at random locations. For coater *B*, bar I had an average rating of 1.5 and bar II of 2.1 and nearly every specimen from bar II had worse average adhesion ratings than those from bar I. For coater *C*, bar I had an average rating of 5.4 and bar II of 2.9 and all specimens from bar I had worse average ratings than those from bar II.

The statistical values in Table 3.2 include some indicators of the variability of the data, such as the standard deviation, coefficient of variation, and range. Standard deviation values ranged from 0.72 to 1.21 and coefficient of variation values ranged from 22.5% to 48%. Standard deviations are greater for bars from coater *C* and bars had greater adhesion ratings (lower adhesion). If standard deviation is divided by mean adhesion and expressed in percentage, the resulting value is the coefficient of variation. As opposed to standard deviations, coefficients of variations are greater for bars from coater *B.* The discrepancy comes from the fact that the magnitude of the mean significantly affects the coefficient of variation. The coefficient of variation is a relative measure of how large the standard deviation is with respect to the mean. The standard deviation is a measure of the dispersion of data in absolute terms and, therefore, provides a better indication of the variability of the readings. Bars from coater *B* had a range of adhesion ratings of 2 and bars from coater *C* had a range of 4.

A statistical analysis indicates that bars with lower adhesion strength had greater variability. This is not surprising since low adhesion may be the result of an inadequate quality control in the coating process. The obvious exception would be a coating with very poor adhesion all along a rebar, in which case there is very little variability of adhesion ratings yet the quality is unacceptable. If adhesion ratings are to be used as quality indicators, both the mean rating and standard deviation have to be examined.

Table 3.1 Adhesion ratings for all specimens using rating system of Table 2.3.

Specimen	Coater *B*				Coater *C*			
	Bar I		Bar II		Bar I		Bar II	
	Side A	Side B	Side A	Side B	Side A	Side B	Side A	Side B
1	1	1	2	2	5	3	3	3
	1	1	3	3	6	4	4	4
	1	1	3	3	7	4	5	3
Average	1		2.7		4.8		3.7	
2	3	3	1	1	6	6	4	3
	1	1	1	2	6	4	3	1
	2	1	3	1	7	4	4	2
Average	1.8		1.5		5.5		2.8	
3	2	2	3	2	5	6	3	2
	2	1	2	1	5	6	2	2
	2	1	3	2	7	5	4	1
Average	1.7		2.2		5.7		2.3	
4	1	2	3	1	6	4	3	3
	1	3	3	1	7	4	3	4
	1	1	3	1	7	5	1	2
Average	1.5		2.0		5.5		2.7	

Table 3.2 Statistical analysis of all individual ratings of Table 3.1.

Parameter	Coater *B*		Coater *C*	
	Bar I	Bar II	Bar I	Bar II
Mean	1.5	2.1	5.4	2.9
Median	1	2	5.5	3
σ	0.72	0.88	1.21	1.08
Cov = $\tilde{\sigma}$Mean (%)	48	42	22.5	37
Range	2	2	4	4

A.D: Average deviation of all ratings
σ: Standard deviation of all ratings

Examination of Table 3.1 shows that a lower number of tests per sample would have resulted in a less precise indication of the overall adhesion of a bar, especially for bars with greater dispersion of data. Small specimens removed from a long epoxy-coated bar, give representation results provided that several tests are performed on each specimen cut from that bar.

It should be noted that both bars from coater *C* came from the same production lot, yet the adhesion of bar I was worse than that of bar II. To properly evaluate a production lot, samples should be obtained from as many different bars as possible so that results are representative of a given lot.

Although average ratings per sample had low variability, Table 3.1 shows that individual ratings may vary significantly within the same specimen. Coating adhesion was not always uniform and usually varied along a bar. Variation of coating adhesion is affected by two factors:

a) Variability produced by the coating application because of inconsistencies of the coating material, uneven surface preparation, temperature differentials, uneven application, or improper curing.

b) Variability produced by adhesion testing because of human error, inaccuracy of the testing method, testing conditions, or sampling procedure.

It is extremely difficult to identify and separate the factors affecting the variability of coating adhesion, making the task of developing and improving the adhesion test particularly complex. It is possible to assume, however, that coating application probably accounts for most of the variability if operator subjectivity is eliminated or reduced from the test.

The issue of test repeatability will be re-addressed in subsequent sections after more test results are presented.

3.3 IMPROVED SPECIAL DEVICE TO CONTROL KNIFE FORCE

For the series of tests reported in sections 3.4, 3.5, and 3.6, the adhesion test device previously used was further improved. The main disadvantage of the device was that very frequent calibration was required. The main change consisted of separating the specimen from the deflecting flexible strip so that the stiffness of the flexible strip was constant. The specimen was mounted and fixed inside a rigid, sturdy wooden assembly supported on metal rollers to allow translation (Figure 3.3). The plywood strip was replaced by an acrylic strip. With the operator exerting pressure with the knife, the whole bar-assembly moves and pushes the flexible acrylic strip until it reaches the desired deflection. The end supports of the acrylic strip were fixed with clamps instead of being simply supported to make the test easier to control. Less deflection (and less translation of the bar-assembly) is needed to achieve the desired force. Calibration procedure was the same as before but the frequency of calibrations was greatly reduced. The improved device was calibrated once per working session.

Another modification in the adhesion test procedure consisted of changing the orientation of the X cuts on the bar surface. The new orientation, illustrated in Figure 3.3, allowed the knife force to be applied normal to the bar in the direction of deflection of the acrylic strip. With the earlier X orientation, the knife was aligned at an angle with respect to the deflection of the plywood strip (Figure 3.1) and made the test

more difficult to perform and control. The drawback of the new orientation of X cuts is that only two flaps (aligned perpendicular to the bar axis) can be tested. The other two flaps are aligned parallel to the bar axis and cannot be tested.

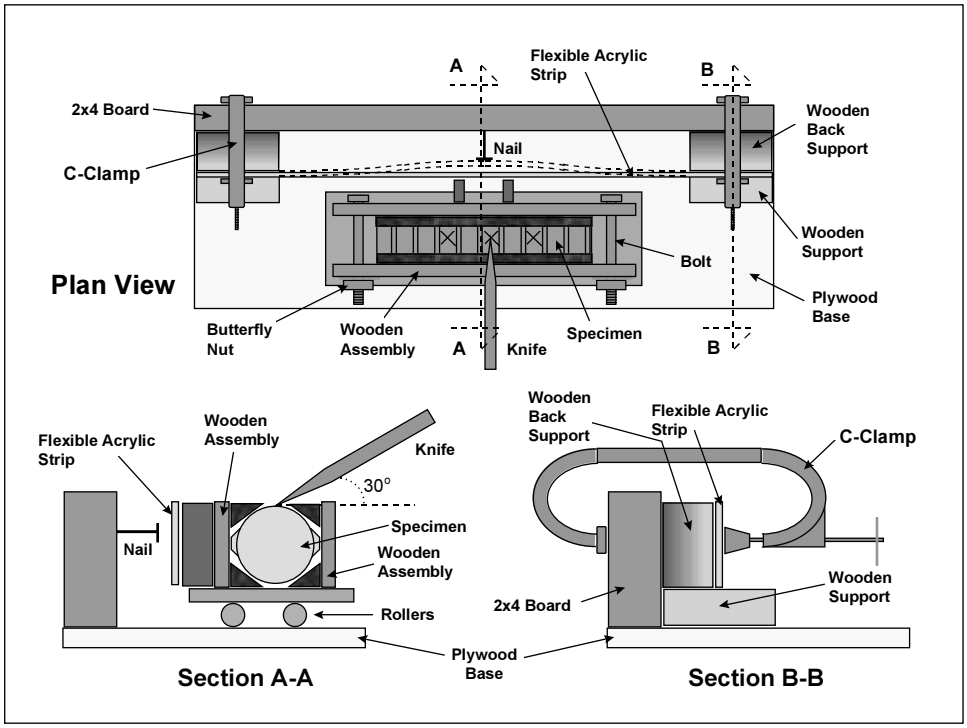


Figure 3.3 Improved device to calibrate knife force.

The modified device was calibrated for a knife force of 3.5 kg. With the knife oriented at 30° with respect to the direction of movement, a 3-kg weight (F_{knife} cos30° = 3.5 cos30° = 3 kg) was used for calibration.

3.4 TIME OF IMMERSION

Most hot water immersion tests have been conducted submerging the specimens for 48 hours. To determine the significance of immersion time, adhesion tests were conducted after several times of hot water immersion. A particular objective of this series of tests was to define an optimal time of immersion.

Study Variables and Test Results

Straight specimens from coaters *B* and *C* were submerged in hot water for the following periods of time: 2, 8, 24, and 48 hours. Specimens were obtained from the same two rebars from each coater as in the previous set of tests. Three samples were cut from one bar end and one sample from the opposite bar end (specimens 5, 6, 7, and 8 in Figure 3.2). Previous test results were used for the 48-hour data and the new samples were immersed for 2, 8, and 24 hours.

The adhesion rating used in previous sections was based on the combination of results from four flaps. Since only two flaps per site were tested, the adhesion rating was modified as shown in Table 3.3 to give a rating based on the combination of results from two flaps.

Since previous test results were used for the 48-hour data, their adhesion rating was re-evaluated according to the modified rating system. The sites were re-evaluated by arbitrarily considering every pair of directly opposite flaps as one test sub-site, resulting in two adhesion ratings per test location of four flaps (Figure 3.4).

Table 3.3 Modified adhesion rating system.

Sub-rating	Description
A	Unable to insert blade tip under the coating
B	Blade tip can be inserted under the coating. Levering action removes small chips of coating but cannot remove the entire coating
C	Blade tip slides easily under the coating and the entire coating can be removed

Adhesion Rating	Description
1	Sub-rating A at 2 flaps (Good adhesion)
2	Sub-rating A at one flap and B at the other flap
3	Sub-rating B at 2 flaps
4	Sub-rating A at one flap and C at the other flap
5	Sub-rating B at one flap and C at the other flap
6	Sub-rating C 2 flaps (Poor adhesion)

Adhesion test results for all specimens are summarized in Table 3.4 and a graph of average adhesion ratings versus time of immersion for each bar is shown in Figure 3.5. Analysis of Figure 3.5 indicates that there was no consistent trend in adhesion ratings with respect to time of immersion. Only bar I from coater *C* showed decreased coating adhesion with longer times of immersion. The remaining bars showed either no significant change or a slight improvement in coating adhesion with longer immersion times. In all specimens, there was little difference between adhesion ratings after 8, 24, and 48 hours of immersion. The large jump between 2 and 8 hours of immersion for bar I from coater *C* may indicate that 2 hours of immersion may not be sufficient exposure.

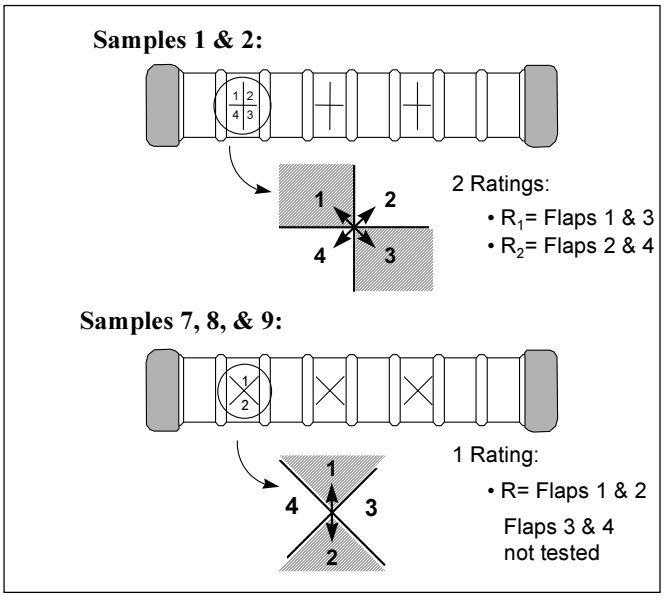


Figure 3.4 Convention for rating test sites using modified adhesion rating system from 1 to 6.

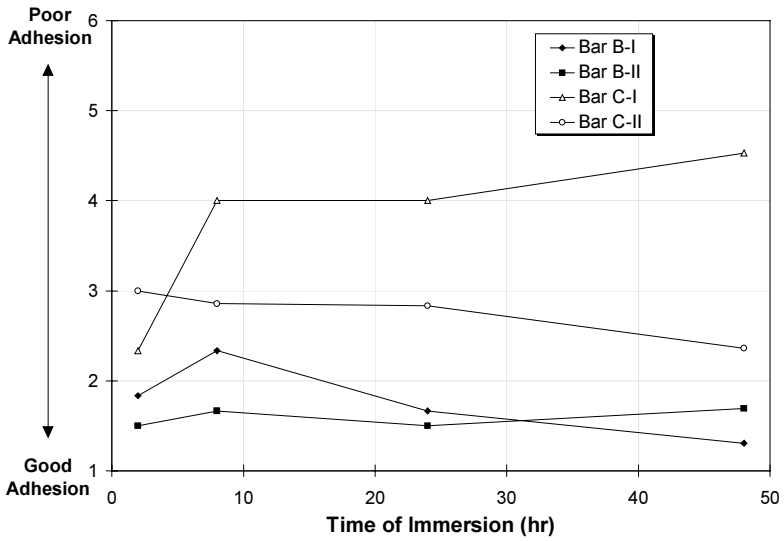


Figure 3.5 Effect of hot water immersion time in coating adhesion.

Table 3.4 Adhesion ratings of specimens immersed at variable times.

Sample	Coater *B*				Coater *C*				Time Immer
	Bar I		Bar II		Bar I		Bar II		
	Side A	Side B	Side A	Side B	Side A	Side B	Side A	Side B	
4	1 , 1 1 , 1 1 , 1	1 , 2 2 , 2 1 , 1	3 , 2 3 , 3 3 , 3	1 , 1 1 , 1 1 , 1	6 , 6 6 , 6 6 , 6	3 , 3 5 , 5 3 , 5	2 , 3 3 , 2 1 , 2	2 , 2 3 , 3 1 , 2	48 hr
Avg.	1.25		1.9		5		2.2		
5	1 1 1	2 2 3	2 2 2	1 1 1	5 5 6	2 3 3	2 3 3	3 3 3	24 hr
Avg.	1.7		1.5		4		2.8		
1	1 , 2 1 , 1 1 , 1	1 , 1 1 , 1 1 , 1	2 , 1 3 , 2 2 , 2	1 , 2 2 , 2 2 , 2	5 , 3 6 , 5 6 , 6	3 , 2 3 , 3 3 , 3	1 , 3 3 , 3 5 , 3	2 , 3 3 , 3 2 , 3	48 hr
Avg.	1.1		1.9		4		2.8		
2	2 , 3 1 , 1 2 , 1	2 , 3 1 , 1 1 , 1	1 , 1 1 , 1 2 , 2	1 , 1 2 , 1 1 , 1	5 , 5 5 , 5 6 , 6	5 , 6 3 , 3 3 , 3	3 , 3 2 , 2 3 , 3	2 , 2 1 , 1 1 , 2	48 hr
Avg.	1.6		1.25		4.6		2.1		
6	3 1 2	2 3 3	1 3 2	2 1 1	5 6 4	3 3 3			8 hr
Avg.	2.3		1.7		4				
7			2 2 2	1 1 1			2 3 3	3 3 3	2 hr
Avg.			1.5				2.8		
8	2 1 2	2 1 3			2 2 3	2 2 3	3 3 3	3 3 3	2 hr
Avg.	1.8				2.3		3		

In Table 3.5, a statistical analysis of mean adhesion ratings of samples with varying immersion times is presented (samples 1, 2, 4, 5, 6, 7, and 8 in Figure 3.2). Table 3.6 includes a statistical analysis of mean adhesion ratings of samples tested after 48 hours of immersion (samples 1, 2, 3, and 4 in Figure 3.2). Samples with different immersion times would be expected to have greater variability than samples with the same immersion time. A comparison of Tables 3.5 and 3.6 shows that this was not always the case.

There were cases where samples with the same immersion time (48 hours) had greater variability than samples from the same bar but with varying immersion times (bars "II" from both coaters).

Table 3.5 Statistical analysis of mean ratings of samples -all times of immersion included.

Parameter	Coater *B*		Coater *C*	
	Bar I	Bar II	Bar I	Bar II
A.D.$_M$	0.299	0.090	0.691	0.201
σ_M	0.370	0.091	0.826	0.241
Range$_M$	1.03	0.19	2.19	0.64

A.D.$_M$: Average deviation of mean ratings
σ_M: Standard deviation of mean ratings

Table 3.6 Statistical analysis of mean ratings of samples tested after 48 hours of immersion.

Parameter	Coater *B*		Coater *C*	
	Bar I	Bar II	Bar I	Bar II
A.D.$_M$	0.25	0.33	0.271	0.396
σ_M	0.312	0.417	0.32	0.491
Range$_M$	0.83	1.167	0.83	1.33

A.D.$_M$: Average deviation of mean ratings
σ_M: Standard deviation of mean ratings

Considering that there is little difference between adhesion ratings of specimens tested after 8 or more hours of hot water immersion, performing a hot water test with shorter immersion has the advantage of reducing the duration of the test compared with the specified MTO procedure. A 24-hour immersion period permits a convenient test schedule. Samples immersed early in the morning can be removed at the same time the next day. After an additional post-immersion period of 24 hours, samples can be tested the following day. This test variable will be re-addressed in the Third Phase (Chapter 4) after more test results are presented.

3.5 EVALUATION OF KNIFE BLADES AND KNIFE FORCE CALIBRATION

Thus far, adhesion tests have been performed with a utility knife with snap-off blades. As mentioned earlier, such blades are difficult to use because the knife has to be held at an oblique angle with respect to the direction of the path that the blade has to follow (along an imaginary line bisecting the flap of coating). For the test operator, this is an awkward and uncomfortable position. The search for a better knife design was an integral part of developing an improved adhesion test. A series of tests was conducted to evaluate several types of knife blades.

Another area of interest was to evaluate the procedure for calibrating the applied knife pressure. The subjective procedure used in the First Phase was compared with the procedure involving the modified test device. It was important to know if test results became less susceptible to human error with the use of the special device.

Study Variables

A series of hot water-adhesion tests was conducted on six samples from 2 coating applicators. Specimens from each applicator came from the same batch. The two variables evaluated were the knife blades and knife force calibration procedures.

Description of knife blades (Figure 3.6)

a) *Utility knife with snap-off blades*: A typical knife consists of connected mini-blades. As a blade tip becomes blunt, a new sharp blade is available by breaking off the blade tip. The knife is economical and widely available.

b) *X-acto knife with # 11 blade*: X-acto knifes are widely used by architects and artists. They have the advantage that a wide variety of blades can be mounted. Blade # 11 is pointed with a triangular shape and has one sharp edge.

c) *X-acto knife with #17 blade*: A chisel blade with long rectangular shape. The blade is flat and has a sharp edge.

d) *X-acto knife with #23 blade*: A pointed blade with a spade shape and two sharp edges.

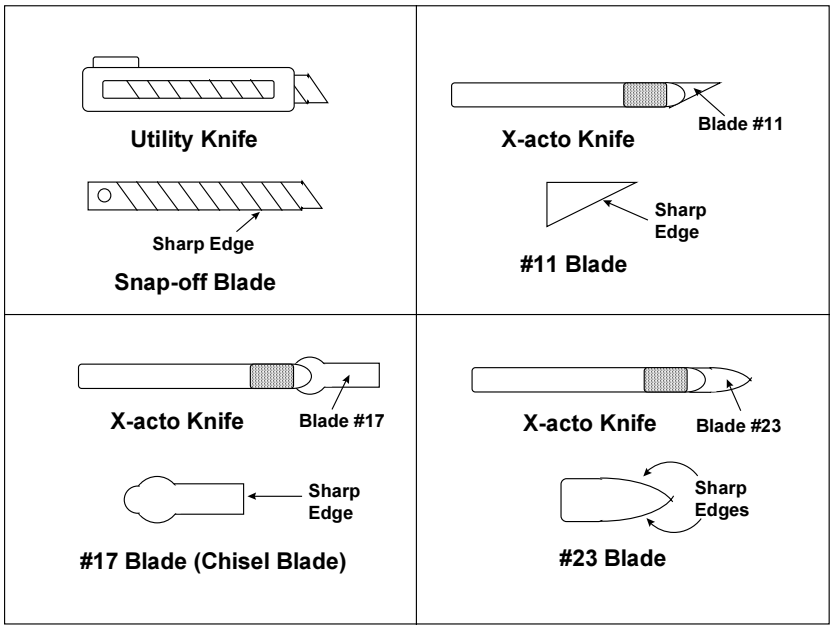


Figure 3.6 Types of knife blades used for adhesion study.

At most test sites, two types of blades were used (one blade type per flap), as illustrated in Figure 3.7 to facilitate comparisons.

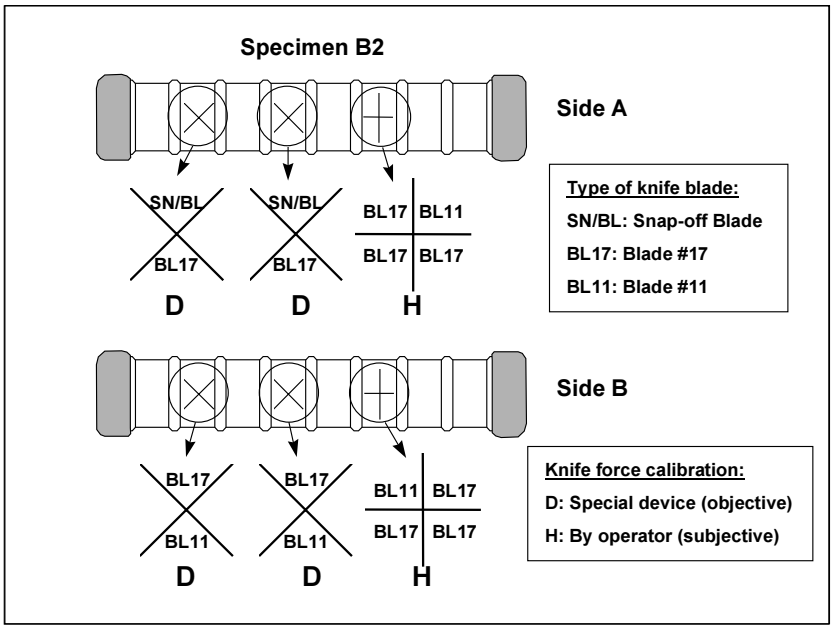


Figure 3.7 Typical specimen for adhesion test using several types of knife blades and force calibration procedures.

Procedures for knife force calibration

a) *Procedure H*: The operator calibrated the applied force by pushing the knife against a digital scale and then emulated that force for the test. The procedure was used before the introduction of the special device.

b) *Procedure D*: The procedure was used in the previous set of tests. The specimen was mounted on the improved special device and when the knife was pushed against the specimen, an acrylic strip was deflected to a predetermined amount.

The two procedures were used on each of the 6 specimens. On each specimen, four sites were tested following procedure *D* and two sites with procedure *H* (Figure 3.7). As shown in the sketch, the X cuts tested with procedure *D* were oriented differently than the X cuts tested with procedure *H*. As has been explained before, the orientation of X cuts shown for procedure *D* made the test easier to perform because the knife moves more parallel to the deflection of the acrylic trip. The drawback was that only two flaps per site are tested. Procedure *H* was not affected by the X orientation and four flaps could be tested. The reason the X cuts for procedure *H* were oriented differently was to facilitate their identification.

Test Results and Discussion

Knife Blades

Table 3.7 contains adhesion sub-ratings for individual coating flaps tested with several types of blades. The description of each sub-rating is included for reference. The #17 blade mounted on an X-acto knife seemed to produce more *B* and *C* sub-ratings than other blades on samples tested with calibration procedure *H*. It should be emphasized that variability of ratings produced by different blades was not greater than the variability observed when only one type of blade was used. No particular blade seemed to give consistently higher or lower ratings.

Table 3.7 Adhesion sub-ratings for individual flaps tested with different blades.

	Procedure *H*				**Procedure *D***			
Sample	**SN/BL**	**BL11**	**BL17**	**BL23**	**SN/BL**	**BL11**	**BL17**	**BL23**
B1					A A	A A		
B2		A A	B B B B B B		A A	B B	A B A B	
B3	A B			A A A A A B	A A			A A
C1	A A	A A			A A	A A		
C2	A B		B B		B C B C		B C C C	
C3	A A			A B	A B A			B B B

SN/BL: Snap-off blade BL17: Blade #17
BL11: Blade #11 Bl23: Blade #23

Sub-rating	**Description**
A	Unable to insert blade tip under the coating
B	Blade tip can be inserted under the coating. Levering action removes small chips of coating but cannot remove the entire coating
C	Blade tip slides easily under the coating and the entire coating can be removed

Evaluation of blades was based on ease of use and cost. The worst blade for adhesion testing was the #17 blade mounted on an X-acto knife. The chisel blade does not have a pointed tip, making it difficult to insert the blade under the coating and, consequently, has a propensity to tear or cut through the coating. The blade was very difficult to use and was expensive.

The #11 blade mounted on an X-acto knife was very long and was not stiff enough to adequately control the knife force. Besides, it only had one sharp edge and the triangular shape was not symmetrical. With

such geometry, the knife had to be aligned at an angle with respect to the path that the blade has to follow under the coating (an imaginary line bisecting the flap of coating). The operator had to perform the test holding the knife in an awkward and uncomfortable position. The blade was also expensive.

The plastic utility knife with snap-off blades, like blade #11, had to be positioned at an awkward angle with respect to the direction the blade had to follow. Its main advantage is that new sharp blades are readily available and it is economical.

The #23 blade on an X-acto knife was found very suitable for adhesion testing. Its symmetrical design with two curved, sharp edges made it possible to position the knife parallel to the path that the blade had to follow. The blade was very stiff and robust, making it easy to control and maintain the knife force. The main drawback was that the blade was very expensive.

All tests in the first phase were conducted with the plastic utility knife with snap-off blades. Most of the tests in the second phase were performed with an X-acto knife with a #23 blade. The plastic knife with snap-off blades was found very suitable for making the X cuts through the coating and was used for that purpose in the second and third phases. The X-acto knife with blade #23 was the basis for a new test knife developed and used for adhesion tests in the third phase.

Procedure for Calibration of Knife Force

Figures 3.8, 3.9, and 3.10 show average adhesion ratings of specimens tested using procedures *H* and *D* for calibrating the knife force. For specimens from coating applicator *B*, there is little difference in adhesion ratings between procedure *H* and *D*. For specimens from coating applicator *C*, much higher ratings were obtained with procedure *D* on two specimens (especially on specimen C2). However, despite some large differences in some specimens, the difference in overall average adhesion ratings produced by procedures *D* and *H* is not significant (2.125 and 1.75 respectively). In fact, if specimen C2 is omitted, the overall average of adhesion ratings would be 1.55 for both procedures.

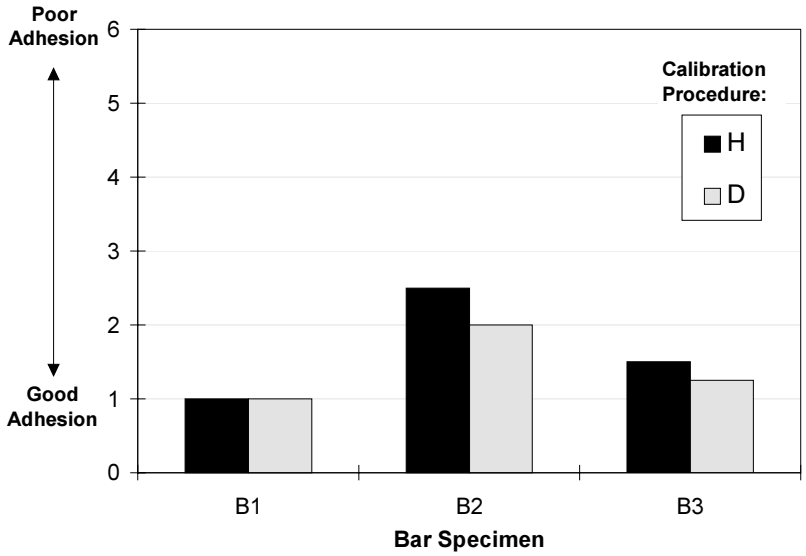


Figure 3.8 Effect of procedures to calibrate knife force on adhesion test results (samples from coater *B*).

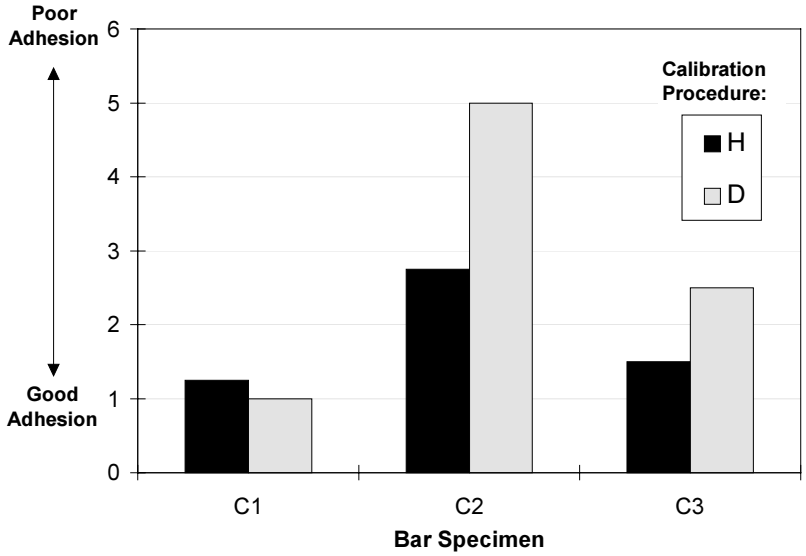


Figure 3.9 Effect of procedures to calibrate knife force on adhesion test results (samples from coater *C*).

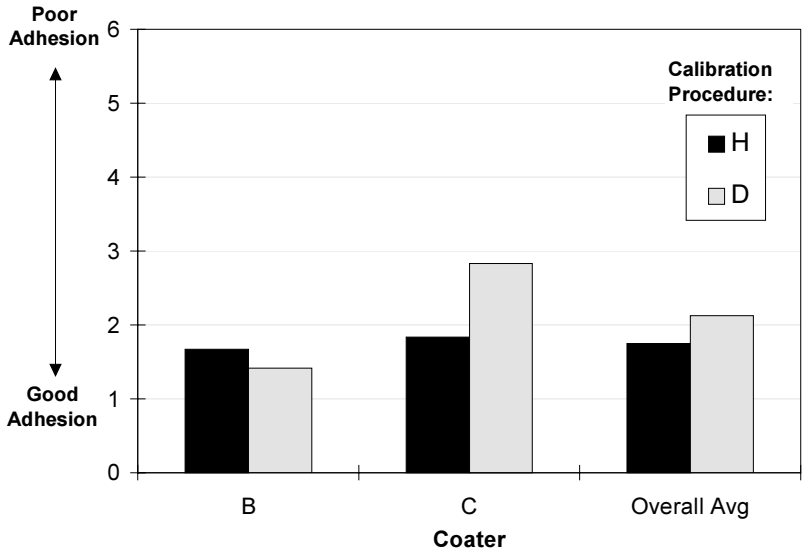


Figure 3.10 Effect of procedures to calibrate knife force on adhesion test results (all samples).

There was more dispersion of adhesion ratings when samples were tested following calibration procedure *D*, as evidenced by the higher standard deviation (Table 3.8). If specimen C2 is omitted, there is less difference in standard deviation: 0.67 for *H* versus 0.76 for *D*. The higher variability of results obtained with procedure *D* could mean that procedure *D* reflects adhesion characteristics better than procedure *H*. It may be possible that with procedure *D*, areas of poor coating adhesion are more easily detected, resulting in a greater variability of adhesion ratings compared to procedure *H*. Therefore, bars with poor quality could be more readily identified by procedure *D*.

Table 3.8 Standard deviation of adhesion ratings on samples tested by calibrating the knife force with procedures *H* and *D*.

Coater	Procedure *H*	Procedure *D*
B	0.78	0.67
C	0.83	1.90
Overall	0.79	1.57

There have been many questions and doubts regarding the validity of adhesion tests, mainly because of the subjectivity involved in the test procedure. One subjective factor that has been widely pointed out is that the amount of pressure applied with the knife is judged by the operator, thus introducing human error. Despite the subjectivity involved in procedure *H* versus the more objective procedure *D*, the overall mean adhesion ratings were similar. Although the number of tests is small, the results seem to indicate that coating adhesion testing can be useful and meaningful even if some subjectivity is involved. With practice, a test operator should be able to calibrate the force and produce reliable test results.

3.6 EFFECT OF TEST OPERATOR

An important concern refers to the variability of results obtained by different test operators. If different operators produce widely different results, adhesion testing would be unreliable. An acceptable test should yield similar results regardless of the operator, especially if adhesion testing is to become a standard procedure in epoxy-coated bar specifications.

Study Variables

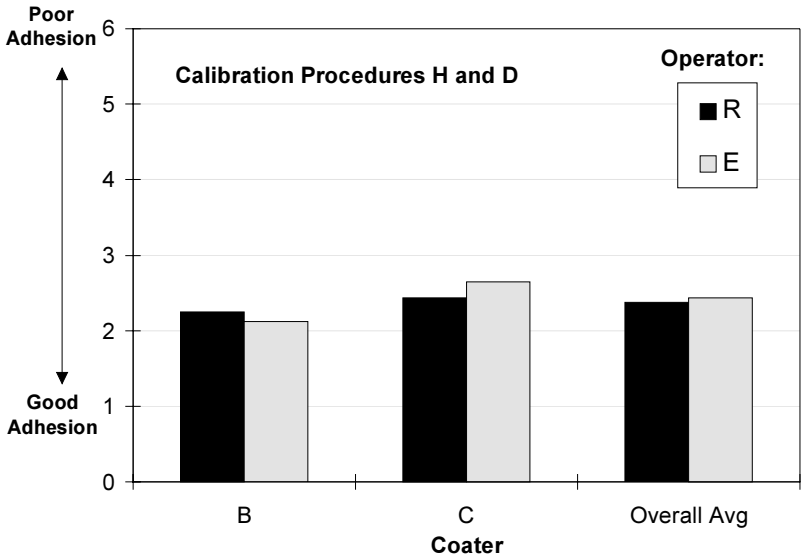
A set of hot water and adhesion tests was conducted on 6 epoxy-coated bar specimens from two coating applicators. Specimens from each coater were cut from the same rebar. Two operators performed adhesion tests on all specimens and each operator followed calibration procedures *H* and *D* to measure and control the amount of force applied with the knife. Adhesion test results were evaluated by each operator using the modified rating system from 1 to 6. It was of interest to verify that the system provides consistent ratings regardless of the person making the evaluation.

Test Results and Discussion

A summary of mean adhesion ratings produced by each operator for each sample is listed in Table 3.9. Mean ratings for both operators were very similar for all samples, with a highest difference of 0.58. In four out of six cases, mean adhesion ratings for operator *E* were higher (lower adhesion) than for operator *R*. As shown in Figure 3.11, the overall difference in mean adhesion ratings between the two operators was only 0.06.

Table 3.9 Mean adhesion ratings for operators *R* and *E*.

Specimen	Operator *R*	Operator *E*	Difference (*E*-*R*)
OP1	2	2.5	+0.5
OP2	3	2.5	-0.5
OP3	2	2.25	+0.25
OP4	2.75	3.33	+0.58
OP5	1.75	1.25	-0.5
OP6	2.75	3	+0.25



Coater *B*: Samples OP5, OP6
Coater *C*: Samples OP1 through OP4

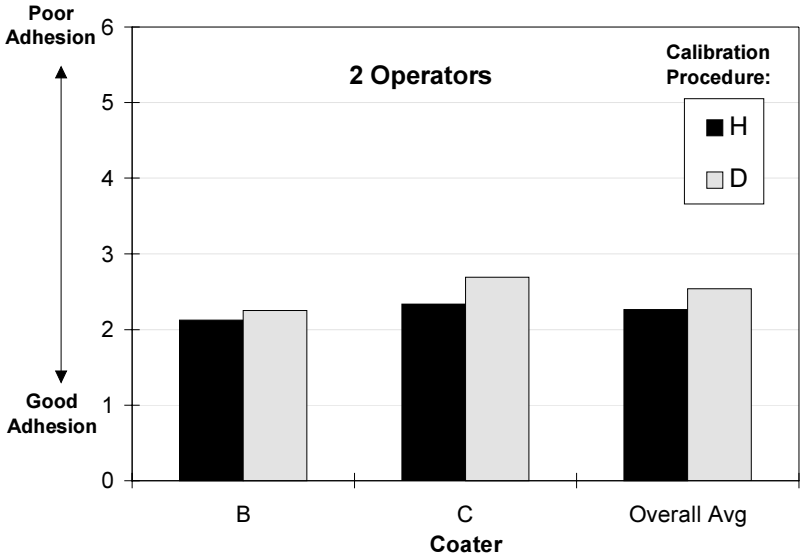
Figure 3.11 Overall mean adhesion ratings for test operators *R* and *E*.

Interestingly, operator *R* seemed to produce more consistent ratings than operator *E*. The overall standard deviation of ratings from operator *R* was 0.71 versus 0.90 of operator *E*. Specimen OP4 accounts for most of the difference. If specimen OP4 is not considered, standard deviation of adhesion ratings for both operators would be identical (0.73).

Adhesion test results performed by two operators are classified in Table 3.10 according to procedure for calibrating amount of knife force. The maximum difference in mean adhesion ratings on any specimen was 0.5. If all individual readings on all specimens are considered, the difference in mean adhesion ratings between the two calibration procedures is reduced to 0.28 (Figure 3.12). Overall, there was no marked improvement in the variability of adhesion ratings by performing adhesion test using the special device to control the amount of knife force (procedure *D*). If test results are analyzed separately by operator, the variability of adhesion ratings by operator *R* was reduced by performing the test with the special device (procedure *D*). However, operator *E* experienced slightly higher variability of adhesion ratings by performing the test following calibrating procedure *D* (Table 3.11).

Table 3.10 Mean adhesion ratings of calibration procedures *H* and *D* (tests performed by two operators).

Specimen	Procedure *H*	Procedure *D*	Difference (*H-D*)
OP1	2	2.5	-0.5
OP2	2.75	2.75	0
OP3	2.25	2	+0.25
OP4	2.33	2.5	-0.167
OP5	1.25	1.75	-0.50
OP6	3	2.75	+0.25



Coater *B*: Samples OP5, OP6
Coater *C*: Samples OP1 through OP4

Figure 3.12 Overall mean adhesion ratings of calibration procedures *H* and *D* (tests performed by two operators).

Table 3.11 Statistical parameters of adhesion tests performed by two operators following two calibrating procedures to control knife force.

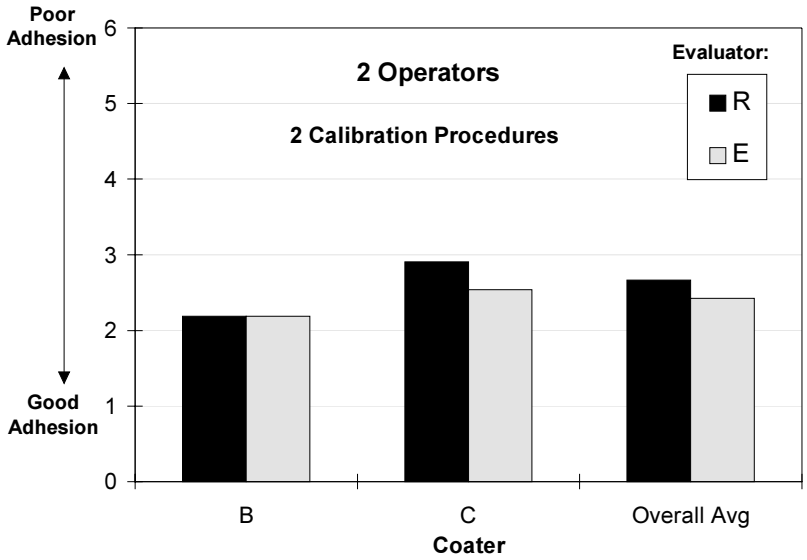
Parameter	Operator *R*		Operator *E*	
	Procedure *H*	Procedure *D*	Procedure *H*	Procedure *D*
Average	2.25	2.5	2.27	2.36
Avg. Dev.	0.73	0.43	0.66	0.69
Std. Dev.	0.83	0.50	0.75	0.95

Average adhesion of specimens rated by two evaluators is listed in Table 3.12. Such specimens were tested by two operators following two procedures to calibrate knife force. The maximum difference in average adhesion ratings was 0.625. Evaluator *R* tended to give higher ratings (lower adhesion) than

evaluator *E*. Evaluator *R* gave higher adhesion ratings to 4 out of 6 specimens. If all individual readings are considered, evaluator *R* gave an overall mean adhesion rating of 2.67 and evaluator *E* of 2.42 (Figure 3.13). Variability of the adhesion ratings given by the two evaluators was very similar. Both evaluators had the same range, mode, and average deviation of adhesion ratings, and the standard deviations were similar (Table 3.13).

Table 3.12 Average adhesion of specimens rated by two evaluators.

Specimen	Evaluator *R*	Evaluator *E*	Difference (*E*-*R*)
OP1	2.875	2.25	-0.625
OP2	2.875	2.75	-0.125
OP3	2.875	2.125	-0.75
OP4	3	3	0
OP5	1.875	1.5	-0.375
OP6	2.5	2.875	+0.375



Coater *B*: Samples OP5, OP6
Coater *C*: Samples OP1 through OP4

Figure 3.13 Overall mean coating adhesion of specimens rated by two evaluators (specimens tested by two operators following two calibrating procedures to control knife force).

Table 3.13 Statistical analysis of coating adhesion of samples rated by two evaluators.

Parameter	Evaluator *R*	Evaluator *E*
Avg. Dev.	0.67	0.67
Std. Dev.	0.86	0.79
Range	4	4
Mode	3	3

Coating adhesion testing was not affected by test operator nor by the calibration procedure to estimate and control knife force. The adhesion rating system seemed to give consistent results regardless of coating adhesion evaluator.

3.7 CONCLUSIONS FROM SECOND PHASE

Important findings in the second phase of the study are categorized and summarized below:

Regarding repeatability of adhesion test:

- There was small variation of average coating adhesion of specimens cut at different locations from the same rebar.
- Small specimens were representative of a long rebar.
- Small specimens were representative of a rebar lot if obtained from different bars in that lot.
- Bars with lower adhesion rates (better coating adhesion) tended to show less variability in adhesion values.

Regarding time of hot water immersion:

- There was no clear correlation between time of hot water immersion and adhesion rating.
- In some cases, specimens from a given lot tested with varying immersion times experienced less variability of ratings than specimens from the same lot subjected to the same time of immersion. In other words, variability of adhesion in a given lot is greater than the variability from different time of immersion.
- Twenty-four hours of hot water immersion was adequate for practical considerations.

Regarding knife blades and calibrating procedure for determining knife force:

- Type of knife and blade did not affect adhesion test results.
- Knife and blade selection was based on ease of use and cost.

- Calibration procedures *H* (knife force subjectively determined) and *D* (knife force objectively determined with special device) yielded similar adhesion ratings and data dispersion.
- Calibration procedure *D* tended to produce a higher variability of ratings. This may indicate that bars with poor quality could be more easily identified with procedure *D*.
- Adhesion testing gave meaningful results even though subjective processes (procedure *H*) were used.

Regarding test operator and evaluator:

- Adhesion testing was not greatly affected by test operators.
    - Average adhesion ratings by two operators were similar.
    - Standard deviation of adhesion ratings by each operator was nearly the same.
- Adhesion ratings were not greatly affected by test evaluators
- Adhesion test results were not affected much by procedures to calibrate knife force when two operators were involved

CHAPTER 4

Third Phase: Refinement of Coating Adhesion Test

Results from the First and Second Phases showed that despite some subjective factors being involved, hot water and adhesion tests can be useful in assessing the overall quality of coating adhesion. Further development of the tests was warranted and was the major thrust for the Third Phase of the study. The main objective was to improve the reliability and practicality of the tests. Particular objectives included the following:

- To study additional variables in hot water immersion (Water temperature, immersion times, post-immersion periods, test operator).
- To analyze the significance of some procedural modifications, such as pre-drilling a hole in the coating before immersion, making cuts in the coating that intersect at variable angles, making rectangular strip cuts in the epoxy coating (instead of X's) for adhesion testing, and performing adhesion tests with and without hot water immersion.
- To develop and test a self-calibrated knife that allows the operator to measure and control the amount of force applied.
- To improve the technique for peeling epoxy coating with calibrated knife.
- To improve the rating system for adhesion evaluation.
- To correlate results of several versions of adhesion tests.
- To correlate results of adhesion tests with existing TxDOT standard methods to evaluate coating adhesion (Bend test, peel test).

The findings and test procedures of Phase 3 of the coating adhesion study can be found in Reference 12.

4.1 BAR PROCUREMENT FOR ADHESION TESTS

Epoxy coated bars used for the First and Second Phases were obtained primarily from three coating applicators. For the Third Phase, epoxy-coated bars were requested from five coating applicators in order to have a wider spectrum of coating qualities. Each coater was identified with the following letters: *U, V, W, Y*, and *Z*. The requested bars from each coater included the following:

- One six feet long piece of deformed steel bar for each size: #4, #6, and #9

- One six feet long piece of plain steel bar for each size: #4, #6, and #9.
- Four bent bars from each of the original rebars where the above pieces were cut. Bars were bent according to TX DOT specification Tex 739-I.

In addition, bars with rigid, nonflexible coatings were requested when available. Details of supplied bars are listed in Reference 12.

Several quality control tests were performed to determine their compliance with ASTM and TxDOT standards. Such tests included visual examination of bent samples, coating thickness measurement, and holiday detection. Bars were divided in one-foot-long segments to record measurements from the above tests. The procedure followed for each of the above tests is described in more detail in Reference 12. Unlike bars for the First and Second Phases, all visible coating damage and imperfections were patched. Although the First Phase test results were not greatly effected by the presence of coating damage, all bars were repaired so they had approximately the same initial coating condition. Holidays invisible to the unaided eye were not patched but the number of holidays occurring at various intervals along the bar was recorded.

Bend Test Observations

Of all bars tested, only the coating by applicator *Y* failed the bend test. All four bent segments from bar Y-2 and three from Y-5 showed some cracking and damage to the coating. Only one bent specimen from bar Y-5 passed the test. As mentioned before, failure to pass the bend test may indicate either a) epoxy coating was too rigid or not flexible enough to pass the test, or b) epoxy coating had poor adhesion to the steel substrate. In most standards, factor (b) is assumed to be the reason for not passing the bend test. Correlation with adhesion tests in subsequent sections helped to clarify the validity of the bend test for determining coating adhesion.

Coating Thickness Measurement

The average coating thickness for each of the bars is shown in Figure 4.1. Each data point represents the average of 24 thickness measurements taken at regular intervals along the bar.[12] According to TxDOT Standard Specification Item 440, thickness values must range between 7 to 12 mils.[44] TxDOT limiting values are shown for comparison. Average thickness values ranged between 8.9 and 17.1 mils, with an average of 12.1 mils. Individual coating thicknesses ranged from 6.5 to 20 mils, with an overall average of 11.8 mils. These averages were at the upper limiting value allowed by specifications, which suggests that a large number of thickness measurements were above the upper limit. Very few measurements were below 7 mils. All bars from coater W and bars V-3 and V-28 had average coating thicknesses above 12 mils.

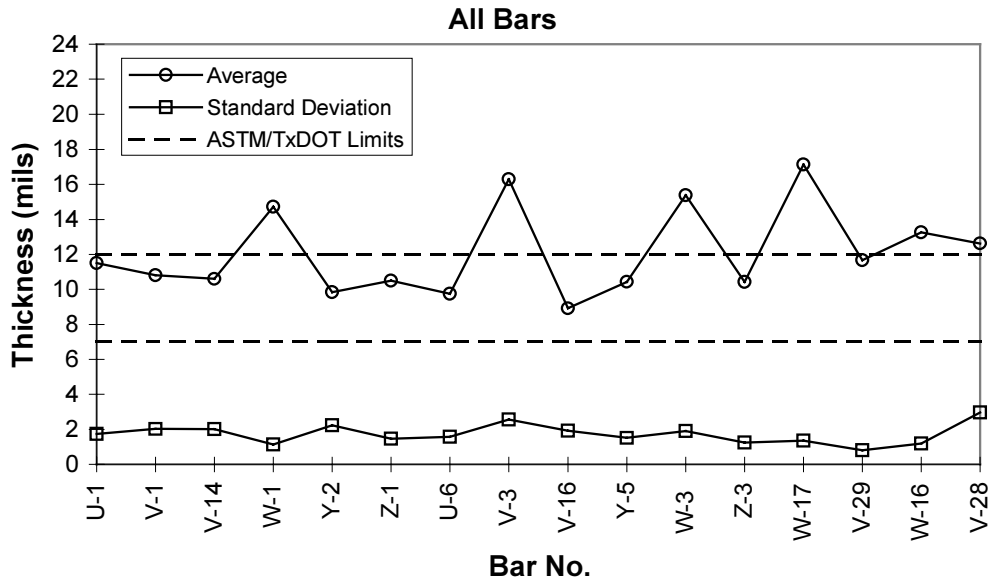


Figure 4.1 Average coating thickness of all bars from 5 coating applicators.

Both ASTM and TxDOT standards also require that 90% of the coating thickness values fall within the range of 7 to 12 mils.[23, 44] Only two bars met this requirement: U-6 and Z-3, with 100% and 92%, respectively, of thickness measurements falling between 7 and 12 mils. Bars Z-1 and Y-5 almost met this requirement, with 88% of readings between 7 and 12 mils. All individual measurements from bars W-1, W-3, and W-17 were higher than 12 mills. If bars from all coaters are considered, 62% of the coating thickness measurements fell within the range of 7 and 12 mils.

Holiday Detection

Figure 4.2 illustrates the average number of holidays detected per linear foot of rebar. The ASTM limit is plotted for comparison. The average number of holidays for all bars ranged from 0 to 5.8 per linear foot. Only three bars did not meet the ASTM standard: V-1, U-6, and Y-5. Bar V-1 had more than 3 holidays per foot and bars U-6 and Y-5 had extensive coating damage in areas close to mill marks. Such damage is not considered as part of the holiday count by ASTM specifications, which specify that such regions must be appropriately repaired.

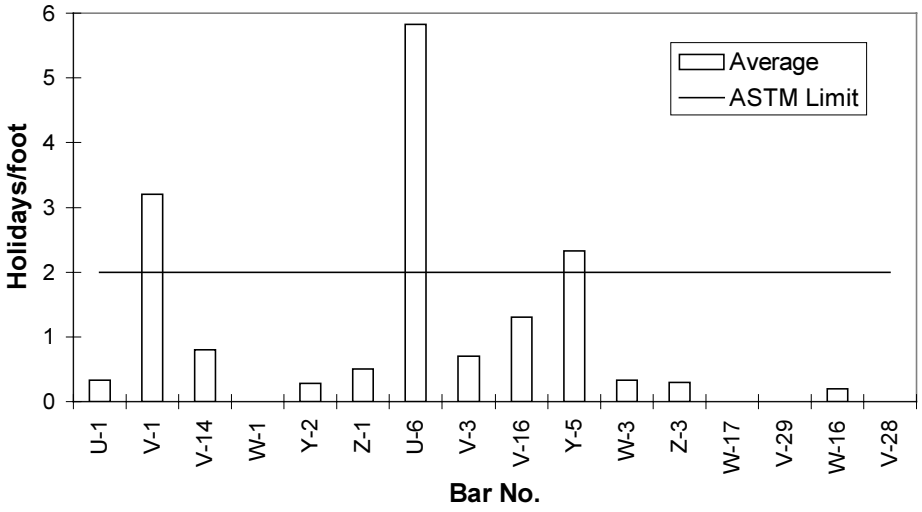


Figure 4.2 Average holiday count for all bars.

4.2 DEVELOPMENT OF CALIBRATED KNIFE

The special device for calibrating and controlling the amount of force applied with the knife had several disadvantages. With this device, adhesion tests could only be performed at the laboratory because it would be impractical to carry the device to coating plants. Another disadvantage was that the rigid assembly holding the bar specimen was built to fit certain bar sizes. To test a wider variety of bar sizes, several rigid assembly holders would have to be manufactured. Even though the device was improved so that calibration was needed only once per test session, the testing process was still time-consuming. Each specimen had to be positioned on the device four times to complete testing at all sites on the bar: 1) Specimen positioned to test on one side, 2) Specimen flipped horizontally and positioned to test in the opposite direction, 3) Specimen rotated about its axis and positioned to test the other side, and 4) Specimen flipped horizontally and positioned to test in the opposite direction. Clearly, this was very cumbersome and tedious, especially for a large number of samples.

Another disadvantage was that the test became more difficult to control as the knife advance around the bar perimeter. The horizontal component of the force decreased and the vertical component increased. This sometimes caused the whole assembly to slip and the operator had to adjust the knife force to continue the test. In a few cases, the blade slipped out or cut through the coating.

Finally, test results from Second Phase of the study showed that there was no remarkable difference in adhesion tests performed with and without the special device. If a better and more practical device cannot be developed, it may be easier and more practical to perform adhesion tests without the device.

With the above considerations in mind, it was desirable to design a device to better measure and control the force applied with the knife. The principle involved in the two previously used devices consisted of

estimating the knife force externally. The deflecting acrylic strip can be considered an external "spring" element that reacts as the bar is pushed against it. Such an external "spring" has a constant stiffness and the amount of force is controlled by how much the spring deforms, that is, how much the acrylic strip deflects.

If an internal spring can be placed inside the testing knife, many of the difficulties associated with the external spring concept can be eliminated. A self-calibrating knife was developed using this principle. An aluminum shaft was machined to exactly encase an X-acto knife and a compression spring (Figure 4.3). To avoid problems of lateral deflection of the spring, the inside diameter at the bottom of the shaft was machined to exactly encase a spring that had a diameter smaller then the diameter of the knife. The bottom portion of the X-acto knife was also machined to fit inside the narrow shaft area encasing the spring (Figure 4.3). During the test, the shaft is held and the base of the X-acto knife compresses the spring. Since the stiffness of the spring is known, the magnitude of the force is determined by measuring the spring compression. The knife surface was tapped to accept a screw and a slot was machined on the shaft surface as shown in Figure 4.3. A screw was inserted through the slot into the knife. The screw served two purposes: 1) To keep the knife from sliding off the shaft, and 2) to hold a small indicator to measure the spring deformation.

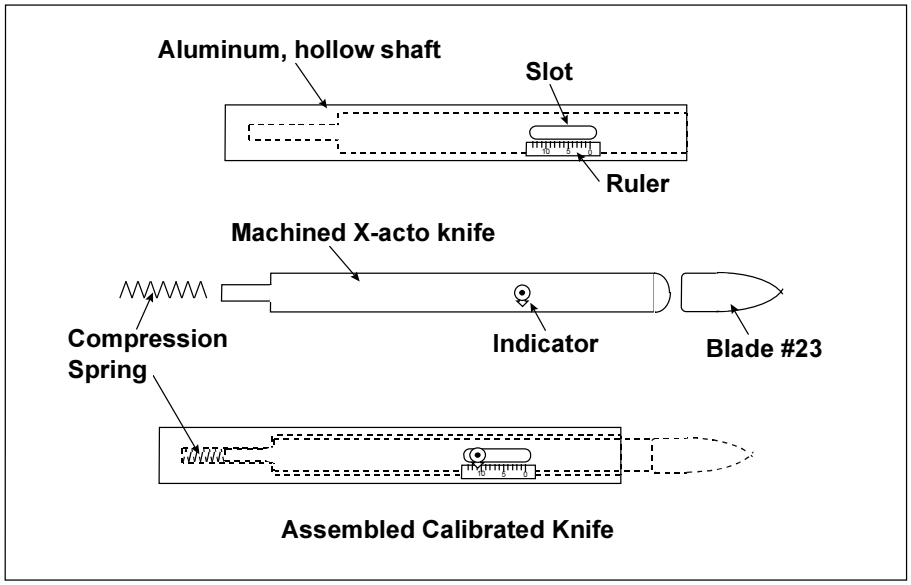


Figure 4.3 Calibrated Knife.

The indicator was secured to the screw above the shaft surface. A millimeter scale was attached next to the slot (Figure 4.3). When the knife is at the initial position (uncompressed spring), the indicator is zeroed. The target force is reached when the spring is compressed to a pre-determined amount.

The are several advantages associated with the calibrated test knife. It is a very simple device, easy to carry, and can be used anywhere (at the coating plant, in the field, at the laboratory). A wide variety of

blade types can be fixed to the sliding X-acto knife. Replacement of blades is easy because the screw restrains the knife from rotating about its axis. The blade holder can be loosened or tightened by turning it in the appropriate direction while the rest of the knife remains gripped. The test knife is very easy to use on any bar size. A major advantage is that the operator is much better able to control the amount of required force. Moreover, the magnitude of the force is no longer influenced by the angle of the knife.

It was not possible to incorporate a practical means of maintaining a constant tangential angle in the calibrated knife. Although the knife angle does not affect the magnitude of the force applied, such angle determines the direction of the knife force. The two hypothetical extremes would be 1) 90° in which case all the force is transferred to the steel, and 2) 0° in which case all the force tends to penetrate the coating layer. An angle of approximately 30° seems adequate in directing most of the force to cut through the coating-steel interface. It is difficult to implement a practical method to control such angle because it must be measured with respect to the tangent of the bar at the point of contact with the blade tip. The knife angle with respect to a horizontal plane must be changed as the knife moves around the bar perimeter to maintain a constant angle with respect to the moving tangent (Figure 4.4).

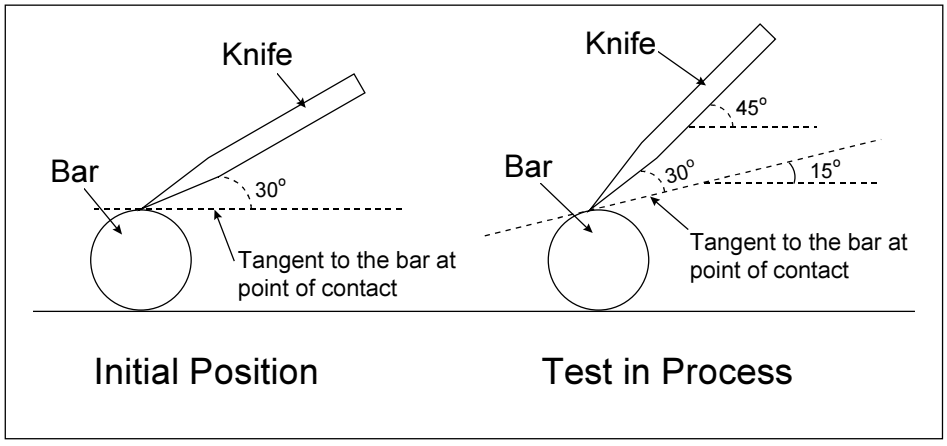


Figure 4.4 Angle of knife during adhesion testing.

An X-acto No. 23 blade (determined previously to be the best choice) was mounted in the test knife and used for adhesion tests. The blade was replaced daily or when its tip became blunt.

4.3 HOT WATER TEST

Preliminary tests

A series of preliminary tests were performed in order to refine the test parameters to be used in the Third Phase of the study. Several of the variables were already addressed in the previous two phases but it was recognized that more tests were needed to validate some of the findings. Some of those variables were not as carefully controlled as they were in the preliminary tests of this phase. In addition, other new variables

were studied and some procedural modifications in the test were introduced. A new adhesion rating system was devised for evaluating test results.

Samples were obtained from one #8 and one #9 epoxy-coated bars. Both bars were obtained from coating applicator *C*. Companion samples 5 inches long were cut from both bars to make a total of 16 samples from each bar. A total of 656 adhesion tests were performed, each test defined as the application of knife force to one of the flaps between two deformations.

Procedural Modifications

Each of the test locations consisted of 2 cuts in the coating that intersect a 45° angle to form an X. In all previous tests, the cuts forming the X intersected at a 90° angle. The angle was changed to make the test easier to perform. A more acute angle allows an easier insertion of the blade tip under the coating flap. Test results may also be simpler to interpret in flaps with an acute angle. It was observed that for several bar sizes and different deformation patterns, two diagonal cuts that extend from the top of one rib to the bottom of the next rib generally intersected at an angle of about 45° (Figure 4.5). The main drawback is that only two flaps, instead of four, can be tested at each X location. As before, X-cuts were made with the plastic utility knife with sharp snap-off blades.

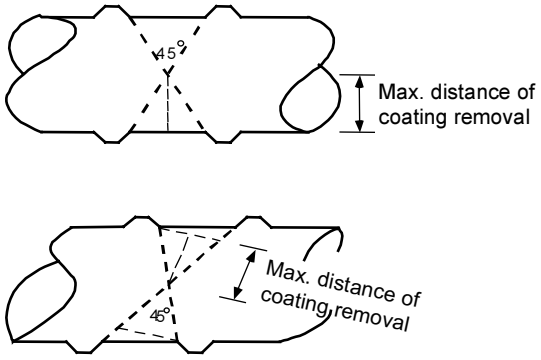


Figure 4.5 Length of cut for bamboo and diagonal deformation patterns.

A second modification consisted of sealing the end of each specimen with a two-part epoxy resin instead of silicone. This resin was much sturdier and more watertight than silicone and prevented water migration under the coating and corrosion of the exposed steel at the ends.

The rating system adopted to evaluate coating adhesion was a function of the length of the line that bisects the triangular flap that is formed between the diagonal cuts (Figure 4.5). A rating was assigned according to the average length of coating removed along the path of the bisecting line (Table 4.1). Measurement of areas of removed coating was not used as a rating criterion because it was easier to measure the length of the section of coating removed. An individual index was given to each flap (before,

an individual index was given to each test location) and all indexes were averaged to yield a specimen adhesion rating. The previous rating systems used did not involve any measurement of removed coating.

Table 4.1 Adhesion rating system for preliminary tests of third phase.

Adhesion Index	Description
1	Difficult to insert blade under coating. Less than 5% of the length is removed.
2	Easier to insert blade under coating. 5% - 25% of length is removed.
3	25% - 50% of length is removed
4	50% - 75% of length is removed
5	More than 75 % of length is removed

Study Variables

The following variables were used for the preliminary tests of Third Phase:

- *Water temperature:* 55°C and 75°C. Previous tests from First and Second Phases were performed at a temperature of 73°C. However, some researchers have used temperatures as low as 55°C when samples were immersed in aggressive media.[6] The objective was find an optimum combination of temperature-immersion time.

- *Time of immersion:* 0, 3, 6, 24, and 48 hours. Tests from Second Phase were performed with times of immersion of 2, 8, 24, and 48 hours. The most significant difference is that samples with no water immersion were included.

- *Post-immersion times:* 0, 3, 6, 24, and 48 hours. Some samples from First Phase were tested after post-immersion times of 24, 40, 72, 90, 120 hours, and 2 months. However, these samples were not carefully controlled test variables.

- *Presence of initial damage:* A 1/8-in. hole was drilled through the epoxy coating into the bar to create an intentionally damaged area. The hole created a more carefully controlled damaged area with the same size, shape, and location in all specimens. Samples from the First Phase had damaged areas of different sizes and shapes and were randomly located. Such damage was present in the as-received bars and was produced during handling and transportation of the bars. Unlike samples from the first phase, adhesion tests were performed at the pre-drilled hole. Previous tests were not always performed at the location of the damaged areas.

- *Knife force:* 3 kg and 4 kg. The higher force was based on the maximum spring compression. The lower force was used to conform to specifications used in previous tests. Tests from the first and second phases used knife forces of approximately 3 and 3.5 kg.

Each side of a sample was tested at four locations using two knife forces and surface conditions (presence or absence of pre-drilled hole), as seen in Figure 4.6.

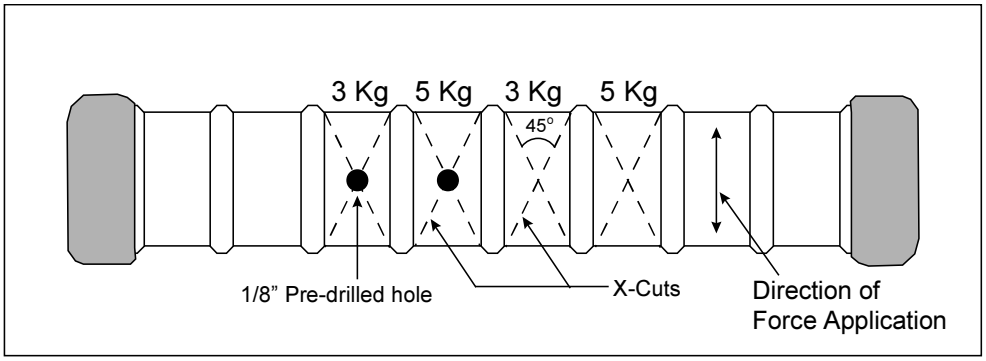


Figure 4.6 Test locations on 5-inch-long epoxy-coated bar specimens for preliminary adhesion tests.

Test Results

Time of immersion

As the time of immersion increased, coating adhesion tended to decrease (adhesion ratings increased) regardless of the temperature of the hot water bath (Figures 4.7 and 4.8). These findings contrast with those of Phase 2, where no consistent trend was found for most samples. Tests in Phase 2 may be less reliable because the procedure used to test samples after 48 hours of immersion was not exactly the same as that for samples tested after other times of immersion (there were differences in the type of calibration device, orientation of X-cut, and number of tests per site). However, other factors might effect the difference of findings between Phases 2 and 3, such as differences in the coating process, coating type, coating porosity, and adhesion test procedure (special device vs. calibrated knife, 90° vs. 45° angle of X-cuts).

An important finding was that adhesion ratings remained fairly constant after times of immersion of 24 hours or longer (Figures 4.7 and 4.8). Tests from phase 2 revealed similar results. In practical terms, this is the most important finding because it confirms that hot water test duration can be effectively reduced from 48 hours (as proposed by MTO) to 24 hours. Times of immersion shorter than 24 hours are not recommended because of the discrepancies found.

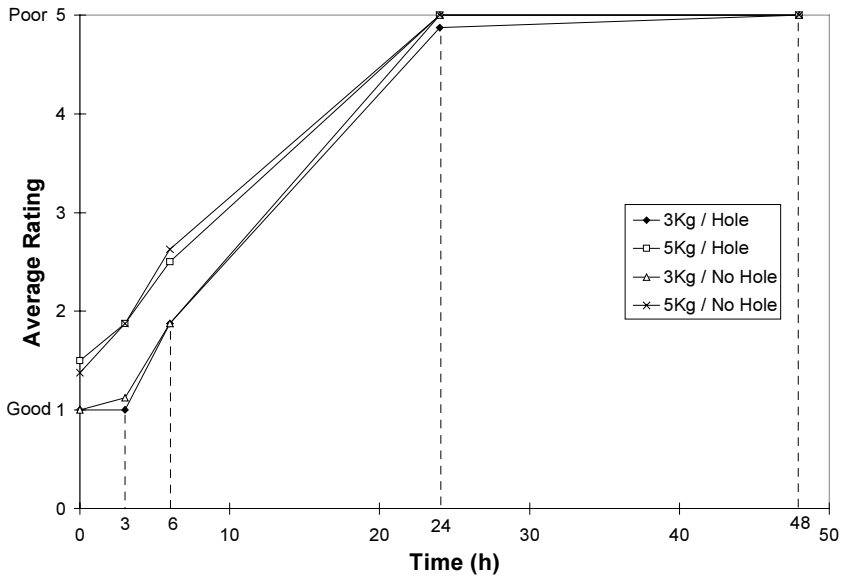


Figure 4.7 Effect of immersion time for bar No. A-1 exposed to 75°C water.

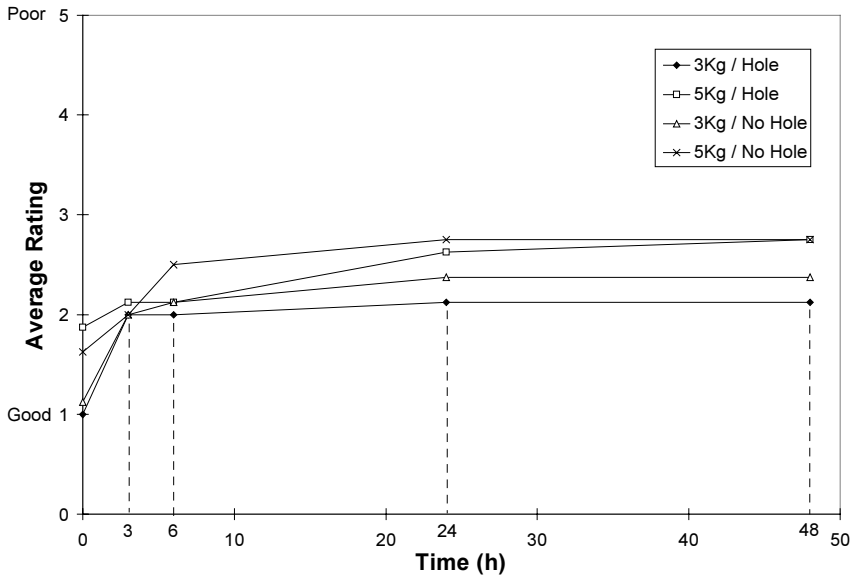


Figure 4.8 Effect of immersion time for bar No. S-1 exposed to 75°C water.

Water temperature

As the temperature of the water bath increased, coating adhesion tended to decrease. A higher adhesion loss was always observed at higher temperatures. For bar A-1, there were samples that showed good coating adhesion at lower temperatures but experienced total loss of adhesion when immersed at 75° (Figure 4.9). For bar S-1, there was little difference in adhesion between 55° and 75° (Figure 4.10). In successive tests, the majority of samples from five coating applicators experienced extensive adhesion loss at exposure to 75° water immersion.

Again, there are some discrepancies between results from the previous two phases and the third phase. A number of samples from previous phases showed good or intermediate coating adhesion after immersion in 75° water. It may be that bars acquired for the third phase were of inferior qualit. Another factor may include differences in test procedure (special device vs. calibrated knife, 90° vs. 45° angle of X-cuts) but these are unlikely to cause such marked difference.

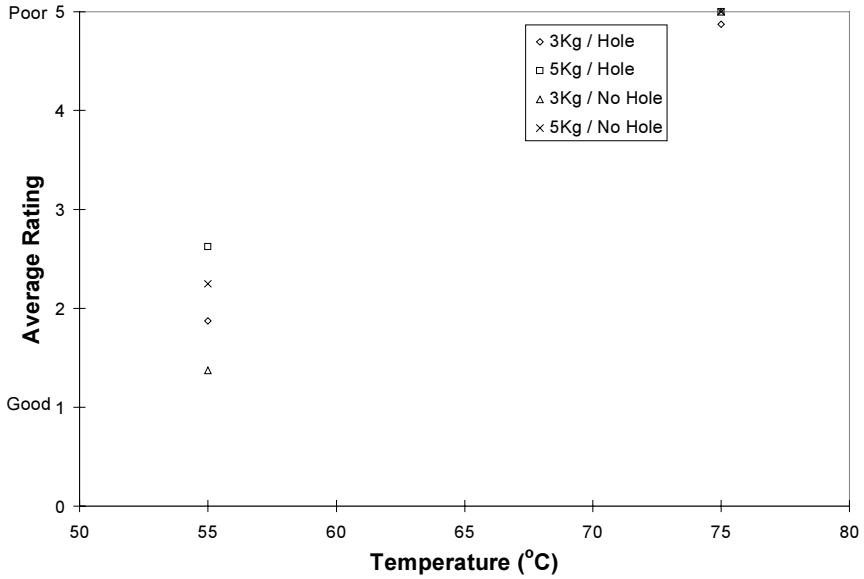


Figure 4.9 Effect of temperature on bar No. A-1 after 24 hours of immersion.

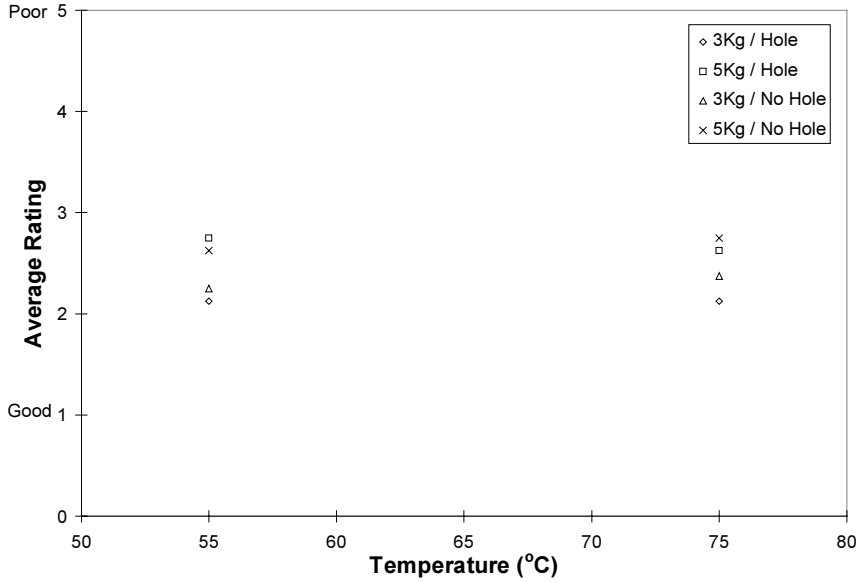


Figure 4.10 Effect of temperature on bar No. S-1 after 24 hours of immersion.

Hot water tests for phase 3 were conducted at a temperature of 55°. Immersion in 75° water may be too harsh for production bars to pass. More tests on bars from a wider variety of coating applicators would be needed to validate this hypothesis.

Presence of damage

Sections with pre-drilled holes experienced slightly higher adhesion loss than sections with undisturbed coating. The presence of the hole allowed migration of moisture and formation of corrosion products even at early stages. All samples exhibited corrosion products in the drilled hole after the hot water bath, even after short immersion times. However, the difference in adhesion ratings between the initial conditions (hole vs. no hole) was very small. In addition, specimens with pre-drilled holes were more difficult to test. No samples were pre-drilled in the remaining tests.

Despite differences in the type of damage and test procedure, results from Phase 1 and preliminary tests of Phase 3 indicated that coating damage before immersion does not greatly affect coating adhesion.

Knife force

Adhesion loss was found to be directly proportional to the applied force (Figures 4.7 through 4.10). In some cases, difference in adhesion ratings between the two forces was as high as one unit (Figures 4.7 and 4.8). Additional tests were performed with two knife forces to further evaluate this variable.

Post-immersion time

With post-immersion periods longer than 6 hours, adhesion ratings tended to remain constant (Figures 4.11 and 4.12). Adhesion ratings from tests in the first phase at varying post-immersion times (equal or greater than 24 hours) did not vary significantly. Based on preliminary results, remaining adhesion tests were performed after post-immersion times of 6 hours or greater.

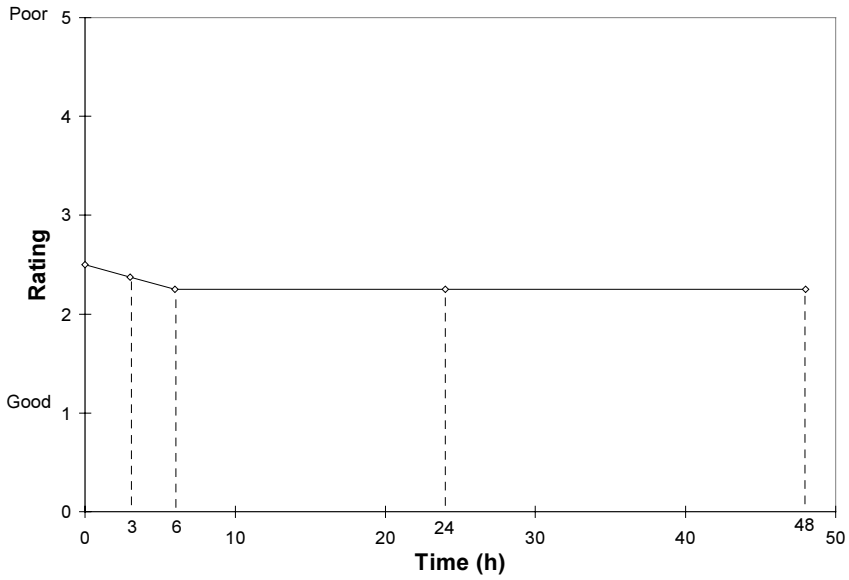


Figure 4.11 Effect of post-immersion period on coating adhesion for bar No. A-1.

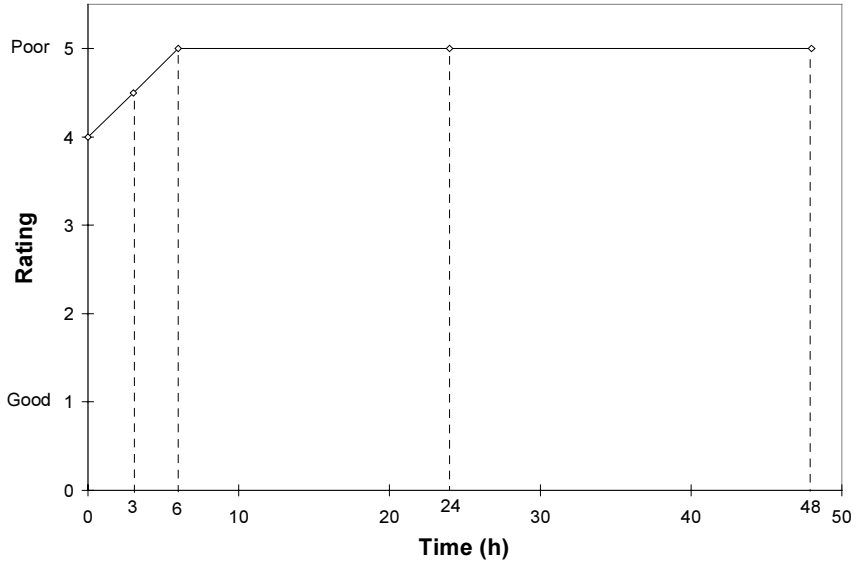


Figure 4.12 Effect of post-immersion period on coating adhesion for bar No. S-1.

Final Adjustments to Hot Water Test

Test Procedure and Variables

From preliminary tests and partial findings as tests progressed, the following variables were selected:

- Temperature of hot water bath: 55°C ± 2°C
- Time of immersion: 24 hours
- Post-immersion time: 6 hours or longer

- Knife force: 3 kg and 4 kg
- Initial coating condition: Undamaged

Samples were obtained from 5 manufacturers as described in section 4.1. One sample was taken from every end and one sample was taken from the middle of the supplied bar. The procedure was intended to take into account possible variations of adhesion along the bar. At all three locations, companion samples were tested by a different operator.

Four 45° X-cuts were made on each side of the bar specimens. Two cuts were tested applying a force of 3 kg and the other two with a force of 4 kg, resulting in eight values of adhesion for every force in each sample. The eight adhesion values were averaged to obtain a mean adhesion rating for the sample. Tests were performed on #6 and #9 deformed bars and #6 and #10 plain bars.

Test Results

Table 4.2 contains adhesion ratings for each bar and each knife force, including the average rating, range, standard deviation, and coefficient of variation. Overall, there was little variability of test results. Standard deviations ranged from 0 to 0.55 for all bars except U-6 (1.01). Figure 4.13 is a graphical representation of values from Table 4.2 when 3 kg of knife pressure were applied. For reference, the mean of standard deviations is represented with a horizontal line. It can be seen that most standard deviations remained close to the 0.4 average regardless of the adhesion rating obtained, which means that there is similar dispersion of adhesion ratings for most coatings.

Neither the standard deviation nor the coefficient of variation correlated with average adhesion ratings. Results from Phase 2 seemed to indicate that bars with lower adhesion strength have greater variability of data, but this was not validated from the larger data base used in this phase. For instance, bar U-1 had very poor adhesion and standard deviation was zero. As stated in phase 2, if adhesion ratings are to be used as quality indicators, both the mean rating and standard deviation of ratings have to be considered.

Table 4.2 Data obtained from hot water tests for each bar.

Bar No.	Bar Size	Minimum Rating*	Maximum Rating*	Average Rating*	Standard Deviation*	Coeff. of Variation*
U-1	#6	5 (5)	5 (5)	5 (5)	0 (0)	0% (0%)
U-6	#9	1.5 (2.5)	4.5 (5)	2.8 (4)	1.01 (0.99)	36% (25%)
V-1	#6	1.5 (1.5)	2.5 (2.5)	2.1 (1.8)	0.42 (0.44)	20% (24%)
V-3	#9	1 (1)	1.5 (2)	1.2 (1.7)	0.25 (0.44)	21% (27%)
V-28	#6	2.5 (3)	4 (5)	3.4 (4.3)	0.55 (0.38)	16% (9%)
V-29	#10	1 (1)	2 (2)	1.3 (1.7)	0.40 (0.33)	31% (20%)
V-14	#6	3.5 (5)	5 (5)	4.5 (5)	0.52 (0)	12% (0%)
V-16	#9	4 (5)	5 (5)	4.9 (5)	0.31 (0)	6% (0%)
W-1	#6	4 (4.5)	5 (5)	4.8 (4.9)	0.34 (0.20)	7% (4%)
W-3	#9	3.5 (4)	5 (5)	4.3 (4.5)	0.45 (0.43)	11% (10%)
W-16	#6	4 (5)	5 (5)	4.9 (5)	0.29 (0)	6% (0%)
W-17	#10	4 (4)	5 (5)	4.8 (4.8)	0.33 (0.33)	7% (7%)
Y-2	#6	1.5 (1.5)	2.5 (3)	2.3 (2.1)	0.34 (0.38)	15% (18%)
Y-5	#9	1 (2)	2.5 (3)	1.6 (2.5)	0.52 (0.48)	33% (19%)
Z-1	#6	1.5 (2)	2 (2)	1.9 (2)	0.23 (0)	12% (0%)
Z-3	#9	1 (2)	3 (3)	1.5 (2.6)	0.54 (0.38)	37% (14%)

* Applied force = 3 Kg (4 Kg)

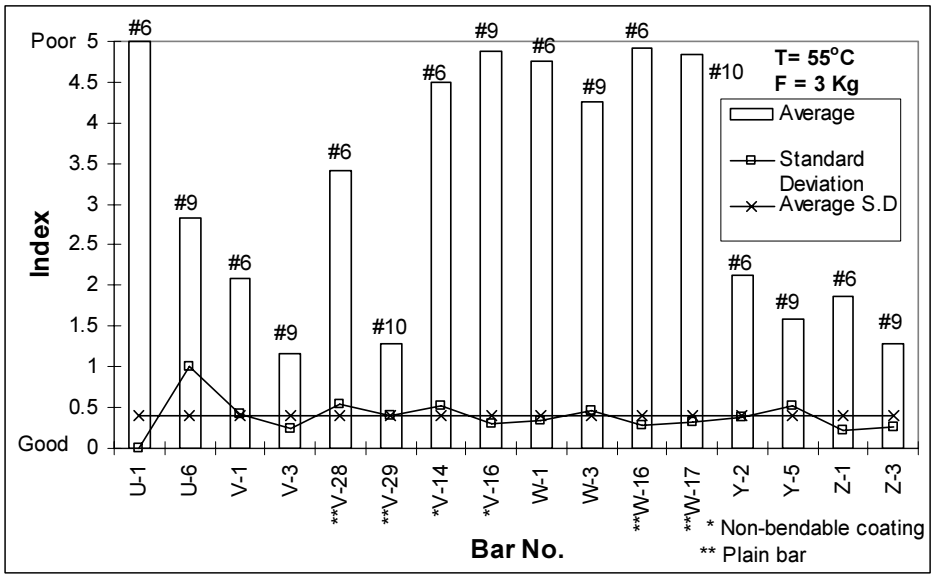


Figure 4.13 Average adhesion rating of all bars in hot water tests.

Although bars from the same coater but different lots generally had similar adhesion ratings, large discrepancies may occur. Bar V-28 had an average rating of 3.4, which is much greater than the average of 2.0 for all other bars coated with the same material by the same applicator and may suggest a less carefully controlled coating operation during production of that particular lot.

Table 4.3 shows values of average adhesion and standard deviation calculated at each location along the bars with a force of 3 kg. The force of 4 kg produced many failures involving tearing or slicing through the coating so calculations at each location are not tabulated. Regardless of the trend in standard deviations at a given location, the majority of adhesion mean values were similar at different bar locations. Average adhesion of most samples was representative of the overall adhesion of a bar as indicated also in results from Phase 2.

Table 4.3 Average and standard deviation of adhesion ratings at three locations along each bar. Applied force = 3kg.

Bar	Bar	Left End		Middle		Right End	
No.	Size	Avg.	σ	Avg.	σ	Avg.	σ
U-1	#6	5	0	5	0	5	0
U-6	#9	3	0.41	3.8	0.87	1.8	0.29
V-1	#6	2.4	0.25	2.1	0.48	1.8	0.29
V-3	#9	1.1	0.25	1.3	0.29	1.1	0.25
V-28 **	#6	3.3	0.65	3.8	0.29	3.3	0.65
V-29 **	#10	1.3	0.29	1.1	0.25	1.5	0.58
V-14 *	#6	4	0.41	4.6	0.48	4.9	0.25
V-16 *	#9	4.6	0.48	5	0	5	0
W-1	#6	4.6	0.48	4.9	0.25	4.8	0.29
W-3	#9	4.3	0.29	4.4	0.48	4.1	0.63
W-16 **	#6	5	0	4.8	0.5	5	0
W-17 **	#10	4.6	0.48	4.9	0.25	5	0
Y-2	#6	2.4	0.48	2.1	0.25	1.9	0.25
Y-5	#9	1.5	0.41	1.8	0.65	1.5	0.58
Z-1	#6	1.9	0.25	1.8	0.29	2	0
Z-3	#9	1.4	0.25	1.4	0.25	1.1	0.25

* Nonbendable ** Plain bar

Nonbendable coatings performed poorly in hot water tests. For the two bars tested, there was almost total loss of adhesion after the hot water bath. This was a disappointing result because nonbendable coatings are claimed to be of superior quality. However, this finding is not conclusive because bars from only one applicator were obtained.

Figure 4.14 shows the average of all adhesion ratings for each coating applicator. As discussed earlier (first phase), such averages permit evaluation of the overall quality of different coating applicators. It is

clear that adhesion correlated well with coating applicator even though other factors, such as coating operator, knife force, bar size, and adhesion within the same bar varied.

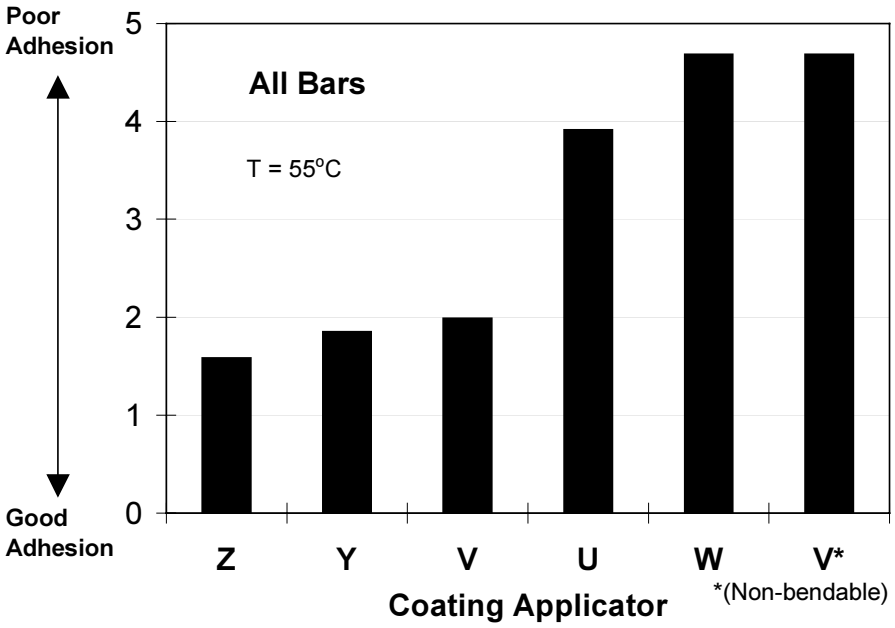


Figure 4.14 Coating applicator performance–Hot water tests.

Effect of Operator

Two operators performed adhesion tests on #9 bars after immersion in 75°C water only (because of time constrains, a second operator did not test bars after immersion in 55°C water). The second operator tested an additional sample from the end of the same bar. For each bar, only one of the samples tested by the main operator M is included in the comparison (the sample located at the same bar end as the sample tested by the second operator). Both operators performed 8 tests for each specimen, one half of the tests with a force of 3 Kg and the other half with a force of 4 Kg.

Test results are summarized in Figures 4.15 and 4.16. Except for bar U-5 tested with 3 Kg, force specimens tested by operator M showed the same or slightly higher average rating (lower adhesion) than those tested by operator E. Average ratings of individual bars tested by both operators were very similar, with a maximum difference of 0.375. If mean ratings from all bars are averaged, the difference of results between the two operators is reduced to 0.06 (3 Kg) and 0.15 (4 Kg), as can be seen in Figures 4.15 and 4.16.

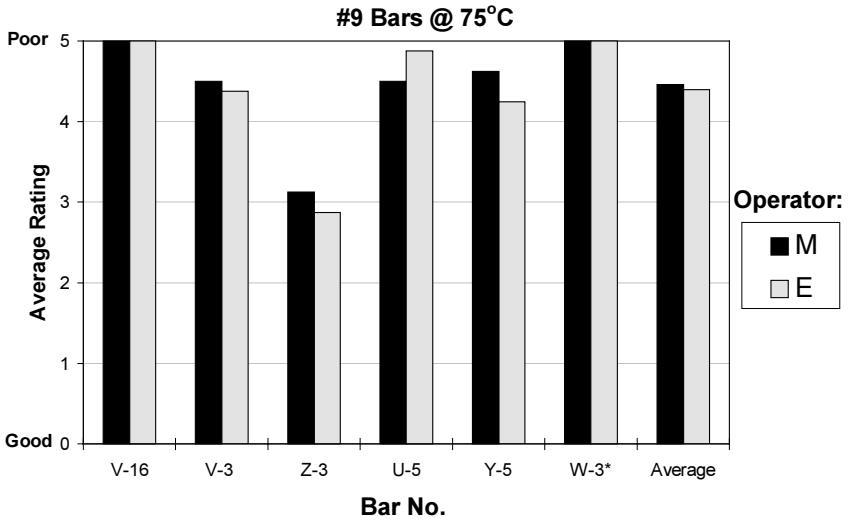


Figure 4.15 Effect of operator (Hot water test, F= 3Kg).

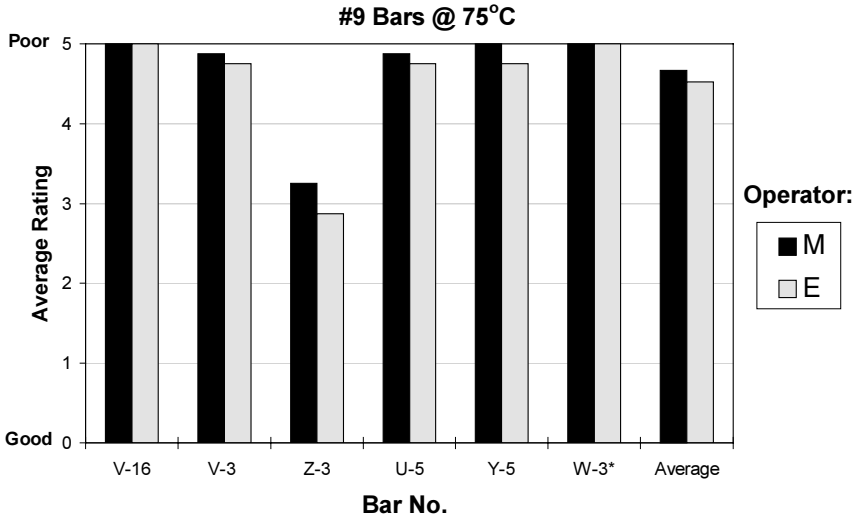


Figure 4.16 Effect of operator (Hot water test, F= 4Kg).

Finally, results from additional tests performed by two operators are shown in Figure 4.17. The tests were performed on specimens from the same bar (P2) at different post-immersion times. Two ratings were higher for operator E (maximum difference of 0.5) and two were higher for operator M. Average ratings for each operator are very similar, with a difference of 0.16.

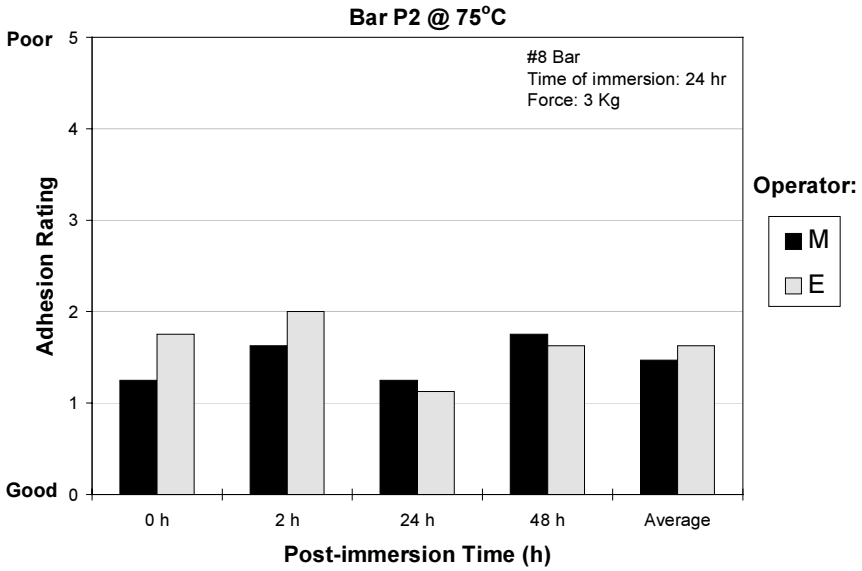


Figure 4.17 Effect of operator (Hot water test on bar P2).

4.4 CONTROLLED PEEL TESTS

Adhesion tests in all Phases 1, 2, and 3 were performed after immersing specimens in a hot water bath. The purpose of the hot water immersion was to accelerate the electrochemical adhesion loss and to provide a measure of the coating adhesion in bars that have been in aggressive environments for an extended period. However, hot water immersion involves an elaborate, time-consuming sampling process.

TxDOT inspectors routinely perform peel tests at coating plants without any prior immersion. Such tests provide a measure of the total adhesion (electrochemical and mechanical) immediately after the bar is coated. At that stage, adhesion is a function of the quality of the coating process and handling at the plant.

The feasibility of performing adhesion tests directly on rebars without conducting hot water immersion was of interest to TxDOT because a simple, quick, and reliable quality control method that could be performed at the coating plant or elsewhere during the construction process was desired. There are several advantages associated with testing at the production line: No laboratory testing is required, results are obtained much faster, and the costs are reduced. Since the test can be performed directly on the coated bar, there is no need to cut samples.

Two test procedures were developed and evaluated: (1) Strip method, and (2) X-cut method. The difference between the two methods lies in the definition of the test area. Evaluation of coating adhesion was not based on a conventional rating system but on direct estimates or measurements, which were different for each method.

A major breakthrough was the technique for peeling the coating during the test. So far, adhesion tests have been performed by applying a shearing force through the coating-steel interface with a knife blade. There are several disadvantages associated with this technique. Inevitably, a portion of the knife force (depending on the actual knife angle) is transferred to the epoxy coating layer and to the metallic surface. Local rough areas on the steel surface and thick coatings may resist the forward motion of the blade. When such resistance is overcome, the blade may suddenly slip off and tear the coating.

The new technique consisted of applying a simultaneous combination of shearing and prying action with the blade. Previously, prying was only used to remove coating that had already been debonded by the shearing action of the blade. For controlled peel tests, prying would become a substantial component of the blade debonding action. Prying of coating was achieved by applying a rotating motion to the testing knife, resulting in an uplifting stress that effectively debonded the coating from the substrate. The magnitude of the shearing force needed to keep the forward motion of the blade was smaller than in previous tests.

Strip Method

Test Procedure

Four cuts were made through the coating to form a 2 x 25 mm rectangular strip at each test location. The strip was parallel to the circumference of the bar. The 2 mm width was determined based on preliminary trials. Narrower strips resulted in debonding of the strip of coating, and wider strips could not be peeled at all. The tip of a utility knife was used to lift the coating at one end of the strip. The tip of the calibrated knife was than inserted under the coating and the knife was positioned at an angle of approximately 30° tangent to the curvature of the bar. A constant force (1 to 2 kg) was applied to the knife maintaining the tip of the blade in the center of the strip. Simultaneously, the knife blade was rotated about its axis as the knife blade moved forward. The amplitude of the rotating motion is illustrated in Figure 4.18. The blade was continuously rotated 30° on each direction from the initial position. The test was stopped when the calibrated knife traversed along the whole length of the strip.

At the end of the test, all loose and debonded material was removed and the surface was examined. The recorded adhesion index consisted of the approximate percentage of coating that remained adhered to the steel. Such values were estimated visually to the nearest 10%. The greater the index, the better the adhesion. Adhesion indexes ranged from 0% (no adhesion) to 70% (good adhesion). The maximum index cannot be 100% because the blade tip removed a very thin strip of epoxy even in the best adhered coating. The maximum index of 70% was based on the actual dimensions of such strips (roughly equal to the width of the blade tip).

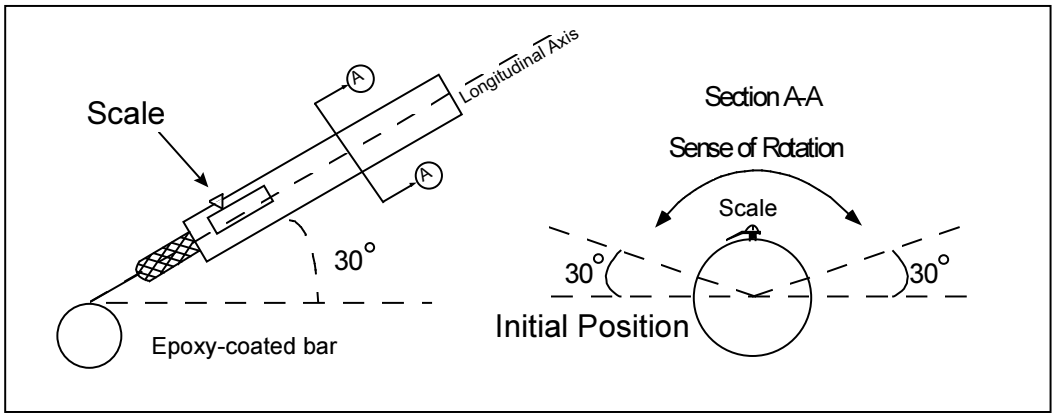


Figure 4.18 Position of testing knife and rotating motion.

The main disadvantage of the strip method was that the initial rectangular cut was very difficult to make and was time-consuming. Once the cut was made, the test was easy to perform. As in previous adhesion tests, the rectangular cut had to be made through the whole coating thickness to the bare steel. Likewise, the blade should be advanced through the coating-steel interface and not through the coating itself. This was verified by a small trail of metal visible through the coating left along the strip.

Adhesion tests were performed at both ends of the rebar and at the middle portion. In each location, two tests were conducted on each side of the bar. A test was defined by the application of knife force to one pre-cut rectangular area or strip. Four adhesion indexes were obtained at each location.

Test Results

Table 4.4 summarizes the test results on bars from five coaters using the same bars as in hot water tests. In some bars, very high coefficients of variation were noted, especially bars with poor adhesion. Average adhesion values were lower than the standard deviation, indicating that coefficients of variation are not meaningful indicators of the dispersion of data. For example, coefficient of variations for bars W-16 and V-29 were 346% and 4%, respectively. Examination of all individual readings reveals that for bar W-16, there were eleven 0% readings and only one 10% reading. For bar V-29, there were eleven 70% readings and only one 60% reading. Obviously, both bars had identical standard deviation of 2.9%. The coefficient of variation for bar W-16 is high because the standard deviation is divided by its much smaller average adhesion value (1% vs. 69% for bar V-29).

Table 4.4 Adhesion indexes from strip tests for all bars in percentage of remained coating.

Bar No.	Bar Size	Max. (%)	Minim. (%)	Average (%)	σ (%)	C.V. (%)
U-1	#6	10	0	4	5.2	124
U-6	#9	60	0	37	22.3	61
U-3	#4	70	60	66	5.1	8
V-1	#6	50	10	22	11.2	51
V-3	#9	70	60	68	3.9	6
V-2	#4	70	50	63	7.8	12
V-14*	#6	30	0	14	7.9	56
V-16*	#9	20	0	12	7.2	62
V-28**	#6	50	0	18	16.4	90
V-29**	#10	70	60	69	2.9	4
W-1	#6	0	0	0	0.0	0
W-3	#9	20	0	8	7.5	101
W-2	#4	10	0	2	3.9	234
W-16**	#6	10	0	1	2.9	346
W-17**	#10	10	0	1	2.9	346
Y-2	#6	50	20	41	10.0	24
Y-5	#9	70	50	61	9.0	15
Y-3	#4	70	50	63	8.7	14
Z-1	#6	70	50	63	6.5	10
Z-3	#9	70	40	55	8.0	15
Z-2	#4	70	50	67	6.5	10

* Nonbendable coating ** Plain bar

Bars with poor adhesion tended to have high coefficients of variation in strip tests and low coefficients of variation in hot water tests. Different interpretations in the rating system for each method produced this discrepancy. In hot water tests, a high rating indicates a poorly adhered coating, while in peel tests by the strip method, a high index indicates good coating adhesion. A higher adhesion index (regardless of whether it represents good or bad adhesion) results in a lower coefficient of variation. As opposed to the coefficient of variation, the standard deviation was not affected by differences in rating systems.

Standard deviation in peel tests by the strip method varied more than in hot water tests. This situation was created by the inherent inaccuracy in reporting the results to the nearest 10% and estimating values visually. A more accurate system would involve measuring the width of the strip at several sections along the strip and calculating an average.

Figure 4.19 shows the average adhesion index and standard deviation of values for each bar. The average of standard deviations is plotted for reference. As in hot water tests, bars from coating applicator U had the largest dispersion of adhesion indexes along the bar. The strip method produced more variation of adhesion strengths among bars from different lots from the same coater than the hot water test.

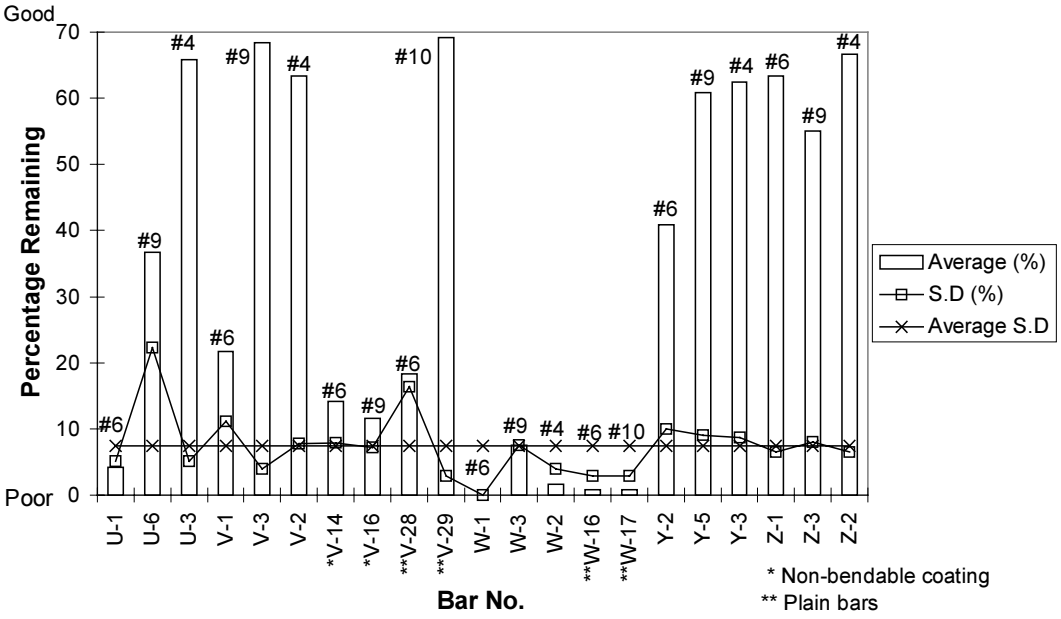


Figure 4.19 Average adhesion index of all bars in strip tests.

All bars from coater W had very poor coating adhesion, in all cases less than 10% of coating remained adhered after the test. Visual examination of these samples disclosed a very dark and scaly residue, possibly the product of improper surface preparation. Again, nonbendable coatings performed poorly compared to bars with flexible coating from applicator V. Interestingly and in contrast to bars from coater W, visual examination of nonbendable samples revealed a very clean steel surface, suggesting that factors other than surface preparation may produce loss of coating adhesion.

For most applicators, #6 bars tended to have poorer coating adhesion than larger (#9 or #10) or smaller (#4) bars.

Figure 4.20 shows the overall average of all adhesion ratings for each coating applicator. The adhesion performance among different coaters was practically the same as in the hot water test, with only a slight difference in the order of the two worst performers.

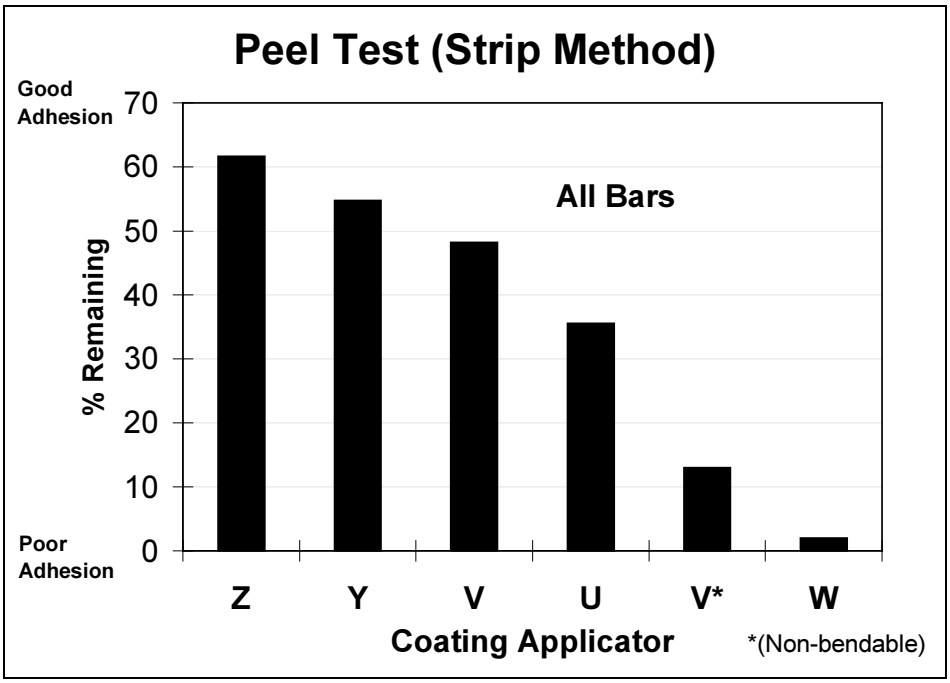


Figure 4.20 Coating applicator performance–Peel tests (strip method).

X-Cut Method

Test Procedure

This test method combines some of the features of the previous two tests. An X-cut, similar to that for adhesion testing after hot water immersion, is made. Peel tests are performed using opposite flaps of the X-cuts. As in the strip method, the calibrated knife is used to apply a combination of shearing force and prying action.

Unlike adhesion tests after hot water immersion, the interior angle of the X-cut was not restricted. After trials on many bars, it was found that when several tests were performed at the same location using the shearing-prying technique, the width of the section where coating broke remained constant, regardless of the angle of the X-cut (Figure 4.21). The uplifting stress applied by rotating the knife tends to break the bond of the epoxy to the steel until the resistance provided by adhesion is larger than uplifting forces. When that point is reached, the coating breaks off or rips apart. The weaker the adhesion, the wider the section at which the coating breaks, regardless of the X-cut interior angle. Therefore, measuring the width of the section where the coating breaks provides an indication of the adhesion strength at that location.

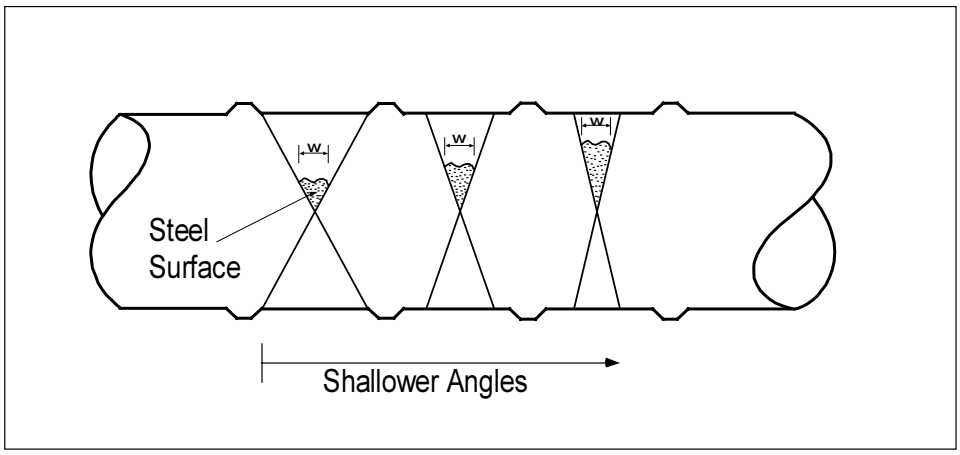


Figure 4.21 Width of final section remains roughly equal as the angle of the X-cut becomes shallower.

The angle of the X-cut can be can be increased or decreased to obtain debonded areas that are easier to measure. If the coating has strong adhesion resulting in very small widths at coating breakage, the next X-cut should have a smaller angle. If adhesion is so weak that the width of section at coating breakage is large, subsequent X-cuts can be made with a larger angle. Cuts with a 45° angle should be tried first.

As in the strip method, adhesion tests were performed at both ends of the bar and at the middle portion. At each location, two tests were conducted on each side of the bar to obtain the same number of readings as in the strip method. A test is defined by the application of knife force on one flap of the cut.

For #4 bars, a "V" cut instead of an "X" cut was used because of the small area between ribs. Twice as many "V" cuts as "X" cuts were made to provide the same number of tests.

Test Results

Results from X-cut tests performed on bars supplied by all coaters are listed in Table 4.5. Adhesion values were based on measurements of the failure zone and ranged from 1.0 mm to 5.0 mm or more. A reading of 5 mm was recorded for all measurements equal to or greater than 5 mm. The rationale was that when section widths larger than 5 mm were reached, the uplifting force of the knife was not effectively transferred to the whole width of the cut and the sharp edges of the blade started shearing or cutting through the coating. Measurements larger than 5 mm become unreliable. Obviously, coating thickness affected this phenomenon. Nevertheless, a 5-mm reading indicated very low adhesion regardless of coating thickness.

Table 4.5 Adhesion measurements from X-cut tests for all bars in millimeters (width of section at coating breakage).

Bar No.	Bar Size	Max. (mm)	Minim. (mm)	Average (mm)	σ (mm)	C.V. (%)
U-1	#6	4.0	2.75	3.3	0.3	10
U-6	#9	4.5	1.75	2.9	1.0	33
U-3	#4	2.0	1.0	1.5	0.3	18
V-1	#6	2.5	1.25	1.8	0.4	25
V-3	#9	1.75	1.0	1.4	0.3	18
V-2	#4	2.25	1.0	1.4	0.4	27
V-14*	#6	5.0	4.0	4.8	0.5	10
V-16*	#9	5.0	4.0	4.8	0.5	10
V-28**	#6	5.0	3.0	4.1	0.7	17
V-29**	#10	1.5	0.5	1.0	0.2	22
W-1	#6	5.0	4.0	4.9	0.3	6
W-3	#9	5.0	3.5	4.1	0.4	11
W-2	#4	5.0	3.0	4.4	0.7	16
W-16**	#6	5.0	4.5	4.9	0.2	4
W-17**	#10	5.0	4.0	4.9	0.3	6
Y-2	#6	1.5	1.0	1.3	0.1	11
Y-5	#9	2.0	1.0	1.8	0.3	19
Y-3	#4	2.25	1.0	1.5	0.4	26
Z-1	#6	1.75	1.0	1.4	0.3	18
Z-3	#9	2.25	1.25	1.6	0.3	17
Z-2	#4	2.0	1.0	1.5	0.3	20

* Nonbendable coating ** Plain bar

Average adhesion index and standard deviation for each bar are plotted in Figure 4.22. Most bars had deviated 15% or less from the average, except bars U-6, V-28, and W-2. All bars from the same coater were within one standard deviation from each other, except bars V-28 and U-3. In general, dispersion of adhesion ratings was less than in the strip method and was similar to that from the hot water test. Figure 4.23 illustrates the average adhesion readings obtained on all bars from each coating applicator. Performance of coaters followed the same order as in hot water tests. Among flexible coatings, those of coater W performed the worst and those of coaters Z and Y performed the best. Nonflexible coatings showed the worst adhesion.

It was evident in Figure 4.22 that no bar size consistently performed better or worse. Comparing performances as rated by hot water test, strip peel test, and X-cut peel test, no particular bar size performed better than others. The data seemed to validate findings from Phase 1, where no consistent trend was found between bar size and coating adhesion.

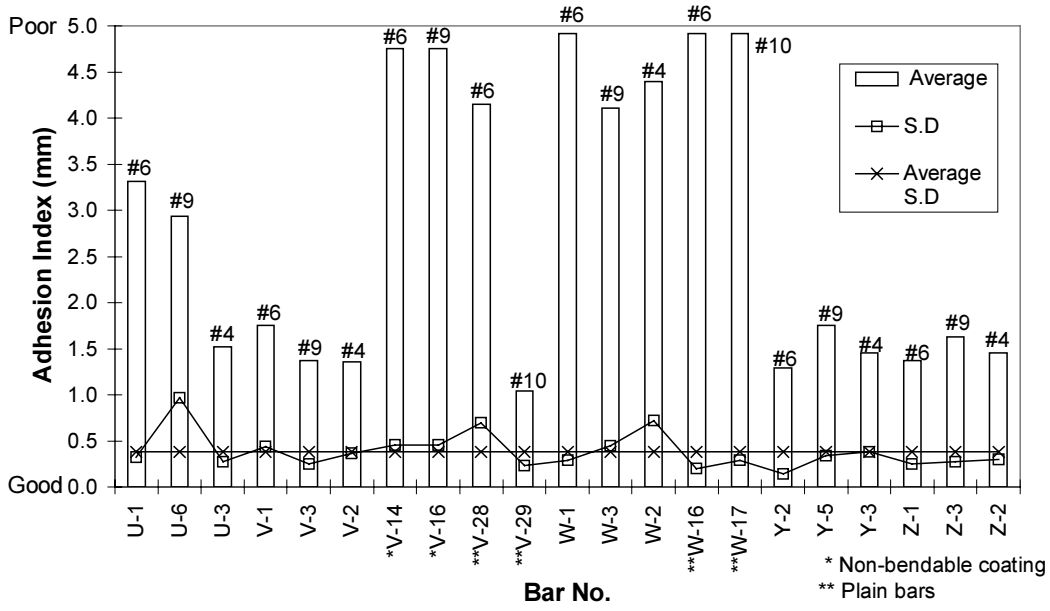


Figure 4.22 Average adhesion index of all bars in X-cut tests.

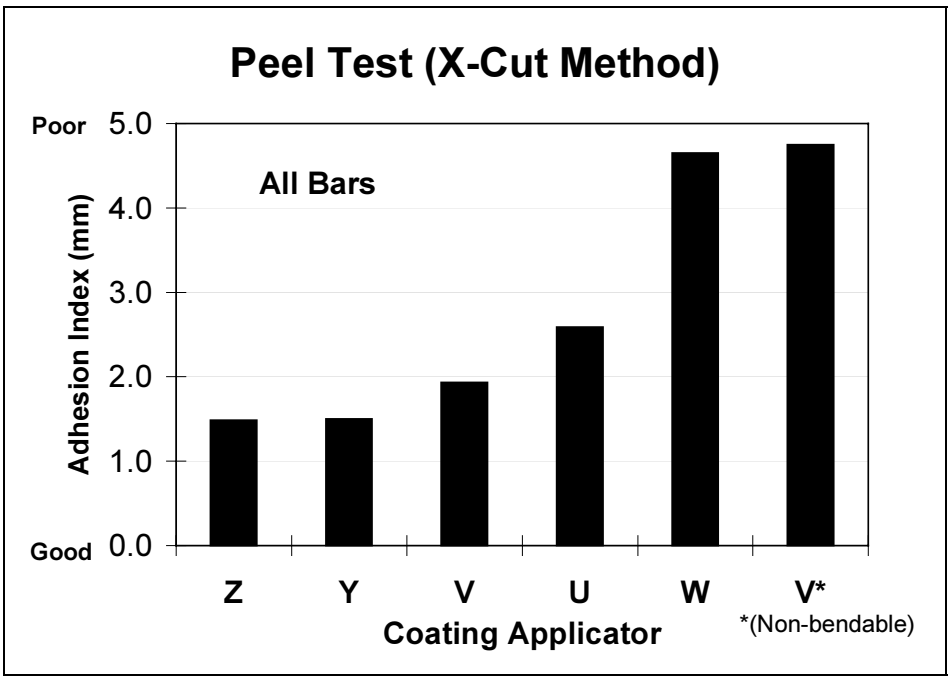


Figure 4.23 Coating applicator performance–Peel tests (X-cut method).

4.5 TXDOT PEEL TEST

The three test methods performed in Phase 3 provide a rating or index that was a good indicator of the coating adhesion of a bar. None of the methods defined a limiting value for acceptance. Such a value will have to be defined and based on field calibrations. Adhesion ratings are likely to be useful mainly for quality control.

TxDOT Peel Test is a subjective adhesion test that does not provide adhesion rating values, but determines only if a coating passes or fails. Test results from the three test methods described above were correlated with results from TxDOT Peel Test. Such correlation may give an approximate acceptance criterion until more definitive evidence is available.

Test procedure

The test was performed following the procedure described in TxDOT test method Tex 739-I presented in Chapter 1 (Section 1.3.4). Tests were conducted at the same locations as for the previous three methods, namely the two bar ends and the middle portion, on both sides of the bar. Only one operator performed all tests to reduce possible variations and subjectivity. An "OK" or "NG" (not good) rating was given to all test location but an overall pass/fail criterion was assigned to the whole bar. If poor adhesion (NG) was found in any of the three tested locations, the bar failed.

Test Results

In Table 4.6, results of TxDOT test method Tex 739-I are given. As expected, all bars from coater W failed the test. Bars with nonbendable coatings also failed the peel test. Rigid, nonbendable coatings were brittle and tended to break during the test instead of being lifted from the steel surface as a whole. Careful judgment had to be exercised to properly evaluate adhesion of rigid coatings.

Table 4.6 Adhesion performance of all bars–Tex 739-I peel test.

Bar No.	Adhesion Rating
U-1	FAIL
V-1	PASS
V-14	FAIL
W-1	FAIL
Y-2	PASS
Z-1	PASS
U-6	FAIL
V-3	PASS
V-16	FAIL
Y-5	PASS
W-3	FAIL
Z-3	PASS
W-17	FAIL
V-29	PASS
W-16	FAIL
V-28	FAIL

4.6 ANALYSIS AND CORRELATION OF ADHESION TEST RESULTS

The three methods for adhesion testing in Phase 3 are compared and analyzed in this section. Each of the three methods has a different rating system. Adhesion values were normalized to a common scale to allow comparisons of results from different procedures. In the rating for strip tests, a low index meant poor adhesion. The opposite was true for hot water and X-cut ratings. Results in strip tests were presented as a percentage of coating remaining after the test and had a maximum value of 70%. The values were subtracted from 70% to transform them to a percentage of epoxy coating that is removed in the test so that a high value indicates poor adhesion and a low value, good adhesion, as in the other two rating systems.

The values were normalized in two steps. The first step involved dividing all readings by the maximum value for each system. This produced a rating system ranging from 0.2 to 1.0 for the hot water and X-cut tests and from 0 to 1.0 for strip tests (before normalization, strip tests had minimum values of 0 while hot water and X-cut tests had minimum values of 1.0). The second step consisted of adjusting the normalized values of hot water and X-cut tests to a common scale from 0 to 1.0 by linear interpolation. Normalized index values approaching unity indicate very poor coating adhesion.

Average normalized adhesion ratings for all bars and all test procedures are plotted in Figure 4.24. The few results of the hot water test at 75°C on #9 bars are also included. Except for hot water tests at 75°C, the values from the three test methods exhibited the same general trends. Adhesion ratings given by the three test procedures were similar for most bars. The largest discrepancies were found for bars U-1, V-1, and Y-2. Even though X-cut and strip tests differed the most in terms of average difference of mean ratings, their mean values were closer to each other than to those for the hot water test in seven out of sixteen bars. No test method consistently gave higher or lower adhesion ratings, although there was a slight tendency for the strip test to give higher values (lower adhesion) in more bars (seven out of sixteen bars). Statistical analysis showed that dispersion of ratings between different test methods was not greater than the dispersion of individual ratings along the bar by one test procedure.

Of the three test procedures, the X-cut method seems to be the most practical. It was easier to perform than the strip method and did not require hot water immersion. There is no practical advantage in immersing samples in 55°C water before adhesion testing. If a more severe test is desired, a hot water test with water temperature of 75°C can be conducted. For adhesion testing after immersion, the X-cut method (shearing and prying) is recommended over the method that employs shearing only.

The three test procedures were correlated with test results from test method Tex 739-I (Peel Test). In Figure 4.25, the results of the TxDOT Peel Test are plotted with the average adhesion index values obtained for each bar. Good correlation is shown between results from the Peel Test and other tests developed in this study. Bars that failed the TxDOT Peel Test generally exhibited poor coating adhesion

by all methods. Only one case (bar V-1) passed the Peel Test but displayed poor coating adhesion by the strip method.

In Table 4.7, limiting values for the three test methods above which bars failed by the Peel Test are listed. Test ratings are presented and normalized for comparison. As noted from the table and from Figure 4.25, there seems to be a well defined adhesion index above which epoxy coatings can be considered to fail the peel tests. The normalized limiting values as given by the three test methods were very close to each other and to their average of 0.47. Of course, a much larger data base would be needed to determine limiting values for use as acceptance criteria.

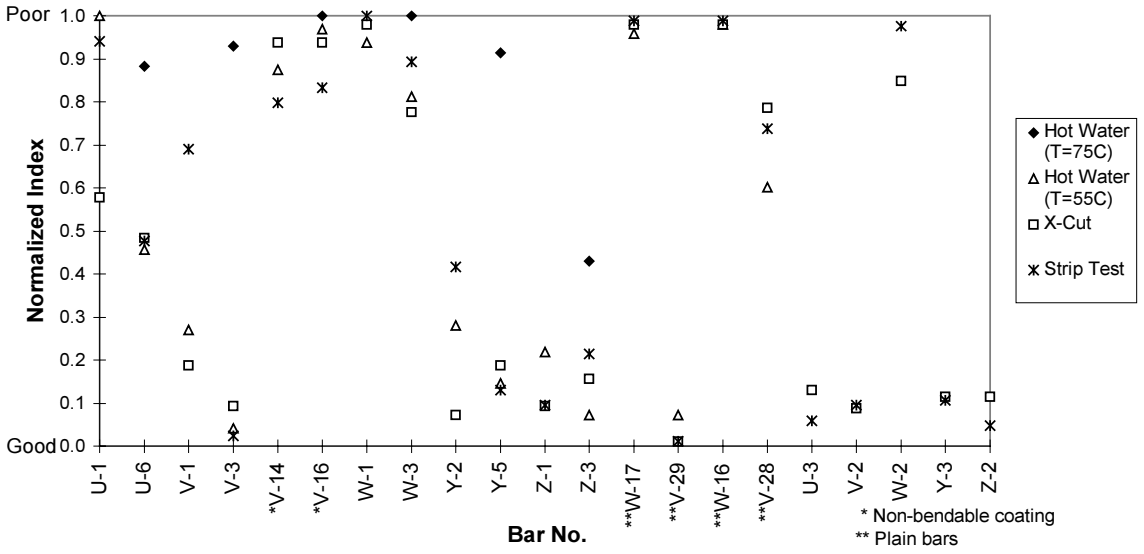


Figure 4.24 Relative adhesion values between all tests.

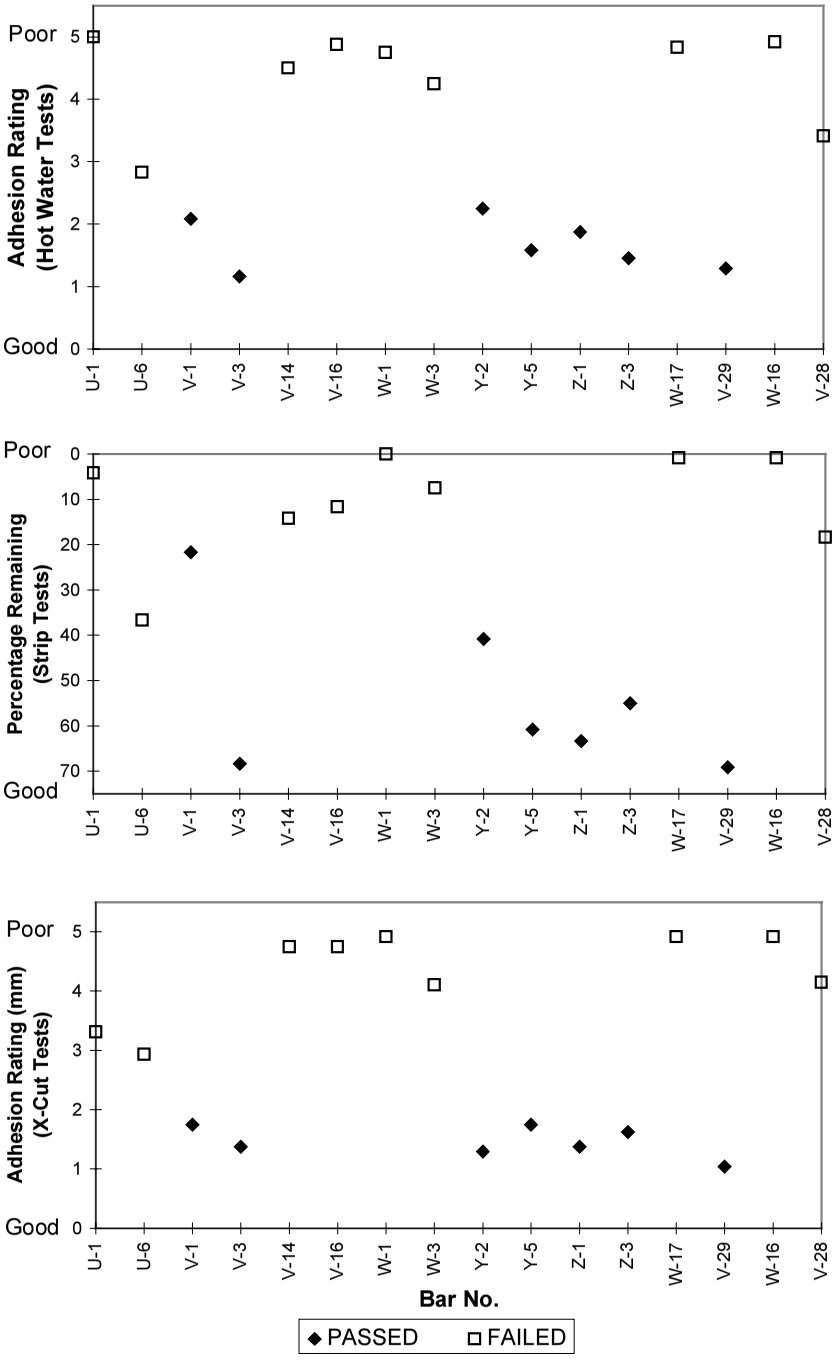


Figure 4.25 Adhesion test results from three test methods compared to TEX 739-I peel test rating.

Table 4.7 Limiting values of all adhesion tests with respect to TxDOT Peel Test.

	Hot Water Test	Strip Test	X-Cut Test	Average
Test Rating	2.8 (Bar U-6)	37% (Bar U-6)	2.9 mm (Bar U-6)	N/A
Normalized Rating	0.46	0.48	0.49	0.47

In Figure 4.26, the results of the bend test performed by the coating applicator (Section 4.1) are plotted with average adhesion index values obtained for each bar using the X-cut test. Clearly, there is poor correlation between the bend test and adhesion. All bars with poor adhesion passed the bend test. The only two bars that failed the bend test showed relatively good adhesion. Bend tests were not reliable indicators of coating adhesion in this study, yet they are often the only tests specified to evaluate coating adhesion.

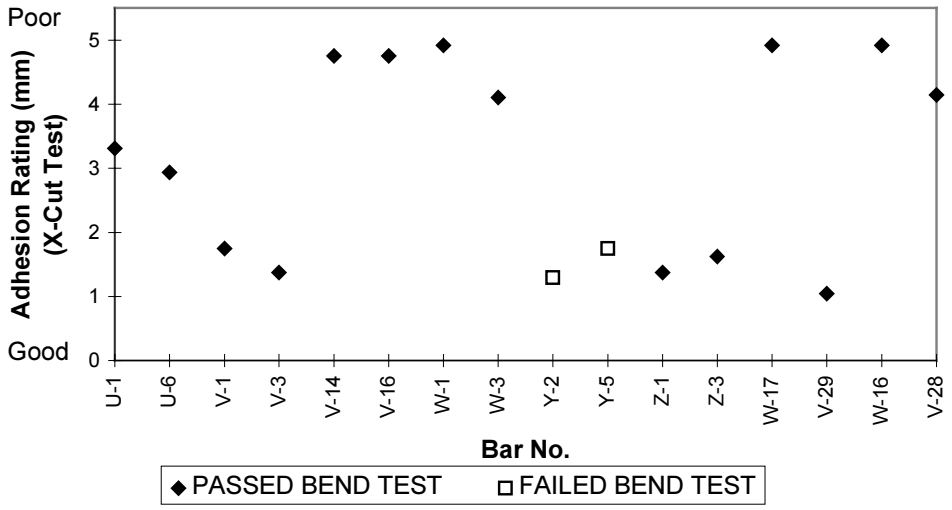


Figure 4.26 Adhesion test results from X-cut method compared to bend test.

In Figure 4.27, the average normalized adhesion index from the three test procedures is plotted against the coating thickness at the test location. As in Phase 1 of the study, no clear relationship was found between coating thickness and adhesion strength for the three tests performed, as evidenced by the large scatter of data in Figure 4.27. There was some tendency for coatings thicker than 14 mils to exhibit poor coating adhesion.

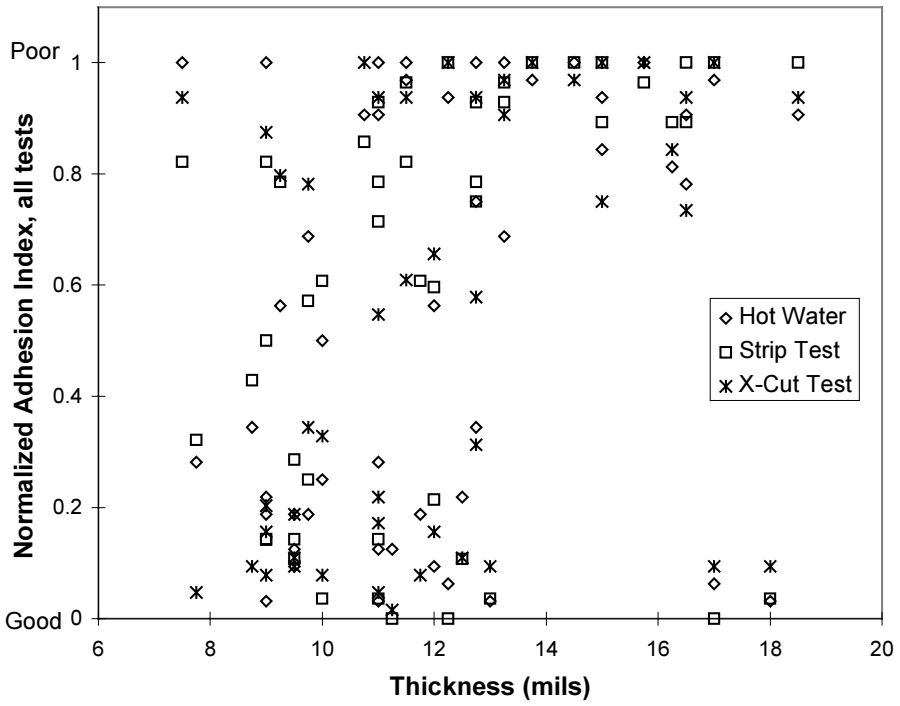


Figure 4.27 Coating thickness -vs- normalized adhesion index.

4.7 NaCl IMMERSION TEST

The role of coating adhesion in the corrosion protection of epoxy-coated bars is not completely understood. In an effort to gain some insight in this subject, a short exposure study was conducted. The study consisted of immersing epoxy-coated bar samples in 3.5% NaCl solution. It should be emphasized that the exposure study does not reflect the corrosive environment of epoxy-coated bars in concrete but provides a way to subject different epoxy coatings to the same chloride environment in a short period.

Test Procedure and Evaluation

Five-inch samples were obtained from the same three locations as for adhesion tests for every supplied bar. Ends of the specimens were sealed with a two-part epoxy resin. Samples were carefully inspected for the presence of defects or holidays. Any coating discontinuity was repaired with a two-part patching material. A 1/8-inch-diameter hole was drilled through the coating on both sides of the bar to expose the steel surface. This intentional discontinuity provided the same initial condition for all specimens and a place for corrosion to initiate.

Exposure consisted of 12 wet/dry immersion cycles; each cycle was 4 days wet and 3 days dry. At the end of the exposure period, specimens were allowed to air dry for 2 weeks before inspection.

Assessment of specimens after exposure consisted of (1) Visual examination of surface condition, (2) adhesion testing using the X-cut peel test at least one inch away from the pre-drilled hole (to test adhesion on a surface free from corrosion products), and (3) peeling of coating around the hole area to inspect the corroded area and to estimate coating adhesion in the zone of chloride attack.

Test Results

Coating adhesion was completely lost in the vicinity of the pre-drilled hole, which corresponded to the corroded area under the coating. A mix of dark brown and dark gray corrosion products filled the pre-drilled hole and the surrounding epoxy coating was rust-stained (Figure 4.28). A radial corroded area under the coating changed in appearance. Closer to the hole, dark gray corrosion products, sometimes with small brown rust areas, were prevalent. Farther away from the hole, corrosion products were dark to light brown. The appearance of the surface away from the pre-drilled hole in bars with good adhesion was shiny and clean, whereas in bars with low adhesion the surface was scaly and dull.

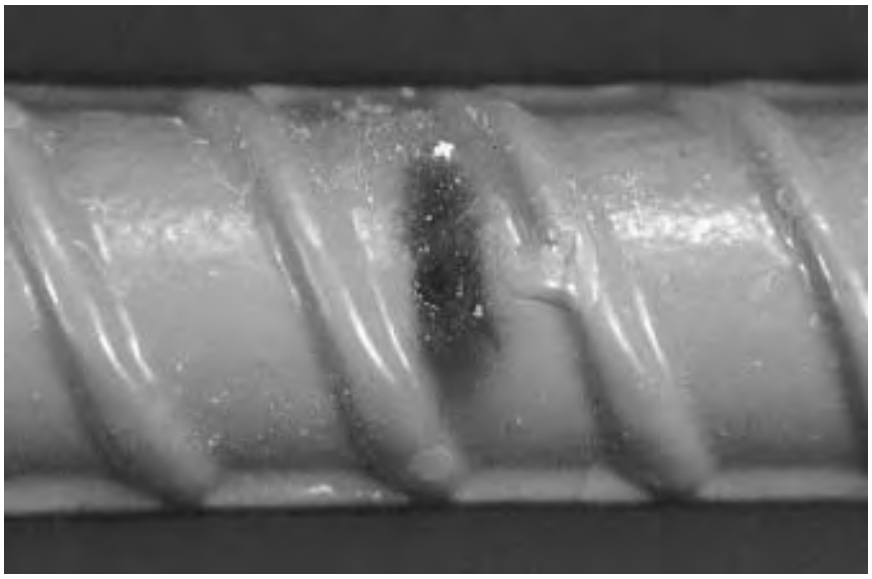


Figure 4.28 General aspect of specimens after immersion.

Average adhesion indexes from X-cut tests performed (at least one inch away from the hole) on all bars are plotted in Figure 4.29. Adhesion indexes for specimens without immersion are also plotted for comparison. In all cases, there was a slight decrease in adhesion as a result of NaCl immersion. This was expected because moisture promoted loss of adhesion of the organic epoxy coatings.

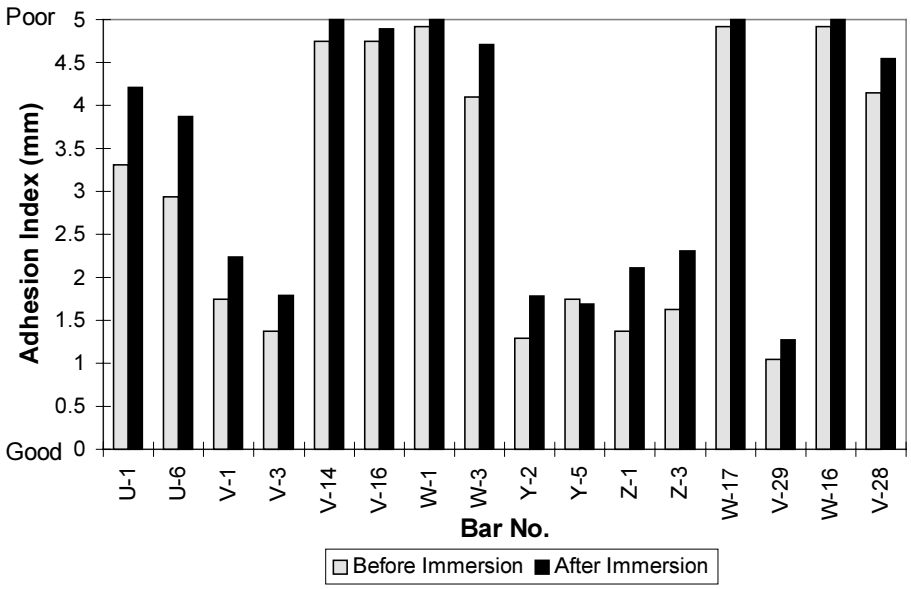


Figure 4.29 Adhesion index before and after immersion for all bars.

It was surprising that most bars with low adhesion before immersion showed less localized corrosion after immersion than those with better adhesion (Figure 4.30). A satisfactory explanation for this phenomenon could not be found. One hypothesis is that bars with varying adhesion strengths developed different anode/cathode area ratios. Very likely, salt solution penetrated easily under the coating in bars with low initial adhesion and made contact with a large portion of the steel surface, resulting in a greater anodic area compared to bars with better adhesion. For similar cathodic areas, the greater the anode the slower the corrosion. However, this hypothesis would be true only if the cathodic areas were of similar size in both cases. The larger steel area available for anodic reactions is also available for cathodic reactions in bars with poor adhesion, resulting in a larger cathode compared to bars with better adhesion. In addition, exposure conditions would tend to become similar with time in both cases because of progressive adhesion loss by moisture. A longer exposure could have produced different results.

Size of corroded area is plotted against adhesion index for all samples in Figure 4.31. There is wide scatter of the data and no clear correlation can be found. Nevertheless, there was a slight trend for bars with lower adhesion to have smaller corroded areas. As adhesion improved, scatter was greater.

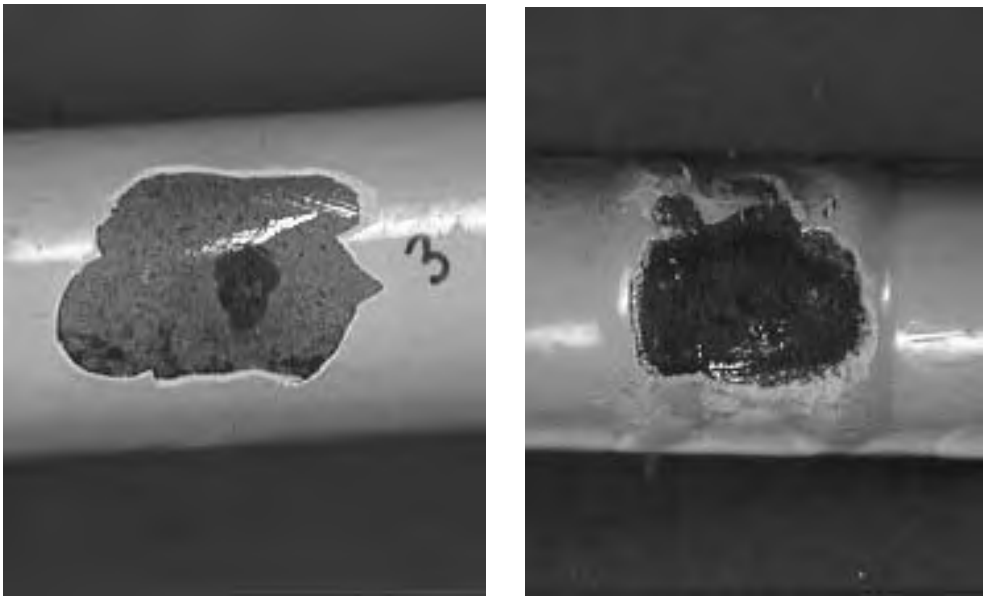


Figure 4.30 Damaged area in samples with good (right) and poor (left) coating adhesion. Note that the dark corroded area on the sample with good adhesion is larger than that on the sample with poor adhesion.

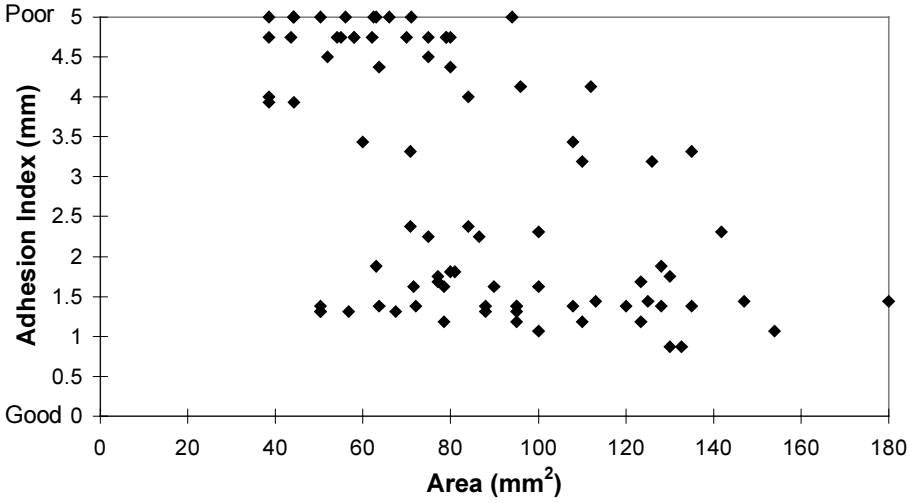


Figure 4.31 Size of corroded area in relation to X-cut adhesion index.

CHAPTER 5

Summary, Conclusions, and Recommendations

5.1 SUMMARY

The role of adhesion in the corrosion protection of coated reinforcement is not well understood. It has been claimed that inadequate adhesion may lead to early failure of the coating protection system. It has also been asserted that adhesion is a measure of quality of the coating application to the steel substrate. The main objective of this study was to develop a reliable, quick, and practical method to evaluate adhesion strength of epoxy coatings. Hot water and adhesion tests were performed on epoxy-coated bars from several coating applicators. A wide variety of variables was studied, aimed at both the development of the tests and at the assessment of their viability for quality control. Practicality and repeatability of tests were especially emphasized.

5.2 CONCLUSIONS

5.2.1 General Conclusions

The hot water and knife adhesion tests developed in this study proved to be a valuable tool for quality control and for in-depth studies of coating adhesion. Hot water and knife adhesion tests were very useful in discriminating and identifying good from bad quality coatings. The tests were relatively easy to perform and did not require special or sophisticated equipment. Most of the subjectivity involved in earlier tests was eliminated or reduced by the development and use of a calibrated knife. Nevertheless, it was shown that the subjectivity of the tests had little or no effect in the detection of coatings with poor adhesion. Test parameters such as knife force calibration procedures, adhesion test method, test operator, type of knife and blade, and test evaluator had little effect in the test results. The coating adhesion study that sample source was the most influential factor for adhesion strength, revealing that the quality of coating application by different coaters can vary greatly and affects coating adhesion. A knife adhesion test procedure is proposed in Appendix A.

An interesting observation was the good agreement between results from hot water-adhesion tests and those from the TxDOT peel test. Considering that the TxDOT peel test is simple and quick to perform, the test would be highly recommended for adhesion evaluation, especially if a calibrated knife is not available. Another important finding was the poor correlation observed between knife adhesion tests and bend tests. Bend tests were not reliable indicators of coating adhesion and gave an indication of coating flexibility. Therefore, the use of bend tests as the only method of evaluating epoxy coating adhesion (as proposed in some ECR standards) is discouraged.

In knife adhesion tests after hot water immersion, straight bars always performed better than bent bars. This finding confirmed that fabrication (bending) of bars weakened coating adhesion, as was found in durability studies. As was already discussed in Chapter 1, all macrocell specimens showed loss of adhesion at bend portions and adjacent straight legs after 2 and 4.5 years of chloride exposure, regardless of the level of corrosion activity. Likewise, coated stirrups in beam specimens showed widespread adhesion loss after one and 4.3 years. On most beams, adhesion loss was slightly more extensive on fabricated bars than on straight bars.

The effect of coating adhesion for adequate corrosion protection is not well understood. Coating powder manufacturers and a number of researchers claim that good adhesion is crucial for satisfactory corrosion protection.[13, 14, 42] It is presumed that a poorly adhered coating will allow unrestricted transport of water, chlorides, and oxygen beneath the coating, causing widespread underfilm corrosion. With the exception of one study at the University of Western Ontario,[3] there has not been a careful and systematic study of the effect of coating adhesion in corrosion protection, especially using coated bars embedded in concrete specimens. It has not been clarified whether it is the amount of damage in the coating or the adhesion of the coating to the steel substrate that governs the rate of underfilm corrosion and coating disbondment. In bar specimens immersed in salt water (discussed in Section 4.7), it was found that specimens with poor adhesion before immersion showed a smaller corroded area than specimens with better initial adhesion before immersion. If the conventionally accepted notion that poor adhesion leads to poor performance is true, then it would be expected that bars with better adhesion before immersion would have corroded less.

5.2.2 Specific Conclusions

Test usefulness

- Hot water and knife adhesion tests can be used to evaluate coating adhesion of epoxy-coated reinforcement. As such, the tests developed in this study are a valuable tool for quality control because they were very useful in discriminating and identifying good from bad quality coatings. Most of the subjectivity involved in earlier tests was eliminated or reduced. The tests were relatively easy to perform and did not require special or sophisticated equipment.

Test procedure

- Coating adhesion can be reliably evaluated by different methods. Test results were not significantly affected by changes in the testing procedure. Adhesion testing proved useful and meaningful even if performed in a subjective way.

- Adhesion testing using the X-cut method combining shearing and prying action with the knife was very practical and reliable.

- Hot water testing at a temperature of 75°C was severe for the bars received. Nevertheless, there were several bars that performed satisfactorily.
- Adhesion testing without immersion provided a measure of minimum quality, and adhesion testing combined with 75°C water immersion provide a measure of optimum quality.

Test parameters

In relation to hot water immersion:

- Temperature of the water bath was the most influential test parameter. A water temperature of 75°C proved to be more severe than 55°C.
- There was little effect of time of immersion for periods longer than 24 hours, presence of pre-drilled holes, and post-immersion periods longer than 6 hours.

In relation to adhesion testing:

- There was little effect of knife force calibration procedures, adhesion test method, test operator, type of knife and blade, and test evaluator.
- Adhesion loss was found to be directly proportional to the applied force of 3 and 4 kg.

Influencing factors

- The following factors had little effect on adhesion test results: Bar diameter, coating thickness and thickness variability, and original coating condition (damaged or undamaged).
- In all cases, straight bars performed better than bent bars.
- Visual examination of the steel surface in samples with poor adhesion revealed, in several cases, a very dark and scaly residue, possibly the product of improper surface preparation. However, in other cases a very clean steel surface was found, suggesting that factors other than surface preparation may produce loss of adhesion.
- Adhesion test results correlated best with sample source (coater).

Test repeatability

- There was a small variation in average adhesion at different locations on the same bar. Average adhesion of samples appears to be representative of the overall adhesion of a rebar.

- Small specimens were representative of the quality of a coating applicator if obtained from many bar lots. Bars from the same coater but from different lots may have similar adhesion ratings, but large discrepancies may occur during the production of certain lots.

- Dispersion and variability of data were independent of adhesion strength. If adhesion ratings are to be used as quality indicators, both the mean rating and standard deviation of ratings must be examined.

- Coefficients of variation may not be meaningful indicators of the dispersion of data. The higher the adhesion index (regardless of whether it represents good or bad adhesion), the lower the coefficient of variation.

- Standard deviation in peel tests using the strip method varied more than those from hot water and X-cut tests. Likewise, peel tests using the strip method produced more variation of adhesion strengths for different lots from the same coater.

Correlation between tests

- There was good agreement between results from the more controlled and objective hot water and knife adhesion tests with those from the more subjective TxDOT peel test.

- From correlation between adhesion tests and TxDOT peel tests, a limiting normalized adhesion rating separating good and poor adhesion was defined in the range of about 0.5 (1.0 indicates poor adhesion, 0 indicates good adhesion). A much larger data base would be needed to define a limiting value as an acceptance criterion for quality assurance.

- There was poor correlation between adhesion tests and bend tests. Bend tests were not reliable indicators of coating adhesion and were more a measure of the coating flexibility.

- Test results from immersion in salt solution were inconclusive. No clear correlation was found between adhesion strength and size of corroded area. Additional long-term research is needed to determine the effect of adhesion strength on corrosion protection of epoxy-coated reinforcement. Presently, there is no clear understanding of the relationship between these two properties.

Miscellaneous

- Nonbendable or rigid coatings were difficult to evaluate. Rigid coatings tended to break and tear when subjected to the shearing action of the knife.

5.3 RECOMMENDATIONS AND IMPLEMENTATION

5.3.1 General Recommendations

The relevance of coating adhesion and its relationship to corrosion performance could not be conclusively evaluated in the present study. Nevertheless, quality control measures to ensure adequate adhesion should be implemented. The rationale is that there are several factors during the coating process that effect adhesion of the final product. Such factors include surface cleaning and preparation, anchor pattern, quality of base steel, temperature during application, and curing time. Poor coating adhesion before the bars are placed in service is usually related to poor application of the coating at the plant.

Hot water and adhesion tests are useful and practical quality control tools for the evaluation of coating adhesion. Test procedures developed in this study are recommended for implementation but additional research must be conducted to substantiate the role of adhesion. In the meantime, acceptance criteria will have to be judiciously established. Since the effect of adhesion strength on corrosion protection is not clearly understood, a very stringent acceptance criterion may not be justified. If time constrains preclude more accurate evaluation, tests involving a higher degree of subjectivity and easy to perform could be implemented. An example of one such test is the TxDOT Peel Test. This research indicated that such a subjective test yielded results similar to those of more objective tests.

The use of bend tests as the only method of evaluating epoxy coating adhesion should be discouraged, as has been proposed in some standards. A combination of bend tests with adhesion tests will enable a better evaluation of the coating quality, assuring good coating flexibility and adequate adhesion strength.

Improved coating formulations incorporating chemical pretreatment of the steel surface can improve the adhesion of the coating and their use is recommended.

5.3.2 Specific Recommendations

1. Implementation of methods developed in this study to evaluate coating adhesion will reduce the subjectivity inherent in prior tests and can be useful for quality control.

2. Of the test methods developed in this study, the X-cut method using a combination of shearing and prying action with a calibrated knife is highly recommended. The only requirements are a calibrated knife, a utility knife, and a properly trained operator. If more resources and time are available, the test can be made more stringent by immersing samples in a 75°C water bath for 24 hours before adhesion testing. If hot water immersion is selected, pre-screening could be conducted at the coating plant by testing bars with the X-cut method. A test procedure is proposed in Appendix A.

3. The TxDOT peel test is simple and quick to perform and is recommended, especially if a calibrated knife is not available.

4. The use of bend tests as the only method of testing epoxy coating adhesion is discouraged.

5. More adhesion tests of nonbendable epoxy coatings are needed to gain a better understanding of their properties and to adjust testing procedures as necessary. Rigid coatings were found to be very brittle and had poor adhesion, but only a few bars were tested in this study.

6. Additional long-term research is needed to determine the effect of adhesion strength on corrosion protection of epoxy-coated reinforcement. Presently, there is no clear understanding of the relationship between these two properties. The effect of coating adhesion on corrosion protection must be established. Acceptance criteria based on adhesion strength alone will not suffice. It is suggested that for future corrosion studies, epoxy-coated bar samples be obtained from the same bars used for durability experiments, and adhesion strength assessed. Adhesion knife tests developed in this study, or cathodic disbondment tests, could be used for that purpose. At the end of exposure studies, adhesion loss and bar surface condition could be compared with the adhesion strength before exposure.

APPENDIX A

Proposed Knife Adhesion Test

A.1 SCOPE

A.1.1 The objective is to evaluate the quality of the adhesion between fusion-bonded epoxy coating and the steel surface of reinforcing bars.

A.1.2 The test provides an indication of the relative quality of coating adhesion after production but may not predict adhesion loss accurately during service.

A.1.3 Although a pass/fail criterion is not provided, consistent poor ratings may be cause for rejection. However, failure to pass the adhesion test does not necessarily mean that the performance of epoxy-coated bars will be unsatisfactory during service.

A.2 SUMMARY OF TEST METHOD

A.2.1 Adhesion testing may be performed after immersion in hot water according to Section A.8 to provide a very harsh test condition and to attempt to simulate the bar condition after long service. The use of hot water immersion is optional and left to the discretion of the testing agency. Adhesion strength is determined by trying to remove a precut area of coating with a test knife.

A.3 APPARATUS

A.3.1 Vise or similar clamping system with protective pads.

A.3.2 A testing knife calibrated to produce a constant force at all test locations, as described in Chapter 4.

A.3.3 X-acto blade #23

A.3.4 Utility knife with sharp blades.

A.4 SAMPLING AND FREQUENCY OF TESTING

A.4.1 Bars should conform to applicable specifications regarding coating thickness and number of holidays.

A.4.2 If hot water immersion is performed, replicate samples from at least three different locations along the bar should be obtained from each production bar tested.

A.4.3 If hot water immersion is not performed, adhesion tests can be performed directly on long production bars not less than three different segments along the bar.

A.4.4 At least two bars of each size from each production lot should be tested.

A.5 ADHESION TEST METHOD

A.5.1 Secure the sample in a vise. The vise clamps should have protective padding to avoid damaging the coating. With a sharp utility knife, cut an X through the epoxy coating. For bars smaller than #6, it may be necessary to make a V-cut to have an adequate testing area. The cut should

penetrate through the entire thickness of the coating so that metal is visible. The interior angle of the cut should be approximately 45°, but it can be modified to obtain more accurate results. Two adhesion tests are performed at the X-cut, one on each flap. Four "X" or eight "V" cuts are made on each sample between deformations (two or four on each bar side, respectively). No cuts should be made within the portions extending 2.5 cm from the bar ends. If testing is performed on long production bars, eight "X" or sixteen "V" cuts are made on each bar segment between deformations (four or eight on each bar side, respectively).

A.5.2 Position the tip of the test knife in the vertex of the flap formed by the "X" or "V" cut, making sure the blade is in direct contact with the steel surface. The knife should be held at an angle of approximately 30° tangent to the curvature of the bar.

A.5.3 Apply a 2 kg force to the test knife while rotating it about its longitudinal axis to create an uplifting effect to the coating. The blade should advance along the bisecting line of the angle formed by the "X" or "V" cut. The test is completed when the epoxy coating inside the test area breaks and is no longer removed in one triangular piece. Remove all lose and unbonded material. Repeat the procedure in all flaps. Use a new blade for each specimen (or bar segment of long production bar) or when the blade becomes dull or damaged.

A.5.4 Measure the width in millimeters of the last section of the epoxy coating flap that was removed before the coating failed. The width is inversely proportional to the adhesive strength of the coating and is termed "rating."

A.5.5 If the width is too small and difficult to measure, reduce the interior angle of the "X" or "V" cut and repeat the test. If the entire flap can be removed completely, increase the interior angle of the flap and repeat the test. Readings of 5 mm or larger are considered to represent poor adhesion and are all taken as 5 mm.

A.6 REPORT

A.6.1 Report the following information:

A.6.1.1 Adhesion rating (width in millimeters of flap at section where the coating failed).

A.6.1.2 Bar source, indicating type of epoxy powder, name of coating applicator, bar size, name of steel mill, and bar lot number.

A.7 INTERPRETATION OF DATA

A.7.1 Ratings of 1 mm or less are indicative of good adhesion.

A.7.2 Ratings of 4 mm or greater are indicative of poor adhesion.

A.8 HOT WATER IMMERSION (OPTIONAL)

A.8.1 Apparatus

A.8.1.1 Water bath with temperature control, circulator, and thermometer. The bath should be capable of heating water to the desired temperature with an accuracy of ± 2°C, and should have a circulator for stirring the water to obtain a uniform temperature.

A.8.1.2 Tap water

A.8.2 Preparation of specimens

A.8.2.1 Specimens 12.5 cm in length and free from bare areas are cut from production bars with a saw.

A.8.2.2 The specimen ends are sealed with an epoxy resin or similar material that provides a watertight seal. The seal should be fully cured before immersing the sample.

A.8.3 Test Method

A.8.3.1 Heat the water bath to a temperature of 75°C ± 2°C.

A.8.3.2 Submerge the specimens inside the bath. It is recommended that bars be suspended so that their entire surface is exposed to the circulating hot water. Samples should be spaced at least 2 cm from each other and from the bath walls.

A.8.3.3 After 24 hours ± 1 hr of immersion in hot water, remove specimens and dry at laboratory temperature (about 23°C ± 3°C) for at least 6 hours before adhesion testing.

A.9 KEYWORDS

A.9.1 Adhesion, knife adhesion test; hot water immersion; fusion-bonded epoxy coating; steel reinforcing bars.

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