Experimental Methods for Evaluating Epoxy Coating Adhesion to Steel Reinforcement

by

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Experimental Methods for Evaluating Epoxy Coating Adhesion to Steel Reinforcement

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Dedication

To my loving parents, how gave me the opportunity to pursue this life long dream

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Abstract

Experimental Methods for Evaluating Epoxy Coating Adhesion to Steel Reinforcement

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Three methods for evaluating adhesion of epoxy coatings to steel reinforcement are presented. These methods are based on applying a shear force in the epoxy/steel interface and recording the observed effect. The results from the three methods are compared by testing sixteen epoxy coated bars from five different coating applicators. Samples include deformed and plain bars, and flexible and rigid coatings. The effect of coating adhesion on corrosion protection is evaluated by correlating the initial adhesion of the coating with the area of concentrated damage found on an intentionally damaged sample immersed in NaCl solution for 12 weeks.

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CHAPTER 1: INTRODUCTION

1.1 BACKGROUND

Reinforced concrete is a material that makes use of the excellent compressive capacity of concrete in combination with the tensile capacity of some reinforcement. The result is a material that can develop high stresses in compression as well as in tension. Presently, the most widely used reinforcement material is deformed steel bars, due in part to their high ductility, adequate tensile strength and low cost.

Several assumptions are made when designing reinforced concrete structures, and most of them are taken for granted. One of this assumptions is that the steel reinforcement will preserve its integrity throughout the entire service life of the structure. If the reinforcing steel were damaged during this period, the capacity of the member to sustain load could be impaired, leading to serviceability problems, and in extreme cases, failure of the system.

Corrosion of the reinforcement reduces the durability of concrete members and is one of the most common causes of deterioration of concrete structures subjected to chloride contaminated environments. The electrochemical process that takes place in the vicinity of the reinforcement not only accounts for damage to the steel bars, but to the surrounding concrete as well. The volume occupied by the products formed during this process is greater than the volume of the original components, therefore high expansive pressures are induced in the neighboring concrete. Depending on the thickness of the cover and the quality of the concrete itself, cracking and spalling of the cover may occur, exposing the reinforcement to the environment with consequent further damage.

Corrosion of steel reinforcement in concrete is a very well understood process that involves the steel, oxygen and an electrolyte. It is logical to assume then, that an effective way of preventing the corrosion process from taking place would be to shield the reinforcing steel bars from the presence of oxygen and electrolytes. Sound, well designed and placed concrete should provide a passive and friendly environment to steel, isolating it from the rest of the products it needed to initiate corrosion. However, in some structures it is very difficult to create a perfect barrier between an aggressive atmosphere and the embedded reinforcement by relying solely on the concrete cover. Highway bridges subjected to freeze-thaw cycles are treated with sodium chloride (an excellent electrolyte in aqueous environments) and other deicing salts to prevent ice from forming on the running surfaces of the pavement, but the presence of chlorides produce a very aggressive environment. Bridges and structures in proximity to marine environments are also subjected to chloride attack. Chloride ions will diffuse through the cover concrete to the level of the steel and promote corrosion.

A number of methods have been proposed to reduce or inhibit the effects of corrosion in steel reinforcement. Some of them involve enhancing the properties of the concrete that surrounds the rebars in order to make it less permeable, but the majority involve treating the steel bar itself. The most common methods of protecting steel reinforcement from corrosion is by means of coatings, which can be of two kinds: Sacrificial or permanent. At present time, epoxy coatings are the most widely used method of protecting steel reinforcement from corrosion in reinforced concrete structures in the United States.

Epoxy coatings are permanent coatings applied to the reinforcement by fusion bonding. The function of the coating is to provide a barrier that will restrict the access of chloride ions which cause the steel to loose its passivity and corrode. Epoxy coating has been in use since the early 1970's, and reports of its behavior and performance have been satisfactory when compared against black or uncoated reinforcement. However, in the 1980's, corrosion induced problems in a number of new bridges containing epoxy coated reinforcement in the Florida Keys have raised a controversy as to the effectiveness of the product. Since then, important research has been conducted by several agencies in order to understand and predict the real performance of epoxy coatings. A long term research project is being carried out at The University of Texas at Austin to evaluate the corrosion performance of the material.

Research has shown that there are a number of factors which decrease the corrosion protection provided by epoxy coatings. Some of these factors are the integrity, thickness and flexibility of the coating, as well as the handling of the steel reinforcement before and after the coating application process. The quality of adhesion of the coating to the steel bar is studied in the project reported herein. The presence of poor bond in the interface between the coating and the steel surface is thought to lessen the corrosion protection provided by the coating. Any discontinuities present in the barrier may permit the ingress of chlorides or carbonation products, which may quickly spread along the bar if there is significant lack of adhesion.

Currently, there is no simple and reliable way of measuring adhesion of epoxy coatings for quality control purposes. Most experiments and tests that have been recommended are affected by some other physical property of the coating, therefore not providing a clear result in terms of coating adhesion.

1.2 OBJECTIVE OF THIS STUDY

Among the existing factors which impair the corrosion protection provided by epoxy coatings, epoxy adhesion to steel is one which has not received a lot of attention in terms of research for better understanding of its role.

Since epoxy coating adhesion appears to be an important factor in the corrosion protection capacity of epoxy coated reinforcement, the primary objective of this study was to devise a standard test to measure the adhesion of epoxy coatings to steel. It should be taken into account that the recommended test must be fairly easy to perform and should produce acceptable and repeatable results.

As a result of the measurement of adhesion, it is possible to study the variability of this parameter along a certain steel bar. This variability can then be traduced into an acceptance criteria which can be used for quality control purposes. Therefore, another objective of this thesis will be to study the variability of the adhesion of the coating in bars supplied by different epoxy coating applicators.

Acceptance or rejection of a shipment of epoxy coated reinforcement should not be based only on the variability of the results, but on the overall quality as well. A last objective of this study will be to correlate, if possible, adhesion and corrosion performance of epoxy coated rebars immersed in a chloride solution in order to provide a better understanding of the importance of adhesion in coated steel.

1.3 SCOPE

This work consists of seven chapters including the present one, where background to the problem has been stated.

Chapter 2 deals with the chemical nature and physical properties of epoxy coatings. Different uses for the material are discussed and the types of epoxy coatings are reviewed.

Epoxy coated reinforcement as a construction material is evaluated on Chapter 3. A short description of the corrosion mechanism of steel in concrete is shown, along with the nature of protection provided by epoxy coated reinforcement. The state of the art in production of the material will be presented, and a complete investigation will be performed on the issue of adhesion in the interface between steel and epoxy barrier.

Chapter 4 is a presentation of the current quality control methods. Even though this study deals mainly with adhesion, all of the measurable properties of epoxy coated reinforcement will be addressed.

Chapter 5 is a detailed description of the experimental procedures used to measure adhesion in the laboratory. Several methods are proposed along with the basic assumptions and theory behind each method.

The results obtained during the experimental phase of the study are presented in Chapter 6. It also contains the correlation between different test methods, while trying to define the best one for quality control purposes. Visual observations of the samples immersed in chloride solution are evaluated in order to provide some understanding of the role played by adhesion of the epoxy coating.

Finally, a summary of the study, along with conclusions and recommendations for future research are presented on Chapter 7.

CHAPTER 2: ORGANIC EPOXY COATINGS

2.1 DEFINITION OF ORGANIC EPOXY COMPOUNDS

The most common coatings used in the industry today to protect steel against corrosion belongs to the family of organic epoxies.

What is generally referred to as an epoxy is a chemical group consisting of an oxygen atom bonded with two carbon atoms, which are themselves already bonded in another ways [1]. Even though there is an immense number of combinations available, the most important from the corrosion protection standpoint are those called α-epoxy or 1,2-epoxy compounds. These consist of a three-member ring, like ethylene oxides and its derivatives (see Figure 2.1). The most widely used epoxy resin for coating reinforcing steel is a type of diglycidyl ether of bisphenol A (see Figure 2.2).

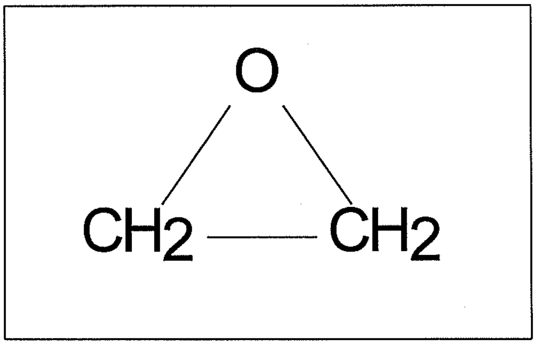


Figure 2.1. Ethylene Oxide or General α-Epoxy Molecule.

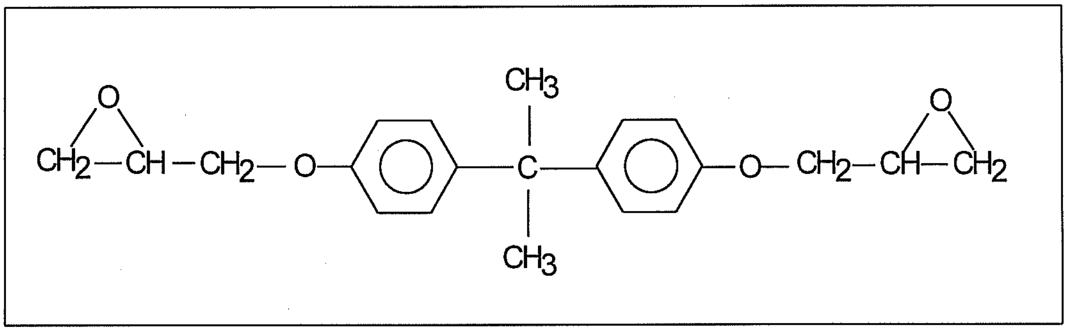


Figure 2.2. Diglycidyl Ether of Bisphenol A.

Two or more molecules from these group and combinations thereof form epoxy resins. The strict definition of the word epoxy applies to the pre-polymerized state of the compound, since after polymerization (in what is called the cured state), most of the epoxy groups may have already reacted. After curing, the compound may have lost its epoxy groups, but is still called an epoxy resin [2]. Also, in order for the compound to be considered an epoxy resin, the combination of α-epoxy groups has to be situated in such a way that it can be converted to a thermoset form which can be used. The term thermoset refers to the material in a cured state, as opposite to thermoplastic, which represents the material in un-polymerized state. Thermosetting polymers are the typical choice in the reinforcing steel coating industry, because after being polymerized, the physical properties are not supposed to change with sudden changes in ambient temperature [3]. Thermoplastic materials would not work well in such conditions; they tend to soften as temperature rises and become brittle in lower temperatures. Good examples of thermoplastic materials are PVC, nylon and polyesters.

There are mainly two forms in which epoxy resins are marketed. One of them is in two-part liquid form, and the other is in powder form. During the reinforcing steel coating process, the epoxy resin is applied in powder form as will be shown in following chapters. Two-part liquid epoxy resins are used as patching material for epoxy coated reinforcement subject to normal use in the field. Bars studied in this project were coated with powder epoxy resins only.

In general, epoxy resin powders consist of four different ingredients: The epoxy resin, pigments, fillers and additives [4]. The additives control physical properties like flexibility, hardness, flow control, etc. Every manufacturer has a certain combination of each of the ingredients to produce a powder with unique properties which will govern the overall quality of the final product. However, some powders produce excellent coating in certain applications while producing poor coating properties in others, so the manufacturer's recommendations must be followed when choosing a specific coating for any application.

The actual production of the powder can be by any of two methods. The first and most economical is to grind all the ingredients to a specific size and then combine them by means of a mixer; in what is called the Dry Blend method. The second method, known as the Fusion Mix method, requires melting the product of the dry blend method, allowing it to dry and then grinding it to a specific size powder. It is easy to note that the second process is costlier, since it involves the first one, but the final product will be of higher quality and more uniform consistency. The quality of the powder will eventually come into play at the different phases of the product's polymerization.

During reaction, the epoxy powder passes through different stages or phases, which are:

a) Melt stage: Immediately after the powder makes contact with the properly cured and heated substrate. The powder melts on contact and flows through the surface of the material.

b) Gel stage: Stage in which the product stops to flow and becomes quite hard.

c) Final curing stage: After application of appropriate temperature, the material hardens completely and there is final cross-linking of the epoxy molecule.

There are many methods for applying the epoxy powders to the substrate material [5], but only two of them are used for coating steel reinforcement. One system uses a fluidized bed, in which a tank is filled with the powder and compressed air or other gases flow through making the contents of the bed resemble a fluid. The previously cleaned and heated steel piece is then "immersed" in the powder in what is known as the "dipping method" of coating. This system is best suited for coating small components, or those having very complicated shapes. The second method of powder epoxy coating is by means of electrostatic spray guns, which provide the powder with an electric charge opposite to that of the base material before leaving the nozzle of the gun. The powder is sprayed from different directions and is then attracted to a heated steel surface, providing a very uniform coat if done properly. The only drawback to this kind of coating application is the high number of variables involved. The physical characteristics of the powder, like the roundness of the particles, will control the amount of charge retained, therefore controlling the thickness and continuity of the coating [6]. The reinforcing steel coating industry uses this method, due to its applicability to in-line production. High standards are set for the properties and physical attributes of the powders in order to make them suitable for electrostatic spray application. The coating process for steel rebar will be reviewed more carefully in Chapter 3. There are other processes for applying epoxy powder to the substrate, like the pouring method, centrifugal casting, rotational coating of pipes, rotational molding, flame spraying and many others. These are used mainly in the automotive and electronic industry and are of no interest in the reinforced concrete field.

2.2 TYPES OF ORGANIC EPOXY COATINGS

There are many classifications by which epoxy resins can be divided, depending on different factors. For example, as was mentioned before, epoxy coatings can be in powder or liquid form. Depending on their chemical composition, they can be epoxy esters, epoxy-alkyds, epoxy-acrylic resins, water borne epoxies, linear epoxies and many others [5]. It is not the scope of this thesis to evaluate or explain the chemical differences between all the mentioned categories, but it is very important to know that each one has different behavior after polymerization. The difference can be used to the coating industries advantage, by being able to produce a coating that will withstand different kinds of treatment, use and abuse.

Throughout this work a very simple classification will be used, based on the flexibility of the coating. This is:

a. "Bendable" or flexible coatings: Those that are applied to the steel reinforcement before any fabrication takes place. Even though there is a tendency for the material to debond from the substrate during normal fabrication, it should maintain its integrity without cracking or flaking.

b. "Non-bendable" or rigid coatings: Coatings which are applied to the steel bar after it has been properly fabricated. Further bending of the bar after the coating has been applied will debond, crack and damage the coating in the vicinity of the bend.

It should be noted that in this classification the rigidity of the coatings has not been related to their thickness, as could be expected, but to the inherent constitutive property of the material. During production of the epoxy resin, properties such as the flexibility can be controlled by the addition of different additives. "Non-bendable" coatings admit less deformation than "bendable" coatings of the same thickness, due in part to a higher modulus of elasticity. Thus, even though thickness will control the stiffness of two coatings of the same kind, it is not taken into account when categorizing different epoxy coating types. Recent developments have shown that rigid coatings can have better resistance to disbondment than flexible coatings, as well as being more difficult to damage in regular field operations [7]. Generally, the powder producer will use a different pigment for each type of coating in order to allow the coating applicator to distinguish between both (see Figure 2.3), and to provide visual warning of the kind of treatment the bar can be subjected to by fabricators and contractors.

Flexible coatings are the most frequently used for coating reinforcing steel and are commonly dyed with green pigments in order to make them easily recognizable.

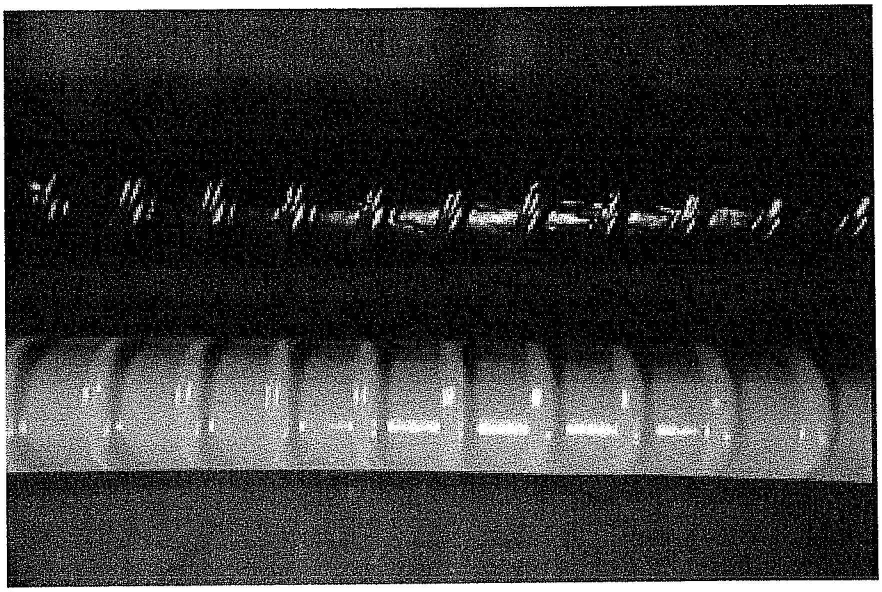


Figure 2.3. Rigid coating (top) vs. Flexible coating (bottom)

The American Society for Testing and Materials (ASTM), provides guidelines for quality control of the different kinds of coatings available for steel reinforcement. They include information on permissible coating thickness, continuity and adhesion, as well as on the condition and preparation of the reinforcing bar before the coating process takes place. Up until 1995, there had been no distinction of specifications for rigid and flexible coatings; they were regarded as the same material and assigned the same standard. ASTM A775 [8] is the original general specification that covers epoxy coated reinforcing steel bars, but it should be noted that the newer rigid coatings for steel fabricated before the coating process must also comply with ASTM A934 [9].

2.3 PROPERTIES AND CHARACTERISTICS OF ORGANIC EPOXY COATINGS

Epoxy resins posses a great number of advantageous properties which makes them the coating material of choice to protect steel reinforcement against corrosion [1,5,10]. The most important are:

a) Easy and fast cure: Curing of the epoxy resin depends on the curing agent selected during the manufacturing process. The epoxy resin powders used in the reinforced steel coating industry are developed to allow for a very fast transformation from gel time to final curing of the compound. Also, the material can be formulated to cure at extreme temperatures as long as the transition temperature of the material is not reached. Both of these properties are very important for the steel coater, because they permit faster production with high quality results.

b) Low shrinkage: During cure, high stresses can develop in thermosetting materials due to temperature gradients between the finished product and the outside environment. This is not the case with epoxy resins. Epoxy resins do not involve water in their formulation and suffer little rearrangement and reorientation in their molecular structure during the liquid or semigelled phases. Having no water to liberate and small internal rearrangement, epoxy resins provide low volumetric changes and internal stresses. The advantage of having low shrinkage is good coating integrity and no loss of bond due to shear stresses in the steel-epoxy coating interface.

c) High adhesive strength: There are numerous theories that account for the phenomenon of adhesion between epoxy resins and other material. For the case of steel reinforcement, adhesion is provided mainly by mechanical interlocking and what is known as chemical or adsorption adhesion. Mechanical adhesion is achieved when the coating resin conforms well to a key pattern impressed on the substrate material. By having low viscosity when in liquid state, epoxy resins easily invade the crevices present in well pre-treated steel, providing good interlock. Chemical adhesion is achieved when there is good molecular contact between the two materials, so adhesion develops because of intermolecular forces generated by the atoms of both materials. In the case of steel and epoxy resins, the presence of pendent hydroxyl groups in the resin interact via hydrogen bond formation with hydroxyl groups found in most metal surfaces, creating adsorption adhesion.

d) Excellent combination of mechanical properties: Epoxy resins have superior strength when compared to other kinds of organic resins, like polyesters and acrylics. As it was already mentioned, this is in part due to their lower shrinkage, which eliminates undesired stresses in the material. Impact resistance for thin film epoxy coatings ranges between 60 and 160 in-lb., which means that care must be taken during handling of the finished products to avoid damage to the coating. Pencil hardness of the product can be as high as 7H if desired.

e) Chemical resistance: Epoxy resins are fairly non-reactive and have low permeability when in a polymerized state making them excellent protectors against chemical attack. Protection of steel in concrete under chloride attack relies on this characteristics to avoid corrosion forming reactions.

Having excellent physical properties does not imply that organic epoxy coatings are free of flaws. One big disadvantage is their low exterior durability. When exposed to direct sunlight or any other source of UV rays, epoxies tend to discolor and become chalky in appearance. This is anyway, a purely aesthetic disadvantage, because the mechanical properties of the material are not affected by exposure. The chalky residue that appears on the surface can be removed by cleaning and wiping. Another inconvenience of epoxy coatings, mostly in powder form, is their short pot life and low humidity tolerance [11]. Epoxy coating facilities must evaluate the optimum rate of consumption in order to have adequate an adequate stock that meets the pot life of the product.

2.4 USES FOR ORGANIC EPOXY RESINS

As time passes , epoxy resins are finding their way into numerous fields of the manufacturing process. Epoxy resins are being used as coatings, fillers, adhesives, tooling and casting materials, and so on. By the year 1991 [2], more than 50% of all the epoxy resins produced in the United States of America were being used as coating materials of different kinds. The next most important use is for electrical applications, with approximately 13%. The rest of the epoxy resins produced were used for several other purposes, with varying proportions (see Table 2.1).

	1990		1991	
	1000 tonnes	**%**	**1000 tonnes**	**%**
Protective Coatings	89	49	84	51
Electrical Applications	25	14	22	13
Reinforced Resins	14	7.5	13	8
Bonding and Adhesives	13	7.5	12	7.25
Flooring	12	6.5	11	6.25
Tooling and Casting	13	7.5	12	7.25
Other	15	8	12	7.25
Total	181	100	166	100

Table 2.1. USA Applications of Epoxy Resins. [2]

In the field of civil engineering, epoxy compounds have been used successfully as adhesives, fillers and protective coatings. Epoxy compounds can be used to bond concrete surfaces together and fill cracks, for example. This is possible because of the high strength that can be achieved, which can be greater than that of the concrete being repaired. A recent development is that of concrete member encapsulation, where a concrete member is vacuum-filled and covered with an epoxy resin in order to provide greater durability for damaged or corroding structures.

The reinforcing steel industry began coating rebars with organic epoxy coatings during the early 1970's. Before that time, they had been successfully used in the coating of steel pipelines and tubing for the petroleum industry, achieving very thin but protective barriers between the metal and the aggressive surrounding environment. Most of the technology used to coat rebars was borrowed from the steel pipe industry, and has not changed very much in the last two decades. Steel pipelines are subject to corrosive conditions comparable to those present in concrete structures where deicing salts or chlorides are present, therefore the chemical formulation of the epoxy powders used for coating both components is very similar.

Due to the versatility of the powder coating process, not only steel reinforcing bars are being coated. Practically every steel component to be used in reinforced steel construction can be provided with an epoxy barrier to protect against corrosion. Some of these components include mechanical splice connectors, shearheads, casings and dowel bars for expansion joints, threaded plain bars for control and construction joints and many others. Several companies are offering epoxy coated steel mesh to be used as shrinkage and creep reinforcement for bridge decks. Also, epoxy coated steel chairs are being used to achieve the proper depth of epoxy coated reinforcement and post-tensioning cables in concrete members and avoiding surface staining.

CHAPTER 3: CORROSION PROTECTION OF REINFORCING STEEL BY MEANS OF EPOXY COATINGS

3.1 CORROSION MECHANISM OF STEEL IN REINFORCED CONCRETE [12,13]

It has already been mentioned that for a corrosion process to occur, there are three components which have to exist to allow the electrochemical reactions to take place: a reactive metal which can oxidize anodically, a reducible substance which provides suitable cathode and an electrolytic medium. It is generally accepted that these three exist in sound concrete structures, and that steel in concrete is protected against corrosion by a condition known as passivity of the reinforcement, not by preventing water and oxygen from reaching the level of the reinforcement steel. The passivity condition is a result of the presence of water and oxygen in sound concrete. Concrete can provide protection only as a barrier against deleterious substances, like chlorides and carbon dioxide, which tend to destroy the passivity of steel.

Before understanding the states of corrosion of steel in concrete it is important to know the most basic electrochemical nature of corrosion. Any metal immersed in an electrolyte can release atoms which pass into solution as positively charged hydrated ions. The site where this release takes place is known as the anode. The remaining free electrons have to flow through the metal until they find an electron acceptor, such as oxygen, that will consume them. The consumption of free electrons takes place at the cathode. The reaction is completed when there is a migration of ions through the electrolyte leading to the formation of soluble or insoluble corrosion products.

As concrete hydrates, the reaction between Portland cement and water produces sodium, potassium and calcium hydroxides, tending to make the existing pore water a very alkaline solution ($pH>13$) which facilitates the formation and maintenance of a protective oxide film on top of the steel surface. The presence of such a protective barrier combined with the elevated pH is what is called the passive state and it reduces the corrosion potential of steel to almost negligible levels, making concrete a very friendly environment for steel reinforcement. The loss of passivity of steel is dependent on the ability of the concrete cover to prevent the access of aggressive substances which will alter the pH of pore water and allow corrosion to take place. The most aggressive agents are atmospheric CO_2 and chlorides.

Carbon dioxide from the atmosphere reacts with calcium hydroxide on the concrete surface through the basic reaction:

$$CO_2 + Ca(OH)_2 \rightarrow CaCO_3 + H_2O$$

As a result , the pH from the concrete pore water changes drastically from 12.5-13.5 to about neutrality. As the outermost surface of the concrete carbonates, carbon dioxide progresses deeper into the concrete until it reaches the level of the steel reinforcement and depassivates the steel. The whole process is slow when concrete is sound and mixes are well designed and placed, but cracks or defects accelerate the penetration of carbon dioxide through the cover.

Chlorides are responsible for nearly all corrosion problems in concrete structures at the present time. They can be introduced in concrete by contamination of the raw material of the mix or by exposure to de-icing salts, seawater, or chloride contaminated soils. If the contamination comes from exposure to chloride bearing agents, these will diffuse through the concrete pores until reaching the level of the steel reinforcement in a sufficient concentration to create a condition where concrete is no longer able to protect the bar from corroding. The presence of cracks will permit a very high chloride concentration in the area surrounding the steel reinforcement, thereby depassivating small areas of the metal (see Figure 3.1).

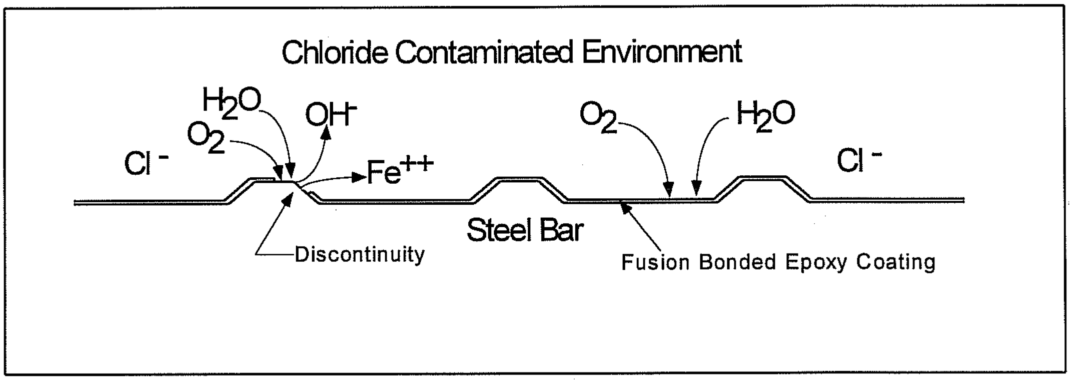


Figure 3.1. Activity at damaged spot on epoxy coated reinforcement

When there is galvanic action between large areas of still passive steel, which act as cathodes and small regions where there is high chloride content and reduced pH, the resulting damage produces pitting. The only requirement for pitting to occur is sufficient oxygen to polarize the anode to a higher potential than that of the surrounding environment. As pitting corrosion progresses, large areas of metal become depassivated, leading to more general corrosion.

Corrosion of steel in concrete is a time dependent process in which two different stages can be recognized.

a) The initiation period, starting from the moment steel is placed in concrete and lasting until steel depassivates. Non carbonated concrete, or concrete not exposed to chlorides will remain in this stage.

b) The corrosion period begins as soon as depassivation of steel commences and lasts until the structure reaches a limiting state where it is no longer considered acceptable under regular service conditions, architectural considerations, or safety standards.

Figure 3.2 illustrates this phenomenon. At the beginning of the service life of the structure the rate of damage is low and depends on the aggressiveness of the surrounding environment. As chemical attack progresses with time, steel looses its passivity. When passivity is lost, the rate of corrosion induced damage increases and it becomes difficult to predict the amount and rate at which damage takes place. Finally, the structure reaches a limiting state where an intervention is necessary to maintain the structure in service.

Various definitions of the limiting state are possible and involve the condition at which the structure is no longer serviceable. Severe loss of cross sectional area of the steel reinforcement and cracking and spalling of the concrete cover due to tensile stresses created by the corrosion process provide evidence of service failure.

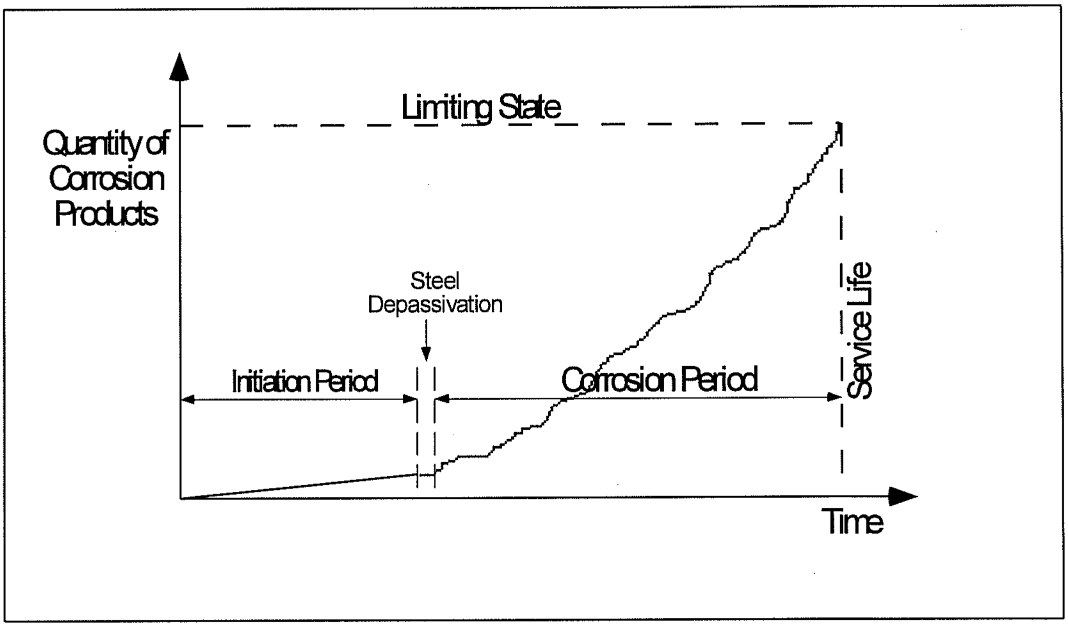


Figure 3.2. Service life components of steel in concrete.

3.2 PURPOSE OF EPOXY COATED REINFORCEMENT [7,14]

An efficient method for preventing corrosion of steel reinforcement embedded in concrete is to prevent penetration of aggressive substances to the level of the steel. When dealing with less severe exposure levels, enhancing the quality of the concrete cover and carefully controlling concrete placement may be sufficient. But, in very aggressive environments like concrete bridges in freeze-thaw zones where de-icing agents are used and in marine structures, the concrete cover alone may control migration of chlorides into the concrete. Permeability of even the best concrete mixes will allow some chlorides to ingress over time. Unlike the carbonation process, chloride diffusion requires the pores of the concrete to be filled with water, which is commonly the case in structures under de-icing salt action or marine structures.

Epoxy coatings are the most efficient and economical barriers commercially available for steel reinforcement. Epoxy coatings conform very well to the profile of the reinforcement bar and have low permeability. As a result, the time required for chloride ions to reach the passive layer of iron oxide surrounding the steel bar is increased and corrosion activity is delayed.

During the coating process, damage or imperfections may occur and are allowed by coating specifications. Such imperfections come in the form of pinholes and minor mechanical damage of the final product. Steel at these imperfections may corrode but the extent of damage is limited. Both the anode and the cathode have to be located in the vicinity of the hole or discontinuity, and the corrosion rate is slowed down due to the low efficiency of the cathodic reaction when compared to the anodic reaction. Epoxy coatings interfere with the existence of a complete corrosion cell and deter the spreading of corrosion from existing corrosion pits.

3.3 PRODUCTION OF EPOXY COATED REINFORCEMENT [3, 8, 9, 15]

The manufacture of epoxy coated reinforcement is a very simple procedure, yet it should be handled with utmost care. It involves the use of regular reinforcing bars as produced by steel mills with no special properties. The American Society for Testing and Materials provides regulations for the production of epoxy coated reinforcement in two different specifications: ASTM

A775 and ASTM A934, which cover bendable and non-bendable coatings respectively. Both processes involve preparing the steel surface, heating, coating and cooling the bar in a line of production. A more detailed explanation of each step is provided below.

3.3.1 Surface preparation

Regular reinforcing steel as provided by steel suppliers is covered by a small film of "mill scale". It is formed of dust and iron oxide resulting from the contact of the exterior steel molecules and oxygen from the atmosphere. This layer is loosely adhered to the bar and provides a very poor anchor for any kind of coating to be applied. In order to eliminate mill scale, abrasive blast cleaning to near white condition is performed (see Figure 3.3). Sand blasting, grit blasting and shot blasting are the most common methods used by the industry. The blasting process has a second function which is as important as removing the mill scale. In order for any coating to adhere to a steel surface, a suitable anchor pattern has to be provided. ASTM requires an average blast profile maximum roughness of 1.5 to 4 mils as determined by replica tape measurements. Recommendations are made about the composition of the blasting material and it is generally accepted that a suitable pattern is obtained by the use of grit or irregularly shaped particles in the cleaning media. After cleaning, the bars must be inspected for the presence of dust or additional mill scale and the process should be repeated until a near white condition and the required anchor pattern are achieved. Bars shall be coated as soon as possible after the cleaning operations, because flash rust tends to appear in the surface soon after blasting. In no case should a clean bar remain outside for more than 4 hours before coating.

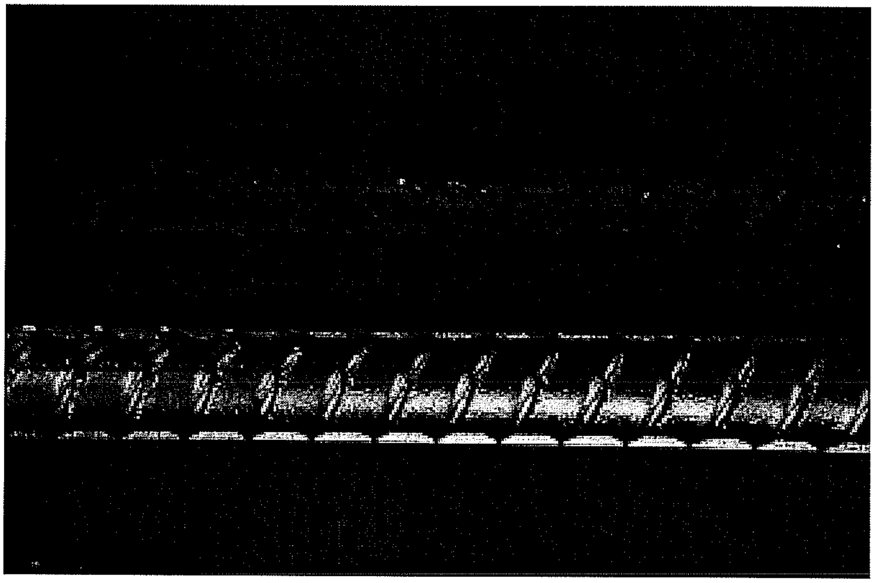


Figure 3.3. Steel bar before (top) and after (bottom) surface treatment

3.3.2 Heating

After the bar has been cleaned, it must be heated to a certain transition temperature which will "melt" the epoxy powder on contact with the surface of the bar. The heating temperature depends on the specific brand of epoxy powder being used, but a range of temperatures should be specified to account for differences and tolerances of different ovens. The common temperatures used for epoxy coating steel rebar range around 80 degrees Celsius. The heating process must be done in such a way that the bar is heated uniformly. The most widely used method is by means of induction ovens (see Figure 3.4), where the bar is rotated as it passes through a cylindrical cavity surrounded by electrical resistors which provide heat to the system. For special parts, like mechanical splices and dowel bars, any oven capable of providing uniform and controlled heat will work. Some small coating industries have successfully used "pizza ovens" for heating steel components. After leaving the oven, the temperature of the steel is checked by using temperature sensing crayons, which melt at a specific temperature.

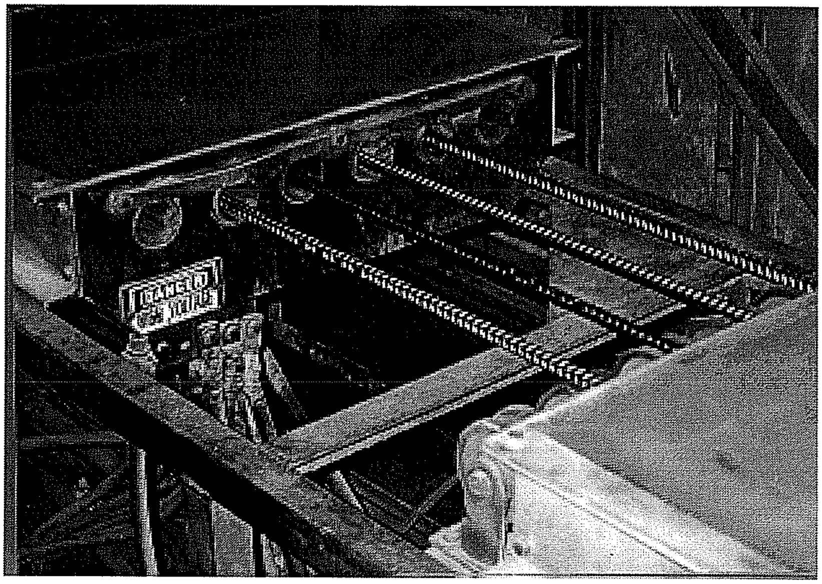


Figure 3.4. Steel rebars entering induction oven after surface pre-treatment.

Improper heating of the steel surface can mean a variety of problems for the finished product. If the surface is underheated, there will not be a complete reaction of the powder, with the corresponding lack of flow and longer gel times. A good indication of underheating is the presence of marks left by the conveyor system tracks. Overheating the surface will cause more severe problems. The epoxy powder will literally burn upon contact, and its physical and chemical properties will change. The coating will be extremely brittle and adhesion will be poor. Good indicators of overheating are discoloration and/or an orange peel aspect of the coating depending of the severity and duration of the exposure to high temperature.

3.3.3 Application of epoxy coating

As it was mentioned in Chapter 2, there are two methods used for application of epoxy powders to steel surfaces, depending on the particular application. The first one, used mainly for small components and those with intricate shapes, is called the hot dip method. It consists of immersing the piece of steel in a fluidized bed. The bed (tank) is filled with epoxy powder through which compressed air is pumped giving it the appearance of a liquid (see Figure 3.5). The method is labor intensive and time-consuming. The quality of the coating is highly dependent on the skill of the worker performing the dipping. The second method, used by almost the entire steel bar coating industry is called the electrostatic spray method. It consists of spraying the heated steel bar with electrically charged epoxy powder particles which are attracted to the metal's surface and enter their gel stage upon contact (see Figure 3.6). The spray method is suitable for automation and requires little labor to achieve good quality coatings.

Both methods require quality control and safety precautions.

- The source of air for fluidized beds and electrostatic spray guns has to be as dry as possible due to the low tolerance of epoxy powders to moisture.

- The voltage needed to correctly charge the particles in an electrostatic spray system range between 15000 to 25000 volts. Epoxy powders are highly flammable, therefore, the whole system has to be perfectly isolated to prevent the presence of sparks.

- Depending on the manufacturer of the epoxy powder, a certain amount of oversprayed epoxy powder can be recycled. Reclaimed powder must be added back to the system only in the specified amounts and mixed as evenly as possible with new powder. Screening and filtering equipment must be used to eliminate large particles and to dry the reclaimed powder before use.

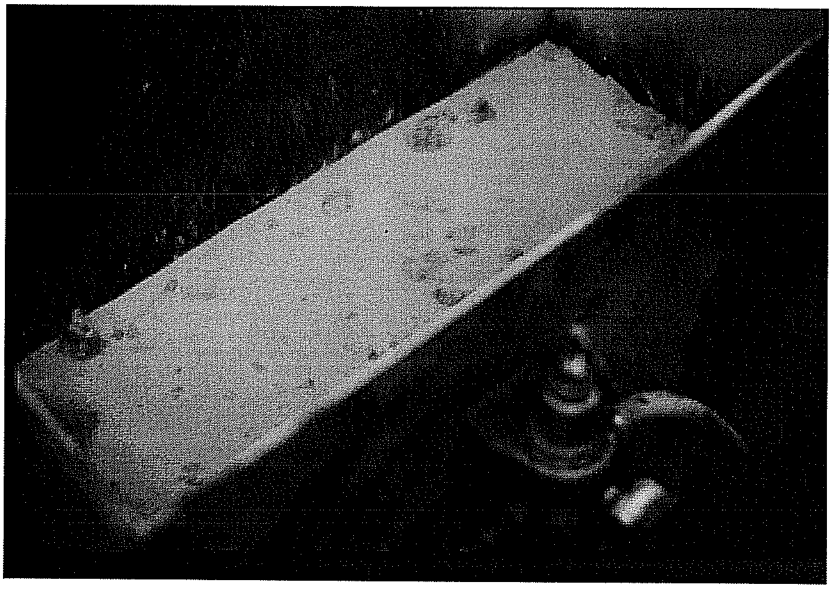


Figure 3.5. Fluidized bed used for dip-coating.

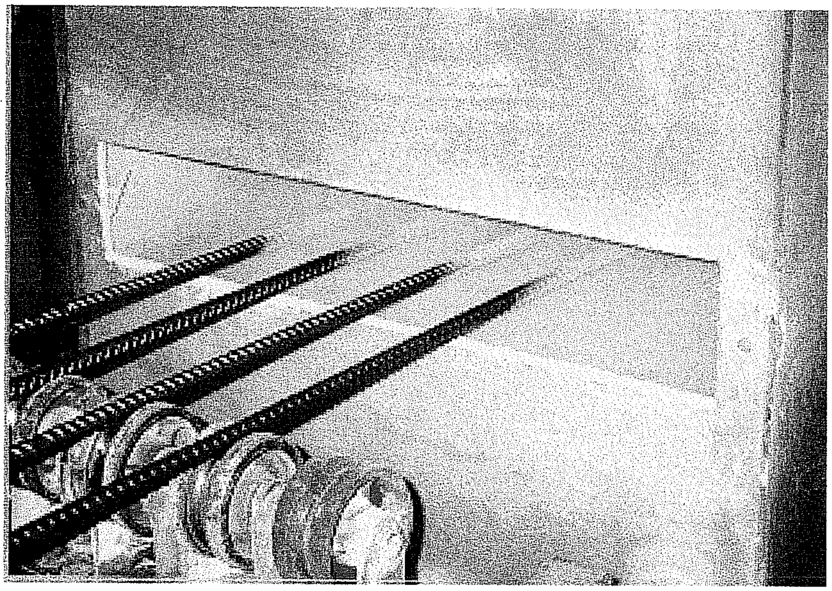


Figure 3.6. Steel bars leaving induction ovens and entering electrostatic spraying chamber

3.3.4 Cooling

The final step of the operation involves allowing the recently coated bar to cool down in order for the polymerization of the coating to be completed. After the steel bar has left the fluidized bed and electrostatic spray, it is rolled down the conveyor system and quenched through a fine mist of water. Smaller components, and those that have been hand dipped can be air dried instead of quenching. The advantage of the quenching process is that the steel bars can be handled immediately into bundles without danger of damage to the coating. It is common practice to have an in-line holiday detection system to check quality of the coating as the bar leaves the powder bed. Spraying the bar with water makes holiday detection easier and more accurate by giving the surface better electrical continuity.

3.4 ADHESION OF EPOXY COATINGS TO REINFORCING STEEL [16]

One of the primary objectives of this study is to find better methods for testing the adhesion of epoxy coatings to steel components used in reinforced concrete construction. Even though several methods can be recommended, it is very important to understand how adhesion works in order to provide efficient quality control tests which will not be affected by other physical properties of the material. The principles behind adhesion of any polymer to a metallic substrate are very complex, however, it is the intent of the following pages to provide an overview of the general phenomenon and the factors which affect it.

There has been great controversy about the role played by adhesion in the corrosion protection offered by epoxy coatings. It is generally agreed that epoxy coatings protect steel by providing a continuous coating which will interfere with the cathodic reactions needed to initiate the corrosion process. Recent studies of concrete prisms have confirmed that in areas where disbondment has occurred there is greater potential for corrosion development, but they also show that not all corrosion can be attributed or associated with loosely adhered coating.

The presence of corrosion products under the coating, resulting from damage to the continuous film, is responsible for loss of adhesion in the areas surrounding the discontinuity. It is therefore important to determine if a better bond between the steel and the coating will deter further loss of adhesion and prevent corrosion formation.

3.4.1 Nature and importance of adhesion of polymers to steel [17, 18, 19]

One of the primary characteristics of epoxy compounds is its excellent adhesion to metals. Very high initial adhesion has been achieved as long as all surface contaminants and weak oxide layers have been removed by any acceptable surface treatment.

The excellent adhesion of epoxy coatings to steel can be attributed primarily to the chemistry of the epoxy compound itself. It was mentioned in Chapter 2 that in the epoxy resin chain and the cured epoxy polymer, high polarity exists due to the presence of aliphatic hydroxyl and ether groups. The unavoidable presence of metal oxides in the surface of treated steel allows the formation of very strong electromagnetic attraction between both materials. The adhesive strength of an organic coating to steel is directly proportional to the hydroxyl group content of the epoxy compound. There is some contribution to adhesion by the formation of chemical bonds among active hydrogen in the metal surface and epoxide groups in the coating. All the chemical interaction that takes place in the epoxy-steel interface is called chemical adhesion.

Depending on the pre-treatment provided to the steel surface, some mechanical adhesion can be attained as well. When a roughened surface is provided or there is the presence of porous oxides on the surface, prepolymeric epoxy resin and curing agents can penetrate into the crevices and pores provided by the pretreatment. This being the case, when polymerization is finished, the coating becomes mechanically embedded in the metal surface or the surface oxide structure. Also, the cavities and pores formed during the surface preparation provide a larger surface area for electrochemical reactions to take place, therefore increasing the adhesive strength of the joint.

3.4.2 Variables affecting adhesion of epoxy coating to steel surfaces [18,19]

3.4.2.1 During the coating process

The first conditions leading to loss of adhesion of epoxy coatings to steel arise during the coating process itself. Some ways adhesion can be diminished during manufacturing are:

a) Failure to provide the steel bar with appropriate cleaning and surface roughening impairs the electrochemical reactions needed for good bond to exist. Weak oxide layers in the steel's surface cannot provide a good anchor pattern for mechanical interlock to take place.

b) Improper heating of the bar will cause the epoxy to have incomplete reaction or degradation. If polymerization is incomplete as a result of underheating, the epoxy will not be able to flow over the surface of the bar and fill the cavities as needed for good mechanical interlock. Degradation of the epoxy is the product of overheating the steel, and it reduces the electrochemical bond between steel and coating by creating new oxide layers in the surface of the steel which will not react with hydroxyl groups in the epoxy.

c) Quenching the finished product too fast can reduce the gel state time of the material. This provides reduced time to produce adequate mechanical bond. Another effect of fast cooling is a high internal stress build up due to the differences in thermal coefficients of expansion between the coating and the substrate.

Assuming good quality control in the coating process and an ideal coating operation, there are still other factors discussed below that affect the adhesion during the service life of the structure.

3.4.2.2 Moisture

It is agreed by nearly all who have studied organic coatings that water is one of the most destructive environments regarding adhesion in metal/polymer systems. The presence of water is a detrimental factor that limits the effectiveness of coating systems by interfering with the adhesion mechanism.

Under completely dry conditions, failure in an epoxy/steel interface generally occurs within the resin itself. This is different than the failure of the system upon exposure to moist environments. Depending on the length of the exposure period, failure tends to change from the resin to the interfacial region between the polymers and the substrate. The presence of water in the interfacial region is believed to be responsible for large reductions in adhesion strength if sufficient exposure time is allowed (see Figure 3.7).

All epoxy coatings have some permeability so that water will be able to reach the interfacial region of the coating eventually. The two ways by which water can reach the interface of the coating are by diffusion through the epoxy or by transport across the interface itself because of discontinuities in the coating.

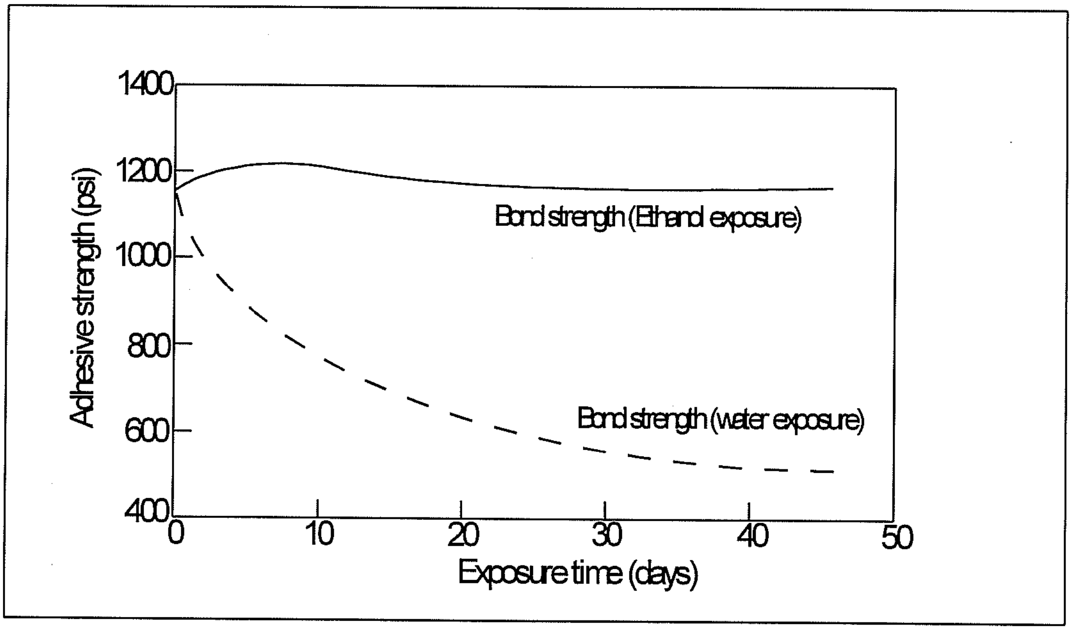


Figure 3.7. Deterioration of bond strength of epoxy coatings in the presence of moisture as compared to Ethanol.

The mechanism by which water promotes loss of adhesion is not completely understood but some excellent theories have been developed.

a) Displacement of epoxy by water: It is already known that electrochemical adhesion in epoxy/steel interfaces depends on strong hydrogen bonds. Water will eventually reach the interface region, and 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. The result will be the disbondment of the coating from the bar with the presence of a thin water layer at the surface.

b) Oxide layer deterioration due to hydration: Another way water can reduce adhesion is by hydrating the oxide layer on top of the steel surface. Generally, metal oxide hydrates exhibit poor adherence to their base metals, therefore, when a weak layer of hydrates is created in the interface, mechanical adhesion is reduced considerably.

c) Corrosion-induced displacement of epoxy: Discontinuities in the epoxy coating can readily produce a small corrosion cell in the area of the damage if the metal is exposed to an electrolyte. The chemical reactions responsible for the corrosion process can cause delamination or deterioration of the epoxy coating itself. The production of hydroxide ions are of interest because they can induce alkali hydrolysis of the resin. As delamination progresses, the cathodic site grows and moves, remaining just ahead of the debonding region and making the corrosion process more effective.

3.4.2.3 High temperatures

Epoxy compounds used for coating steel reinforcement are applied to the bar in the form of a thermosetting powder which flows as soon as a transition temperature is reached. Temperatures above the recommended application temperature tend to deteriorate the physical composition of the epoxy and render it useless. After the powder is applied, the epoxy polymerizes and forms a continuous coating with the same properties as the original compound. Should the final product be exposed to a temperature close enough to the transition temperature, the coating will become soft and flowable, making it very prone to damage. The condition worsens in the presence of moisture, because the loss of adhesion becomes irreversible due to causes already mentioned. It is interesting to note that the presence of high temperature alone will cause momentary loss of adhesion, but as soon as the epoxy cools down again, adhesion can be regained. This is easily demonstrated by placing an epoxy coated rebar in hot water and placing an identical sample from the same rebar in an oven at the same temperature. After a period of 24 hours, both samples are allowed to cool down and then tested for adhesion by any known method. There is high probability that the sample placed in the oven will have the same adhesion it had before starting the test, while the immersed sample will have a considerable reduction in adhesion strength. It is important to note that high temperatures, as long as not high enough to burn the epoxy, will only produce loss of adhesion when moisture is present. A moist environment alone will produce loss of adhesion over time, but temperature helps to accelerate the disbondment process.

3.4.2.4 Presence of initial damage

Discontinuities in the coating are not responsible for adhesion loss by themselves. They act as "open doors" for deteriorating agents to enter the steel/epoxy interface and produce loss of bond. Damage in the coating as small as pinholes are large enough for water, moisture and diluted chemical substances to make their way under the epoxy film. An epoxy coated rebar in a corrosive medium can have many corrosion cells located in the sites of any damage or discontinuity. As corrosion progresses, adhesion loss is intensified. New corrosion products formed in the steel/epoxy interface are responsible for total loss of adhesion in the corroded regions.

3.4.3 Determination of adhesion strength in epoxy coated reinforcement [8, 9, 20, 21, 22, 23, 24]

Evaluating adhesion of a coating to a surface is not a simple process. It depends on the properties and shapes of both materials, the forces needed to break the expected adhesion, the amount of time available, the accuracy of the method and the operator. In the case of epoxy coated reinforcement, one important question has to be answered: Is the adhesion of the coating to the bar adequate to prevent the fast growth of the cathodic region and the corrosion process?. Most authors agree that adhesion plays a very important role in protecting a coated steel surface against corrosion. Therefore, having an effective quality control method for testing adhesion may provide a good estimate of the performance of the coated bar during its service life.

There is currently no standard method for testing adhesion that has been accepted by a majority of testing agencies. The ASTM only makes mention of bend tests when dealing with adhesion of the epoxy coating. Some other tests have been proposed, but so far none have been efficient enough or their results have had an acceptable degree of objectivity. Some of the tests widely performed have the flaw of being affected by some other property of the material besides adhesion, which is the case of the bend test. Other methods depend highly on the judgment of the operator and provide very subjective results. A review of the most common methods used presently to test adhesion follows.

3.4.3.1 Hot water tests

Measuring loss of adhesion is one way to take advantage of the capacity of hot and moist environments of deteriorating the bond of epoxy/steel joints. Hot water tests involve immersing a sample of epoxy coated rebar in water or any other solution at a temperature higher than the surrounding air, but lower than the glass transition temperature of the given epoxy coating. The length of immersion, temperature and cooling period vary depending on the specification. After immersion, any one of several kinds of tests are performed, ranging from mere visual inspections to knife peel tests.

In 1989, the Swiss Federal Highways Office started performing a hot water immersion test for adhesion quality control. According to this method, test elements are stored for 7 days in hot water, at a constant temperature 10°C below the established glass transition temperature of the material. The ends and damaged or uncoated spots with an area larger than 25 mm^2 should be coated with a suitable repair material before immersion. After the immersion period, the samples are inspected visually for the presence of new damage or further damage in the repaired spots. Depending on the degree of new damage observed, the bar is given a damage rating that goes form S0 to S5 (see Table 3.1). Only a certain number of locations of any damage type are acceptable and failure to comply with the limit is cause for rejection.

Damage Type	Description (Significance)
S 0	No damage (evaluated by visual equipment)
S 1	Microscopic damage, recognized only by visual equipment (magnifying glass or microscope)
S 2	Damage that can be recognized by the eye, without visual equipment
S 3	Clear visual damage (0.1 1.0 mm^2)
S 4	Clear visual damage of larger dimensions (> 1mm^2)
S 5	Surface failure of coating (over 5mm^2)

Table 3.1. Damage type rating according to Swiss Federal Highways Office standards.

The Ontario Ministry of Transportation has a more specific hot water immersion test. It involves submerging 125mm samples in water at a temperature of 73±2 °C for a period of 48±2 hours. After the immersion period, the samples are allowed to cool at a temperature of 23±3 °C for a period of 24±2 hours. The following step involves cutting an "X" through the coating at six locations in each sample (three in the bottom, three in the top) making sure the cut extends through the coating such that metal is exposed. The length of each cut should not be smaller than 10mm or the distance between deformations and the two cuts necessary for forming the "X" should intersect at an angle as close to 90° as possible. The actual test consists of placing the point of a stout blade in the intersection of the cut and applying a force of approximately 3 Kg at a shallow angle with respect to the surface of the bar. The force should be maintained until the coating resists insertion of the blade. The amount of disbonded material due to the intrusion determines an adhesion rating (see Table 3.2).

Rating	Significance
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 slices easily under the coating and the entire coating can be removed at one or more sections

Table 3.2. Adhesion rating according to Ontario Ministry of Transportation.

According to the Canadian guidelines, the basis for acceptance of a lot of epoxy coated reinforcement is to have more than 95% of the sections provide ratings of 1, and no adhesion rating larger than 2.

Several other variations of the hot water test have been implemented. Many of them involve using different solutions instead of pure water, but always at high temperatures to accelerate the adhesion loss. Some of the most popular solutions used are sodium chloride, potassium hydroxide, sodium hydroxide and combinations thereof. The effect of using these solutions is twofold. Sodium chloride is a very corrosive environment for steel, and the hydroxides elevate the pH to a level in which the solution resembles chloride-contaminated mature concrete.

The main drawback on hot water tests is that they are of relatively long duration and samples must be shipped to the testing location were a suitable hot water source is located.

3.4.3.2 Peel or knife tests

The vast majority of the adhesion tests in use at present time involve the use of a knife with which different kinds of actions are applied. Most make use of the shearing action provided by a knife blade. Some tests, like the Canadian method mentioned before, involve using the point of the blade to apply a shearing action and for prying under the epoxy coating after a hot water bath has taken place in order to measure adhesion. The general idea of performing knife tests, by themselves or associated with any other tests, is a very common method of measuring adhesion which has found reasonable acceptance with the epoxy coating industry and state authorities.

The way a knife test works depends on the initial conditions of the test. If the samples have been exposed to moist and hot environments which have affected their electrochemical adherence, applying a force with the point of a knife is intended to break the remaining mechanical adhesion provided by the roughened surface of the metal. On samples that are tested in the plant with no special treatment before the knife test, the shearing stress produced by the knife has to overcome the combined electrochemical and mechanical adhesion. Thus, it would be logical to assume that the first condition is a better measure of adhesion during the service life of the bar because epoxy coatings have a tendency to loose their electrochemical adhesion in moist environments (like those present where epoxy coated reinforcement is used) over time. However, it could also be argued that both components of adhesion will be present in the first years of life of the structure and both should be taken into account.

Regardless of the kind of test being used, the intent is to apply a given shear stress at the interface between steel and epoxy coating. It is expected that the coating will lift from the substrate until a point is reached where the adhesion strength is larger than the applied shear stress. At that point, the knife will no longer progress under the coating or will cut through the epoxy coating itself.

In the Texas Department of Transportation Manual of Testing Procedures, test method Tex-739-I, introduces the "peel test" as an adequate means of measuring coating adhesion. The text of the standard method as presented by Texas DOT is as follows:

> 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.

Some difficulties arise when using this method for measuring adhesion. First of all, the correct placement of the knife before starting the force application depends on the operator's interpretation of the norm. The same can be said about the amount of force to be applied and the criteria for acceptance. If the test were to be performed in exactly the same way by all operators, the results might be sufficiently accurate to provide a basis for acceptance or rejection. A problem also turns out when making knife tests without performing predetermined cuts in the epoxy because the results will reflect the stiffness of the coating more than its adhesion to the steel surface. Peel tests as described above will be performed in the experimental phase of this project and compared against the results obtained with other proposed methods.

3.4.3.3 Bend Tests

The only method of testing epoxy coating adhesion to steel reinforcement mentioned in the ASTM standards is the bend test, which is permissible only on flexible or bendable epoxy coatings. For rigid coatings, cathodic disbondment methods are recommended.

Bend tests consist of bending a piece of epoxy coated bar around a specific size mandrel. The diameter of the mandrel, the bend angle after rebound and the time to completion of the test are dictated by the diameter of the coated bar (see Tables 3.3 and 3.4 and Figure 3.8). In this particular figure, no cracking is visible on the coating, even though adhesion was poor. The bending apparatus and mandrels must be protected with rubber sleeves in order to avoid mechanical damage to the coating. All test methods require the longitudinal deformations to be placed in a plane perpendicular to the mandrel radius.

After the bending operation has been completed, no cracking or splitting of the coating should be observed by the unaided eye in the outside radius of the bend, where high tensile stresses were produced. Also, no wrinkling should be present in the inside of the bend.

The main drawback of bend tests is that all the distinguishable patterns observed after the test, like splitting and cracking, are as dependent on coating flexibility as they are of coating adhesion. A flexible, but loosely adhered coating can pass the bend test without any immediate signs of distress. Days after the test has been carried out and the lot has been accepted, the coating may crack. A simple way to overcome this problem would be to observe the results some time after the bending operation has taken place.

Bar No.	**Mandrel Diameter (in)**	**Bend Angle (After Rebound, degrees)**	**Time to Completion (s)**
3	3	180	15
4	4	180	15
5	5	180	15
6	6	180	15
7	7	180	45
8	8	180	45
9	9	180	45
10	10	180	45
11	11	180	45
14	17	90	45
18	23	90	45

Table 3.3. Bend test requirements according to ASTM A-775

Bar Size	Mandrel Size	Bend Angle (After Rebound, degrees)
#3 through #8	6d	180
#9, #10 and #11	8d	180
#14 and #18	10d	120

Table 3.4. Bend test requirements according to Tx. DOT test Method Tex-739-I

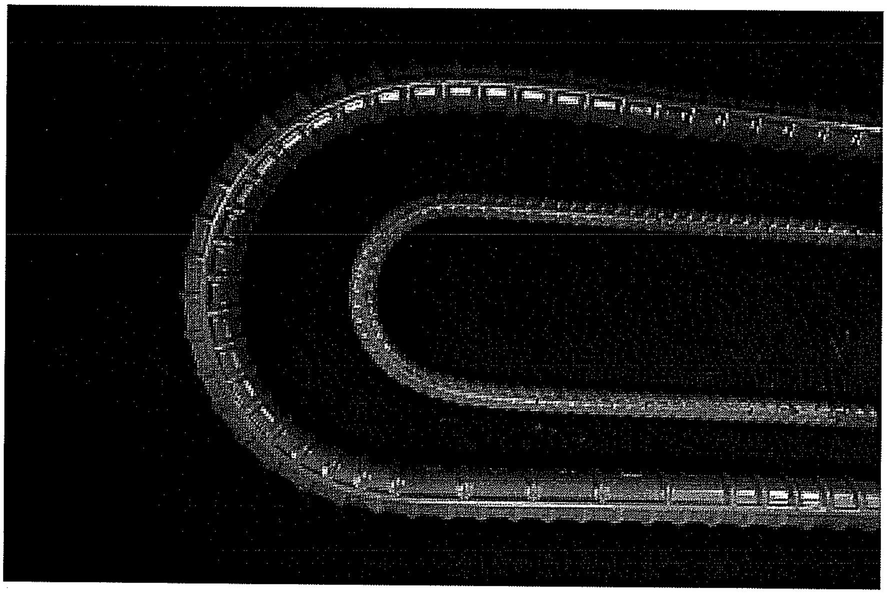


Figure 3.8. Appearance of steel bars after performing a bend test. Note the difference in mandrel diameters.

3.4.3.4 Cathodic Disbondment Tests

Another method used to measure adhesion of epoxy coatings on different coated applications is by cathodic disbondment.

The basic premise behind cathodic disbonding is subjecting the coating on the test specimen to electrical stress while immersed in a highly conductive, alkaline electrolyte. Before starting the test, the coating is intentionally damaged. After a prolonged time of exposure, which may go from 30 to 90 days, the samples are removed from the solution and new reference holidays are created in a zone not exposed to the electrolyte. Adhesion is measured by comparing the effect of attempting to lift the epoxy coating with a sharp thin knife in both the original holidays and the reference ones.

ASTM G-8 provides standard test methods for cathodic disbondment of pipeline coatings, and recently, ASTM A934 incorporated cathodic disbondment as a means of measuring coating adhesion in rigid epoxy coatings on steel reinforcement. The traditional bend test performed in flexible coatings is not suitable for rigid coatings, as the name of the material implies. Hot water and knife tests, however, can be implemented for rigid coatings.

3.5 QUALITY CONTROL OF EPOXY COATED REINFORCEMENT

Quality control of epoxy coated reinforcement bars is an involved process that goes from initial qualification of the epoxy powder to quality control of the final coated product. The acceptance criteria of a shipment of epoxy coated bars depends on mainly three factors:

a. Coating thickness
b. Holiday Detection
c. Coating adhesion

The first two will be explained in more detail in the following pages. The third one, which is the main subject of this thesis, received independent attention in part 3.4 and will continue to be studied in upcoming chapters.

3.5.1 Thickness measurement

Most regulatory agencies have set upper and lower limits on the allowable thickness of epoxy coatings. The lower limit is set due to durability and corrosion performance concerns. The thinner an epoxy coating is, the lower its capacity to withstand mechanical damage and contaminant intrusion will be. The upper limit is set to maintain a certain mechanical anchorage capacity in the steel bar. If the coating becomes too thick, the effective surface of the deformation of the bar is diminished and mechanical bond is impaired.

The most valuable methods for measuring thickness of coatings involve the use of nondestructive instruments based on a magnetic principle. The most typically used thickness measurement gauges are based on the pull-off principle and thickness is read on a movable dial. The advantage of such instruments is their light weight, ease of operation and portability.

Thickness measurements must be made in straight sections of the bar, avoiding the deformations and mill marks, where values tend to be larger. Also, different agencies have imposed different allowable limits.

The ASTM A 775 acceptance criteria is based on the fact that 90% or more of all the recorded thickness measurements lie between 7 to 12 mils, taking into account that any single measurement below 5 mils should be considered cause for rejection. A single recorded reading is taken as the average of three individual readings obtained between three consecutive deformations, and at least five measurements must be made evenly spaced along the bar.

The Texas Department of Transportation test method Tex 728-I allows the use of single point pull-off gauges. The limit values of thickness are contained in TxDOT Standard Specification 440, and also range from 7 to 12 mils. Also, a minimum of five readings must be taken along the bar and no more than 10% of the readings may fall outside the specified range.

3.5.2 Holiday detection

The process of scanning the surface of the coating for the presence of discontinuities is known as holiday detection. Discontinuities consist of visible damage and pinholes not detectable by the unaided eye. Holiday detectors based on electrical continuity principles are used to detect the presence of such pinholes. The coated bar is electrically connected to the ground pole of a 67.5V voltage source. Then, a wet sponge connected to the positive side of the circuit is passed along the surface of the coating. If any discontinuity is present during the operation, the wet sponge makes electrical contact with the steel and closes the circuit. The detector then produces an audible signal to alert of the presence of the defect.

Mechanical damage is often a result of rough handling of the finished product, while pinholes are a product of improper coating procedures. There are limits on the amount of pinholes a steel bar can have per foot of length, again depending on different regulatory agencies.

ASTM standards allow no more than two holidays per linear foot of bar. These deficiencies do not take into account those produced by mechanical damage, only pinholes invisible to the naked eye. Mechanical damage to the coating should be patched according to the coating manufacturer's recommendation.

CHAPTER 4: EXPERIMENTAL PROGRAM AND PROCEDURES

The experimental program involved different adhesion tests which were developed or modified from existing procedures. The basis for each procedure is presented here. The goal was to examine correlations between tests and to recommend a suitable test method for quality control for adhesion of epoxy coatings.

Epoxy coated bar samples were requested from five different coating applicators (to be provided from production runs). The request for steel included the following:

- One piece coated deformed bar (6 ft long) in each size (#6 and #9)
- One piece coated plain bar (6 ft long) in each size (#6 and #9)
- For each straight piece of bar (mentioned above) four bent bars were provided, obtained form the same original bar as the straight pieces. Bars were bent according to TX DOT specification Tex 739-I

Also, the coating applicators were asked to supply straight bars with a rigid coating applied, if such coatings were available from their plants.

The inconvenience of delivering small orders of material made response from the coaters somewhat slow and in some cases the coating applicators failed to provide the bars as specified. Table 4.1 provides a list of the materials as supplied.

		COATER				
PRODUCT	**ORDERED**	**U**	**V**	**W**	**Y**	**Z**
Straight deformed bars	1#4 1#6 1#9	O.K	O.K	O.K[a]	O.K	O.K
Bent deformed bars	4#4 4#6 4#9	4#4 4#6 3#9	4#4 4#6 2#9	O.K	O.K	O.K
Straight plain bars	1#6 1#9	---	O.K[b]	O.K[a]	---	---
Bent plain bars	1#6 1#9	---	O.K[b]	O.K	---	---
Straight deformed bars (rigid coating)	1#4 1#6 1#9	---	O.K	---	---	---
Straight plain bars (rigid coating)	1#6 1#9	---	O.K[b]	---	---	---

O.K = Product supplied as ordered
a = Coater only supplied lengths of 3’9”
b = Coater provided #10 bars instead of #9

Table 4.1. As-supplied materials for experimental program

Most of the bars were received in some form of protective case or wrapping to reduce damage during shipment. Some coaters provided casings which were individual plastic sleeves and offered very good protection, while others merely placed the coated steel bars in a bundle and wrapped them in packing material.

4.1 QUALITY CONTROL OF AS-RECEIVED BARS

Upon arrival, each one of the submitted steel bars was measured, classified (by coating type, deformation pattern, etc.) and labeled. The label presented the manufacturer's letter designation and a number which signifies the bar number. This bar number was maintained throughout the project. Other parameters were added as they became necessary.

After labeling, the compliance of each bar with ASTM and TX DOT standards was determined. First, a visual observation of the bent samples to determine adhesion loss was done as recommended by ASTM A775 and Tex 793-I. The coating on the outside of the bend was inspected for cracking or splitting, and the area inside the bend was examined both visually and by hand for the presence of wrinkles in the coating. A magnifying glass was used if no imperfection was noted by the unaided eye. It must be remembered that the bent bars came directly from the coating applicator and the precision and dependability of the bend test operation could not be checked for compliance with the specifications.

The thickness of the epoxy coating on each bar was measured with the aid of a MIKROTEST approximating thickness detection gauge. The gauge operates on magnetic principles; the thickness of the coating is proportional to the strength of a magnetic field which keeps the gauge probe in contact with the surface. The probe is pulled away from the surface until its own weight makes it loose contact and at that point thickness can be measured. The thickness detection process was done in the following manner:

- The steel bars were divided in one foot long sections
- Thickness measurements were taken at two points in every section on each side of the bar. The points measured were those corresponding to ¼ and ¾ of the length of the section

- The average thickness of the coating was calculated by obtaining the mean of all the obtained values. Also, the standard deviation and coefficient of variability were obtained.

The process was performed in a clean area, free of magnetic interference from large pieces of metal or electronic equipment, and after the bar was cleaned to eliminate all dirt, grease or any other obstruction. In deformed bars, the test was performed in the straight region between lugs (see Figure 4.1). The coating thickness was compared with the limiting values specified in TxDOT Standard Specification Item 440. According to the standard, thickness values must range between 7 to 12 mils.

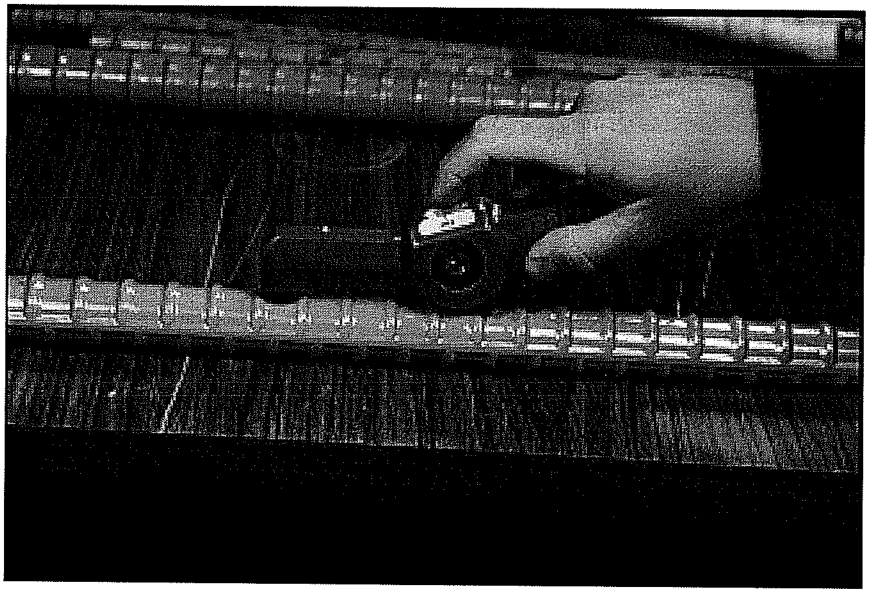


Figure 4.1. Thickness measurement on a deformed steel bar.

The holiday detection process was the last step in assessing the quality of the steel as-received. The objective was to determine the number of pinholes and discontinuities present in the steel bars. A pinhole is an imperfection in the coating not discernible to the unaided eye. All those imperfections visible without the use of visual aids were patched according to the manufacturer's specifications before starting any tests. A 67.5 Vdc holiday detector was used for detecting discontinuities. Holiday detection was done in the following manner:

- The bars were marked to identify one foot long sections (the same ones used for thickness measurement)
- All imperfections visible to the naked eye were patched
- The holiday detection probe was passed over the surface of the bar. Every signal from the detector was checked carefully to identify all possible pinholes at a location
- The number of pinholes in each section was recorded .

The number of pinholes in every bar was compared to the values specified in ASTM A775. The average number of pinholes per unit length was also obtained. During the test, care was taken to maintain good electrical contact between the probe and the steel surface by keeping the sponge wet at all times and by providing a clean, uncoated location for electrical grounding of the circuit. Special attention was paid to detecting discontinuities around steel bar deformations and mill marks, because such locations are more prone to pinholes and damage.

4.2 DEVELOPMENT AND DESCRIPTION OF ADHESION TEST METHODS

Considering current techniques, there does not appear to be a test method that provides a reliable means to quantify adhesion. The basic assumptions behind the tests used here are based on earlier research on the properties of epoxy coatings and were not altered. Two test approaches were examined:

a) Hot water controlled peel tests

b) "Non-immersion" controlled peel tests

Both types of tests are used today in some form or another. For this program, they were only modified to make them more precise and repeatable. Also, both of the test varieties make use of a knife or blade with which a specified force or action is applied to obtain the adhesion rating of the coating. A major problem in the current procedures has been the difficulty in applying a controlled force to the knife.

As a first step in the process of developing a reliable test method, a test knife was developed. The logical solution was to construct a spring loaded apparatus that comprises an X-ACTO hobby knife inside a hollow tube were the spring is housed (see Figure 4.2). A small scale on the side of the knife permits the measurement of the deformation in the spring which is proportional to the applied force. After this tool was developed, it was necessary to find a suitable blade. A symmetrical blade with a relatively high stiffness was needed for performing these tests. Various commercially available blades were tried. The best blade available for this purpose was found to be the X-ACTO No. 23 blade. For good results, the blade must be replaced daily or after a given number of tests.

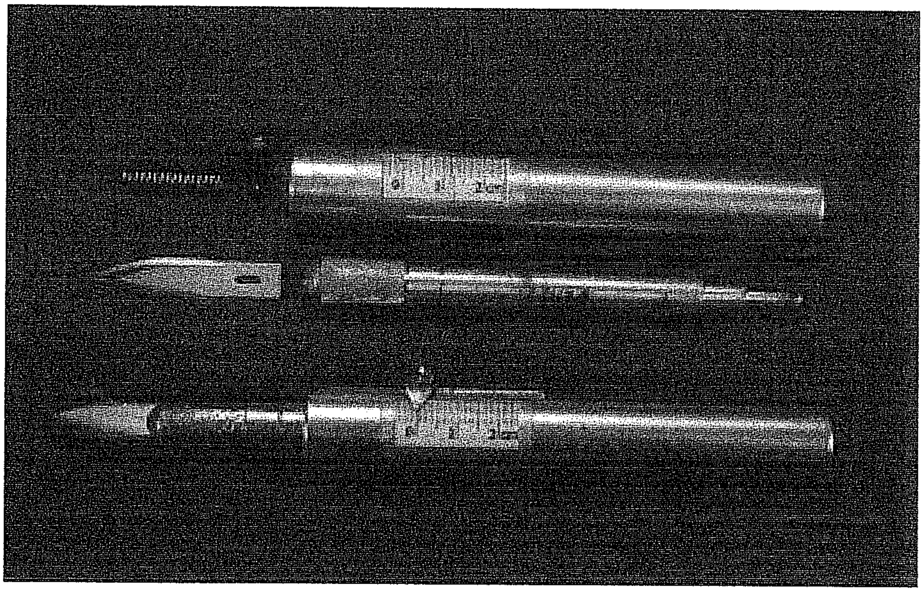


Figure 4.2. Adhesion test knife. Disassembled (top) and assembled (bottom)

Three test methods to measure coating adhesion were developed and will be described in the following pages. One of them is a hot water test, while the other two are controlled peel tests that can be performed in the field.

4.2.1 Hot water test

The hot water test developed in this thesis is a modification of the Ontario Ministry of Transportation's test procedure. The objective was to make an already excellent method more dependable and precise. The largest difference between the original test method and the one proposed herein was the ability of the operator to measure the applied force. Also, some preliminary testing took place to study the effect of different variables on the test results and be able to obtain a final set of test parameters.

4.2.1.1 Basic Assumptions

Hot water tests are based on the principle that high temperatures and moisture accelerate the loss of adhesion between steel and epoxy coatings. To obtain a constant level of moisture the bars were completely immersed for the duration of the test. A hot water bath was built in order to have a temperature controlled environment with as little change as possible. The hot water bath consisted of a Styrofoam ice chest lined with plastic covering to avoid leaks. A water heater/circulator was placed on the lid of the chest along with a thermometer for easy inspection of the inside temperature (see Figure 4.3). The thermostats maintained the water temperature at about ±2°C of the test temperature, which was assumed acceptable for this experiment.

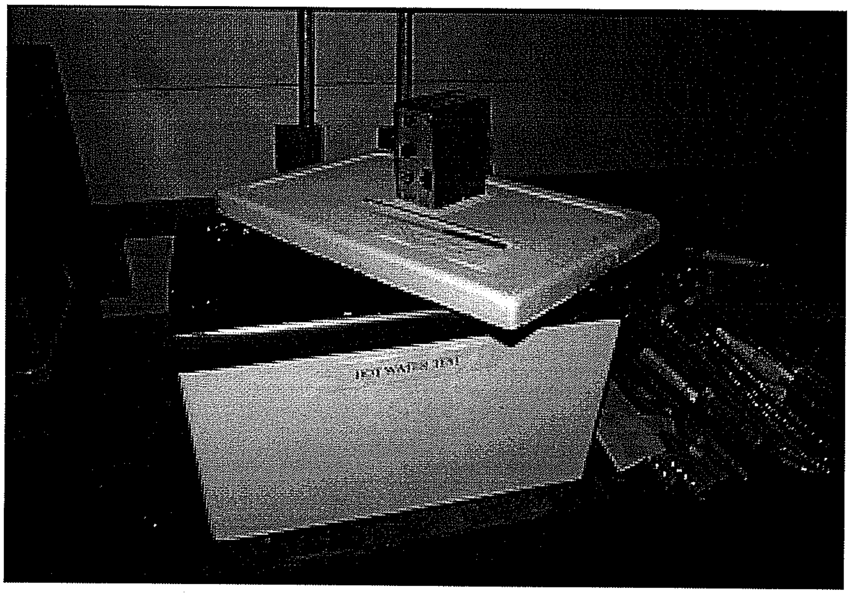


Figure 4.3. Hot water bath used for adhesion tests

All hot water tests were performed in a room with an air temperature of approximately 72°F so that samples could dry and cool in a constant environment. However, failure of the air conditioning system in the room negated this condition.

For hot water testing, the same glass transition temperature was assumed for all epoxy coatings. All the coatings were exposed to the same water temperature for the same period of exposure.

Another assumption concerned the amount of damage present in the sample at the time of exposure. Only those discontinuities large enough to be seen by the unaided eye were patched. Even though the samples had varying amounts of pinholes before the test, this was not taken into account in evaluating the results. The holiday detection performed earlier was done only to verify if the bars comply with current specifications.

4.2.1.2 Preliminary study of variables and selection of test parameters

When performing hot water tests, a significant number of variables must be taken into account to obtain the final test parameters. The variables under consideration were:

- Water temperature: 55°C and 75°C. Almost all of the available information regarding hot water tests includes temperatures in the range of 70°C to 80°C. However, some researchers have used temperatures as low as 55°C when samples are immersed in aggressive media. The idea was to correlate the effect of different temperatures and different immersion times in order to find the optimum combination.

- Time of exposure to hot water: 0, 3, 6, 24 and 48 hours. The objective was to find the time at which there is no significant change in the adhesion loss of the epoxy coating. Again, there was the possibility of finding the same results at different temperatures with different times of immersion.
- Post-immersion periods: 0, 3, 6, 24 and 48 hours. These times allow the test to be performed within an 8 hour work shift. The times considered allow the test to be completed within a normal work shift if samples are immersed early in the morning.
- Presence of initial damage: The presence of intentional damage on the sample before immersion was also taken into account. Some tests reported in the literature showed worse adhesion if the samples were damaged prior to exposure as compared to undamaged samples. To create a "constant" damaged location, a 1/8" hole was drilled through the epoxy into the bar. With larger holes it was more difficult to use the testing knife, while smaller diameters were more difficult to drill.
- Applied force: 3Kg and 4Kg. The higher force was based on the strength of the testing knife. The lower force was used to conform to specifications used in earlier tests.

One #8 and one #9 epoxy coated bars were used for the preliminary tests. The #8 bar was a leftover from previous experiments and the #9 bar was a new bar but not taken from those mentioned in Table 4.1. Companion 5 inch samples were cut from both epoxy coated bars. In total, 16 samples were taken from each bar to cover all the possible immersion times and temperatures. Cooling time tests were performed later. The ends of each sample were sealed using a two part epoxy compound to prevent water migration under the coating and corrosion of the exposed steel at the ends. After the hot water bath, all samples were allowed to dry for a 24 hour post immersion period before conducting adhesion tests. Each side of the sample was tested at four locations using two different forces and surface conditions (pre-drilled hole/no pre-drilled hole), as seen in Figure 4.4.

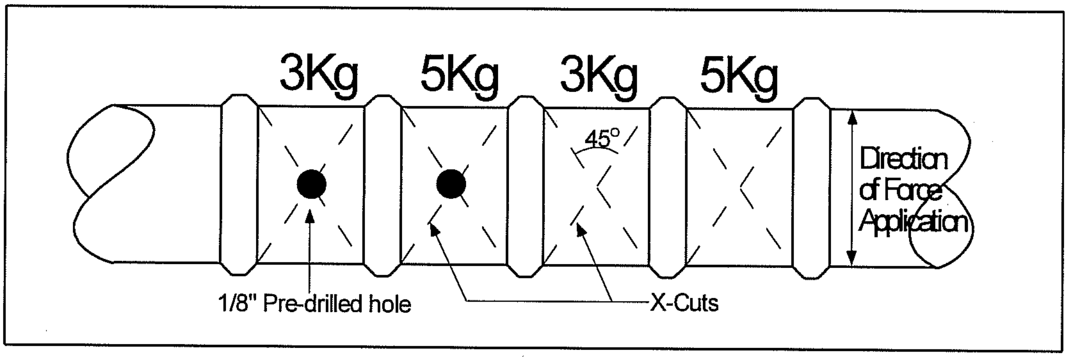


Figure 4.4. Test locations on 5” long epoxy coated rebar sample for preliminary adhesion tests

After drying for 24 hours, the specimens were safely secured to a table vise in order to have a stable working platform. An X-cut with 45° “flaps” was made through the epoxy coating in each of the four testing locations per side of the sample as shown. A sharp utility knife, drawn steadily across the surface was used to make the cuts. Special care was taken to cut through the epoxy to the bare metal. It was noted that for various sizes of bars and different deformation patterns, two diagonal cuts spanning from the top of one lug to the bottom of the neighboring lug generally produced angles about 45°. After the cuts were made, the sample was placed and securely clamped in a table vise.

The test itself consisted of inserting the end of the calibrated knife into the tip of the flap created by the 45° cuts at the interface between the epoxy coating and the steel surface. A known force was then applied to the knife which was held at an angle of approximately 30°, tangent to the curvature of the bar (see Figure 4.5).

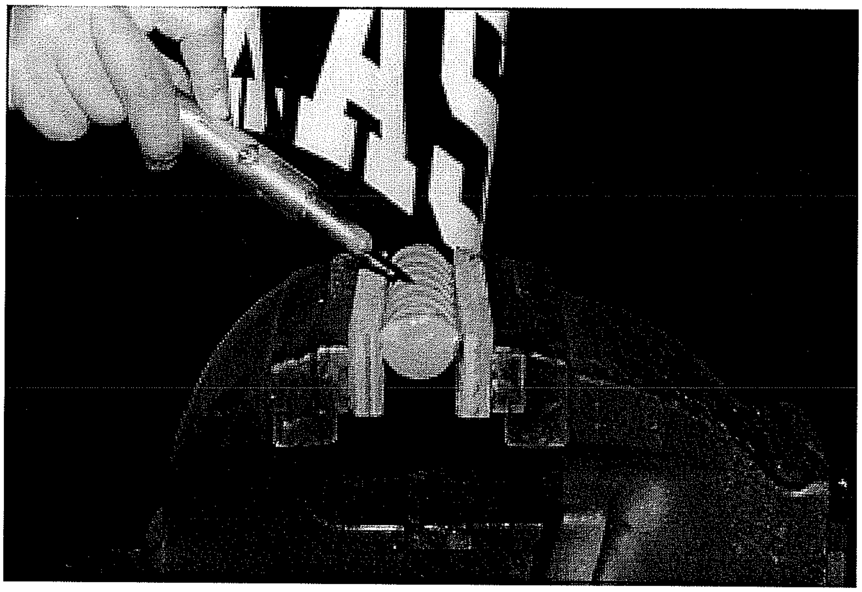


Figure 4.5. Correct force application position for hot water tests.

The test was stopped either at the end of 35 seconds of constant force application with no advance of the knife or when the knife slipped and cut through the epoxy coating. After the test was stopped, all the loose material was removed from the test area and an adhesion index value was assigned to every cut. Two tests (readings) could be made in every location. Even though the ends of the sample were perfectly sealed, cuts were made at least ½ inch from the ends of the specimen to allow for the effect of some possible water migration.

The rating system adopted to evaluate the epoxy coating adhesion was a function of the length of the bisecting line formed in the flap between the cuts (see Figure 4.6). This rating system was selected due to practical reasons. Mainly, because it is easier to measure the length of the section than the area of coating removed. Table 4.2 presents the rating system as used for the preliminary tests.

INDEX	BEHAVIOR OF COATING
1	Difficult to place blade under coating. Less than 5% of the length removed.
2	Easy placement of blade under coating. 5% - 25% of length removed.
3	25% - 50% of length removed
4	50% - 75% of length removed
5	> 75% of length removed

Table 4.2. Rating system for preliminary tests

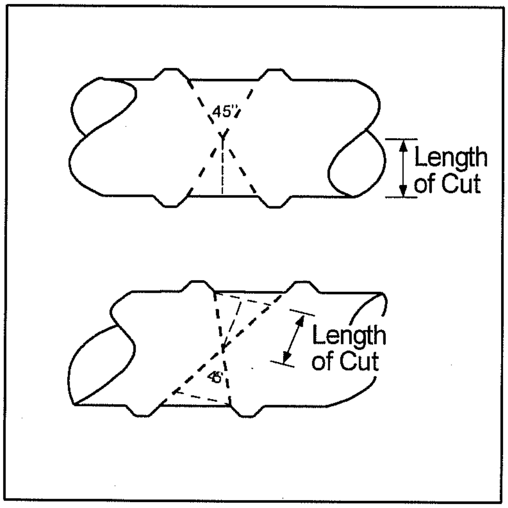


Figure 4.6. Graphical definition of length of cut for bamboo and diagonal deformation patterns

A total of 576 adhesion tests were performed in this series of preliminary tests, each considered as the application of force in one of the flaps between two consecutive deformations.

After this series of tests, a second series was run in order to find the most suitable post-immersion period to be used for the final test. Using the optimum values of temperature, immersion time, force, and initial condition obtained from the first series of tests, a threshold value of drying time was researched. A total of 80 adhesion tests were performed in companion samples for 5 different post-immersion times.

From this preliminary analysis, the final parameters for the hot water test were obtained. Some of the trends found were the following:

a) As the time of immersion increased, there was a tendency for adhesion to decrease (index value increases) regardless of the temperature of the hot water bath. This effect could be attributed to a more effective migration of moisture under the coating. Water migration could have been aided by the presence of small pinholes or by porosity of the coating, which is not impermeable. In all the samples, however, adhesion ratings remained fairly constant after immersion periods of 24 hours or longer (see Figures 4.7 and 4.8).

b) As water temperature increased , adhesion loss tended to increase. A higher adhesion loss was always observed at higher temperatures. Some of the samples that showed good adhesion at lower temperatures had a total loss of adhesion (number rating of 5) when exposed to 75° water immersion (see Figures 4.9 and 4.10)

c) Tests performed on sections with holes drilled before immersion tended to yield slightly higher index values (higher adhesion loss) than those in which the coating was undisturbed. The presence of a hole marks a discontinuity in the epoxy, which allows for the migration of moisture, as well as the formation of corrosion products even in the early stages of the process. All samples exhibited corrosion products in the drilled hole after being removed from the hot water bath, even at short immersion times. Specimens with pre-drilled holes proved more difficult to test, and the difference in rating between the two initial conditions was so small that this practice was dismissed for practical reasons.

d) As expected, adhesion loss was found to be directly proportional to the applied force. The difference in rating between the two different forces was as much as one index point higher in some cases as seen in Figure 4.9 , so subsequent tests were performed with both applied forces to evaluate this effect in a larger number of bars.

e) There was no conclusive trend in the adhesion of samples with post cooling periods shorter than 6 hours. However, as time increased from the 6 hour mark, there was a tendency for the rating number to stay constant (see Figures 4.11 and 4.12).

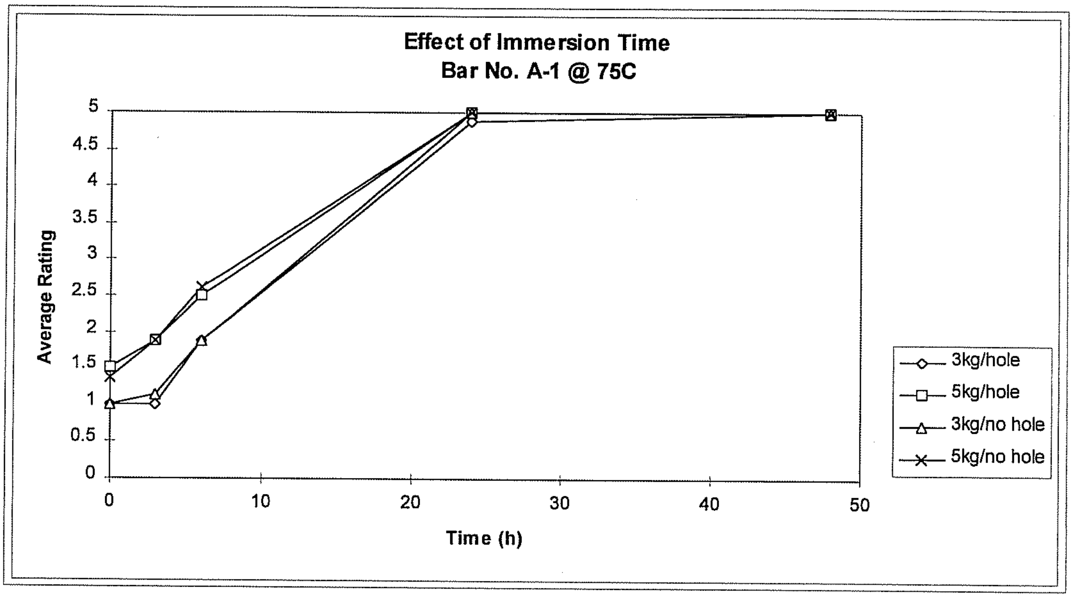


Figure 4.7. Effect of immersion time for bar No. A-1 exposed to 75°C water

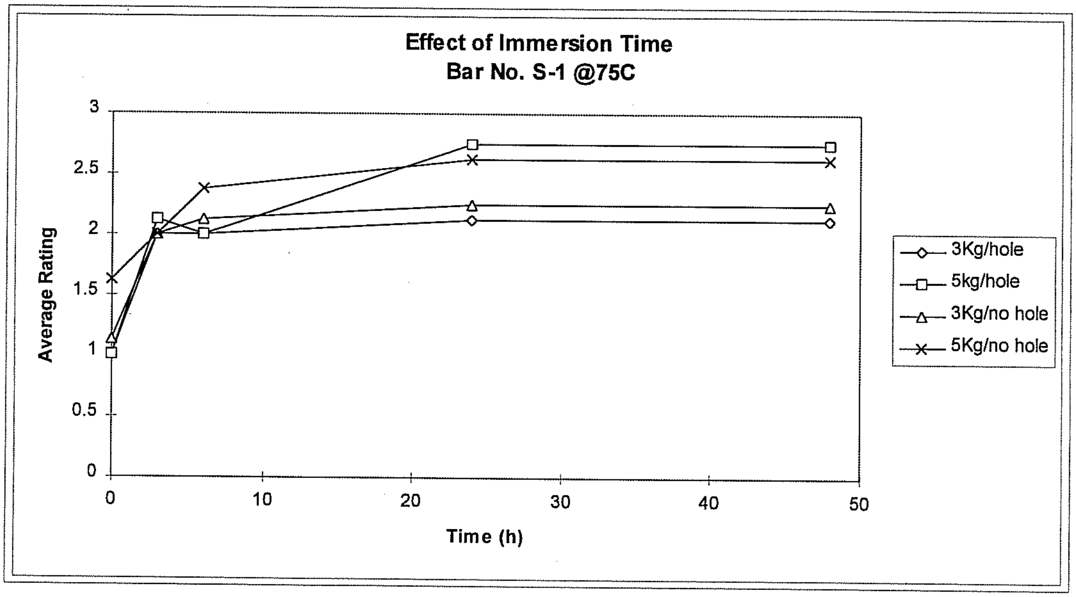


Figure 4.8. Effect of immersion time for bar No. S-1 exposed to 75°C water

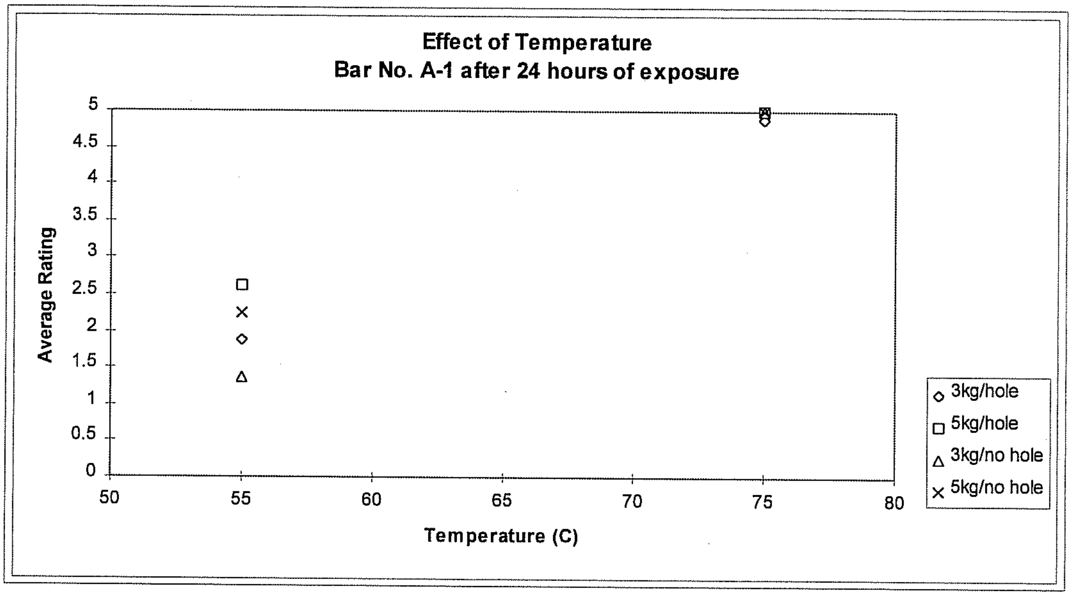


Figure 4.9. Effect of temperature on bar No. A-1 after 24 hours of exposure

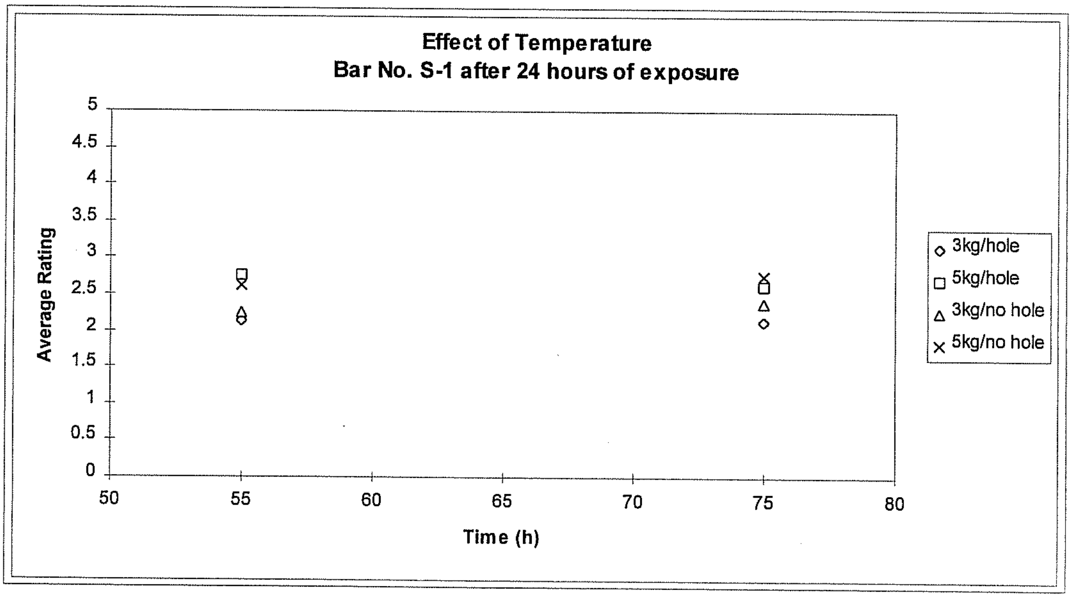


Figure 4.10. Effect of temperature on bar No. S-1 after 24 hours of exposure

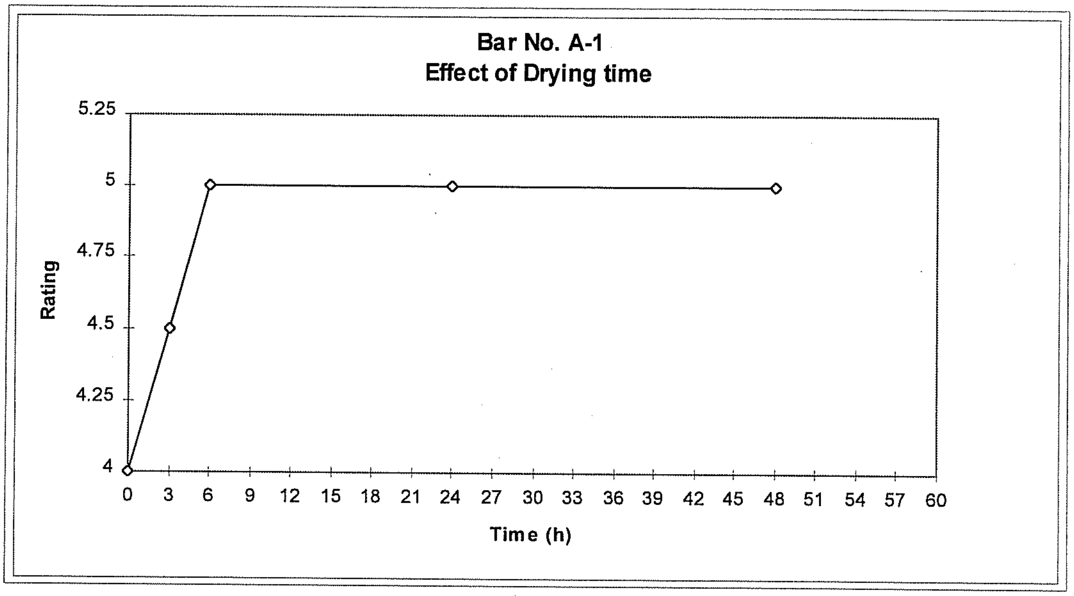


Figure 4.11. Effect of post-immersion period on adhesion rating for bar No. A-1

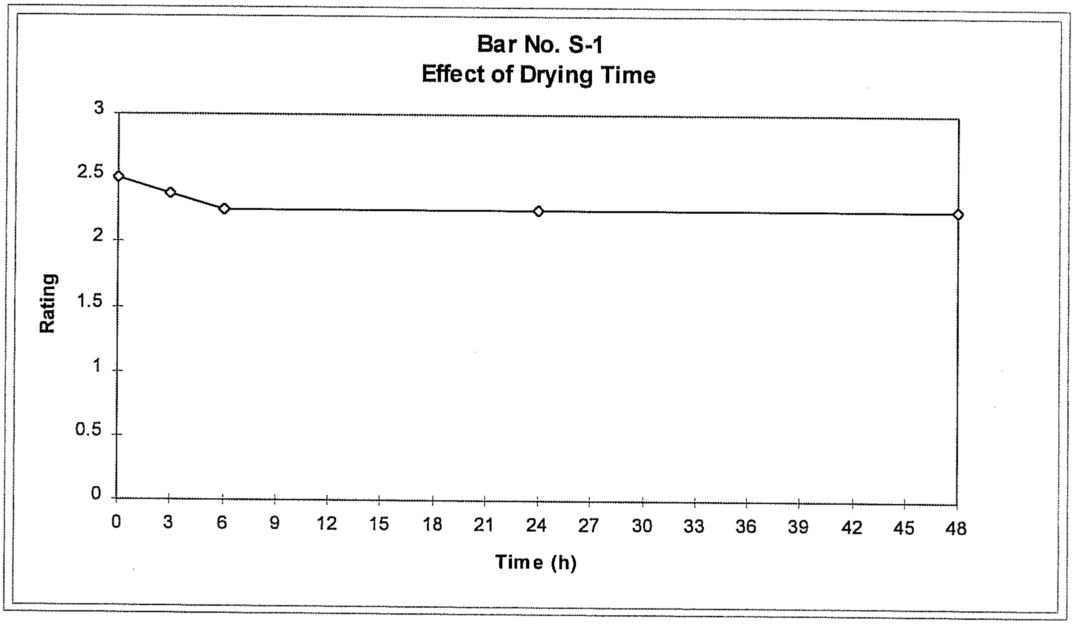


Figure 4.12. Effect of post-immersion period on adhesion rating for bar No. S-1

4.2.1.3 Final hot water test method

The purpose of the preliminary tests was to obtain values of parameters to be used in optimizing the hot water test from the point of view of time, repeatability, and coating condition. Variable values selected are as follows:

Temperature of hot water bath:	55°±2°C
Time of immersion:	24 hours
Post-immersion period:	6 hours or longer
Applied force:	3Kg and 4Kg
Initial condition of coating:	Undamaged

The main test program to evaluate coatings from different applicators and powder manufacturers proceeded in essentially the same manner as the preliminary tests. Sample size was adequate for performing the test, and the testing knife proved to be efficient in applying known forces with reasonable accuracy. Also, the rating system utilized in the preliminary tests offered a very wide range of adhesion conditions, so it was retained.

The sampling procedure consisted of taking samples from three different locations of the supplied bar. One sample was taken near each end and one from the middle of the bar. This sampling procedure would take into account possible variations of the coating adhesion along the rebar. For all the three locations, companion samples were prepared for testing by a different operator.

Four 45° "X" cuts were made on each side of the bar sample. Two cuts were tested with an applied force of 3Kg and the other two with a force of 4Kg. Eight values of adhesion were obtained for every force in each sample. The eight adhesion values were averaged to obtain a mean adhesion value for the sample. The standard deviation and coefficient of variation were obtained for adhesion values in each location (bar sample). The overall standard deviation and coefficient of variation along the bar were also obtained. These differences in coating adhesion between separate locations in the bar provide a good indication of the quality control of the applicator and of the testing procedure used.

It must be pointed out that in some instances, particularly when very thin coatings are involved, there was a tendency for the knife to rip through the epoxy coating before the end of the test was reached. This sudden effect was caused by the knife cutting through epoxy layers and causing delamination of the material. The test result was confirmed by re-applying the test force at the position where the coating failed and the knife slipped. Any additional debonding occurred was taken into account in the result.

For all tests, a sharp utility blade was used to make the X-cuts through the epoxy coating thickness without damaging surrounding coating. Also, the blade used in the testing knife was changed daily, or as soon as wear or loss of sharpness was detected on its tip. This was a very important issue, because a blunt blade tended to give lower numerical rating indexes (meaning better adhesion) than a sharp, well defined blade.

4.2.2 Controlled peel test (Strip Method)

The controlled peel test presented here was a "non-immersion method". It was intended to provide a simple, easy, and reliable quality control method applicable to in-plant testing. The advantages of testing at the production line are that (1) no laboratory testing is required, (2) results are almost immediate, and (3) costs are reduced. There is no need to cut samples because the test can be carried out directly on the bar. The method was labeled the strip method because of the particular shape of the test area defined in the epoxy coating. The locations tested in this manner would have to be properly repaired unless the entire bar was scrapped.

4.2.2.1 Basic assumptions

After electrochemical adhesion is lost, the adhesion capacity of the coating is maintained by the mechanical bond existing between the steel and the coating. Any action that diminishes the electrochemical adhesion before the test is performed takes this effect into account. The hot water test provides a good measure of the coating adhesion in bars that have been in aggressive environments for an extended period. On the other hand, peel tests measure the total adhesion present in the bar after it is coated. Immediately after application of the epoxy, adhesion is a function of care and quality control of the coating process. Any imperfection in the cleaning operation can lead to loss of electrochemical or mechanical components of adhesion.

Peel tests measure adhesion strength as a function of the total contribution of both the electrochemical and mechanical adhesion components. It is probably not a bad assumption to say that a poorly applied coating will have bad adherence due to a non-existent amount of both components of adhesion, therefore a test procedure that involves breaking the bond between the surfaces without any previous treatment is acceptable.

In the peel tests, a known shearing force is applied to the epoxy-steel interface (in the same way used for hot water tests) while applying a rotating motion to the testing knife. The effect of this motion is to provide an uplifting stress that tends to separate both materials. If the shearing force and rotating motion are kept constant, the amount of coating removed is inversely proportional to the adhesion between the epoxy and the steel surface.

A test area must be defined by isolating a part of the coating from the adjacent coating. This is done by cutting through the epoxy with a knife in a predetermined shape. As it was the case in the hot water test, the cut must go through the coating to the bare steel and must be accomplished without disturbing the coating in the test area.

4.2.2.2 Sampling and test procedure

The test was performed in three locations along the bar length, both ends and the middle. In each location, two tests were done on each side of the bar. A test consists of the application of force in one defined test area. The result was four adhesion measurements for each location.

The test procedure consisted of making a 2 mm wide by 25 mm long cut on each test location. The resulting strip was the test area. The width limitation was set taking into account the blade of the testing knife. Several trials were done in order to find a width of coating that would not peel completely (very narrow) or not peel at all (very wide) for most cases. After the cut was made, one end of the strip was lifted with the aid of a sharp utility knife. The tip of the testing knife was then placed between the epoxy coating and steel surface. A constant force was applied to the knife at an angle of approximately 30° tangent to the curvature of the bar, maintaining the tip of the blade in the center of the strip. The magnitude of the force was taken as the force required to maintain forward motion during the test, which for most cases oscillated in a the range of 1Kg to 2Kg. At the same time, a rotating motion is applied to the knife in order to provide an uplifting force. This motion consisted of rotating the knife about its longitudinal axis. Starting with the blade in horizontal position, it was continuously rotated 30° from the initial position on each direction, like presented in Figure 4.13. The test stopped when the testing knife advanced along the whole length of the strip of epoxy coating (see Figure 4.14).

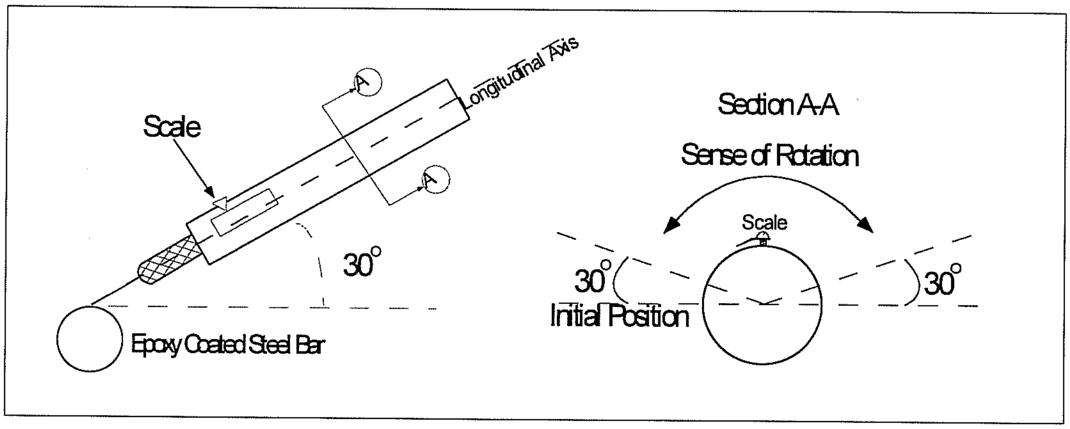


Figure 4.13. Position of testing knife and definition of rotating motion

After the test was finished, all loose and unbonded material was removed and the "stripped" surface observed. The value recorded was the approximate percentage of coating in the test area that remained adhered to the steel. For example, in Figure 4.14, the recorded value would be approximately 60% .

Visual observations are not precise but can be estimated to the nearest 10%. The percentage obtained varied directly proportional to the adhesion strength of the coating. A value of 0% means essentially no adhesion. It is important to note that there is no 100%, because the movement of the blade removes a thin strip of epoxy even in very good coatings. The width of such a strip is approximately that of the tip of the blade, which yields a maximum remaining percentage close to 70% for the best adhesion ratings.

Figure 4.14. Sample after performing strip test

A disadvantage of the strip method is the difficulty in defining and cutting the test area. The method proved to be easy to perform once the cuts were made. Similar to the hot water tests, care must be taken to assure that the blade is moving in the epoxy/steel interface and not in the epoxy coating itself. In the case of the strip method, this was easily verified because as the blade advanced, there should have been a small trail of visible metal left behind even if the quality of the coating was very good.

4.2.3 Controlled peel test (X-cut method)

4.2.3.1 Basic assumptions and sampling

The final method devised for this study was a combination of the first two. It is also considered a peel test since there is no immersion or pre-treatment prior to testing. The difference between the strip and X-cut methods lies in the definition of the test area. The test area for the X-cut method consists of a cut similar to the one created for the hot water immersion tests, however, there is no restriction in the interior angle of the flap formed in the cut. It was observed in many bars that for the same location, when applying a force in the same manner as was done in the strip test, the width of the section where the coating breaks or rips at the end of the test is roughly the same regardless of the angle in the cut. The reason for this can be explained theorically. As the force is applied to the coating through uplifting created as the knife rotates, the coating starts lifting from the steel substrate until there is enough resistance provided by its adhesion and the coating breaks off or rips apart. At the location where the coating breaks, the width of the section in which the coating could still be lifted as a whole (see Figure 4.15) is an indication of the adhesion strength of the coating in that location. This test is very flexible, because it allows the operator to start the test with any arbitrary angle for the X-cut and depending on the adhesion of the coating, the angle can be increased or decreased in order to obtain results that can be more easily interpreted.

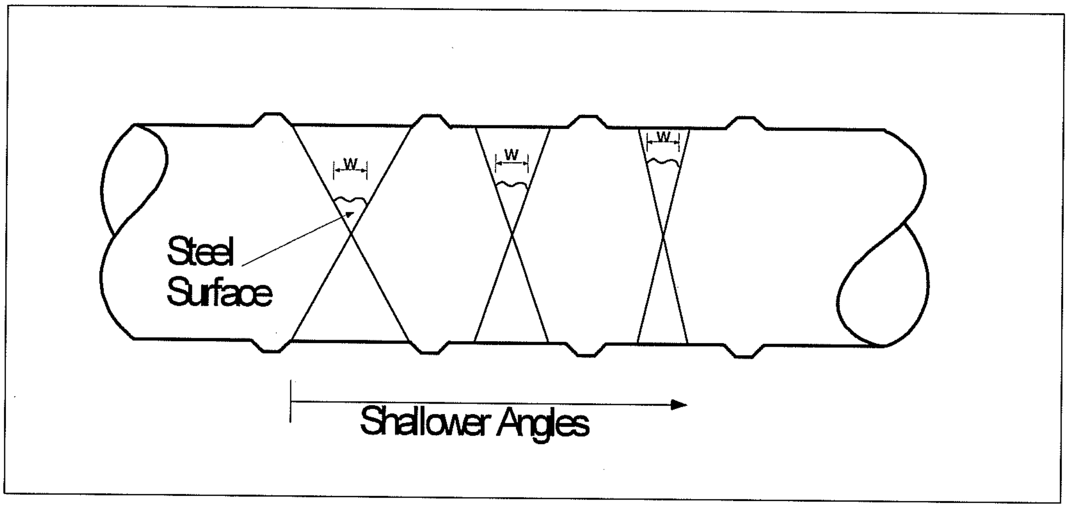


Figure 4.15. Width of final section remains roughly equal as the angle of the X-cut becomes shallower

The test procedure is exactly the same as the one used in the strip test, and the same assumptions about "non-immersion" tests hold true in this case. The test was performed at the same locations in each of the bars, and in order to obtain the same number of readings as in the strip method, two tests were carried out on each side at each location. Each test is defined as the application of a force on one flap of the cut.

The results of the test were given as the width in milimeters of the section were the epoxy coating failed due to sufficient adhesion strength. This occurs when the epoxy adhesion strength is larger than the uplifting stress applied. After testing all locations, an average value of adhesion could be found and the variability of the values was investigated.

The test was performed on #6 and #9 bars, which are large enough to make X-cuts and test in each direction (two flaps). For smaller bars, it is possible to use a “V” cut instead of an “X” cut and perform the test in the same fashion. The only caution is that is necessary to carry out the same number of adhesion tests as required for the X-cut. This is an advantage over the strip method, where performing the test on bars smaller than #6 is very difficult due to space constraints.

4.2.4 Texas Department of Transportation Test Method Tex 793-I

In order to provide an acceptance criteria for the three methods presented before, peel tests as suggested by the Texas Department of Transportation were executed in the same locations of all the tested bars, at both bar ends and the middle portion on both sides. The test procedure was carried out according to test method 739-I, as presented in Chapter 3 of this thesis.

The results of this method are highly dependent on the operator, so the same person performed all the tests in order to avoid variability and subjectivity in the results.

An accepted/not accepted rating was used to assess the overall adhesion of the bars. Even though a result was given for every test location individually, the not accepted rating involved finding poor adhesion at any location tested on a given bar.

4.3 IMMERSION OF EPOXY COATED BARS FROM DIFFERENT COATING PLANTS IN 3.5% SODIUM CHLORIDE SOLUTION

4.3.1 Purpose

The intent of this part of the research was to obtain a better understanding of how adhesion strength relates to corrosion protection of epoxy coated bars under aggressive chemical attack.

To provide a very harsh environment for the samples, a solution of NaCl at 3.5% was chosen. This chloride concentration was selected because it has worked satisfactorily in previous corrosion studies carried out in The University of Texas at Austin. It is clear that such a solution does not represent the condition of the surroundings of a steel bar embedded in concrete. Such condition will change from initial service conditions to those created by exposure conditions, de-icing chemicals, etc. However, exposing the samples to the same aggressive environment provides an idea of how different epoxy coatings withstand chloride exposure.

As for the adhesion tests, samples for immersion testing were taken from the same three locations of every supplied bar. Specimens were 5 inches long and the exposed ends were sealed with a two part compound to avoid the effect of having a steel surface exposed to the solution. Before immersion, every sample was carefully inspected for the presence of defects or holidays. Such coating imperfections were sealed with the use of two part patching material supplied by the powder manufacturer. After making sure the surface of the coating was free of discontinuities, a 1/8in. diameter hole was drilled through the coating on both sides of the bar, such that the underlying steel was exposed. The purpose of these holes was to have a controlled discontinuity that was easily monitored and where the corrosion process could start. The samples were suspended in tanks filled with NaCl solution. To optimize the corrosion process, wet/dry cycles were used to allow easy access of oxygen ions to the cell during dry periods. Due to time constraints, the total length of exposure was limited to 12 weeks, with repeating 4 day-wet/ 3 day-dry cycles. The solution was replaced two times during the exposure period, generally after substantial amounts of corrosion products settled to the bottom of the tanks

5.3.2 Evaluation of samples after exposure period

After the exposure period was finished, the bars were allowed to air dry for a period of 2 weeks before inspection. The specimens were then evaluated visually to asses the effects of the solution immersion test.

Each sample was carefully inspected for the presence of any damage to the coating, in the form of corrosion, cracks or rips, blisters and any other possible sign of deterioration.

The second step of the evaluation process was to measure the adhesion strength in each sample. This value was later compared to that obtained from the corresponding bar location with no chloride immersion. Adhesion was measured using the X-cut controlled peel test (section 4.2.3). Adhesion was tested at least one inch away from the intentional discontinuity (1/8in. hole) created in the samples to avoid the total loss of adhesion caused by the appearance of new corrosion products. The intent was to measure how adhesion strength changed in a coated bar with discontinuities when exposed to high moisture conditions

After adhesion measurements were taken, the epoxy coating was peeled back around the drilled hole with a utility knife. In order to avoid damaging the steel surface around the hole, the peeling operation was initiated away from the hole and carefully peeling toward it. Peeling extended to the point where clean steel (no evidence of corrosion) was uncovered, or until the coating proved difficult to peel.

The next step in the evaluation process consisted of examining the area adjacent to the discontinuity in the coating; assessing coating adhesion, appearance of corrosion products, and approximate size of damaged area. The adhesion of the coating in the vicinity of the hole was estimated from the difficulty offered by the epoxy to the peeling operation. In most cases, no adhesion existed in the areas affected by the corrosion process, even in samples from locations that exhibited good adhesion before immersion. Loss of adhesion in such areas is promoted by easier access of moisture and formation of new corrosion products.

Finally, the area affected by corrosion was compared against the adhesion value for the corresponding location in the bar before any immersion tests.

No definite conclusions can be reached from the results of the chloride exposure, because only a limited number of coatings and applicators were included in the test. However, the results are interesting enough to encourage further investigation regarding the effect of epoxy coating adhesion on the corrosion protection of reinforcing steel.

CHAPTER 5: TEST RESULTS

In this chapter, results of the three proposed adhesion testing methods are presented for five different coating applicators. Also, observations from the visual inspection of the samples immersed in chloride solution are discussed.

Each different coating applicator was assigned a letter name in order to be easily identified. The letter names used were U, V, W, Y and Z. The number following the letter identifies different bars obtained from the same coater. The names assigned to the bars used for the experimental project were:

- #6 deformed bars:	U1, V1, V14 (non-bendable), W1, Y2 and Z1
- #9 deformed bars:	U6, V3, V16 (non bendable), Y5, W3 and Z3
- #10 plain bars:	W17 and V29
- #6 plain bars:	W16 and V28

5.1 QUALITY CONTROL OF AS-RECEIVED BARS

5.1.1 Bend test observations

Each bar provided by the coating applicators was accompanied by four bent pieces from the same original bar. Visual observation of the bent pieces provides a measure of adhesion quality.

Only the coating by applicator Y failed the bend test. All the bend tests performed on bar Y2 showed some cracking and damage to the coating due to the bending operation. Only one of the four bend tests provided for bar Y5 passed the test, while the other three pieces showed the same kind of coating flaws as bar Y2.

The presence of damage on two bars from different lots of the same coating applicator may be the result of:

a. Epoxy powder used for coating the bars that is not flexible enough and cracks due to the high stresses developed during the bending operation, or

b. An inherent mistake in the coating operation translates into poor bond between the coating and the steel substrate. Many regulatory agencies have suggested this as the primary factor..

In further tests, the adhesion strength of the bars that failed the bend test will be obtained by the three different methods proposed in this project. The reliability of the bend test can only be proved if adhesion strengths coincide with the result of the test. Should any particular coater pass the bend test while obtaining lower adhesion strengths than those obtained for coater Y, the validity of the bend test as an adhesion measuring test can be questioned.

5.1.2 Coating thickness measurement

The average coating thickness for each of the as-received bars is shown in Figure 5.1. The value plotted in this graph is the average of 24 thickness measurements taken at regular intervals along the bar as explained in Chapter 4. The Tx DOT allowable thickness limits are plotted for comparison.

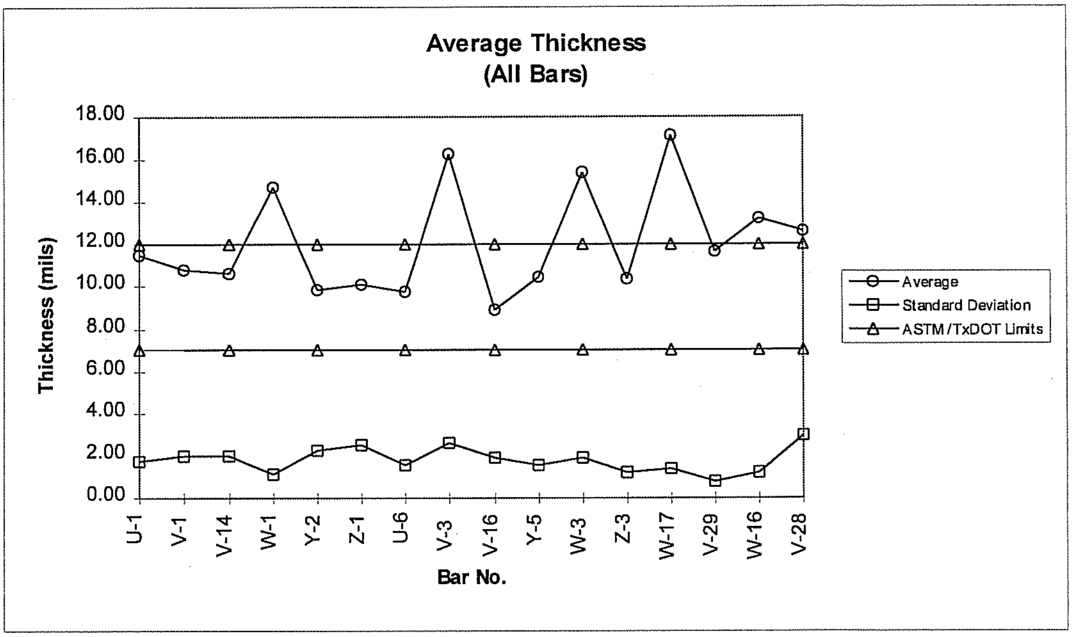


Figure 5.1. Average thickness of all bars

Tx DOT and ASTM standards require 90% of the coating thickness values to fall within the specified range of 7 to 12 mils. In order to achieve this goal, a coefficient of variation of no more than 25% with respect to the mean (8.5 mils) should exist in the coated bar for a normal distribution of coating thickness. Coefficients of variation larger than 25% indicate poor quality control or unequal coating distribution during coating procedures. Variability coefficients of the as-received coating thickness are presented in Table 5.1 along with average coating thickness for both sides of the bar, overall bar average and standard deviation.

Side to side variations of coating thickness are important to detect improper cooling of epoxy coated bars. Figure 5.2 shows average coating thickness measurements from both sides of the bars. Bars U-6, V-3, V-16, W-3, W-16 and V-28 had side to side variations of thickness higher than 15% of the average. This difference could indicate higher than normal coating temperatures or early quenching of the bar.

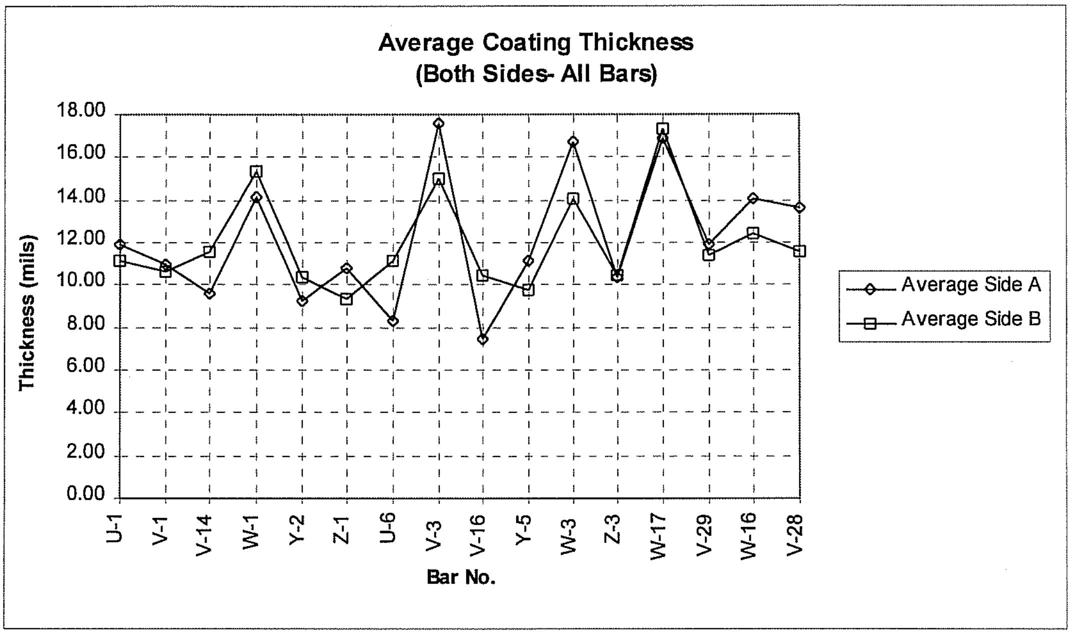


Figure 5.2. Average thickness on both sides of coated bars

Thickness values from the 5 different coaters ranged between 8.92 and 17.13 mils, with an average of 12.08. mils. This average value suggests a high number of samples with coating thickness higher than the upper limit specified by the standards. None of the individual readings from all the bars was below the specified limit of 7 mils. All the bars received from coater W as well as bars V-3 and V-28 had average coating thickness measurements higher than the upper limit allowed by the specifications. In the case of bars from coater W, this increased thickness was appreciable and could be determined by simple inspection of the bars.

	AVERAGES				
Bar No.	Side A (mils)	Side B (mils)	Overall (mils)	S.D (mils)	C.V (%)
U-1	11.88	11.13	11.50	1.74	15.17
V-1	10.96	10.67	10.81	2.03	18.74
V-14	9.63	11.58	10.60	2.01	18.96
W-1	14.13	15.31	14.72	1.14	7.74
Y-2	9.29	10.38	9.83	2.23	22.72
Z-1	10.79	9.38	10.08	2.51	24.93
U-6	8.33	11.17	9.75	1.59	16.29
V-3	17.58	15.00	16.29	2.57	15.78
V-16	7.42	10.42	8.92	1.93	21.63
Y-5	11.13	9.75	10.44	1.52	14.57
W-3	16.69	14.06	15.38	1.91	12.43
Z-3	10.33	10.50	10.42	1.25	11.98
W-17	16.92	17.33	17.13	1.37	7.98
V-29	11.92	11.42	11.67	0.81	6.92
W-16	13.94	12.44	13.19	1.20	9.08
V-28	13.67	11.58	12.63	2.96	23.47

Table 5.1. Variability of coating thickness of as-received bars

5.1.3 Holiday Detection

Figure 5.3 shows the average holiday count per unit length of the as-received bars. ASTM holiday count limits are also plotted in the graph for comparison purposes.

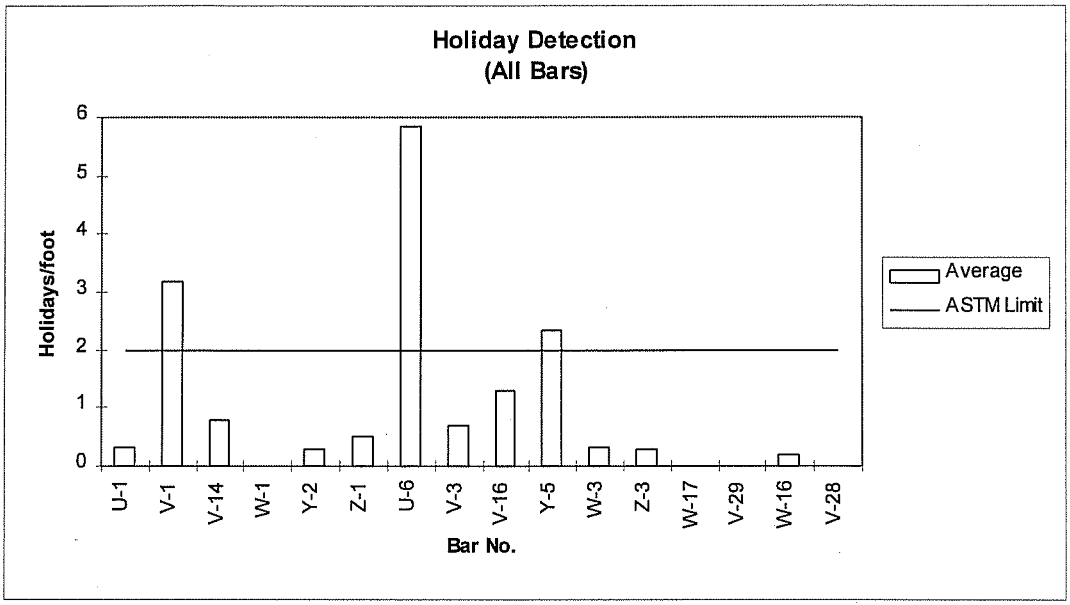


Figure 5.3. Average holiday count for all bars

Holiday counts for the five different coating applicators ranged between 0 and 5.8 holidays per foot, with a total average of 1.0 holiday per foot.

Of the three bars that had holiday counts larger than allowable, only bar V-1 was damaged in form of holidays. Bars U-6 and Y-5 had extensive coating damage in the region adjacent to mill marks. Coincidentally both bars were shipped in bundles with other bars without protection from abrasion or mechanical damage. ASTM regulations do not consider such damage as a part of the holiday count and only specify that such regions must be appropriately patched.

5.2 HOT WATER TESTS

The results obtained from the hot water tests are provided separately for each coating applicators. Due to time constraints, tests were performed only on

#6 and #9 deformed bars and #6 and #10 plain bars. Table 5.2 and 5.3 include values obtained for each bar and for both testing forces, including the average adhesion strength, range, standard deviation and variation coefficient. Figures 5.4 through 5.19 show values of adhesion measured at each location with a force of 3Kg. Plots representing one #6 bar and one #9 bar coated by the same epoxy coating applicator are presented on each page. The force of 4 Kg produced many failures of different coatings, so the results obtained for all locations will not be included in the graphics. For each location, four values of adhesion are shown, representing the average of the readings taken in both flaps of the X-cut. The average and standard deviation for each location are also plotted in each graph.

Bar No.	Bar Size	Minimum Rating	Maximum Rating	Average Rating	Standard Deviation	Coefficient of Variation
U-1	#6	5	5	5	0	0%
V-1	#6	1.5	2.5	2.1	0.4	20%
V-14	#6	3.5	5	4.5	0.5	12%
W-1	#6	4	5	4.8	0.3	7%
Y-2	#6	1.5	2.5	2.2	0.3	15%
Z-1	#6	1.5	2	1.9	0.2	12%
U-6	#9	1.5	4.5	2.8	1.0	36%
V-3	#9	1	1.5	1.2	0.2	21%
V-16	#9	4	5	4.9	0.3	6%
Y-5	#9	1	2.5	1.6	0.5	33%
W-3	#9	3.5	5	4.2	0.5	11%
Z-3	#9	1	3	1.5	0.5	37%
W-17	#10	4	5	4.8	0.3	7%
V-29	#10	1	2	1.3	0.4	31%
W-16	#6	4	5	4.9	0.3	6%
V-28	#6	2.5	4	3.4	0.5	16%

Table 5.2. Data obtained from hot water tests for each bar. Applied force =3Kg

Bar No.	Bar Size	Minimum Rating	Maximum Rating	Average Rating	Standard Deviation	Coefficient of Variation
U-1	#6	5	5	5	0	0%
V-1	#6	1.5	2.5	1.8	0.4	24%
V-14	#6	5	5	5	0	0%
W-1	#6	4.5	5	4.9	0.2	4%
Y-2	#6	1.5	3	2.1	0.4	18%
Z-1	#6	2	2	2	0	0%
U-6	#9	2.5	5	4.0	1.0	25%
V-3	#9	1	2	1.7	0.4	27%
V-16	#9	5	5	5	0	0%
Y-5	#9	2	3	2.5	0.5	19%
W-3	#9	4	5	4.5	0.4	10%
Z-3	#9	2	3	2.6	0.4	14%
W-17	#10	4	5	4.8	0.3	7%
V-29	#10	1	2	1.7	0.3	20%
W-16	#6	5	5	5	0	0%
V-28	#6	3	5	4.3	0.4	9%

Table 5.3. Data obtained from hot water tests for each bar. Applied force =4Kg

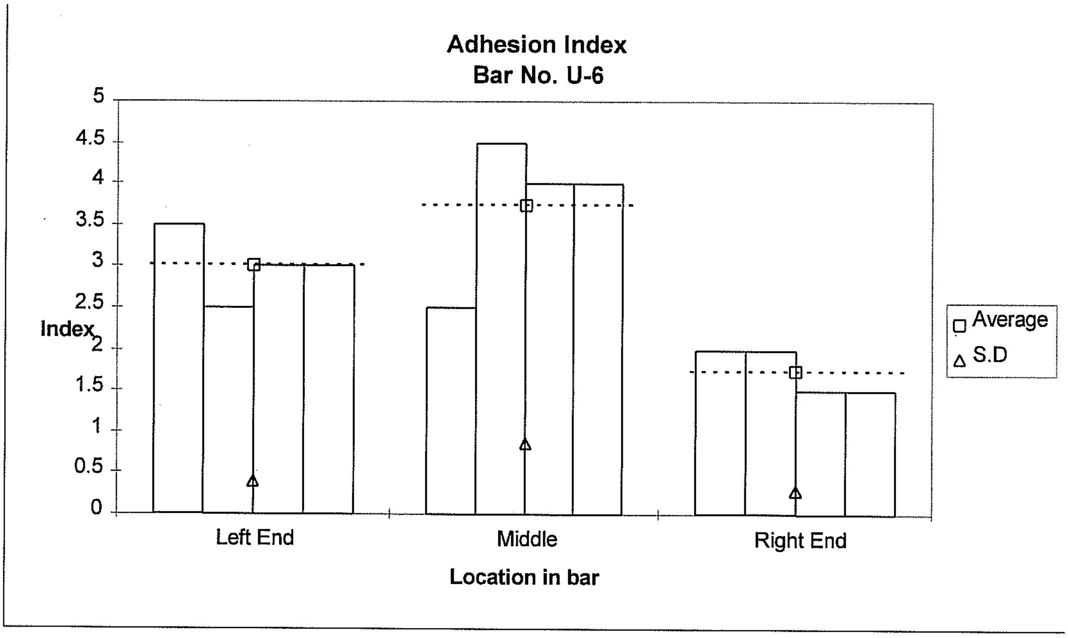


Figure 5.4. Adhesion rating for bar No. U-6

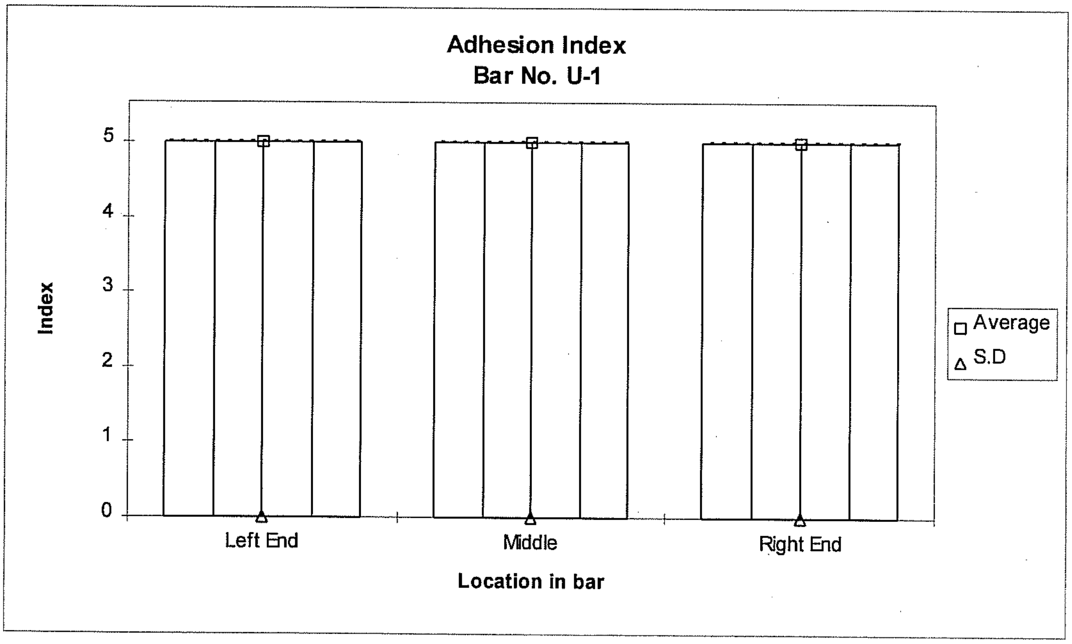


Figure 5.5. Adhesion rating for bar No. U-1

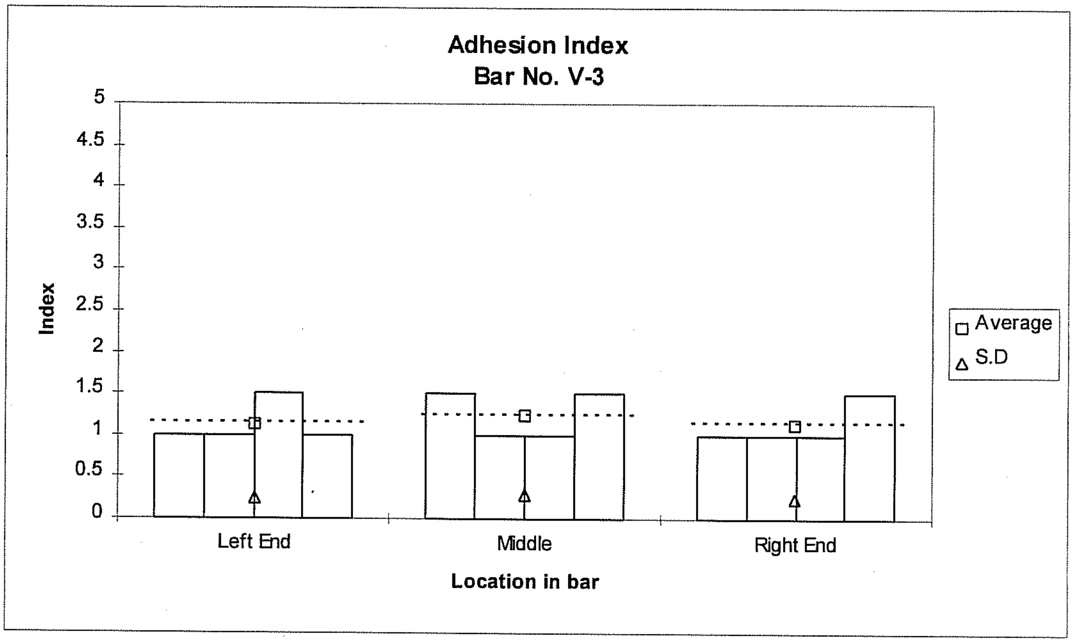


Figure 5.6. Adhesion rating for bar No. V-3

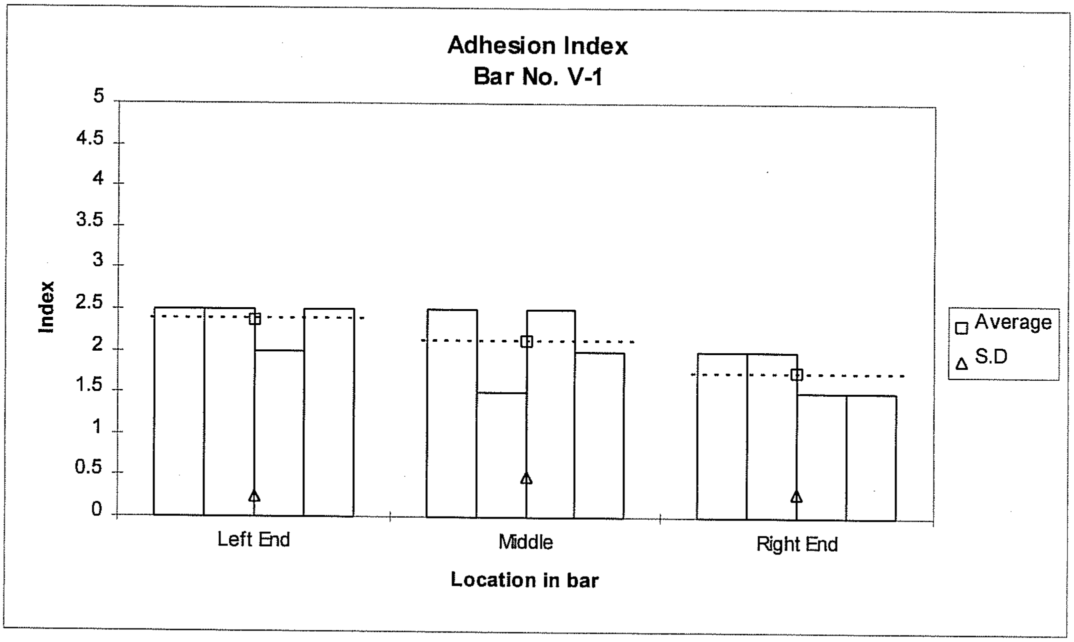


Figure 5.7. Adhesion rating for bar No. V-1

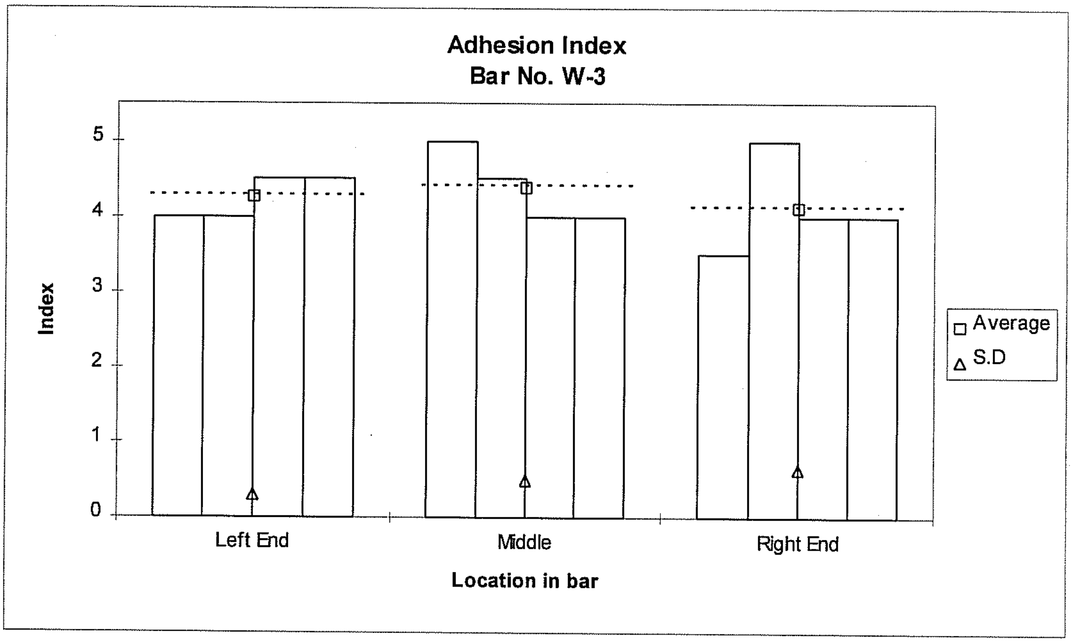


Figure 5.8. Adhesion rating for bar No. W-3

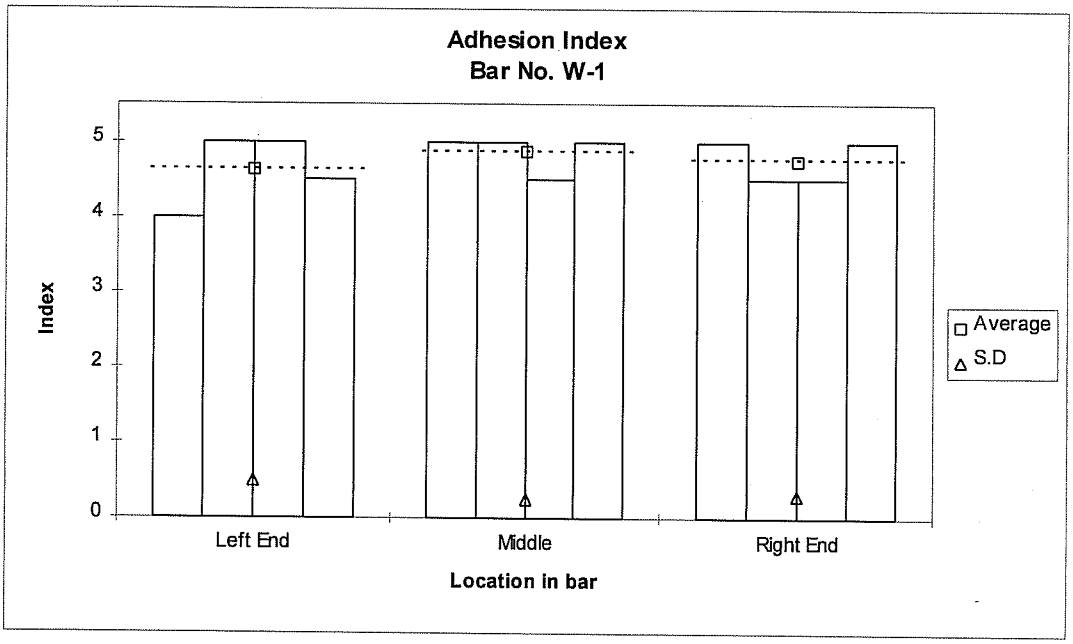


Figure 5.9. Adhesion rating for bar No. W-1

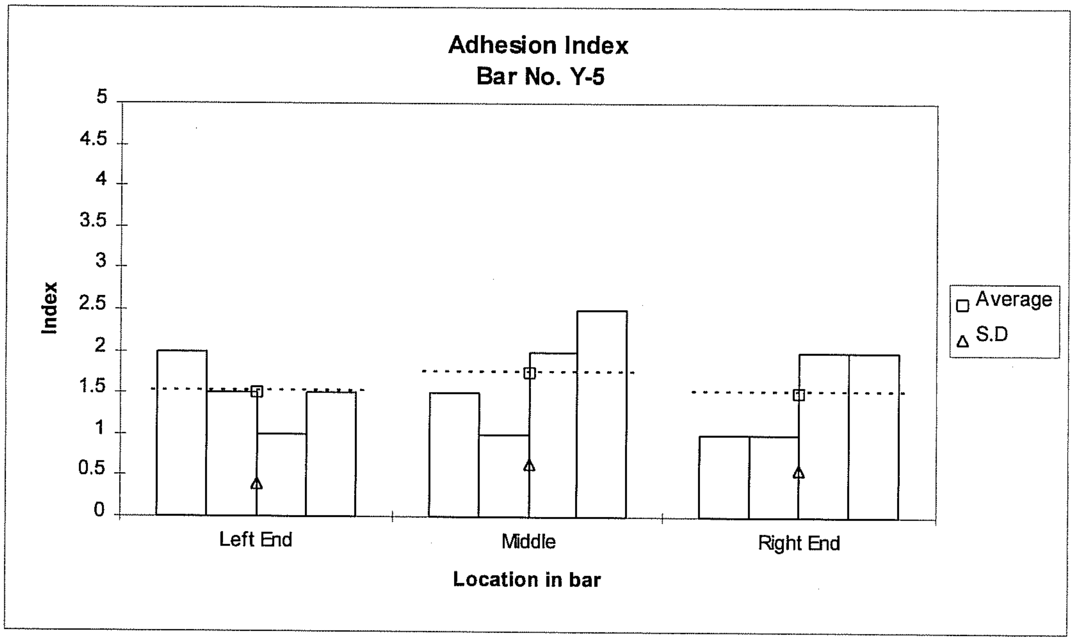


Figure 5.10. Adhesion rating for bar No. Y-5

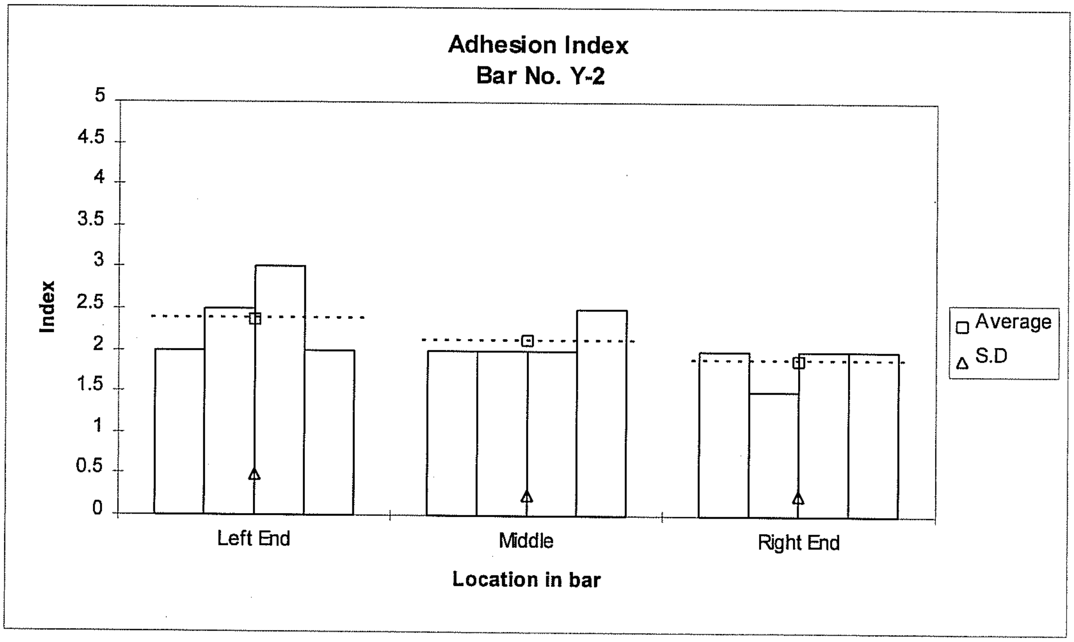


Figure 5.11. Adhesion rating for bar No. Y-2

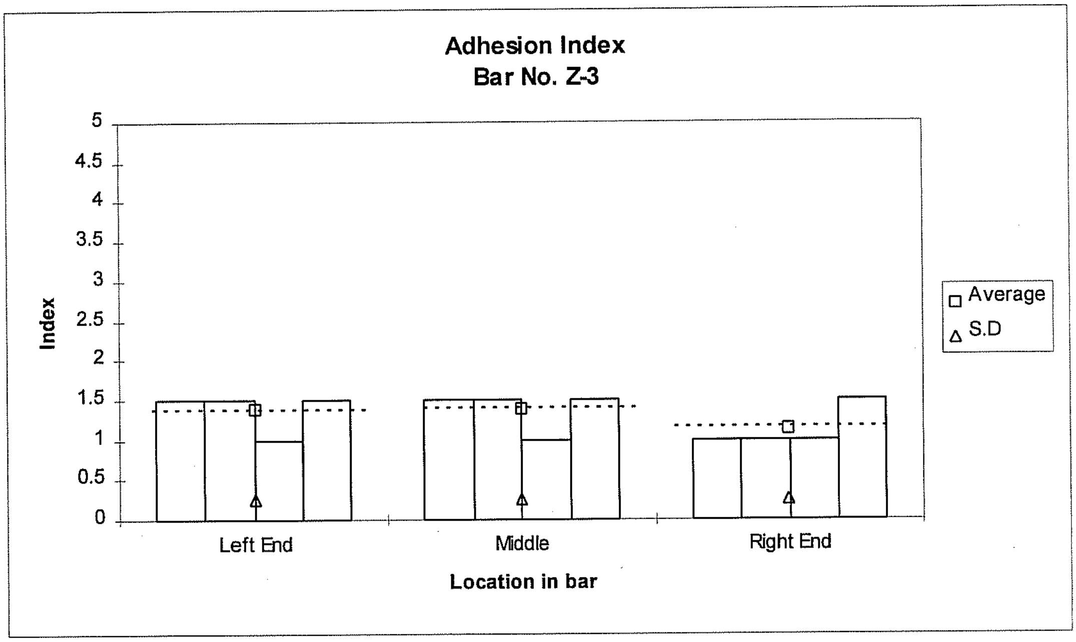


Figure 5.12. Adhesion rating for bar No. Z-3

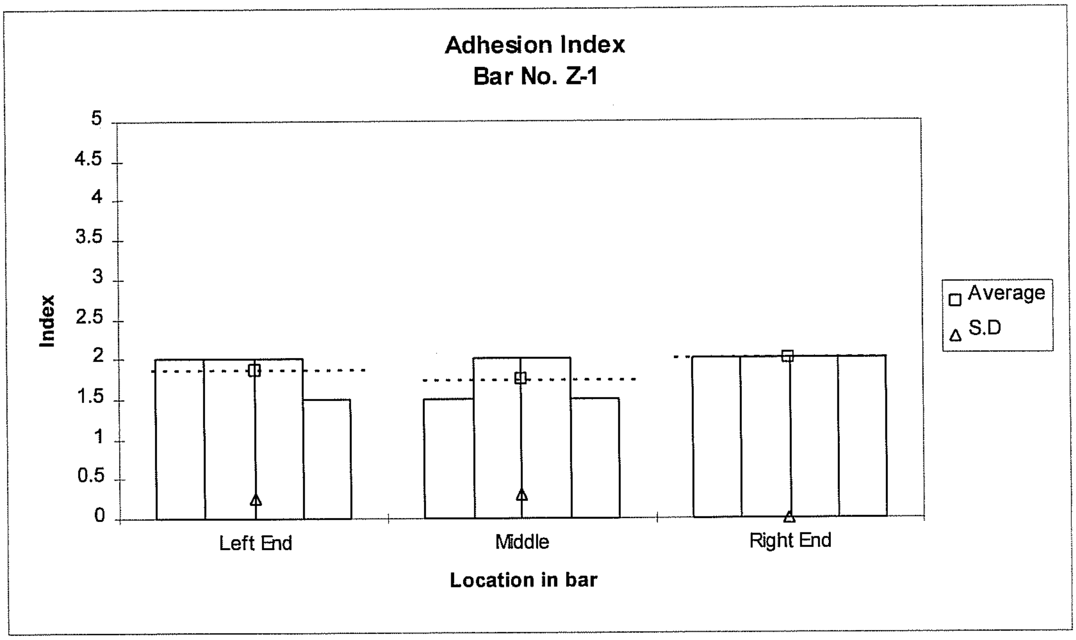


Figure 5.13. Adhesion rating for bar No. Z-1

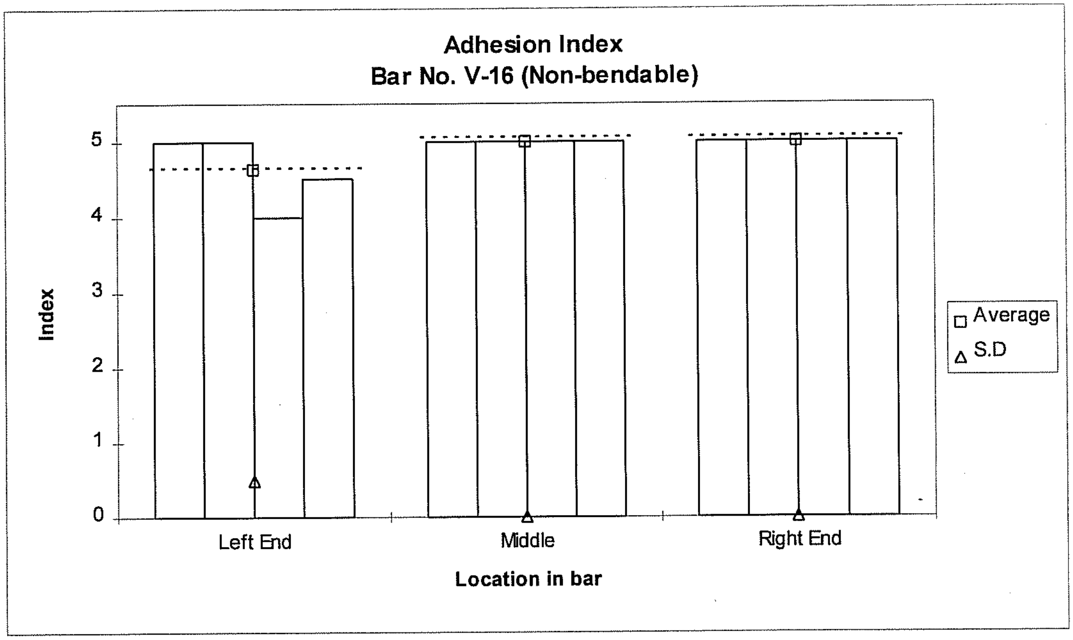


Figure 5.14. Adhesion rating for bar No. V-16

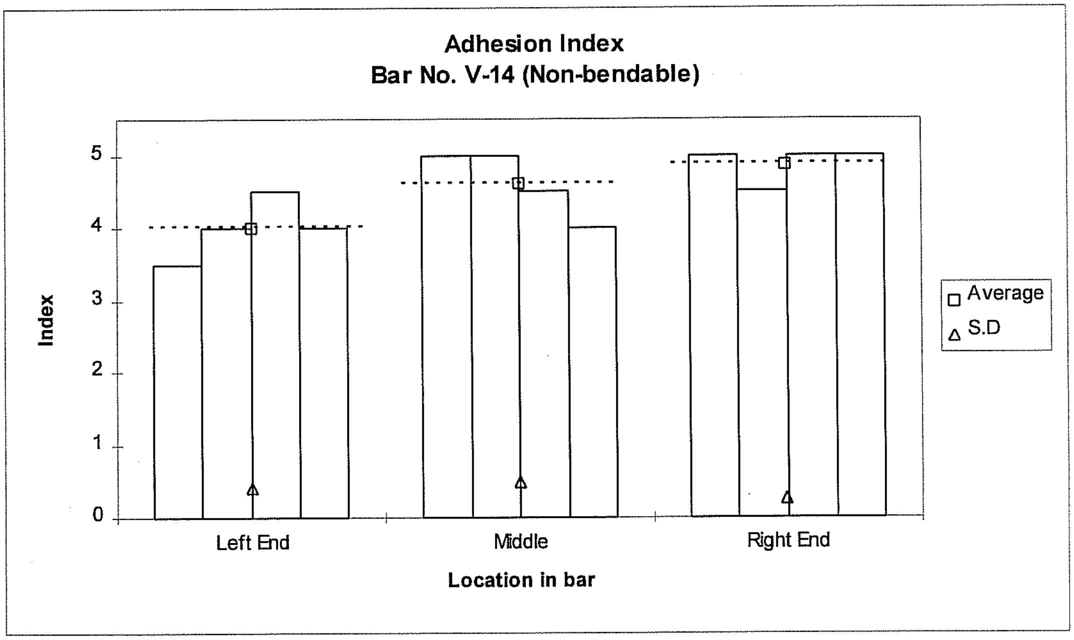


Figure 5.15. Adhesion rating for bar No. V-14

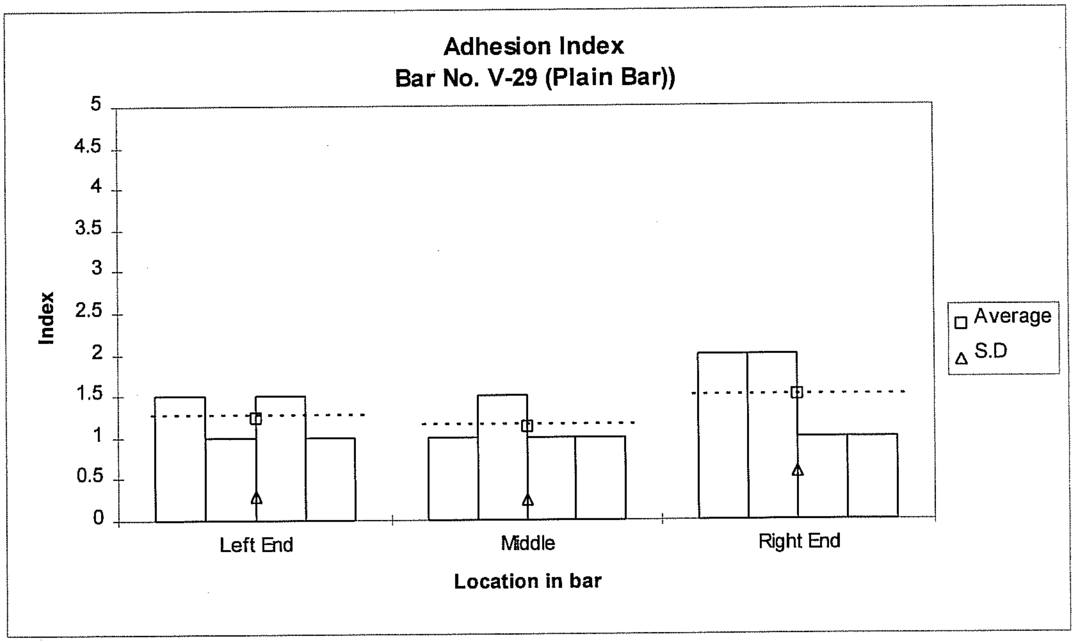


Figure 5.16. Adhesion rating for bar No. V-29

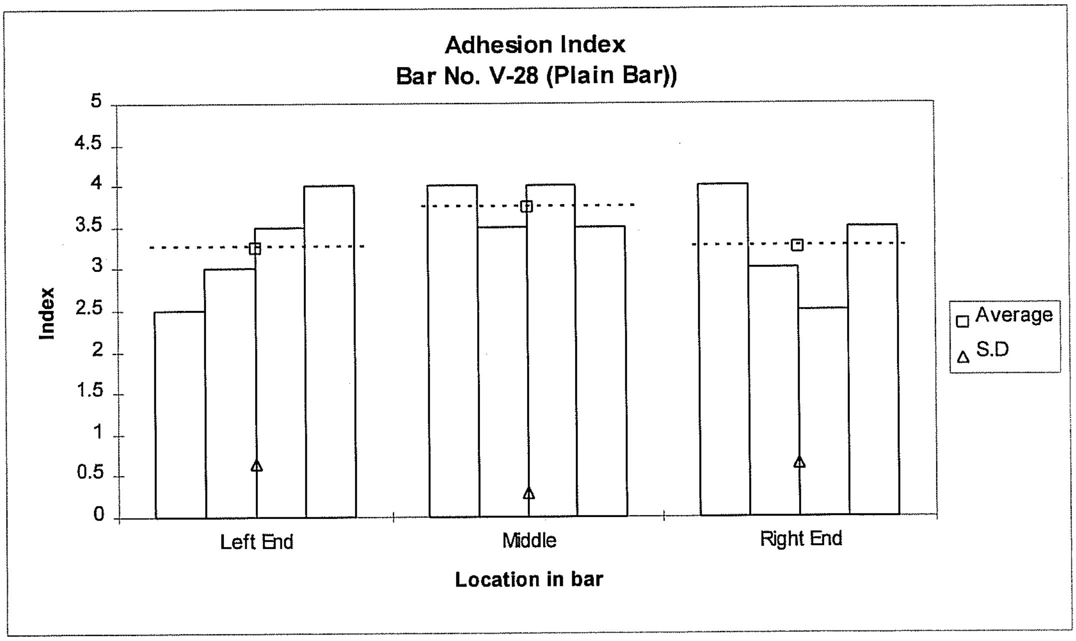


Figure 5.17. Adhesion rating for bar No. V-28

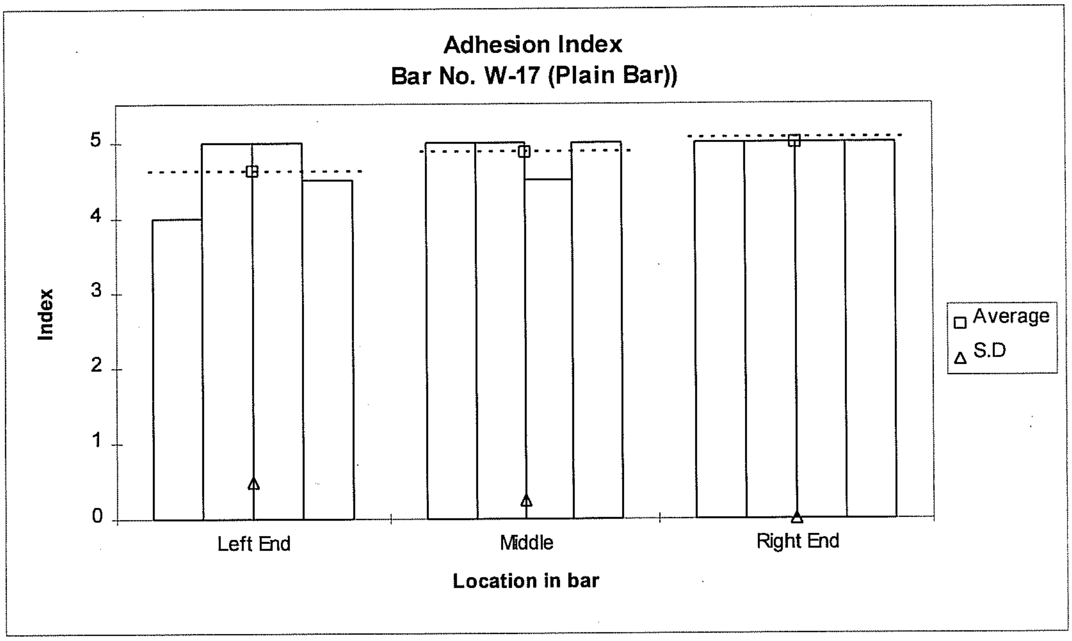


Figure 5.18. Adhesion rating for bar No. W-17

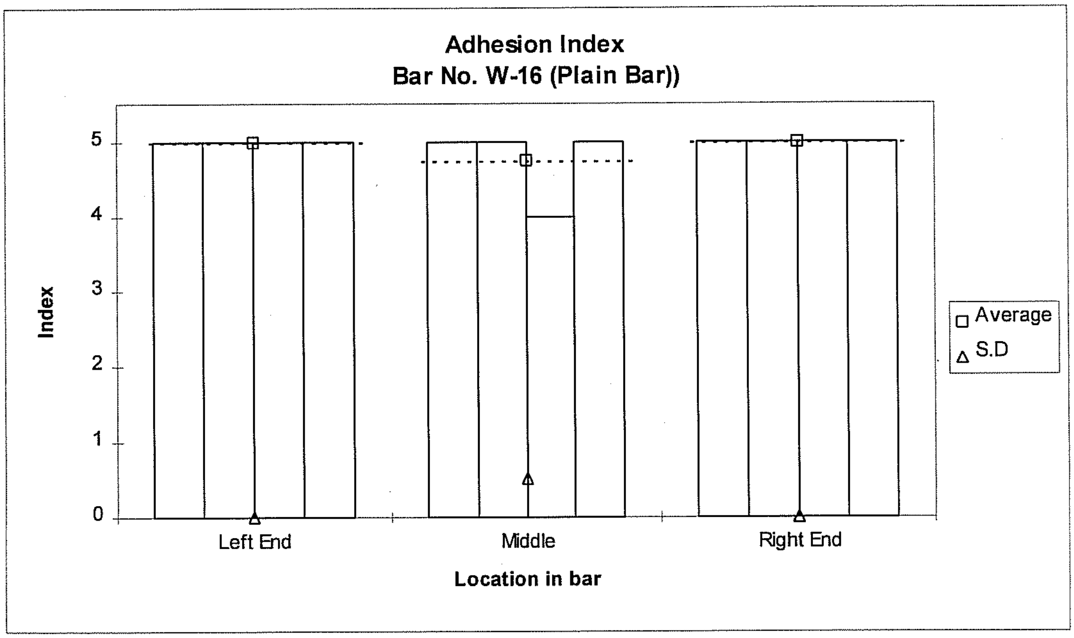


Figure 5.19. Adhesion rating for bar No.W-16

It is possible to note from the preceding plots and tables that the coefficients of variation of adhesion values for each bar vary from a low value of 0% up to a high value of 37%. The coefficient of variation of coating adhesion in epoxy coated bars is affected by two factors:

a. Variability in the coating adhesion of the bar as produced by the coating applicators. This means inconsistencies in the coating, uneven surface preparation, temperature differentials and any other factors that affect coating adhesion. This can be considered the coefficient of variation due to production.

b. Variability obtained during adhesion testing operations or within-test coefficient of variation. This depends on human error, inaccuracy of the testing method, testing conditions and others.

There is no way of separating the factors that affect the overall coefficient of variation in order to prove the accuracy of the testing method. This is because a coated bar can have severe changes in adhesion strength over very short distances along the bar. Performing two adhesion measurements in the area between adjacent deformations may improve the coefficient of variation. Unlike other materials that have homogeneous properties when coming from the same batch, epoxy coated bars from the same lot can have highly variable adhesion strengths.

It is possible however, to understand that many factors affect coating adhesion and that application techniques probably account for most of the variability.

It was noted that lower adhesion indices had higher coefficients of variation. As the adhesion index provided by the test increases, adhesion strength decreases and the coefficient of variation also decreases due to the high average adhesion rating. The only physical meaning of this observation is that bars with lower adhesion indices have larger variations in terms of the average. Standard deviation, on the other hand, ranges from a low of 0 to a high of 1.0 units and most of the higher values of standard deviation correspond to bars with lower adhesion strengths. However, as can be noted in Figure 5.20, the standard deviation remained lower of fairly over (about 15%) the average of 0.40 regardless of the adhesion index obtained, which means almost the same dispersion of adhesion values is being obtained for all coatings. The only exceptions were bars U-6 and U-1 from the same coating applicator, and bar V-28. Both bars from coater U presented high individual index value measurements, and bar U-6 presented large adhesion variability. This could be expected because applicators producing epoxy coated steel with low adhesion strengths are assumed to be less careful in coating application operations and have larger production variability. Bar V-28 suggests a defect in the coating operation during production of that particular lot, because its average rating of 3.4 is fairly large with respect to the average value of 2.0 obtained from all bars coated by that same applicator.

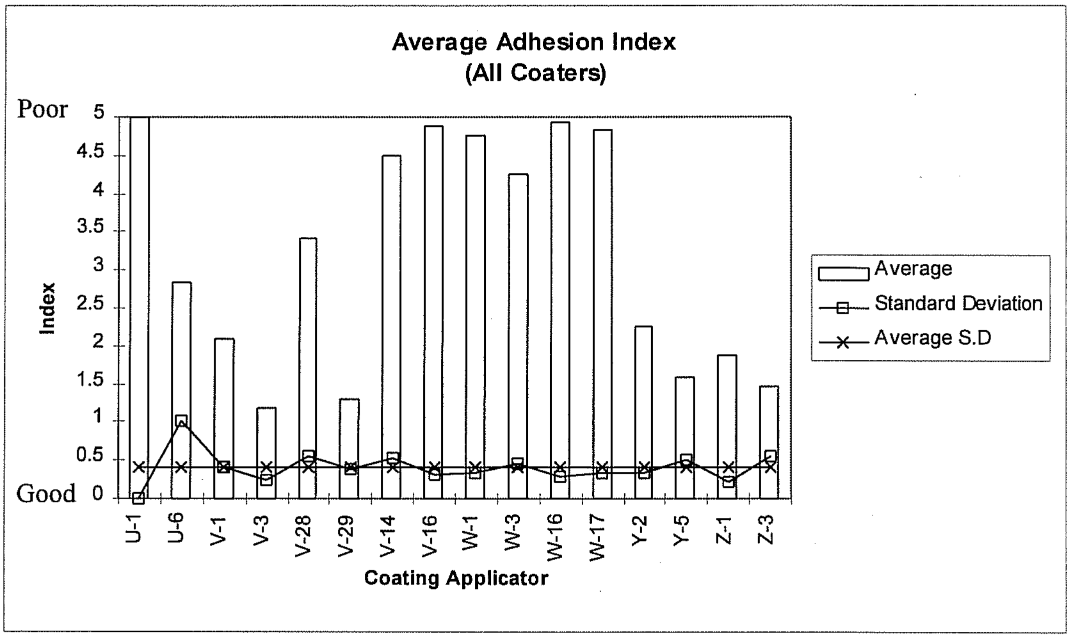


Figure 5.20. Overall adhesion rating of all coaters

Another important observation was that for two bars from different lots from the same applicator, adhesion indices were similar. However, due to the way the evaluating system was devised, it can be expected that smaller bars from the same coater will yield slightly higher values.

Non bendable coatings, performed poorly in hot water tests. It should be remembered that only two samples from the same applicator were tested. For both samples, almost total loss of adhesion was recorded as a result of the adhesion testing.

The average adhesion index values of all bars from the same coating applicator are presented in Table 5.4. The results are presented in decreasing order of adhesion strength.

Coater Designation	**Adhesion Index (All Bars)**
Z	1.67
Y	1.92
V	1.98
U	3.92
W	4.68
V (non-bendable)	4.69

Table 5.4. Coating applicator performance - Hot water tests

5.3 CONTROLLED PEEL TEST (STRIP METHOD)

Table 5.5 presents the results obtained from strip tests on bars supplied by all coaters. For comparison, the same number of tests were performed at each location as conducted for hot water tests.

Bar No.	Maximum (%)	Minimum (%)	Average (%)	S.D (%)	C.V (%)
U-1	10	0	4	5.1	123
U-6	60	0	37	22.3	61
V-1	50	10	22	11.1	51
V-3	70	60	68	3.9	6
V-14	30	0	14	7.9	56
V-16	20	0	12	7.2	61
W-1	0	0	0	0.0	0
W-3	20	0	7	7.5	100
Y-2	50	20	41	10.0	24
Y-5	70	50	61	9.0	15
Z-1	70	50	63	6.5	10
Z-3	70	40	55	8.0	14
W-17	10	0	1	2.9	346
V-29	70	60	69	2.9	4
W-16	10	0	1	2.9	346
V-28	50	0	18	16.4	90

Table 5.5. Data obtained from strip tests for all bars

Very high coefficients of variation indicate large variation in terms of the average adhesion in some bars. The standard deviation varied more than in the hot water tests, probably due to the inherent inaccuracy in reporting the results to the nearest 10%. An interesting observation is that bars with lower adhesion strengths (represented by low numerical values of percentage remaining) can have average adhesion values lower than the standard deviation. This suggests large dispersion of adhesion values in terms of the average. In the case of strip tests, the values of adhesion obtained from bars supplied by the same coater were outside the mean and one standard deviation range found in hot water tests. One reason for errors is the fact that the total percent remaining was taken as the overall portion of epoxy coating that remained adhered to the steel substrate after the test was performed. A more accurate way to measure it might have been to take the percent of the width of the strip that was removed at a specified number of sections along the strip and to obtain an average, as in the X-cut tests. This approach would also provide an indication of coating adhesion changes along the length of the strip. However, by measuring an overall value, it was possible to determine how the procedure correlates with other more accurate methods.

Figures 5.21 through 5.36 show the adhesion values in terms of percentage remaining for each bar. The results are presented for each location along the bar, as was done for hot water tests.

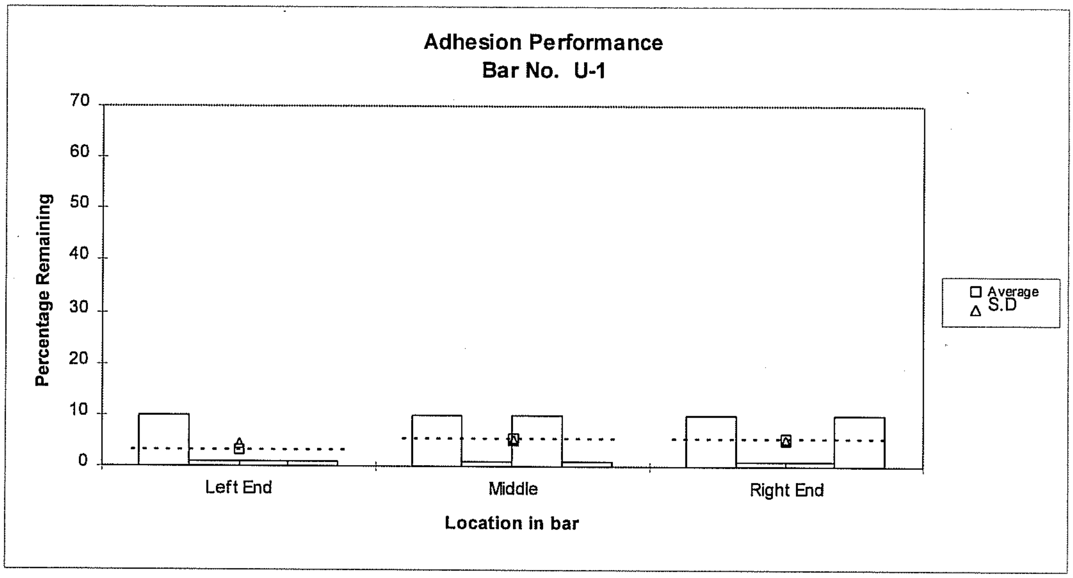


Figure 5.21. Adhesion performance of bar No. U-1

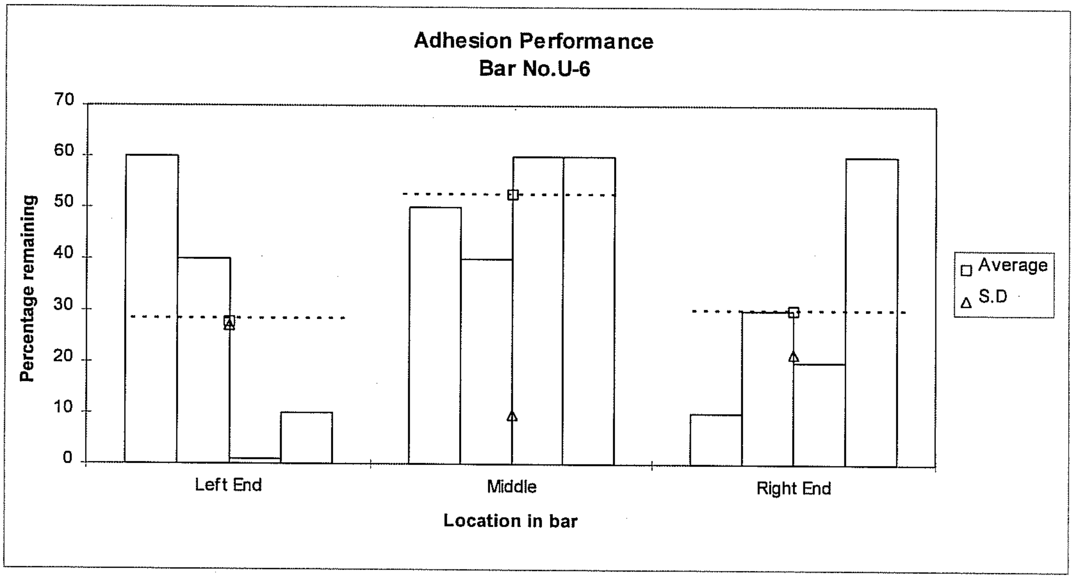


Figure 5.22. Adhesion performance of bar No. U-6

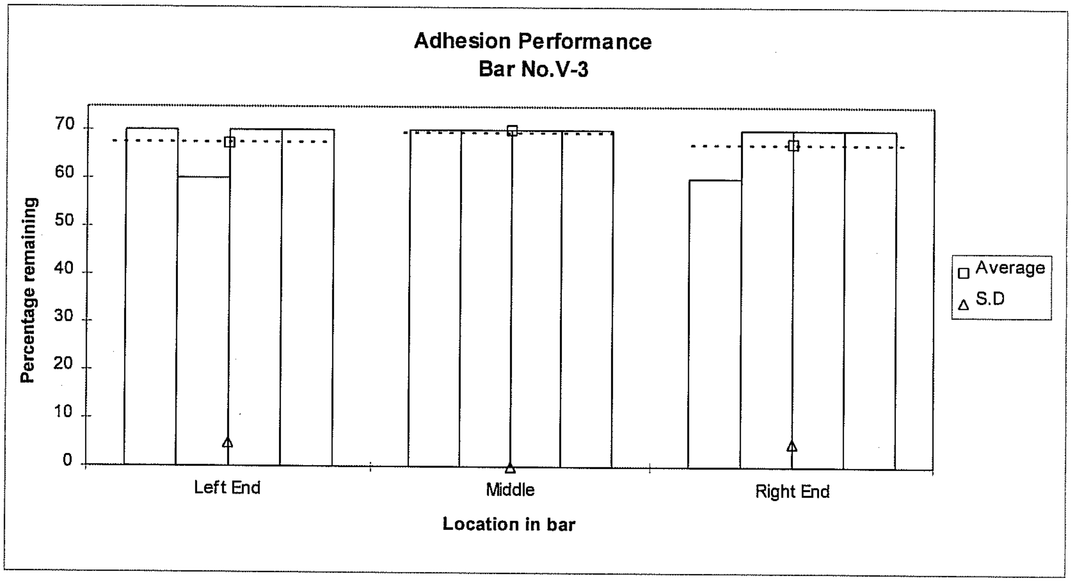


Figure 5.23. Adhesion performance of bar No. V-3

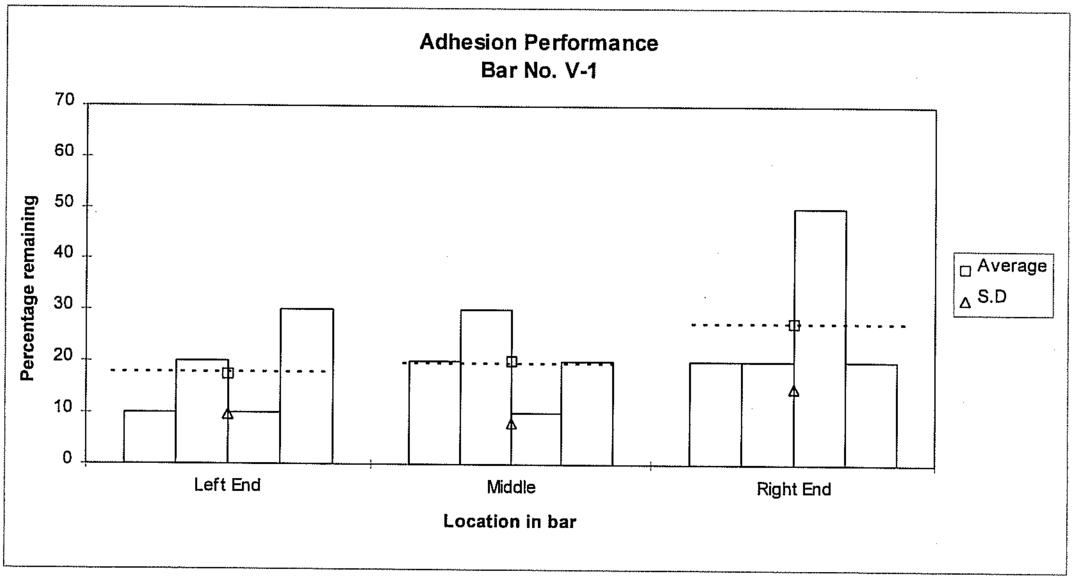


Figure 5.24. Adhesion performance of bar No. V-1

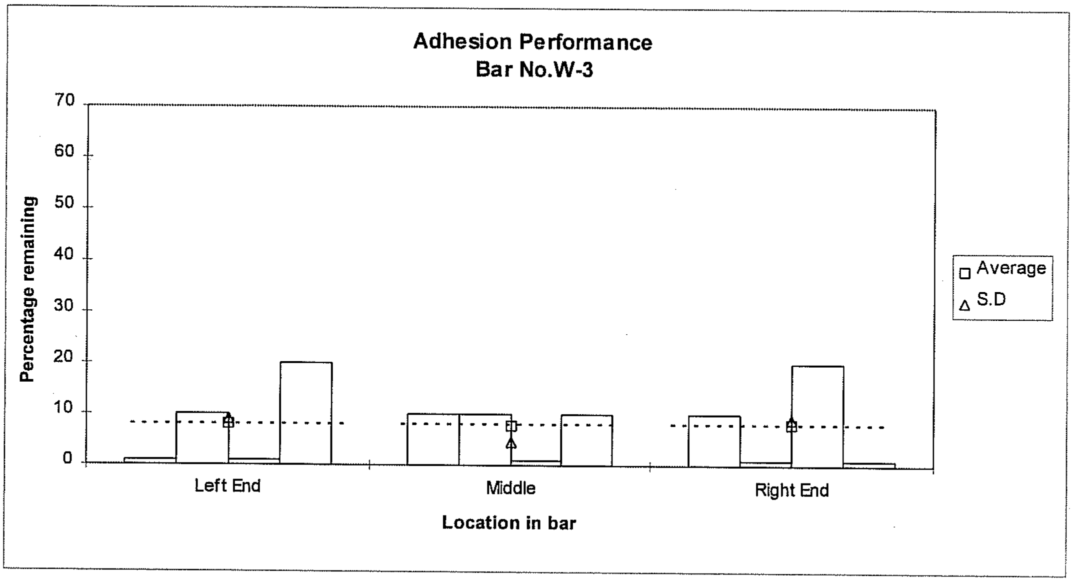


Figure 5.25. Adhesion performance of bar No. W-3

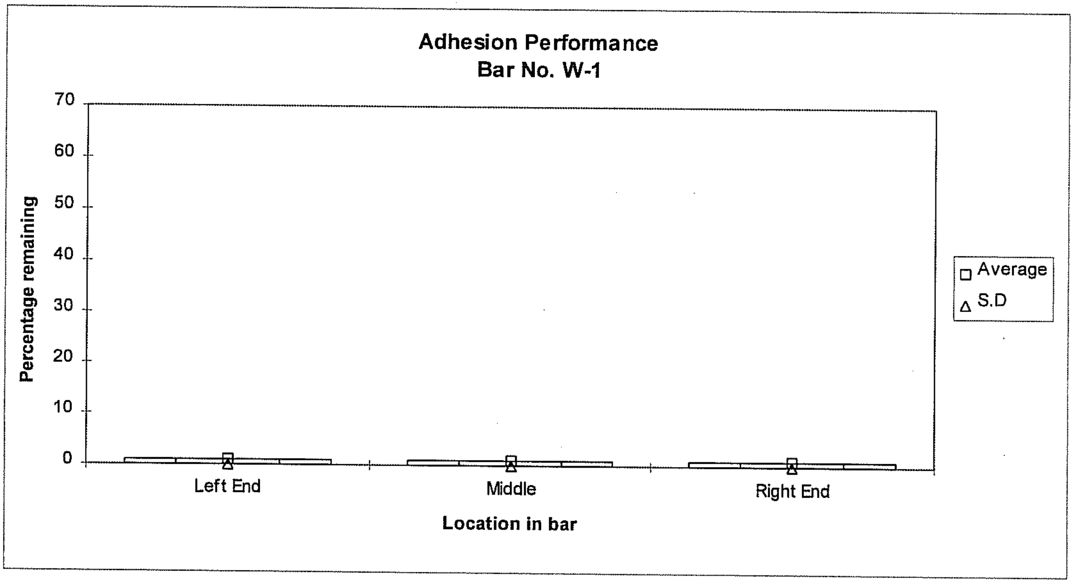


Figure 5.26. Adhesion performance of bar No. W-1

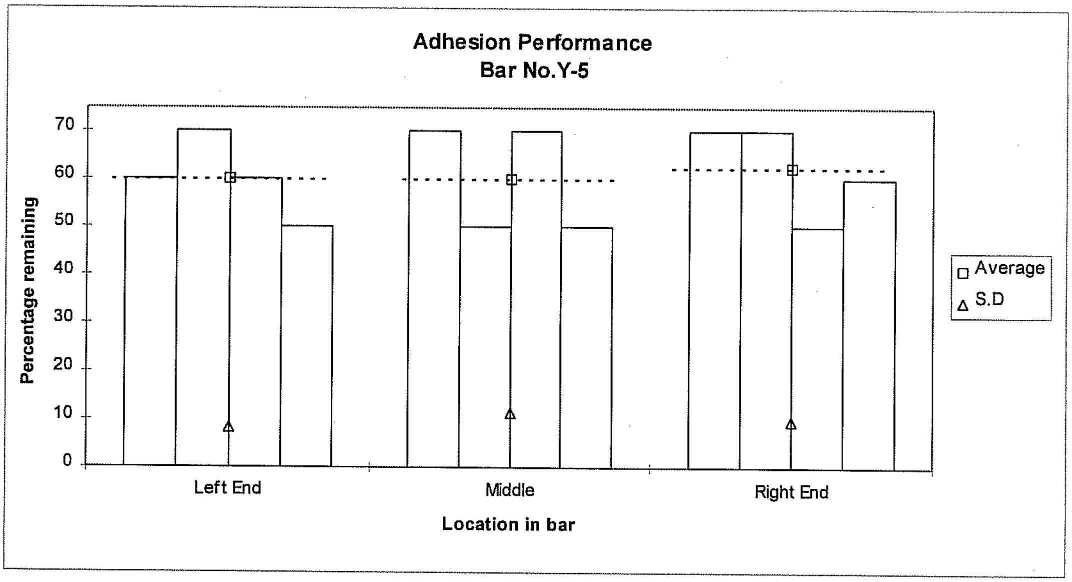


Figure 5.27. Adhesion performance of bar No. Y-5

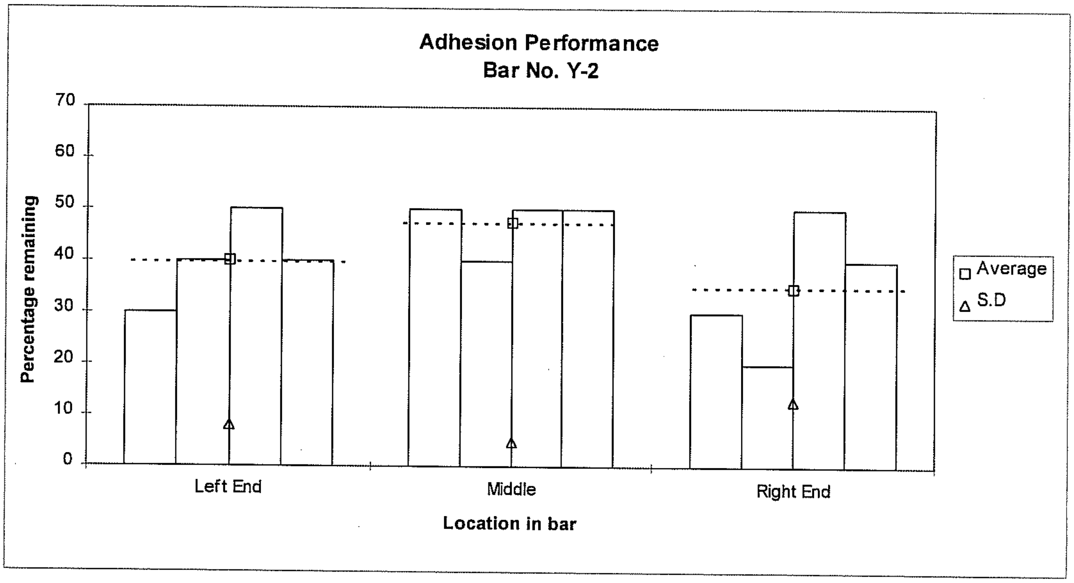


Figure 5.28. Adhesion performance of bar No. Y-2

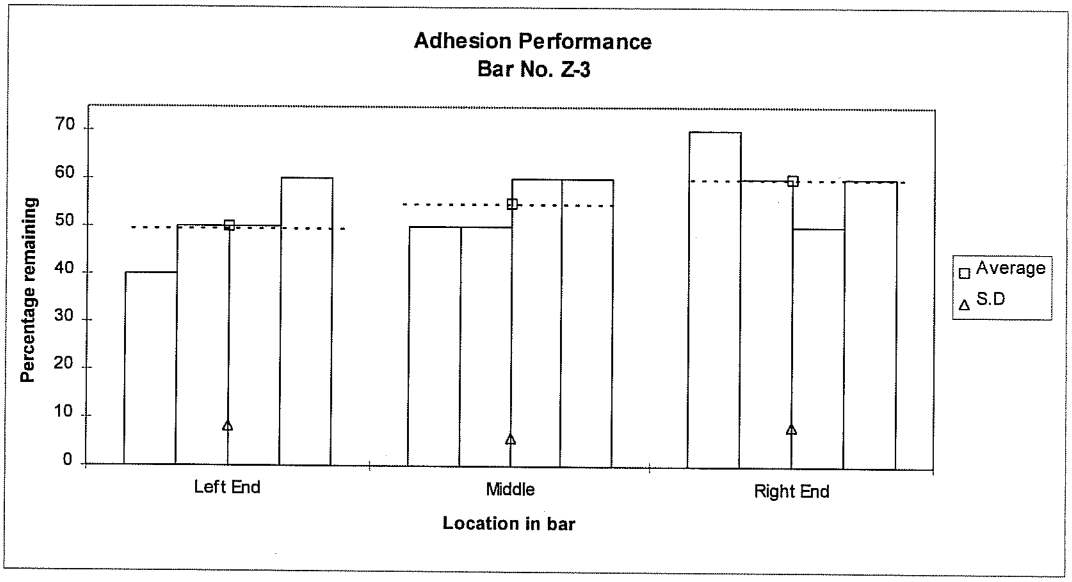


Figure 5.29. Adhesion performance of bar No. Z-3

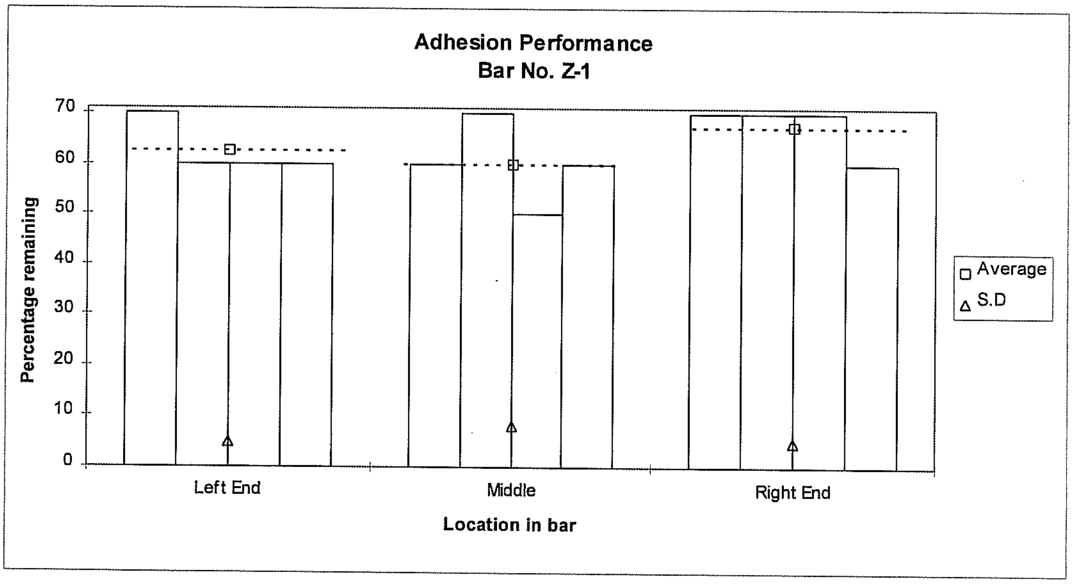


Figure 5.30. Adhesion performance of bar No. Z-1

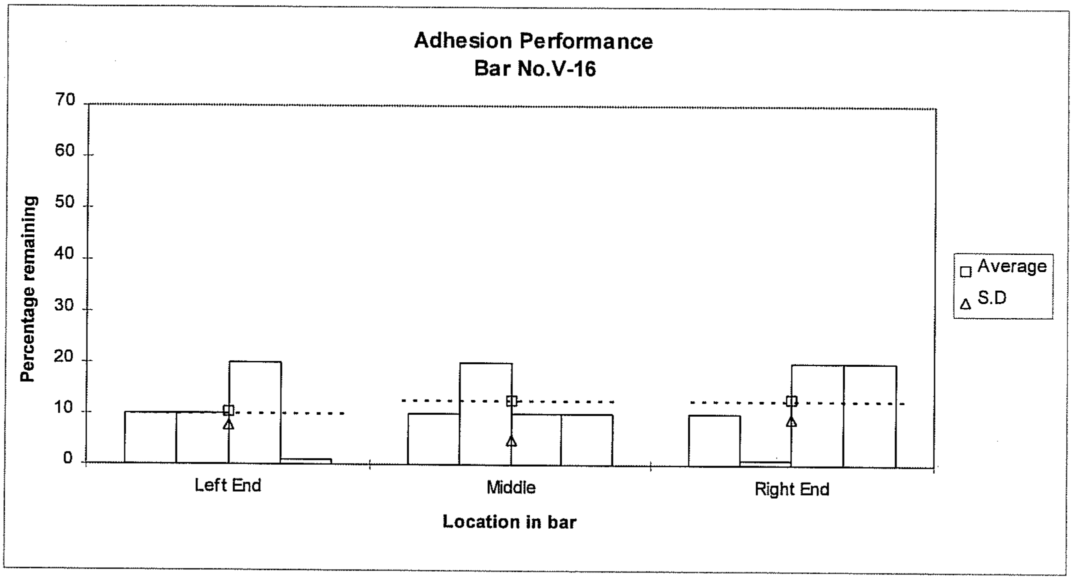


Figure 5.31. Adhesion performance of bar No. V-16

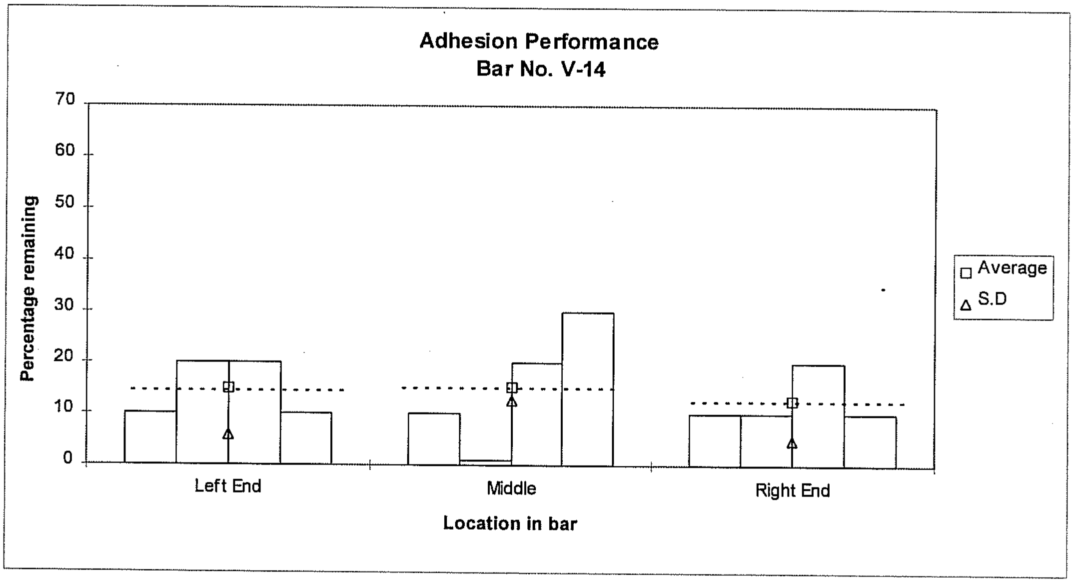


Figure 5.32. Adhesion performance of bar No. V-14

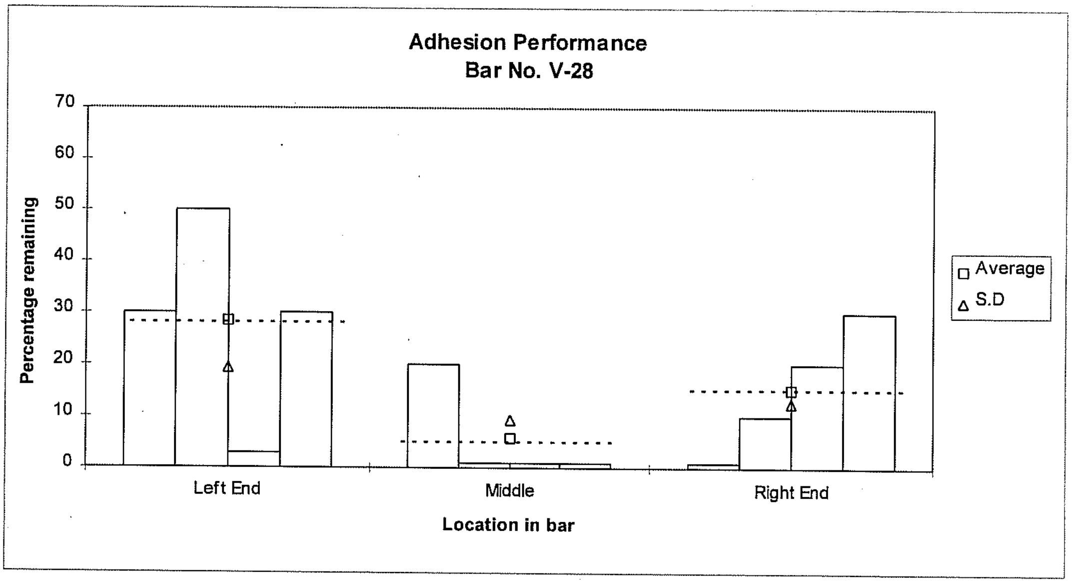


Figure 5.33. Adhesion performance of bar No. V-28

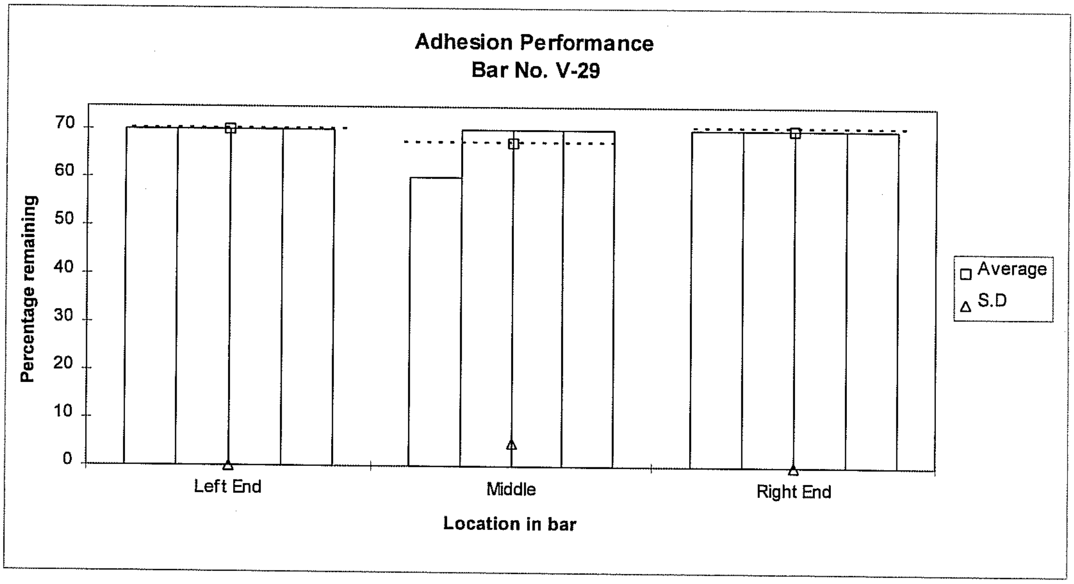


Figure 5.34. Adhesion performance of bar No. V-29

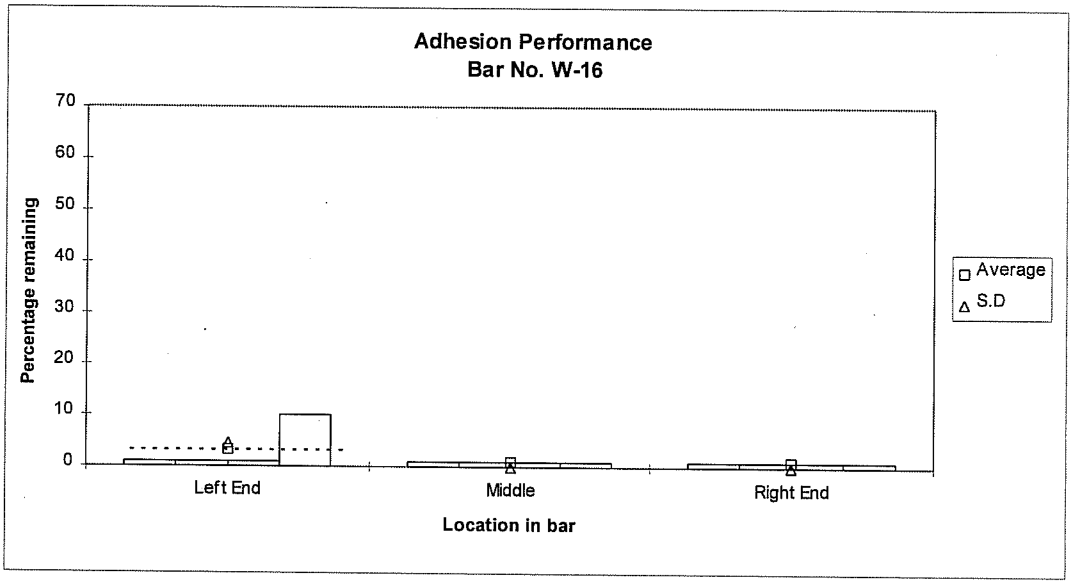


Figure 5.35. Adhesion performance of bar No. W-16

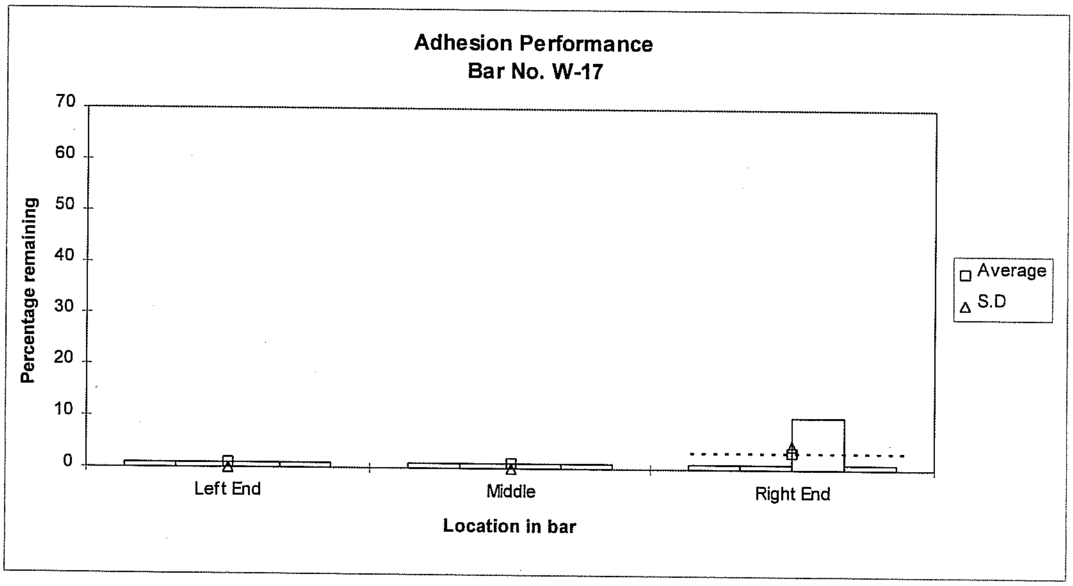


Figure 5.36. Adhesion performance of bar No. W-17

The average adhesion obtained from all bars can be seen in Figure 5.37, along with the standard deviation for each bar.

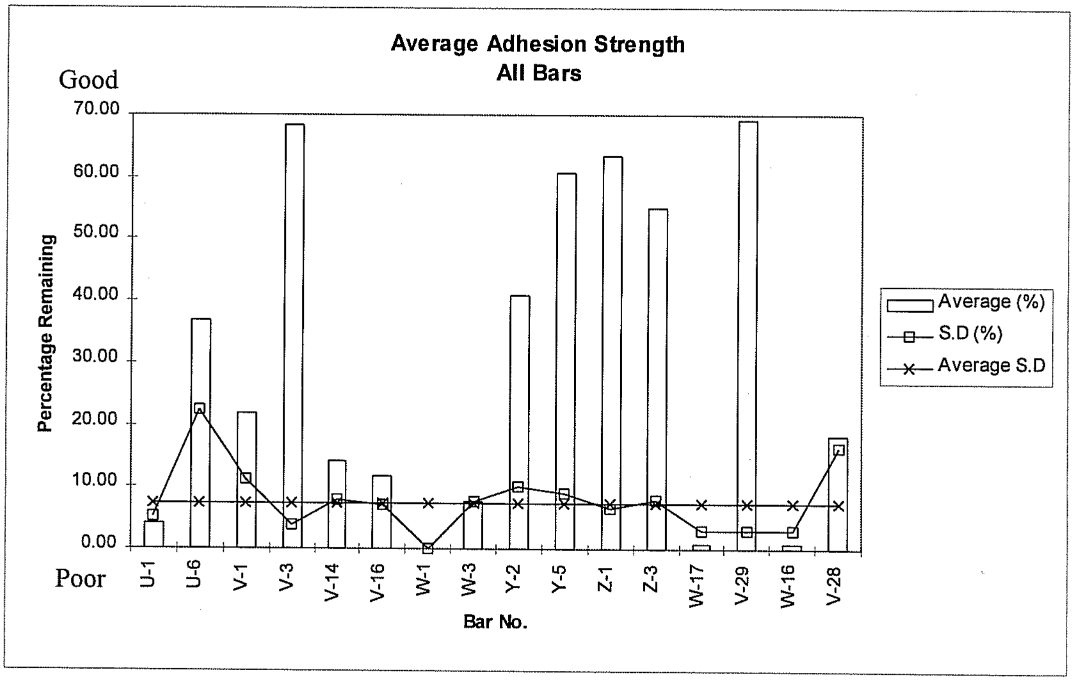


Figure 5.37. Average adhesion performance of all bars in strip tests

As in the hot water tests, rebar produced by coater U had the largest dispersion in adhesion along the bar, as well as variable lot-to-lot adhesion strengths. All bars from coater W had very low adhesion strengths, in all cases below 10%. After the strip tests were performed, visual examination of the surface of the steel in these samples showed a very dark and scaly residue which could be assumed to be the product of improper surface preparation.

Again, as in the hot water tests, non-bendable coatings performed poorly in terms of adhesion strength when compared to bars with flexible coatings provided by the same applicator. However, in contrast to coater W, bars with non-bendable coatings from coater V had very clean steel surfaces, suggesting that adhesion loss may be produced by causes other than poor surface preparation.

The average adhesion of all bars from the same coating applicator are shown in Table 5.6. The results are presented in order of decreasing adhesion as measured by strip tests.

Coater Designation	**Adhesion Performance (All Bars)**
Z	59 %
Y	51 %
V	44 %
U	20 %
V (non-bendable)	13 %
W	2 %

Table 5.6. Coating applicator performance - Strip tests

5.4 CONTROLLED PEEL TEST (X-CUT METHOD)

The results obtained from performing X-cut tests on bars supplied by all coaters are listed on Table 5.7.

Bar No.	Maximum (mm)	Minimum (mm)	Average (mm)	Standard Deviation (mm)	Coefficient of Variation (%)
U-1	4	2.75	3.3	0.3	10
U-6	4.5	1.75	2.9	1.0	33
V-1	3	1.25	1.8	0.6	31
V-3	1.75	1	1.4	0.2	18
V-14	5	4	4.8	0.4	10
V-16	5	4	4.8	0.4	10
W-1	5	4	4.9	0.3	6
W-3	5	3.5	4.1	0.4	10
Y-2	1.5	1	1.3	0.1	11
Y-5	2	1	1.8	0.3	19
Z-1	1.75	1	1.4	0.2	18
Z-3	2.25	1.25	1.6	0.3	17
W-17	5	4	4.9	0.3	6
V-29	1.5	0.5	1.0	0.2	22
W-16	5	4.5	4.9	0.2	4
V-28	5	3	4.1	0.7	17

Table 5.7. Adhesion performance of all bars during X-cut tests

Adhesion ratings ranged from a low of 1.0 mm to 5.0 mm or more. The largest adhesion readings taken were 5 mm, which corresponds to measuring 5mm or larger widths. The reason for not taking any larger measurements is because at that point the uplifting force created by the knife is not being effectively transferred to the whole width of the cut and results are no longer reliable. This was evident in several tests where the coating fractured instead of lifting when values beyond 5mm were reached. The thickness of the coating plays an important role in this matter, but as there were large differences in coating thickness, the same limit value was assumed for all coatings. Regardless of coating thickness, a 5mm reading indicates very low adhesion.

In Figures 5.38 through 5.53, the values of adhesion as measured by the X-cut test are presented for all bars. Average values of adhesion and standard deviation are plotted on each of the three locations tested.

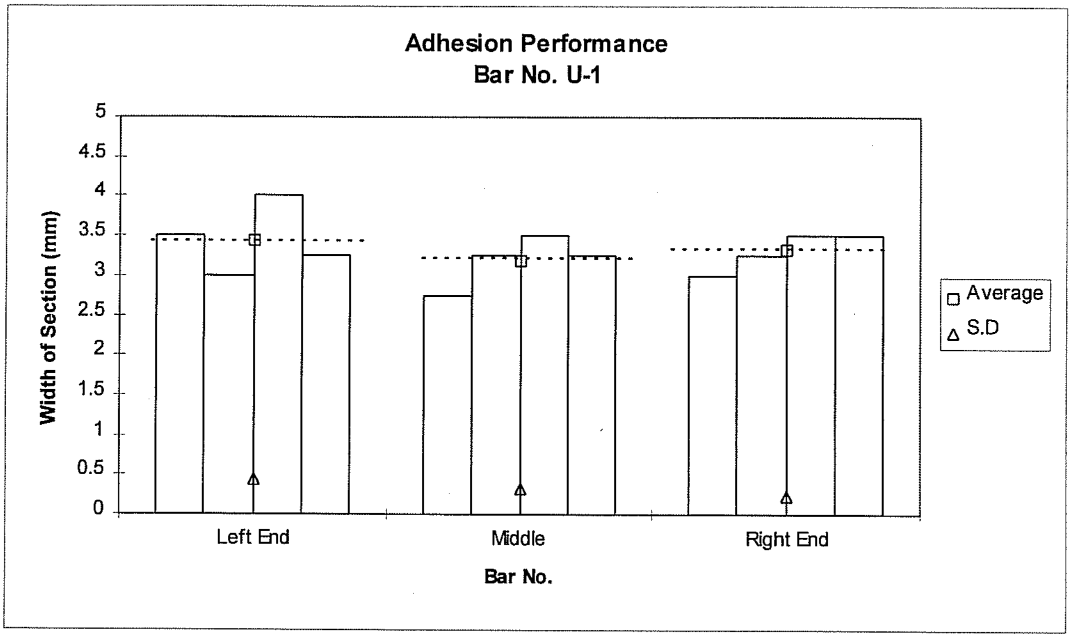


Figure 5.38. Adhesion performance of bar No. U-1

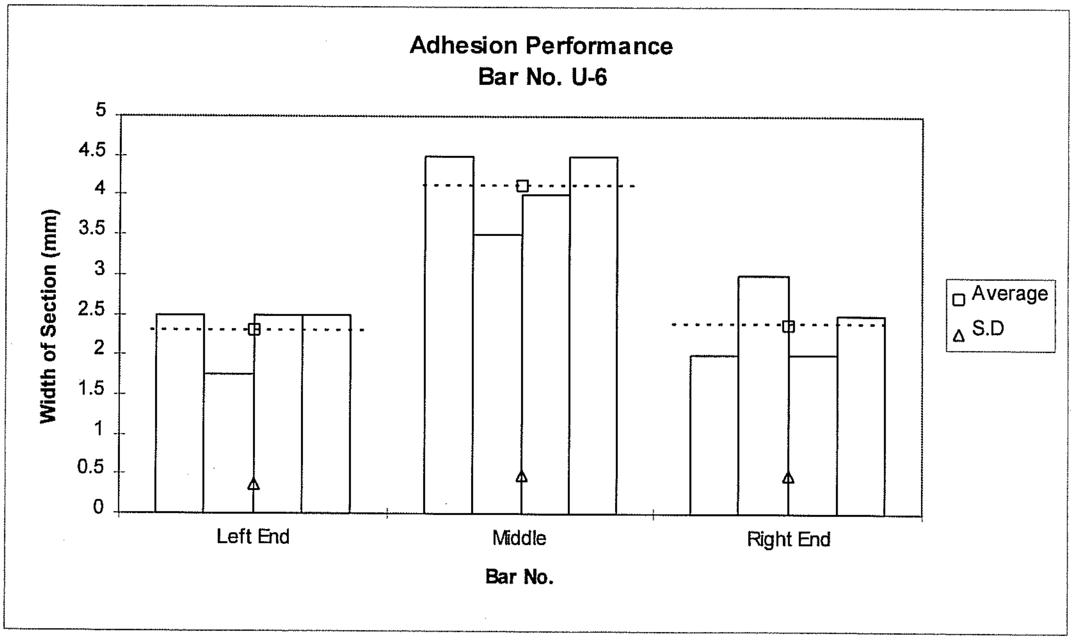


Figure 5.39. Adhesion performance of bar No. U-6

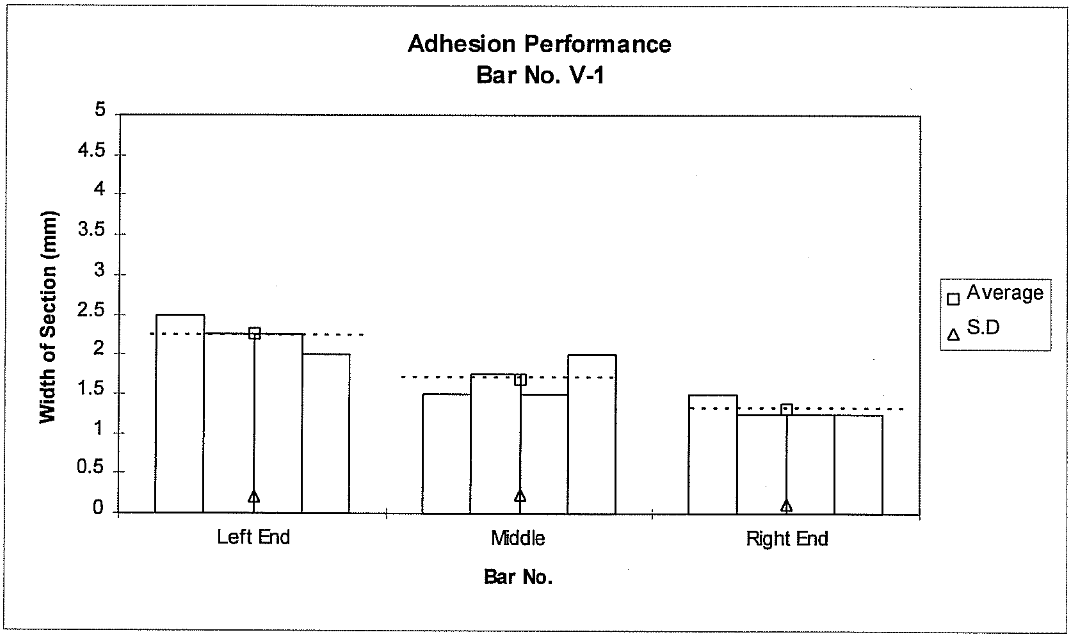


Figure 5.40. Adhesion performance of bar No. V-1

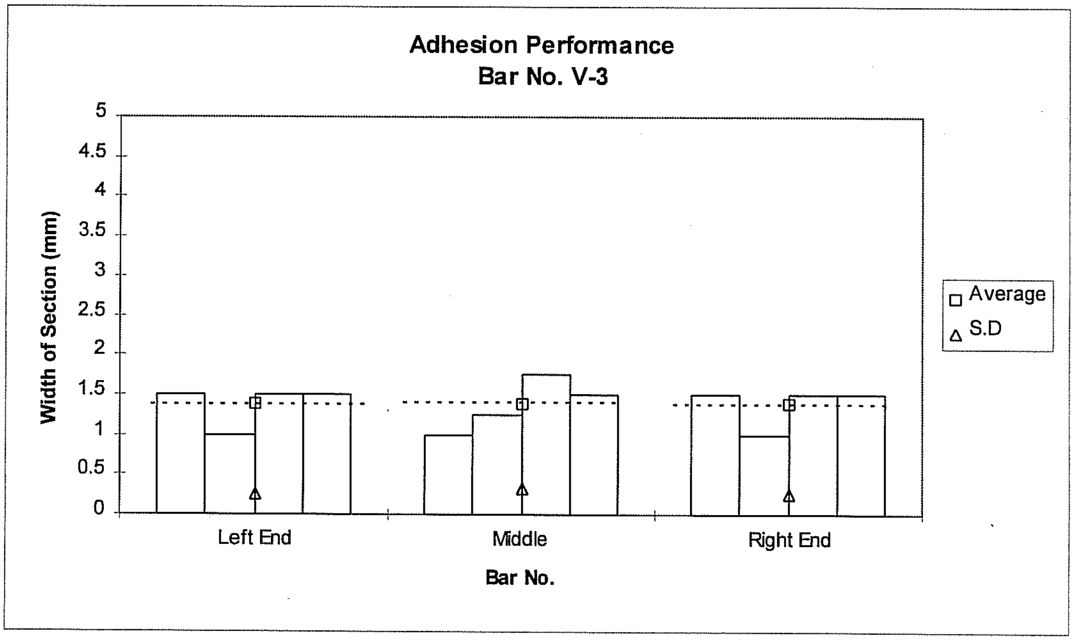


Figure 5.41. Adhesion performance of bar No. V-3

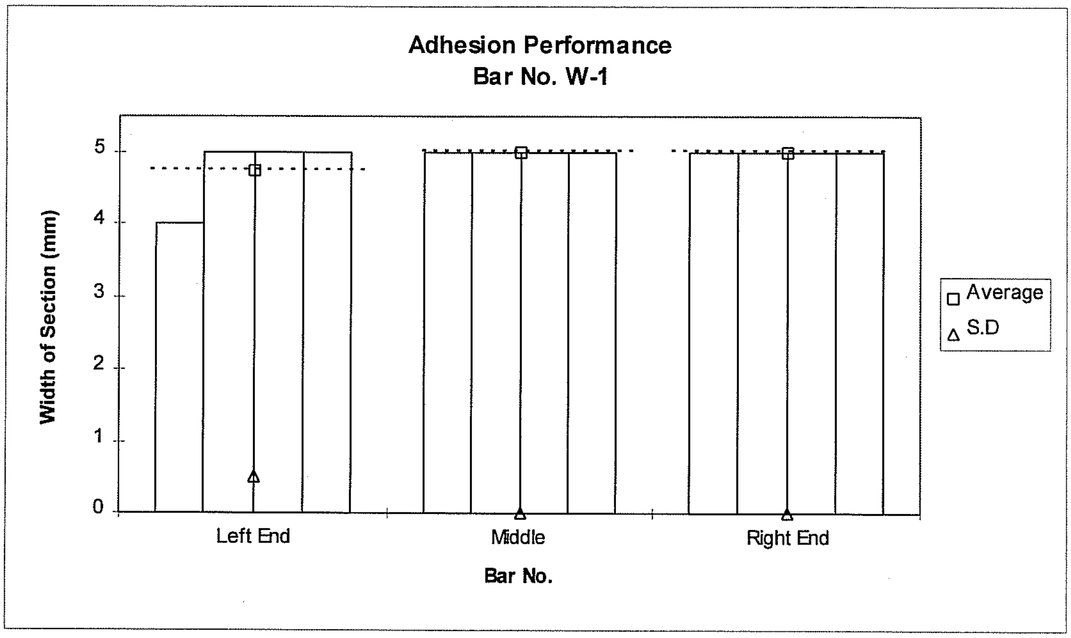


Figure 5.42. Adhesion performance of bar No. W-1

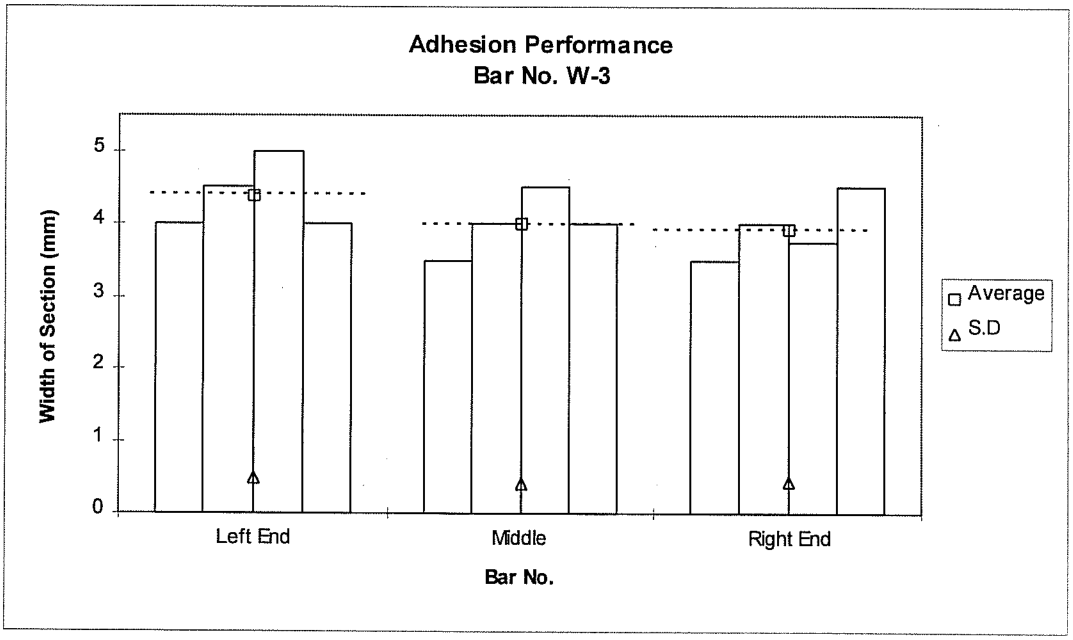


Figure 5.43. Adhesion performance of bar No. W-3

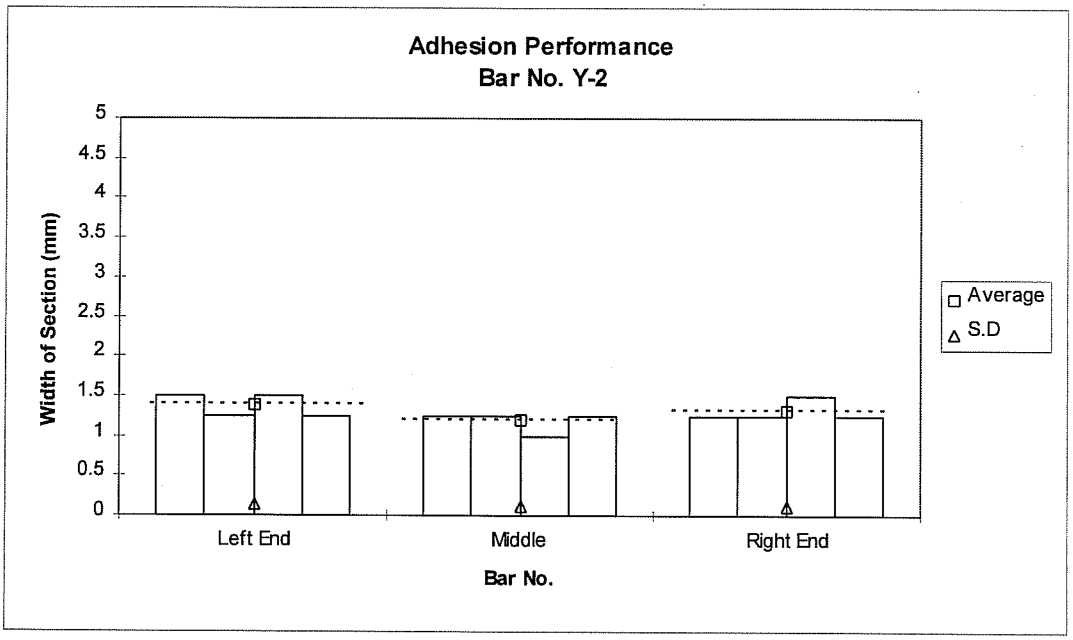


Figure 5.44. Adhesion performance of bar No. Y-2

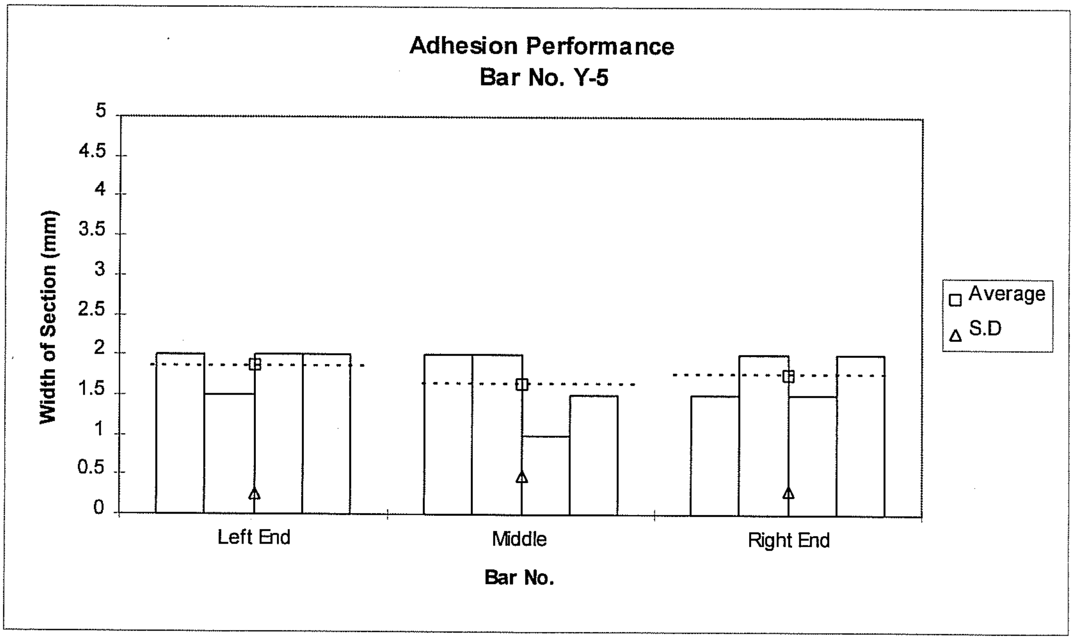


Figure 5.45. Adhesion performance of bar No. Y-5

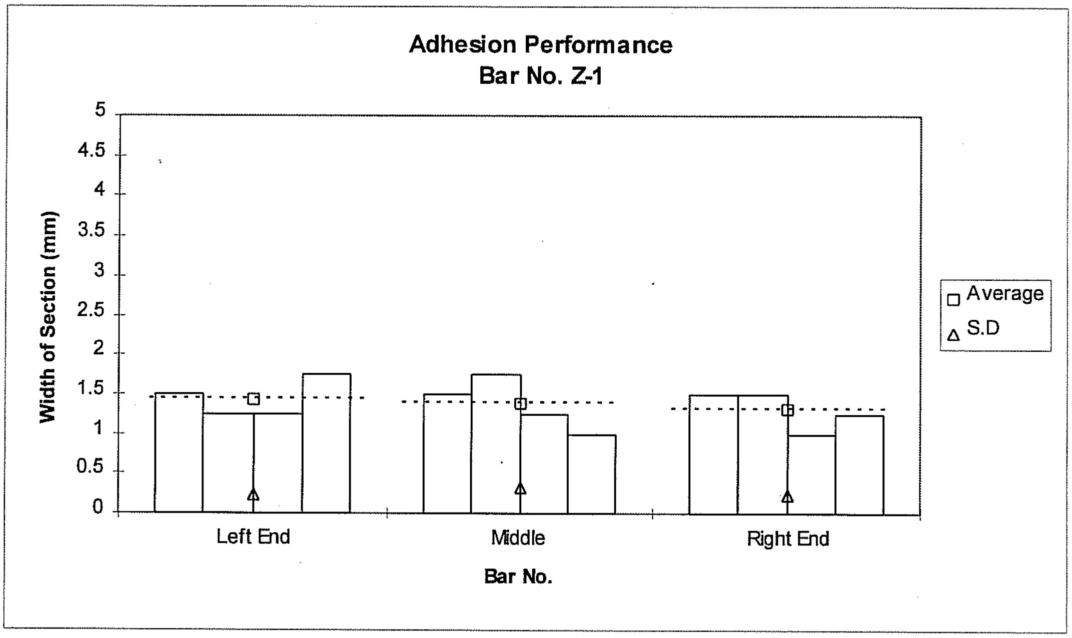


Figure 5.46. Adhesion performance of bar No. Z-1

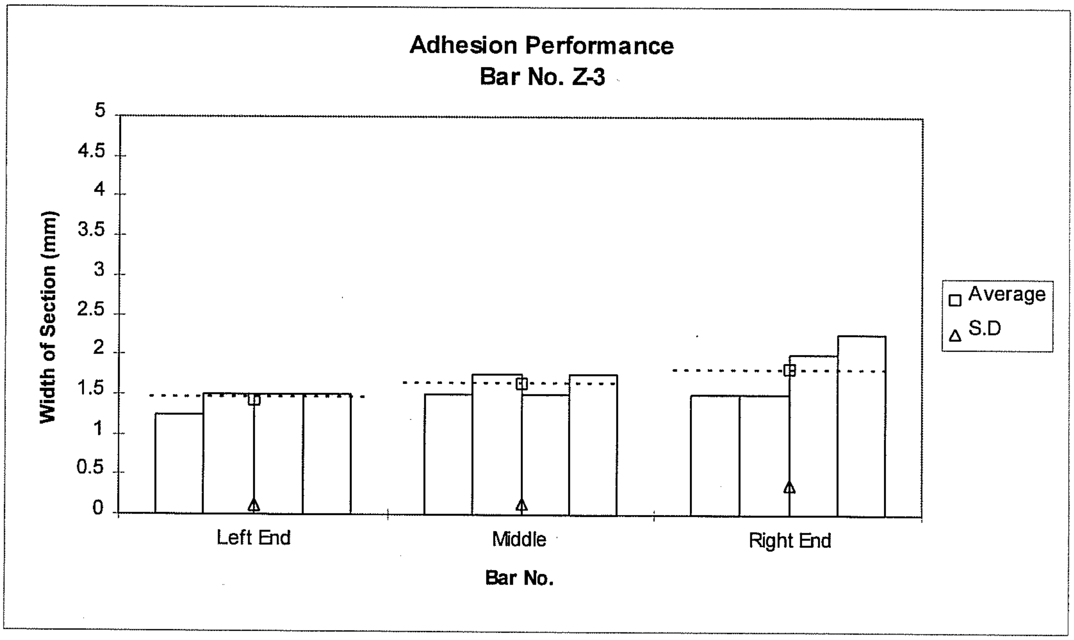


Figure 5.47. Adhesion performance of bar No. Z-3

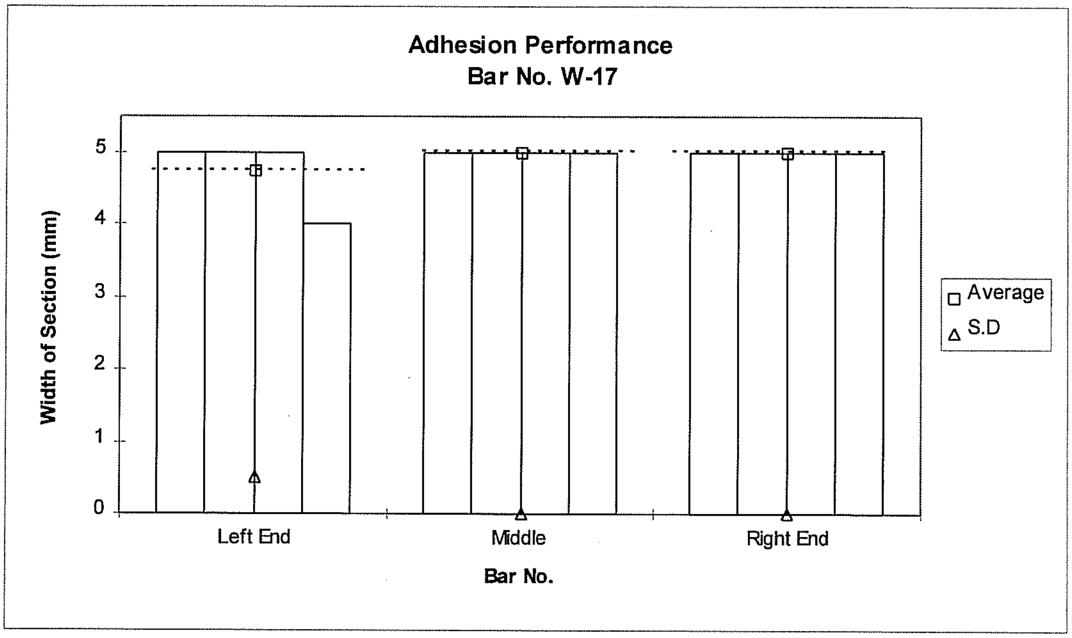


Figure 5.48. Adhesion performance of bar No. W-17

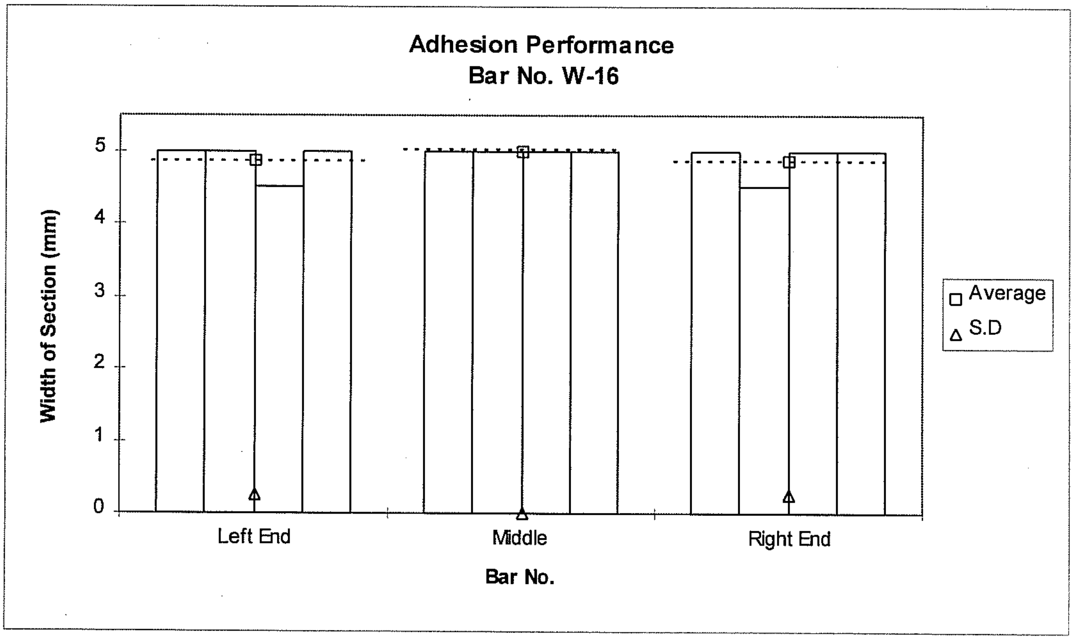


Figure 5.49. Adhesion performance of bar No. W-16

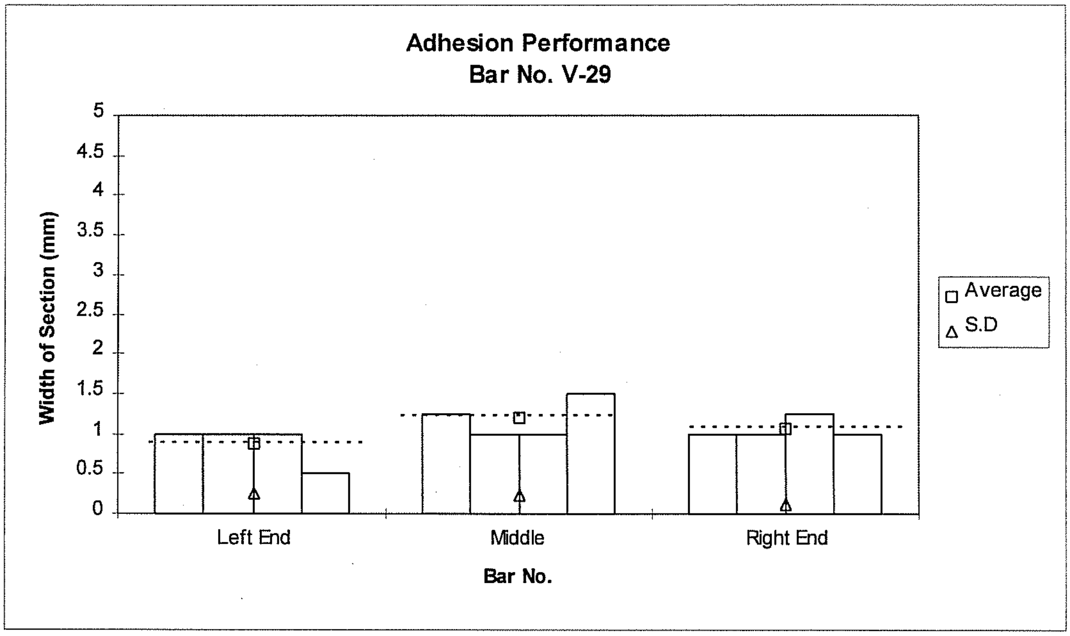


Figure 5.50. Adhesion performance of bar No. V-29

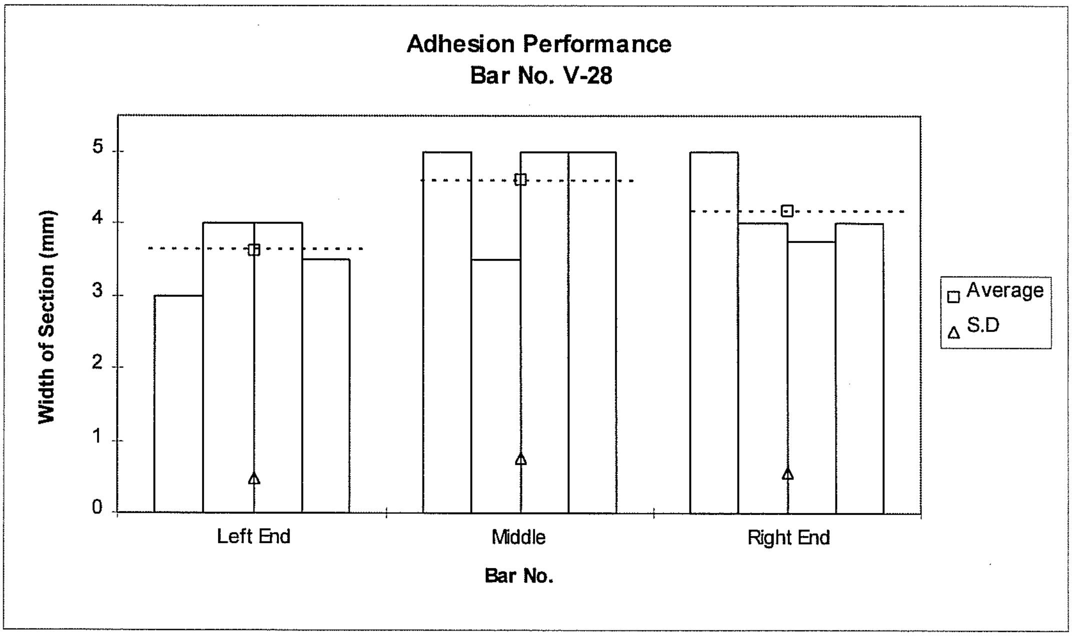


Figure 5.51. Adhesion performance of bar No. V-28

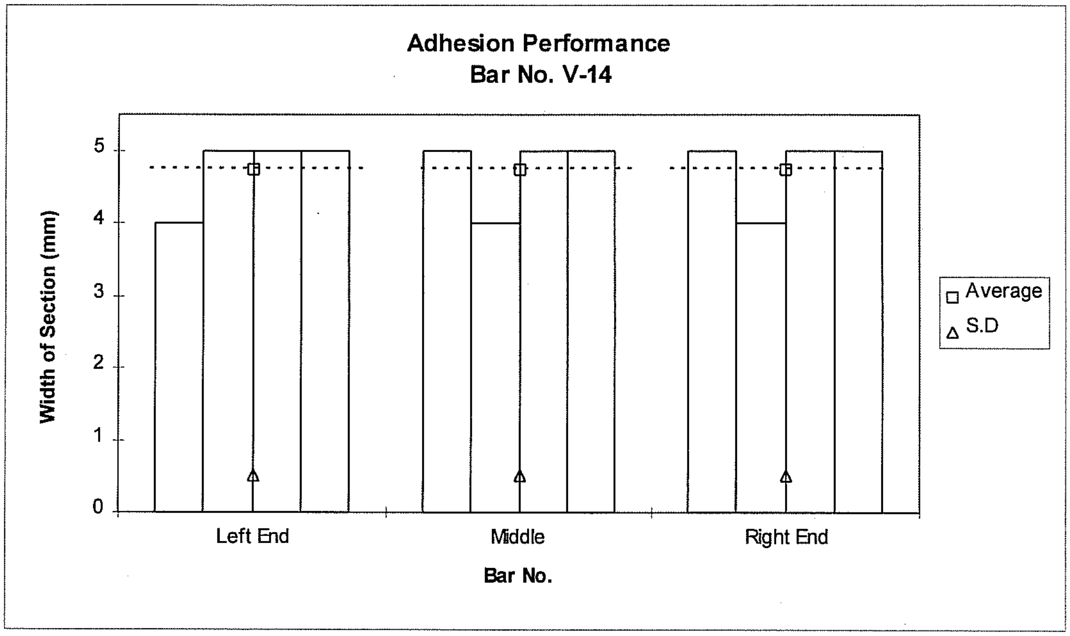


Figure 5.52. Adhesion performance of bar No. V-14

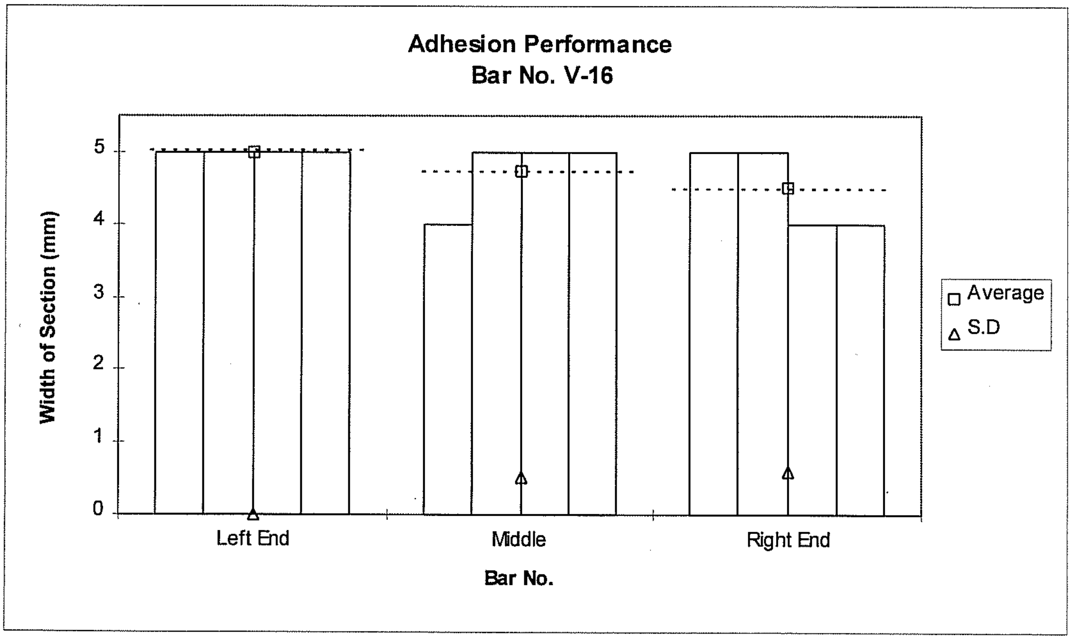


Figure 5.53. Adhesion performance of bar No. V-16

Individual adhesion readings from all bars ranged from 0.5mm to values larger than 5mm with an overall average of 3.1mm. Standard deviation values in the bars ranged from 0.14 to 0.97 as presented in Figure 5.54. The average standard deviation for all bars was 0.38 mm and most bars had standard deviations lower or not more than 15% over the average, with the exception of bars U-6 and V-28. With the exception of bar V-28, all bars from the same coater were within one standard deviation from each other. This is the same case found during hot water tests and provides a measure of production uniformity in a coating application plant.

The best adhesion was obtained in bar V-29, with an average of 1.0 mm. Bar V-28 from the same applicators, provided the lowest adhesion rating in flexible coatings for that particular applicator. Average adhesion values for coater V range from 1.0 mm to 4.1 mm (V-28), with an average of 2.1 mm for all tests. The average value suggests that most readings for the applicator lie close to the 1 to 2 mm range, while the value from bar No. V-28 is an outlier that may suggest further investigation of that particular lot of bars.

The lowest adhesion was obtained in all bars from coater W, with an average adhesion performance of 4.71mm. Table 5.8 lists the average adhesion readings obtained on all bars from the same coating applicator.

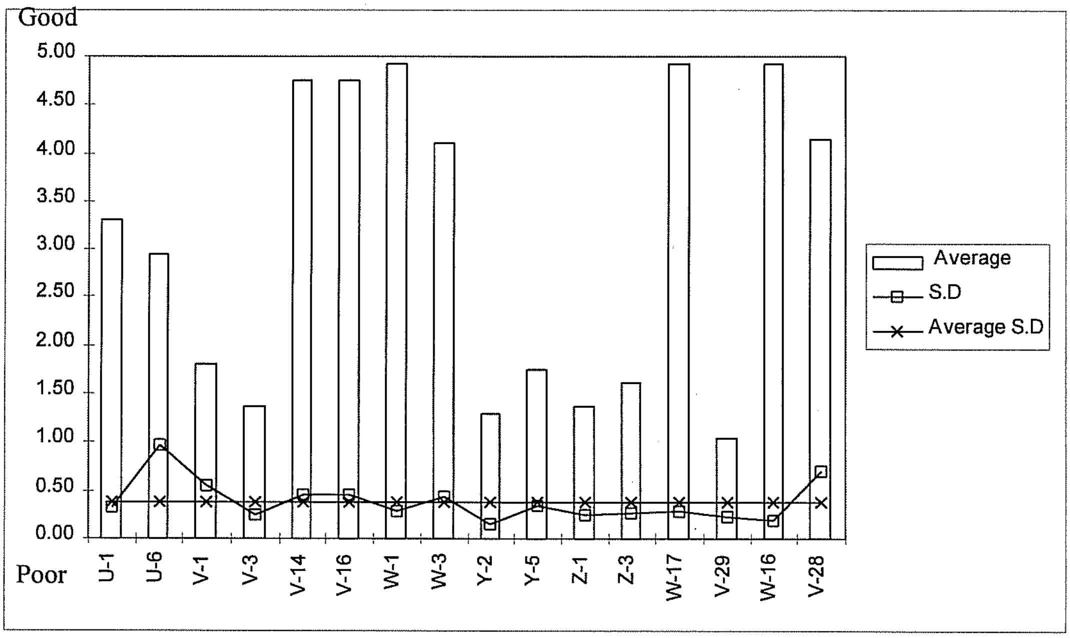


Figure 5.54. Overall adhesion performance of all bars by the X-cut method

Coater Designation	Adhesion Performance (All Bars)
Z	1.5 mm
Y	1.5 mm
V	2.1 mm
U	3.1 mm
W	4.7 mm
V (non-bendable)	4.8 mm

Table 5.8. Coating applicator performance - X-Cut tests

5.5 PERFORMANCE OF DIFFERENT COATERS AS MEASURED BY TXDOT TEST METHOD TEX 739-I

As mentioned in Chapter 4, all bars from the five different coaters were tested for adhesion as recommended by test method Tex 739-I. The results from these tests are presented in Table 5.9. Individual evaluations were carried out in each of the three locations on every bar and the rating system is based on a pass/fail criteria. The fail rating involves finding bad adhesion in any or all the tested locations of each bar.

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

Table 5.9. Adhesion performance of all bars - Tex 739-I peel tests

No attempt is made to establish an order of better to worse adhesion due to the subjective nature of such a process. The adhesion performance of the coating is only rated by how easy or difficult it is to lift sections of coating from the bar, and not by how much force is used during the process.

As expected, all bars from coater W failed this peel test. The most probable cause of failure was bad surface preparation before coating operations. Also, both bars with non-bendable coatings failed the test. In the case of non-bendable coatings, care had to be taken because rigid coatings tend to be brittle and break during the test, instead of lifting from the steel surface in one piece. Failure to understand such behavior can lead to a wrong determination of coating adhesion.

5.6 COMPARISON OF TEST RESULTS

In Figure 5.55 the average index for each location is plotted against the thickness of the coating at the tested location. This was done for the three test procedures developed. No relationship was found between thickness of the coating and adhesion strength for any of the three kinds of tests performed on the bars.

In order to be able to compare values of adhesion from different tests, results were normalized by dividing them by the highest adhesion values possible for the test. As the normalized index values approach unity, the adhesion strength is reduced.

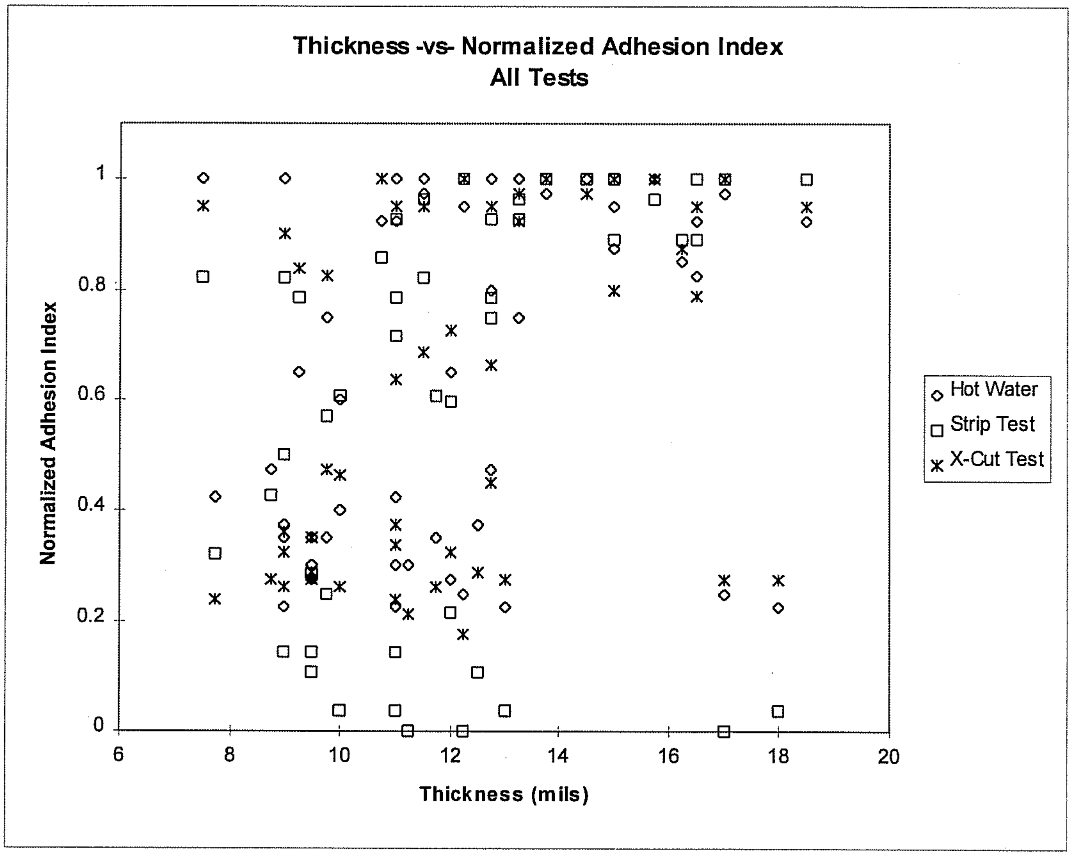


Figure 5.55. Thickness - vs- Normalized Adhesion Index plot

Due to the different rating systems used for each test presented in the prior sections, individual normalized test results for each bar can be compared graphically against the results of another procedure. For this purpose, the normalized rating obtained from the average of the three tests will be assumed to provide the "exact" adhesion strength for each bar. The objective is to find a trend in the results, or in other words, to investigate if the relative adhesion between different bars is maintained when using different test procedures.

Since the hot water test and the X-cut method have rating systems which provide a low numerical value for bars with good adhesion and a high numerical value for bars with poor adhesion, the results from the strip test had to be presented in the same manner to obtain good graphical correlation. This was done by taking the results, which were in terms of the percentage of epoxy coating remaining in the strip, and subtracting them from the maximum possible value of 70%. The resulting value represents the percentage of epoxy coating that is removed.

Figure 5.56 shows the average adhesion values for all bars as measured by all test procedures. In the figure, all boxed bar numbers correspond to bars that failed the Tex 739-I peel test. Table 5.10 shows the difference between adhesion ratings obtained by each method when compared to the average of the three. The comparison is made in terms of the percent difference between the average and the normalized rating for each bar. It was observed that bars with good coating adhesion (between 0.20 and 0.4 normalized rating) had the largest percent differences when compared to the average of the results from all tests. This difference could be attributed to the fact that in strip tests, a perfect rating means that only a thin strip the width of the blade is removed. As the result is given only by visual inspection, the test is assigned a rating of 70%. Any variation in the width of the thin strip lifted by the knife would lead to a high estimate of the percentage of coating remaining at the end of the test. This effect tends to lower the average of all tests and produce larger percent differences when compared to more accurate test methods. A better correlation of results is observed for all adhesion ratings when using the X-cut test. The results from the X-cut method are inherently more exact than the strip method because they are based on an exact measurement rather than a visual observation. Another reason for differences is that each test was performed in different areas of the same section of the bar, and adhesion strength can change in very short distances along the bar. One such case is bar V-1, which in both tests had standard deviations larger than the average, suggesting large scatter of adhesion along the bar.

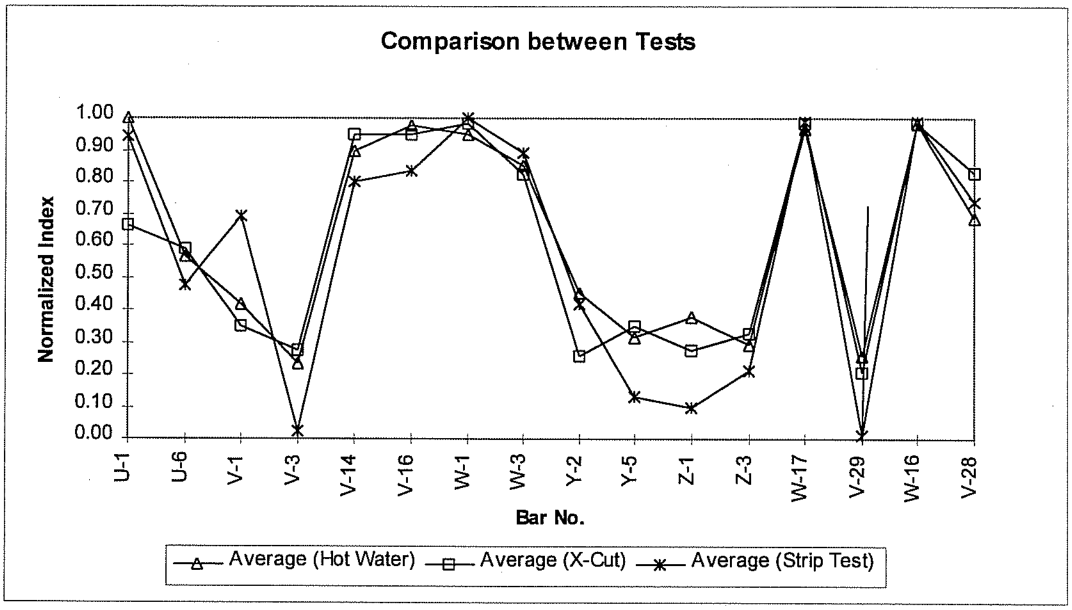
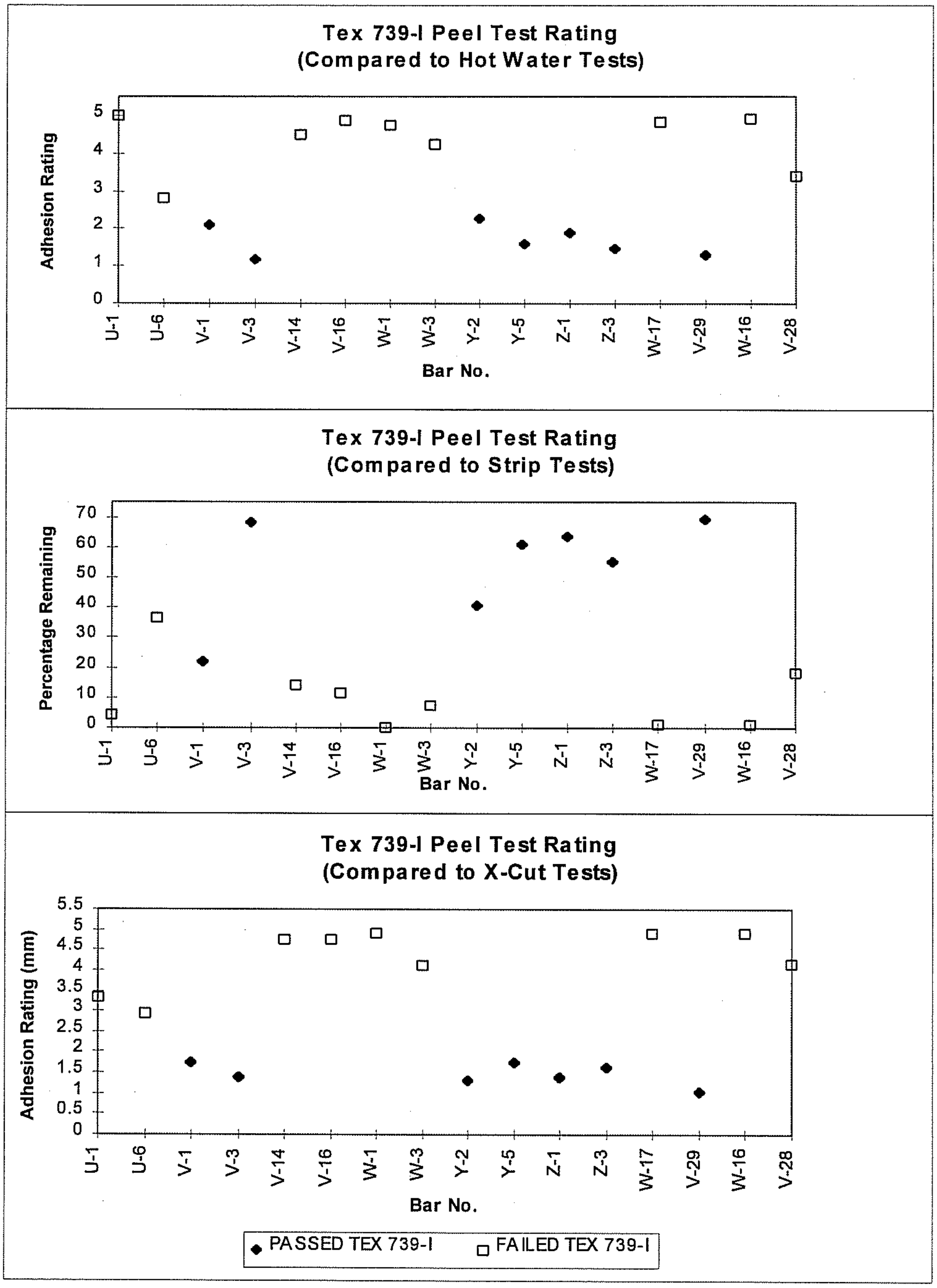


Figure 5.56. Relative adhesion values between all tests

	Percent Difference		
Bar No.	**Hot Water Test**	**Strip Test**	**X-Cut Test**
U-1	15.2	23.7	8.4
U-6	4.2	8.1	12.3
V-1	14.2	28.0	42.2
V-3	31.5	55.1	86.6
V-14	2.0	7.6	9.6
V-16	6.0	3.3	9.4
W-1	2.8	0.6	2.3
W-3	0.5	3.9	4.5
Y-2	20.0	31.1	11.1
Y-5	19.0	31.6	50.7
Z-1	51.0	10.7	61.7
Z-3	5.3	17.3	22.6
W-17	1.3	0.4	0.9
V-29	62.0	30.6	92.5
W-16	0.1	0.2	0.3
V-28	8.9	10.5	1.6

Table 5.10. Percent difference between results of all tests compared to the average

All test methods were later compared against the results obtained from performing test method Tex 739-I. Figure 5.57 presents results of this peel test plotted with the average adhesion index values obtained from each bar. In only one case (bar V-1) there was a sample that passed the peel test but was inadequate when evaluated by the other procedures. When tested by the strip method, the normalized rating obtained was almost 30% higher than the average of the other methods, so the value can be assumed as an outlier.



Tex 739-I Peel Test Rating (Compared to Hot Water Tests)

Tex 739-I Peel Test Rating (Compared to Strip Tests)

Tex 739-I Peel Test Rating (Compared to X-Cut Tests)

Figure 5.57. Adhesion test results compared to Tex 739-I peel test

Disregarding the value obtained for bar V-1 in the strip method, Table 5.11 presents the limiting rating values for the Tex 739-I peel test with respect to the other three tests. These are values above which bars failed the peel test. Test ratings are presented in their regular rating and normalized form.

	Hot Water Test	**Strip Test**	**X-Cut Test**	**Average**
Test Rating	2.83 (Bar No. U-6)	36.67% (Bar No. U-6)	2.94mm (Bar No. U-6)	N/A
Normalized Rating	0.56	0.48	0.59	0.55

Table 5.11. Limiting values of all adhesion tests with respect to Tex 739-I

As can be noted from the Table and Figures through , there is a reasonably well defined adhesion index above which epoxy coatings fail the peel tests. In the case of this project, the upper bound value of adhesion corresponded to a normalized rating of 0.48 as measured by the strip test. However, more accurate results are obtained by using the hot water and X-cut methods, which provide upper bound values in the range of 0.56-0.59, which are close to the average normalized rating of 0.55. A more elaborate study, involving a larger number of coating applicators could provide a better estimate of the correct value above which adhesion strength is marginal according to Tex 739-I.

5.7 VISUAL OBSERVATIONS FROM SAMPLES IMMERSED IN SODIUM CHLORIDE SOLUTION

In all samples immersed in NaCl solution, total adhesion loss was found in an area surrounding the pre-drilled hole. This area corresponded to the size of the corrosion damaged area under the coating. The general aspect of all samples before removal of the epoxy coating was very similar. The pre-drilled hole was filled with a mix of dark brown and dark gray corrosion products. The oxygen availability in this region aids the continuing corrosion process, helping ferrous oxide turn into ferric oxide. In most cases, the whole depth of the hole was filled with such products. As the epoxy coating was being removed, the appearance of the corrosion product under the coating changed in a radial fashion according to the oxygen availability under the coating. In the vicinity of the hole, dark gray corrosion products were encountered in all samples, sometimes accompanied by small patches of brown rust. Farther away from the hole, corrosion products started became dark to light brown (see Figures 5.58 and 5.60). There was a gradual transition to a point where the surface of the steel remained bright as it was before immersion in the solution and adhesion was evident. Figure 5.59 shows the average adhesion values obtained from performing X-cut tests (at least one inch away from the hole) on all bars after immersion compared with adhesion values before immersion. In all cases there was a slight decrease in adhesion as a result of immersion. This was expected, because the moist environment is a key factor in the loss of adhesion strength of organic epoxy coatings.

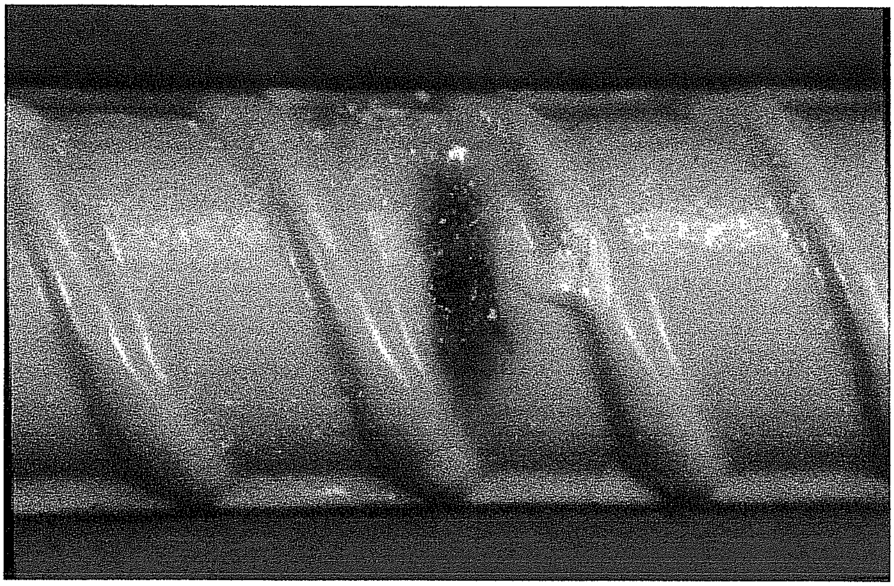


Figure 5.58. General aspect of samples after immersion

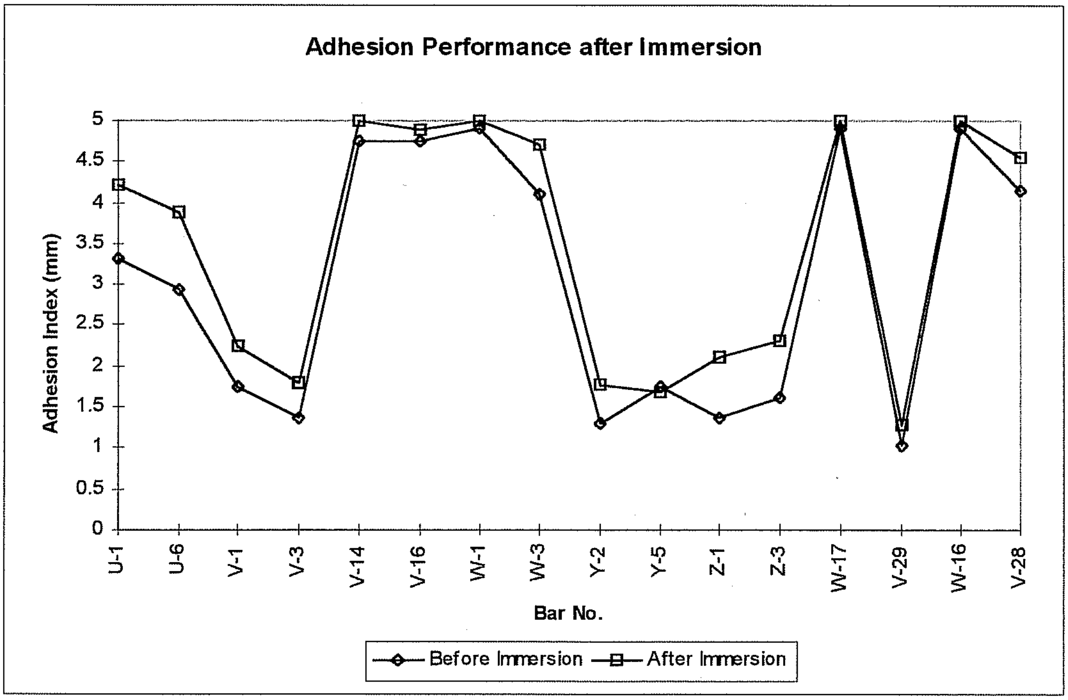


Figure 5.59. Adhesion index before and after immersion for all bars

Another observation was that bars with low adhesion before immersion presented less localized damage after immersion than those with good adhesion (see Figure 5.60). Also, bars with low adhesion tended to show darkening of the steel surface along the sample.

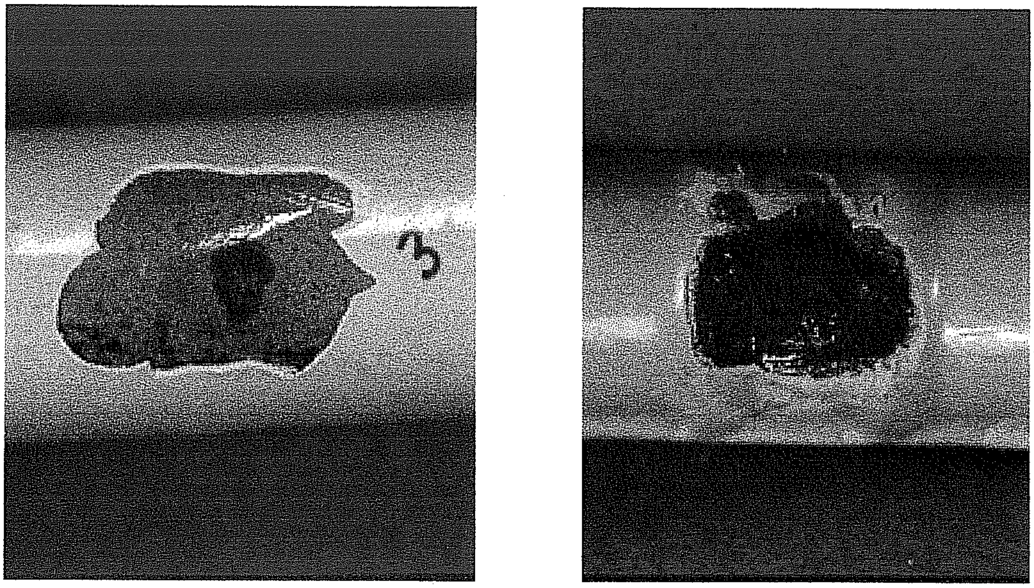


Figure 5.60. Damaged area in samples with good (right) and bad (left) adhesion

In bars with low initial adhesion, the solution is allowed to ingress under the coating and make contact with the whole surface of the steel along the sample. The low oxygen availability in zones away from the hole slow the corrosion process and only a light rust film forms on top of the surface. In bars with high initial adhesion, corrosion damage becomes localized in the vicinity of the pre-drilled hole and the corrosion process only advances as adhesion is gradually lost.

Figure 5.61 presents the size of the damaged area in the vicinity of the whole plotted against the adhesion for all samples. Note that only the size of the most notable damage surrounding the hole was measured. Even though no clear correlation can be found from these values, it is possible to observe that samples with lower adhesion tend to have less concentrated damage around the pre-drilled hole. There is more scatter in the data for samples with better adhesion strength.

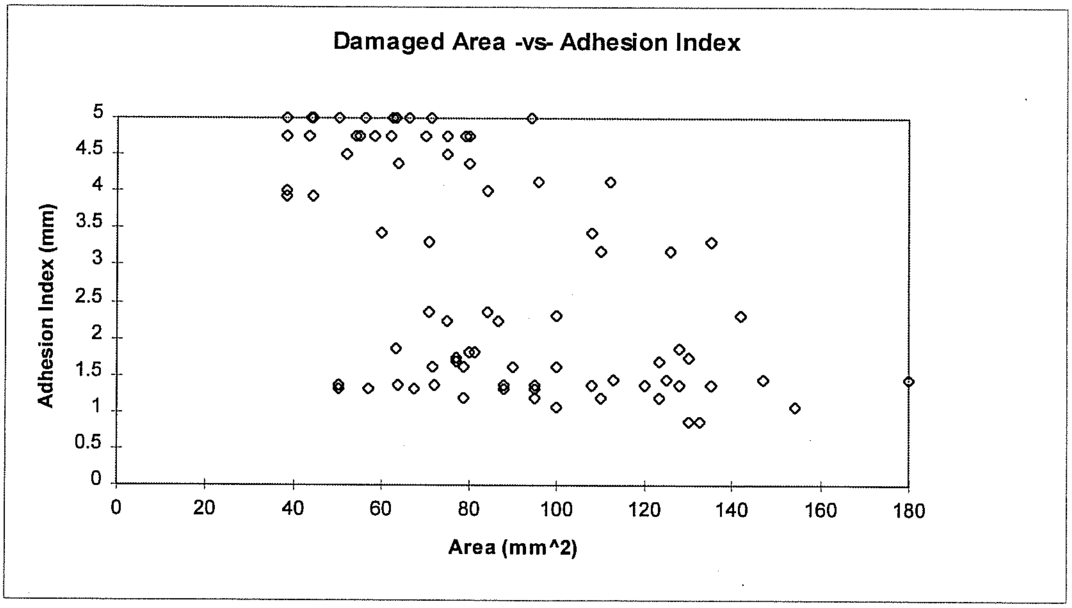


Figure 5.61. Size of damaged area in relation to X-cut adhesion index

CHAPTER 6: SUMMARY AND CONCLUSIONS

6.1 SUMMARY

The role played by adhesion of epoxy coatings in the corrosion protection capacity of steel reinforcement is not very well understood. No extensive research has been devoted to the subject and one of the main causes is the lack of reliable ways to measure adhesion before and after exposure tests have taken place.

The principal objective of this study was to develop a reliable and accurate method of evaluating adhesion strength of epoxy coatings. The development of such a method should satisfy the needs for quality control and qualification purposes, therefore it should be simple and provide consistent results in a relatively short time.

Three different methods for testing coating adhesion were proposed. All of them consist of applying a certain shear stress at the epoxy/steel interface and assigning an adhesion index based on the observed effect. The main differences between the three test methods are the initial condition of the test sample, the definition of the test area, and the applied force. Based on the initial condition of the sample, two different kinds of peel tests were proposed: hot water tests and non-immersion tests.

After the test procedures were developed and refined, sixteen bars from five different coating applicators were tested. The different test methods were performed in three locations on each of the supplied bars and an average adhesion index was determined for each method. The intention was to compare the adhesion values obtained from each test. As different test methods were assigned different rating systems, the results were normalized against the values corresponding to the worst adhesion (in each method) in order to make the comparison. A normalized rating of 1.0 means low adhesion. Adhesion increases and the normalized index approaches zero. The most important observations from these comparisons are that the three methods provided very similar results, with the exception of limited cases in which the standard deviation of adhesion strengths along the bar was very high. Variation in the readings depend on variability inherent to the testing procedures, and variations in the epoxy coating applicator quality control.

All results were compared to the specification used by Tx DOT to rate adhesion in epoxy coated bars. There appears to be a clear limiting value after which adhesion is very low and fails the Tex-739-I test. This value corresponds to a normalized rating between 0.48 and 0.59. More extensive research involving a larger number of coating applicators and coating adhesion strengths is required in order to obtain a more precise value.

Coating thickness measurements were taken at specific points along the bars and compared with the adhesion index obtained from each test at each location. The results show large scatter in the data and no clear correlation between the coating thickness and adhesion strength of the coating.

Intentionally damaged samples from each location of the tested bars were immersed in a NaCl solution for a period of twelve weeks, with alternate wet and dry cycles. Even though this condition is not representative of the service state of an epoxy coated bar embedded in concrete, the intention was to evaluate how coatings with different adhesion strengths perform when exposed to the same aggressive environment. The results are inconclusive, due in part to the short exposure period and the exposure conditions. It was noticed that bars with good adhesion presented very localized damage in the vicinity of the intentional discontinuity, with the adjacent steel surface remaining unaffected. On the other hand, bars with low adhesion exhibited localized damage of smaller dimensions around the intentional discontinuity and general steel surface degradation under the coating. However, no clear correlation was found between adhesion strength and area of damage. Any acceptance criteria developed for adhesion tests should take into account the effect of concentrated or distributed damage along the bars. In this sense, it remains very important to perform long term tests to determine the relative importance of concentrated or distributed corrosion damage.

6.2 CONCLUSIONS

From the experimental procedure performed in this project, the following conclusions were reached:

1. It is possible to measure adhesion strength of epoxy coatings to steel reinforcement by means of simple tests. The only precautions needed to achieve a good measurement are to have a well defined testing area and a reliable method of measuring applied shear stresses. Hot water

and non-immersion peel tests proved to be efficient for adhesion quality control.

2. Normalized results obtained by hot water peel tests are similar to those obtained by non-immersion peel tests. However, due to a different force application and initial conditions they should be interpreted differently. Hot water tests provide an effective method of rapidly simulating the long term effects on coated rebar embedded in concrete. Gradual loss of adhesion has been observed over time for bars embedded in a concrete environment. Non-immersion tests on the other hand, give an indication of adhesion at any time before placement in concrete.
3. Strip tests had the largest scatter of results for a given bar. Also, strip tests tended to provide unconservative readings due to the subjective nature of the rating system. As a consequence, the average normalized adhesion index for each location was lowered. A way to solve this problem would be to change the rating system of strip tests. Measuring the width of the section of epoxy coating removed instead of an overall percentage would provide more accurate results and, if done in more than one location along the strip, could provide an indication of adhesion variability along the strip.
4. No immersion X-cut tests were the easiest to perform and provided reliable results in most situations, except where very thin or non-bendable coatings were used. The results showed less scatter because the procedure involved a direct measurement for establishing the

adhesion index. This test method does not involve hot water immersion, therefore can be completed in shorter times. The testing area was easily defined, so in-plant quality control operations should be simplified.

5. The TxDOT test method Tex-739-I peel test provides a good estimate of relative adhesion between bars from different applicators, given the same operator performs all the tests. However, the high subjectivity involved in the interpretation of the results makes it less desirable for quality control. Independent operators can use any of the tests presented in this thesis to calibrate the results obtained from Tex 739-I, in order to make it more repeatable. Routine bend tests used for quality control were shown to give little meaningful information regarding adhesion and are useful primarily for a measure of coating flexibility.
6. It was found that when using a normalized rating system, limiting values between poor and good adhesion range from 0.48 to 0.59 depending on the test procedure used. In this normalized rating system, a value of 1.0 indicates poor adhesion, and a value of 0 indicates very good adhesion.
7. Non-bendable or rigid coatings were difficult to evaluate due to their high stiffness. Rigid coatings tended to break when subjected to the shearing action of the knife, as opposed to flexible coatings which were lifted as one piece until their adhesion strength was reached.
8. No correlation was found between coating thickness and adhesion strength of the coating, regardless of the test method used.

6.3 RECOMMENDATIONS FOR FURTHER RESEARCH

More research is needed in order to have a better understanding of the effect of adhesion strength in corrosion protection capacity of epoxy coated reinforcement. The objective of this project was to provide a means to measure such adhesion strengths. With this valuable tool, industry quality control and in-depth adhesion studies can be carried out. Some recommendations that arise from this project are:

1. Implement the use of methods like the ones presented in this thesis to measure coating adhesion in epoxy coated reinforcements. This methods reduce the subjectivity inherent to prior tests and provide very good relative adhesion values between bars from different sources.
2. Carry out research studies to predict the effect of adhesion strength in corrosion protection of epoxy coated reinforcement. Such research is needed to provide a clear relationship between adhesion strength and corrosion of epoxy coated bars. Acceptance criteria may need to be based on the effect of distributed or concentrated damage, and may not rely on adhesion strength alone.
3. Discourage the use of bend tests as the only method of testing epoxy coating adhesion, as proposed by some standards. The combination of bend tests with "peel tests" like the ones used in this project will provide a better evaluation of the quality of epoxy coatings, assuring good coating flexibility and adequate adhesion strength.

4. Promote more detailed investigation of adhesion of non-bendable epoxy coatings. Even though the methods presented in this project will provide a reasonable idea of their adhesion strength, only a very small number of bars were tested. More detailed analysis is encouraged.

APPENDIX A: IMPLEMENTATION OF ADHESION TESTS

From the experience obtained during the development of this research project, it is suggested to use the X-cut no-immersion test as a quality control tool for quality control of epoxy coated reinforcement. This test is recommended due to its relative ease of execution, low scatter of test data and applicability to in-plant testing. It is assumed that epoxy coated bars being tested meet all the necessary thickness and continuity of coating requirements.

To obtain a representative concept of coating adhesion along a bar, it is recommended to perform the test at least in three locations on both sides of the coated bar. Two adhesion readings should be taken on each side of every location to reduce the possibility of outlying results. Adhesion readings should be taken at least one inch away from the ends of the bar and trying to avoid mill marks or sections with excessive damage.

The procedure for obtaining each adhesion reading is the following:

1. Place the sample in a flat surface and, if possible secured with a vise to prevent it from moving. Using a sharp utility knife, make a cut through the epoxy coating in the shape of an "X". For bars smaller than #6, it may be necessary to make the cut in the shape of a "V" to have an adequate testing area. The interior angle of the cut should be in the vicinity of 45°, and it can be later modified to obtain more accurate results. Make sure the cut goes through the entire thickness of the coating. Each X-cut allows two adhesion readings to be performed, one in each flap formed by the cut. This cut is going to define the test area.

2. Place the tip of the testing knife (described in section 4.2) 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. Failure to have direct contact with the steel may cause the blade to cut through the coating layer when force is applied. The knife should be held at an angle of approximately 30° tangent to the curvature of the bar.

3. Apply a 2Kg force to the testing knife while rotating it about its longitudinal axis to create an uplifting effect in the coating, like it was explained in 4.2.2.2. The blade should advance along the bisecting line of the angle formed by the "X" or "V" cut. The test is stopped when the epoxy coating inside the test area breaks and is no longer removed in one triangular piece. When this happens, the adhesive strength of the epoxy coating is greater than the shearing stress applied by the knife, therefore the coating fails. After the coating fails, the force application is stopped and all loose or unbonded material is removed.

4. Measure the width in milimeters of the last section of the epoxy coating flap that was removed before the coating failed. This width is inversely proportional to the adhesive strength of the coating.

5. If the width is difficult to measure, and effective method of obtaining more accurate results is by reducing the interior angle of the "X" or "V" cut and repeating the test. Similarly, if the whole flap is lifted without failure, the interior angle of the flap can be increased in order to obtain a reading. Readings of 5mm or larger are considered to represent poor adhesion, and are all taken as 5mm. The reason for this is because after 5mm, the uplifting force created by the blade is not being transferred effectively to the whole width of the testing area.

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Vita

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