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**DURABILITY OF AN INORGANIC POLYMER CONCRETE COATING**

By

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## ABSTRACT OF THE THESIS

### **DURABILITY OF AN INORGANIC POLYMER CONCRETE COATING**

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The objective of the research program reported in this thesis is to evaluate the durability of an inorganic polymer composite coating exposed to freeze/thaw cycling and wet-dry cycling. Freeze/thaw cycling is performed following ASTM D6944-09 Standard Practice for Resistance of Cured Coatings to Thermal Cycling and wet/dry cycling is performed following guidelines set forth in a thesis written by Ronald Garon at Rutgers University. For both sets of experiments, four coating mixture proportions were evaluated. The variables were: silica/alumina ratio, mixing protocol using high shear and normal shear mixing, curing temperatures of 70 and 120 degrees Fahrenheit and use of nano size constituent materials. The mix with highest silica/alumina ratio was designated as Mix 1 and mixes with lower ratios were designated as Mix 2 and Mix 3. Mix 4 had nano silica particles. Four prisms were used for each variable including control that had no coating.

The performance of the coating was evaluated using adhesion strength measured using: ASTM D7234 Test Method for Pull-Off Strength of Coatings on Concrete Using Portable Adhesion Testers. Tests were performed after every five consecutive cycles of thermal conditioning and six consecutive cycles of wet-dry exposure. Results from the

thermal cycling and wet-dry testing demonstrate that all coating formulations are durable. The minimum adhesion strength was 300 psi even though a relatively weak base concrete surface was chosen for the study. The weak surface was chosen to simulate aged concrete surfaces present in actual field conditions. Due to the inherent nature of the test procedure the variation in test results is high. However, based on the test results, high shear mixer and high temperature curing are not recommended. As expected nano size constituent materials provide better performance.



### **Disclaimer Statement**

The contents of this report reflect the views of the authors who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official views or policies of Rutgers University. This report does not constitute a standard, specification, or regulation.

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## Table of Contents

Abstract of the Thesis .....	ii
Disclaimer Statement .....	iv
Acknowledgements .....	v
Table of Contents .....	vi
List of Tables .....	viii
List of Illustrations .....	ix
Chapter 1 - Introduction	
1.1 - Scope and Objective of This Study .....	1
Chapter 2 – State of the Art	
2.1 - Introduction .....	3
2.2 - Matrix .....	4
2.3 – Organic Matrix .....	4
Epoxies .....	5
Acrylics .....	5
Urethanes .....	6
Polyureas .....	6
2.4 – Inorganic Matrix .....	7
Alumino-Silicates .....	7
2.5 – Composite Coatings .....	8
2.6 – Durability of Engineering Coatings .....	8
2.6.1 – Degradation of Polymeric Materials .....	9
2.6.1.1 – Swelling and Dissolution .....	9
2.6.1.2 – Covalent Bond Rupture .....	9
2.6.2 – Freeze-Thaw Resistance of Engineering Coatings .....	10
2.6.3 – Carbon Fiber Reinforced Polymer Coatings .....	11
2.6.4 – Alumino-Silicate Based Coatings .....	12
2.6.5 – Engineering Coatings Used for Adhesive Purposes .....	13
2.6.6 – Nano-Material Composite Coatings .....	15

Chapter 3 – Testing Methodology	
3.1 - Introduction .....	17
3.2 – Concrete Substrate .....	17
3.3 – Inorganic Polymer Matrix.....	18
3.4 – Durability Testing.....	21
3.5 – Freeze-Thaw Cycling.....	22
3.6 – Wet-Dry Cycling.....	26
3.7 – ASTM Testing of High Performance Coatings .....	29
3.8 – Adhesion Testing .....	30
3.8.1 – Potential Failure Modes .....	30
3.8.2 – Pull-Off Adhesion Apparatus .....	32
3.8.3 – Adhesion Testing Procedure .....	33
Chapter 4 – Durability Under Wet-Dry Testing	
4.1 - Introduction .....	35
4.2 – Experimental Evaluation.....	36
4.3 – Adhesion Test Results.....	38
4.4 – Discussion of Test Results .....	41
4.5 - Summary.....	43
Chapter 5 – Durability Under Freeze-Thaw Testing	
5.1 - Introduction .....	52
5.2 – Experimental Evaluation.....	53
5.3 – Adhesion Test Results.....	55
5.4 – Discussion of Test Results .....	58
5.5 - Summary.....	60
Chapter 6 – Conclusions .....	70
References .....	77

## **List of Tables**

Table 3.1 – Coating Mix Distribution.....	18
Table 3.2 – Coated Specimens Identification .....	19
Table 3.3 – Wetting and Drying Cycling Testing Schedule .....	28-29
Table 6.1 – Summary of Coated Specimens .....	75-76

## **List of Illustrations**

Figure 3.1 – Heating Chamber for Curing Process and Thermal Cycling.....	25
Figure 3.2 – Metal Shelves Inside Heating Chamber with Specimens Curing.....	25
Figure 3.3 – Schematic of Wet Dry Testing .....	27
Figure 3.4 – Primary Failure Modes .....	31
Figure 3.5 – Adhesion Testing Apparatus .....	33
Figure 4.1 – Example of a Concrete Adhesion Failure.....	37
Figure 4.2 – Example of a Mortar Adhesion Failure .....	37
Figure 4.3 – Example of a Coating Adhesion Failure .....	38
Figure 4.4 – Example of an Epoxy Adhesion Failure.....	38
Figure 4.5 –Average Strength of Mix 1.1 (Specimen 459).....	44
Figure 4.6 –Average Strength of Mix 1.2 (Specimen 426).....	44
Figure 4.7 –Average Strength of Mix 1.3 (Specimen 408).....	45
Figure 4.8 –Average Strength of Mix 1.4 (Specimen 402).....	45
Figure 4.9 –Average Strength of Mix 2.1 (Specimen 425).....	46
Figure 4.10 –Average Strength of Mix 2.2 (Specimen 407).....	46
Figure 4.11 –Average Strength of Mix 2.3 (Specimen 410).....	47
Figure 4.12 –Average Strength of Mix 2.4 (Specimen 432).....	47
Figure 4.13 –Average Strength of Mix 3.1 (Specimen 423).....	48
Figure 4.14 –Average Strength of Mix 3.2 (Specimen 455).....	48
Figure 4.15 –Average Strength of Mix 3.3 (Specimen 422).....	49
Figure 4.16 –Average Strength of Mix 3.4 (Specimen 430).....	49
Figure 4.17 –Average Strength of Mix 4.1 (Specimen 453).....	50

Figure 4.18 –Average Strength of Mix 4.2 (Specimen 456).....	50
Figure 4.19 –Average Strength of Mix 4.3 (Specimen 414).....	51
Figure 4.20 –Average Strength of Mix 4.4 (Specimen 439).....	51
Figure 5.1 – Example of a Concrete Adhesion Failure.....	54
Figure 5.2 – Example of a Coating Adhesion Failure .....	54
Figure 5.3 – Example of a Mortar and Coating Adhesion Failure.....	54
Figure 5.4 – Example of a Concrete Failure With Voids Detected .....	59
Figure 5.5 – Example of a Coating Failure With Voids Detected .....	60
Figure 5.6 – Example of the Interfacial Transition Zone (ITZ).....	60
Figure 5.7 – Average Strength of Mix 1.1 (Specimen 451).....	62
Figure 5.8 – Average Strength of Mix 1.2 (Specimen 446).....	62
Figure 5.9 – Average Strength of Mix 1.3 (Specimen 427).....	63
Figure 5.10 – Average Strength of Mix 1.4 (Specimen 405).....	63
Figure 5.11 – Average Strength of Mix 2.1 (Specimen 449).....	64
Figure 5.12 – Average Strength of Mix 2.2 (Specimen 448).....	64
Figure 5.13 – Average Strength of Mix 2.3 (Specimen 458).....	65
Figure 5.14 – Average Strength of Mix 2.4 (Specimen 457).....	65
Figure 5.15 – Average Strength of Mix 3.1 (Specimen 454).....	66
Figure 5.16 – Average Strength of Mix 3.2 (Specimen 436).....	66
Figure 5.17 – Average Strength of Mix 3.3 (Specimen 437).....	67
Figure 5.18 – Average Strength of Mix 3.4 (Specimen 406).....	67
Figure 5.19 – Average Strength of Mix 4.1 (Specimen 416).....	68
Figure 5.20 – Average Strength of Mix 4.2 (Specimen 411).....	68
Figure 5.21 – Average Strength of Mix 4.3 (Specimen 412).....	69

Figure 5.22 – Average Strength of Mix 4.4 (Specimen 424)..... 69



## **Chapter 1**

### **Introduction**

#### **1.1 Scope and Objective of This Study**

The durability of construction materials is extremely important to civil engineers. Harsh environmental exposures, such as changing weather cycles, are major contributors to the degradation of America's infrastructure. In order to combat this degradation, high performance composite materials have been studied over the last 40 years as a means to repair or prevent structural degradation. High performance surface applied coatings are designed to protect and/or repair concrete and other building materials from cracking and further structural degradation. However, a lack of data and applicable research [1] on the durability of composite materials has hindered their use in industrial applications. New materials must be able to demonstrate the ability to withstand rigorous durability exposure while maintaining their repair or protective properties before they can warrant industry support and be used in comparative life cycle cost analyses.

In a previous study [2], a fiber-reinforced inorganic polymer composite was formulated and evaluated as a protective coating and repair material for transportation structures. The composite matrix used for this research was an inorganic alternative to organic matrices that have been used in the past. This inorganic matrix is comprised of potassium, aluminum, and silicate (a combination of silicon and oxygen). The composite polymer displays a high tolerance to heat, is water-based, and is permeable enough to prevent vapor pressure buildup underneath its surface. The inorganic polymer mixes used

for this study will be tested for durability while being exposed to cycles of thermal conditioning as well as cycles of wetting and drying.

Thermal cycling, designed to replicate exposure to a temperate climate with harsh winters and hot summers, will be conducted according to ASTM D6944-09 Standard Practice for Resistance of Cured Coatings to Thermal Cycling. For this study, a complete thermal cycle will consist of at least 8 hours of freezing at 5°C followed by 8 hours of heating at 95 - 105°F. The specimens will undergo adhesion testing after every five consecutive cycles in order to assess the composite polymer's adhesion strength as affected by the temperature changes.

Wetting and drying cycles, designed to replicate exposure to a rainy climate, will be performed using a custom built chamber with programmable timers designed to automatically switch from wetting to drying. The cycling consists of 3 hours of immersion in water followed by 3 hours of air drying with the help of an industrial fan. Adhesion testing will be performed after every six full cycles of wetting and drying through the 36<sup>th</sup> cycle as well as after 138 cycles.

## **Chapter 2**

### **State of the Art**

#### **2.1 Introduction**

Surface applied composite materials undergo some form of environmental conditioning no matter what climate they are used in. Environmental degradation can be especially problematic to surface applied materials due to the complex nature of the bond between the composite material and the substrate material. In addition, the bond [3] between reinforced polymer coatings and concrete can significantly affect the strength of the composite concrete system. Every composite material has different material properties and therefore would exhibit very different reactions to environmental and adhesion testing. This difference reflects the complex nature of bond stresses that are dependent on the stiffness and thickness of the composite system. Therefore, it is important to determine the bond strength of a specific composite material that is undergoing environmental degradation.

This chapter gives an overview of high performance coatings as they pertain to the protection of concrete and other building materials with a focus on durability. Concrete protective coatings being used today can be categorized as either organic or inorganic. The organic coatings discussed are epoxies, acrylics, urethanes, and polyureas. The inorganic coatings discussed are cement based or alumino-silicate based. Composite fiber reinforced coatings will also be discussed.

This chapter will also discuss the durability of engineering coatings. Since the majority of coatings are polymer based, an overview of the degradation process of polymers will be reviewed. The research topics investigated for this chapter are the freeze-thaw durability of high performance coatings, the durability of carbon fiber reinforced polymer coatings, the durability of alumino-silicate based coatings, the durability of engineering coatings used as adhesives for polymer fabrics or plates, as well as the use of nano-materials to increase the durability of composite coatings.

## **2.2 Matrix**

The matrix defines the characteristics of a particular coating material. For engineering coatings, the two primary matrices are organic and inorganic. Organic coatings are typically carbon polymer based and are ideal for applications that require impermeability. [4] The most widely used organic coatings are epoxies, acrylics, and urethanes, however, more advanced polymer hybrids such as polyureas are also commercially available.

Inorganic coatings in the past have primarily been cement based; however, research has led to entirely new subsets of inorganic coatings including alumino-silicate based high performance coatings. Inorganic coatings are ideal for applications that require the release of vapor pressure from the substrate after application.

## **2.3 Organic Matrix**

Organic matrices have been successfully used for engineering applications for decades. They are typically applied as a two-component system including a resin and a hardener that are mixed together prior to use. [4] Proper surface preparation of the

substrate is crucial in developing sufficient bond strength between the coating and the substrate material. Organic epoxies, acrylics, urethanes, and polyureas will be discussed in this section.

### **Epoxies**

Epoxies are part of a subgroup of polymeric materials called thermosetting polymers. [5] Upon formation, thermosetting polymers turn into permanently hard materials. They display excellent adhesion properties as they remain hard even after heating. Only extreme temperatures cause any noticeable degradation.

Typically, epoxy coatings [6] are used for their resistance to chemical degradation. They are comprised of two components, an epoxy resin and a co-reactant or hardener. Epoxy resins range from moderate to high chemical resistance as well as moderate to low flexibility. Hardeners primarily consist of either an amine or polyamide.

Amine-cured epoxies [7] demonstrate superior chemical resistance compared to polyamide-cured epoxies. Polyamide epoxies, on the other hand, display superior water resistance and bond strength. The main disadvantage of epoxy coatings, however, is its inability to allow vapor transmission from the concrete. This creates gas build up underneath the surface of the coating creating bubbles, blistering, and potential delamination.

### **Acrylic Coatings**

Acrylics are part of a subgroup of polymeric materials called thermoplastics. As opposed to thermosetting polymers, thermoplastics become soft when exposed to high temperatures and become hard when exposed to lower temperatures. These processes are

both reversible and repeatable. Permanent degradation can occur when thermoplastics are exposed to extremely high temperatures.

Acrylics can either be oil based or water based [8], although for industrial applications water based acrylics are more common. They also exhibit good bonding to concrete while maintaining breathability. This prevents vapor buildup between the coating and the concrete. Acrylics are safe and easy to use while reducing the permeability of the concrete.

### **Urethane Coatings**

Polyurethane coatings [7] are typically thicker than epoxy and acrylic coatings and are ideal for engineering applications that require superior abrasion resistance. They exhibit high durability resistance to chemicals as well as UV light exposure.

Polyurethanes are also much more flexible than epoxy coatings which are hard and brittle.

### **Polyurea Coatings**

Polyurea coatings are some of the most physically desirable of all of the organic polymer coatings. They exhibit exceptional environmental characteristics including the ability to release zero volatile organic compounds (VOC) making it ideal for industrial applications including potable water facilities. [9] Polyurea coatings also offer remarkable elongation up to 500% [10] as well as superior flexibility. Polyurea systems are ideal for waterproofing of concrete and steel water tanks.

The main disadvantage of polyurea coating systems is in the application process. Since polyurea coatings dry within seconds of application, only specialty plural

component spray applicators can be used for the majority of commercial polyurea coatings. Plural component sprayers are high-tech airless spray pumps that combine the two components of the coating at the tip of the spray gun and apply the material as a super heated and super pressurized mist to the substrate. This application process requires specialized contractors that have extensive experience using this type of equipment.

## **2.4 Inorganic Matrix**

Inorganic matrices have primarily been cement based coatings and mortars before the development of an alumino-silicate based matrix. This new matrix is ideal for applications that require a smaller particle size than that used in the cement based coatings. [4]

### **Alumino-Silicates**

The emergence of inorganic polymers began in the early 1990s with the classification of a new material called Geopolymers. Research began in the early 1970's following a number of devastating building fires in France including one in a nightclub [11], the Club Cinq-Sep, which killed 146 people. What developed from subsequent research [12] was a set of amorphous three-dimensional semi-crystalline structures that were resistant to igniting, burning, or releasing smoke even after prolonged heat exposure. This new material also demonstrated the ability to retain 67% of its original flexural strength after exposure to a simulated large fire. Various inorganic reinforcements have been shown [13] to be compatible with the polymer matrix including SiC fibers.

The original Geopolymers were designed for aircraft structures; however, it has since been modified to be used in the construction industry for repair and protection of building materials. This derivative contains a cementing component consisting of a potassium alumina-silicate, or polysialate silox. Polysialates [14] have the general empirical formula:  $M_n\{-(SiO_2)_z-AlO_2\} \cdot wH_2O$ , where «M» is a cation such as potassium, sodium or calcium, «n» is a degree of polycondensation and «z» is 1, 2, or 3.

## **2.5 Composite Materials**

Typical composite materials consist of two phases, a continuous matrix phase that surrounds a dispersed phase. [5] The matrix phase can be organic or inorganic as discussed previously, whereas, the dispersed phase can be particles or fibers used as reinforcement for the surrounding matrix. In order to determine the properties of the composite material, it is necessary to understand the properties of the two phases that comprise it. The composite materials investigated in this report are under the class of materials termed fiber reinforced polymer composites. This type of composite consists of a dispersed phase of reinforcing fibers and a matrix consisting of a polymer resin, a “high molecular-weight reinforcing plastic.”

## **2.6 Durability of Engineering Coatings**

Engineering coatings designed for outdoor use require superior durability and performance characteristics. This section covers the durability, including the freeze-thaw durability and wet-dry durability, of various engineering coatings. A brief overview of the different forms of degradation that occur in polymeric materials subjected to environmental conditioning will first be discussed.



### **2.6.1 Degradation of Polymeric Materials**

Polymeric materials [5] undergo degradation when exposed to environmental conditions. As opposed to metals which undergo an electrochemical reaction termed corrosion, polymers undergo physical and chemical reactions termed degradation. The primary forms of degradation for polymers are swelling and dissolution when exposed to wetting and covalent bond rupture due to exposure to extreme heat.

#### **2.6.1.1 Swelling and Dissolution**

Swelling and dissolution are forms of degradation that result from polymers undergoing wetting. Swelling occurs when a liquid solute is absorbed by a polymeric material and proceeds to displace the molecules of the polymer. This causes an expansion of the material leading to a decrease in the secondary intermolecular forces within the polymer. This process lowers the glass transition temperature while making the polymeric material softer and more ductile. When the polymeric material is completely soluble, dissolution occurs. In a sense, swelling can be considered a partial dissolution process.

#### **2.6.1.2 Covalent Bond Rupture**

Scission, or the rupture of covalent bonds, is a form of polymeric degradation that occurs when exposed to elevated temperatures or high heat levels. This process results in a separation of molecules at the location of rupture as well as a release of gases which can in turn lead to a decrease in molecular weight. Polymeric materials, which rely on high strength to weight ratios, can be extremely sensitive to changes in molecular weight.

This reduction in molecular weight may adversely affect some physical and chemical properties of the polymer: specifically its strength and resistance to chemical degradation.

### **2.6.2 Freeze-Thaw Resistance of Engineering Coatings**

High performance composite coatings have been researched as a way to protect concrete and other substrates from environmental degradation as well as improve performance of the composite system. Commonly used commercial surface-applied coatings are monofluorophosphate-based (MFP) inorganic and organic inhibitors, which are amine or aminoalcohol (AMA) based [15]. Common corrosion inhibitors are AMA based coatings that work to reduce the permeability of hardened concrete by acting as a barrier for water and more corrosive chemicals. Testing has shown that specimens covered with this coating demonstrate increasing freeze-thaw resistance. This can be attributed to the coating acting as an impermeable membrane for the concrete which in turn prevents water to enter the concrete and freeze once inside. This property, however, also does not allow air vapor or water to exit the concrete once the coating is applied which can cause problems for the coating's adhesive strength.

Similar freeze-thaw testing [16] on the durability of two commercial fiber reinforced polymer materials that can be used as protective coatings or as standalone materials revealed a 20 to 30% decrease in flexural strength after 300 cycles. Testing of the dynamic modulus of elasticity of the composite materials during the cycling resulted in significant variance of the data. The authors concluded that degradation of the specimens was due to expansion of moisture during the freezing cycle, resulting in loss in

strength, rigidity, and toughness as well as damage to the fibers and the bond between the fibers and the matrix.

### **2.6.3 Carbon Fiber Reinforced Polymer Coatings**

High performance composites used as repair materials must demonstrate superior durability in order to withstand the environmental exposure that led to the substrate's degradation. The bond between carbon fiber reinforced polymers (CFRP) and masonry substrates reveal [17] that exposure to freeze-thaw cycling as opposed to wet-dry cycling results in substantially less resistance to shear forces. Moderate degradation occurs after 48 cycles with extreme surface degradation occurring after 96 cycles.

A similar durability [1] study performed on three different commercially available CFRP strips revealed significant degradation of the bond between the strips and the concrete substrates after environmental exposure. Specimens were exposed to 20 cycles of freezing followed by thawing at 100% humidity. The formation of micro-cracks resulting from temperature changes helped create a more brittle failure of the FRP material. Results showed that the carbon fiber reinforced polymer plates bonded to the concrete deteriorated after being exposed to elevated temperatures above 140°F, although this may also be attributed to the degradation of the adhesive epoxy used to attach the plates. It was concluded that the adhesive failures demonstrated only a marginally reduced flexural capacity whereas failures associated with the FRP degradation exhibited a significantly reduced flexural strength. Based on the results of the bond tests, there was little evidence that the environmental conditioning had an adverse affect on the properties of the concrete substrates. Deterioration was often more pronounced on bond properties,

possibly due to the critical nature of the adhesives used for the CFRP plates or resin layer used for the CFRP fabrics.

#### **2.6.4 Alumino-Silicate Based Coatings**

Potassium alumina-silicate polymers have been studied [18] as an alternative to typical organic reinforced polymers. This inorganic polymer was proposed as an alternative to traditional organic polymers that have been used as bonding agents for carbon fiber reinforced polymer (CFRP) wraps. Composite wraps are surface applied sheets made of carbon fibers that can be applied in several layers as a way to increase the flexural strength of a concrete beam. Testing of the reinforced concrete beam showed that the inorganic polymer helped to increase the flexural strength of the beam by as much as 50%. The previously studied organic polymer also helped to increase the flexural strength of the concrete beam by 50%.

However, the difference in the performance of the two polymers appeared in the manner in which each reinforced concrete beam failed. The failure mode of all reinforced concrete beams coated with the inorganic polymer was by rupture of the fabrics and not by failure of the inorganic adhesive. This is in contrast to the results of a previous study which demonstrated a failure mode of the delamination of the CFRP wrap using an organic adhesive. This type of failure mode is considered unacceptable for engineers as it underutilizes the full potential of the composite wraps as well as leads to a brittle failure of the beam. This has indicated that the inorganic polymer provides superior adhesion both to concrete surfaces as well as the planes of the carbon fiber wrap compared to the traditional organic polymer adhesive.

Research [2, 19] performed at Rutgers University has demonstrated the effectiveness of a potassium alumina-silicate matrix as a repair and protective coating for damaged infrastructure. A field study conducted in 2008 was performed on a transportation structure damaged by corrosion from saltwater. Results from the project demonstrated that both inorganic and organic composite polymers could be used for protecting and sealing joints between different construction materials including concrete, steel, aluminum alloy, ceramic tiles, and sheet rock. The coating was also successfully used on concrete surfaces that had been deteriorated by the saltwater exposure.

A study in 2004 investigated the durability of inorganic polymer matrices in both lab experiments and field experiments. Testing of the material under wetting and drying conditions revealed that samples did not degrade even after 100 cycles of exposure. Plain concrete specimens coated with the inorganic polymer demonstrated no decrease in flexural strength after 100 cycles of wetting and drying. Coated concrete samples exposed to scaling conditions experienced only a 3% decrease in flexural strength compared to specimens left unexposed.

#### **2.6.5 Engineering Coatings used for Adhesive Purposes**

High performance composite materials have been developed as a way to adhere FRP materials to concrete or other substrates. Epoxy based adhesives have been shown to decrease in effectiveness after cycles of freeze-thaw exposure. An underwater curing epoxy adhesive [20] used for an FRP jacket designed as a wood pile repair experienced a decrease in shear strength after being exposed to 20 cycles of freezing and thawing. It

was concluded that the exposure to freeze-thaw cycles resulted in a change in the failure mode from predominantly adhesive type to a combination of adhesive and cohesive type.

Testing [21] on an epoxy based adhesive used as a bonding agent for concrete substrates produced similar results. Flexural testing of concrete prisms bonded together with an organic epoxy was performed after undergoing cycles of freezing and thawing. The results showed that the adhesive strength of the epoxy coatings decreased after 40 cycles with decreasing bond strength directly proportional to the number of freeze-thaw cycles. The adhesive strength was also directly related to the thickness of epoxy applied to the concrete. It was determined that when exposing the test samples to high temperatures, the thicker the application of epoxy, the worse the concrete specimen performed in load bearing tests. Between the 40<sup>th</sup> and 60<sup>th</sup> cycles, the flexural strength of the concrete prism was reduced by as much as 49%. Results demonstrated the need for a composite material to act as a bonding agent that could withstand the effects of freeze-thaw cycling.

A repair coating called Aqua-Advanced-FRP [22] was developed to be used for underwater structural repairs in conjunction with an underwater epoxy called aqua primer adhesive (APA). Results from dry testing showed that the adhesive strength of the epoxy nearly tripled the adhesive strength of epoxies currently in use. Testing [23] on the FRP coating bonded to the substrate with the aqua epoxy revealed that the adhesive strength increased as the length of the bond increased up to a certain minimum required bond length. Separate adhesion testing [24] on externally bonded fiber reinforced polymer plates revealed that increasing the width of the plate bonded to the concrete decreased the

shear strength of the composite. Thermal cycling was performed on the polymer in order to determine the coefficient of thermal expansion of the coating. The tests demonstrated that the thermal coefficient of the FRP was smaller than that for concrete or steel indicating that the coating could withstand temperature changes better than the substrate material.

#### **2.6.6 Nano-Material Composite Coatings**

Nano-engineering is a relatively new concept that involves developing products from its basic structure and building that structure up to the final product. In order for a construction material to be classified as nano-engineered it must contain nano-particles that can be controlled during the manufacturing process. In essence, a material that contains particles the size of a nanometer is not a product of nanotechnology unless the amount and location of the particles in the finished product can be controlled by the developer [25].

The majority of engineering coatings currently being used are developed with particles with dimensions on the order of a few micrometers. New research, however, has focused on the possibility of coatings being developed with particles an order of magnitude smaller: as tiny as a few nanometers. Nano-composites, a material that combines nanoparticles with other inorganic polymers such as alumino-silicates, have been shown to produce durable coatings that can withstand high temperature exposure similar to traditional inorganic polymer coatings. These nano-composites demonstrate increased toughness and create a more durable coating surface with improved mechanical

and electrical properties. Nano-particles also have shown the capability of producing self-cleaning as well as self-healing properties in high performance coatings.

Two recent studies [26, 27] have begun to demonstrate the potential of nano-engineered coatings as a durable alternative to micro-polymer coatings by investigating the effect that nano-composites have on the permeability of concrete. Accelerated weathering tests conducted on nano-composites consisting of nano-clay mixed with commercial resins were conducted. The results showed that the addition of the nanoclay particles reduced the permeability of the hardened concrete by as much as 69% when exposed to a sodium chloride spray. It was concluded that the addition of the nano-particles helped to fill the micro-voids on the surface of the hardened concrete more effectively than typical polymer composite coatings.



## **Chapter 3**

### **Testing Methodology**

#### **3.1 Introduction**

This chapter will provide an overview of the research that was performed for this report. It will address all stages of the research testing including the preparation of the concrete substrates, four different inorganic coating mixes, the environmental conditioning process as well as the durability testing procedure. This testing process is further broken down into freeze-thaw testing and wet-dry testing. The procedures and schedule that was followed throughout the research will also be outlined. The chapter will conclude by discussing the evaluation process for the coated specimens.

#### **3.2 Concrete Substrates**

Plain concrete prisms were created as substrates for this project. Each concrete substrate was a 3" x 3" x 8" rectangular prism dimensioned in order to maximize the usable surface area for adhesion testing. The average compressive strength of the concrete was 4700 psi.

Mixing of the concrete prisms was performed in the Rutgers University civil engineering laboratory. The formwork was built as a grid of #2 SPF capable of molding 60 prisms. The concrete was poured into the formwork and consolidated with a mechanical vibrator then troweled to a smooth finish. After 24 hours of curing inside the laboratory under a plastic sheet, they were demolded and moved to the moist curing

chamber. The specimens were moist cured for more than 28 days before they were coated with the inorganic polymer. Of the 60 prisms made, 17 were used for each of the thermal cycling and wetting and drying tests. The following mix design was used to make 60-3" x 3" x 8" prisms as well as three - 3" x 6" cylinders for compression:

W/C ratio for a 4000 psi concrete mix design = 0.464

Summary:

Cement:	17.39%
Water:	8.07%
Sand:	29.5%
Gravel:	45.03%
Check Sum	100%

### 3.3 Inorganic Polymer Matrix

Previous studies identified mix proportions that yielded high flexural stress resistance with low shear stress resistance and low flexural stress resistance with high shear stress resistance. By adjusting the proportions, the mix can be tuned to a specific flexural/shear resistance. The mixes identified for adhesion testing included four of the adjusted combinations as shown:

	<b>Flexural Resistance</b>	<b>Shear Resistance</b>
<b>Mix 1:</b>	High	Low
<b>Mix 2:</b>	Moderate	Moderate
<b>Mix 3:</b>	Low	High
<b>Mix 4:</b>	Same as mix 2 substituting nano materials for micro materials	

**Table 3.1** Coating Mix Distribution

The four coating mixes were formulated for this research project to be applied to a total of 16 concrete prisms. The same formulations were used for the thermal cycling as well as the wetting and drying cycling. Four specimens were used for each individual mix. Two specimens for each mix design were prepared with a high shear mixer, manufactured by Blendtec, while the other two were prepared with an ordinary mixer, manufactured by Ninja. One specimen from each mixing method was cured at 120°F while the other specimen was cured at 70°F. One additional specimen was left without a coating as a control.

The coatings were made by pouring all the wet ingredients into the mixer followed by the dry ingredients. The mix sequence consisted of 60 sec on high power followed by 120 sec of rest followed by another 60 sec of mixing. The coatings mixed using the high shear Blendtec were hot to touch and even produced steam. These mixes had a very short pot life and required repeated mixing with the spatula by hand in order to prevent a skin from forming on the surface. The coatings from the normal shear Ninja were easier to apply and had a longer pot life. Table 3.2 below summarizes the specimens used for this project.

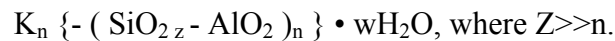
	<b>Mix Number</b>	
	<b>High Shear Mixer</b>	<b>Normal Shear Mixer</b>
<b>70°F Curing</b>	A	B
<b>120°F Curing</b>	C	D

**Table 3.2** Coating Mix Identification

The inorganic matrix used for this project is a polysialate composed of aluminum and silicon dioxide or silica. Wet dry testing [28] performed on this composite has established an optimum ratio of silica to alumina between 18 and 20.

The following is a summary of earlier findings on this material:

- The cementing part of the coating, originally developed for use in aircraft structures, was subsequently modified for use as a coating material. The cementing part is a potassium alumina-silicate, or polysialate silox with the general chemical structure:



- The matrix used in the composite is inorganic, can withstand temperatures up to 1000°C, and is not affected by UV radiation. Fire tests show that the flame-spread index is zero.
- The resin is prepared by mixing a liquid component with silica powder. Fillers and hardening agents can be added to the powder component. The two components are mixed to the consistency of paint.
- The system is water-based and has no toxic substances. No toxins are released during the application or curing.
- The pot life varies from 30-minutes to 3-hours for compositions that cure at room temperature.
- The base coating material is white and hence other color schemes can be easily formulated using pigments. Various color schemes, including concrete and brick color coatings have been successfully developed.

- The system is compatible with brick, concrete, wood, and steel.
- The coating can be applied with minimum surface preparation.
- The permeability of the coating is much less than the permeability of concrete but it allows the release of vapor pressure build up. Therefore, the coating does not delaminate from the parent surface.

A previous study [19] has determined a set of guidelines for the field use of this surface applied inorganic polymer:

- The coating can be applied in the ambient temperature range of 40 to 90° F. At temperatures higher than 80°F, the pot life might be less than 2 hours.
- The coated surface should be protected from direct rain or running water for the first 24 hours.
- The coating should not be subjected to freezing in the first 24 hours.
- The coating can be applied to new or weathered concrete surfaces that have exposed aggregates.
- The surface should be pre-wetted. Loose and oily materials should be removed. Light dust will not reduce the adherence of the coating material.

### **3.4 Durability Testing**

Composite coatings must exhibit tremendous durability when exposed to harsh weather or degrading chemicals. High performance surface applied coatings are commonly exposed to 100% humidity, saltwater, thermal cycling, as well as wetting and drying. In regions of the United States that experience very cold winters as well as very warm summers, a major concern for buildings would be exposure to cycles of freezing,

thawing, and heating as well as cycles of wet weather followed by dry weather. Effective surface applied coatings must be able to withstand the most severe environmental conditions in order to garner industry support.

Determining the durability of high performance composite materials, however, can be extremely challenging. Laboratory tests need to be representative of environmental service conditions and should be performed in a way that minimizes the possibility of errors. The two durability tests conducted for this research were thermal cycling and wet-dry cycling.

### **3.5 Freeze-Thaw Cycling**

Thermal cycling involving exposure to elevated temperatures can also offer challenges. When exposing composite materials to elevated temperatures at or above its glass transition temperature, the properties of the material may change and therefore would not be representative of its field performance unless those temperatures are typical in service. Moderately high temperatures, on the other hand, can cause a post-curing effect on the material which could initially counteract the effects of the freeze-thaw testing.

When performing testing on high performance coatings, it is important to understand the effects of degradation on both the polymer and concrete substrate. Freeze–thaw conditioning affects concrete depending on the permeability and air content of the design mix. Thus, test results may reflect degradation of the substrate in addition to degradation of the coating.

Damage done to composite materials due to freeze-thaw cycles are caused by a number of different factors including matrix hardening, microcracking, and fiber-matrix bond degradation. [29] Studies have shown that the temperature effects on composite structures resulting from freeze-thaw conditions can potentially result in debonding of laminates. Degradation on the concrete substrate can also have an adverse effect on the interface between the concrete and the composite coating. A gap analysis performed in 2003 on composites in engineering stressed that the development of coatings that would serve as protective layers for the bulk composite against external influences including environmental conditions, intended, and accidental damage would be crucial for the future of composite materials.

Thermal stress application using ASTM D6944-09 [30] Standard Practice for Resistance of Cured Coatings to Thermal Cycling is a method to determine the effect of weather changes on the adhesion of the dry film coating. This standard has two methods for testing, one in which the specimens are immersed in a liquid and one in which the freeze/thaw testing is performed completely dry. Test Method A involving liquid immersion, requires four hours of heating at 122°F followed by four hours of tap water immersion at 77°F and finally 16 hours of freezing at 5°F. Test Method B, on the other hand, requires only 8 hours of heating at 122°F followed by 16 hours of freezing at 5°F. Both test methods are flexible in that they allow for the specimens to be held in the freezer for more than 16 hours if needed without adversely affecting the testing.

Thermal cycling was performed based on guidance from ASTM D6944-09 Standard Practice for Resistance of Cured Coatings to Thermal Cycling. This testing

method was followed due to the fact that qualitative results were considered more important than quantitative results. ASTM C666 Standard Test Method for Freeze-Thaw Testing of Concrete was considered to be a more quantitative test method as well as being a more appropriate test of the concrete substrate as opposed to the coating material. ASTM D6944 also allowed us to perform the cycling without having an all encompassing freeze-thaw chamber.

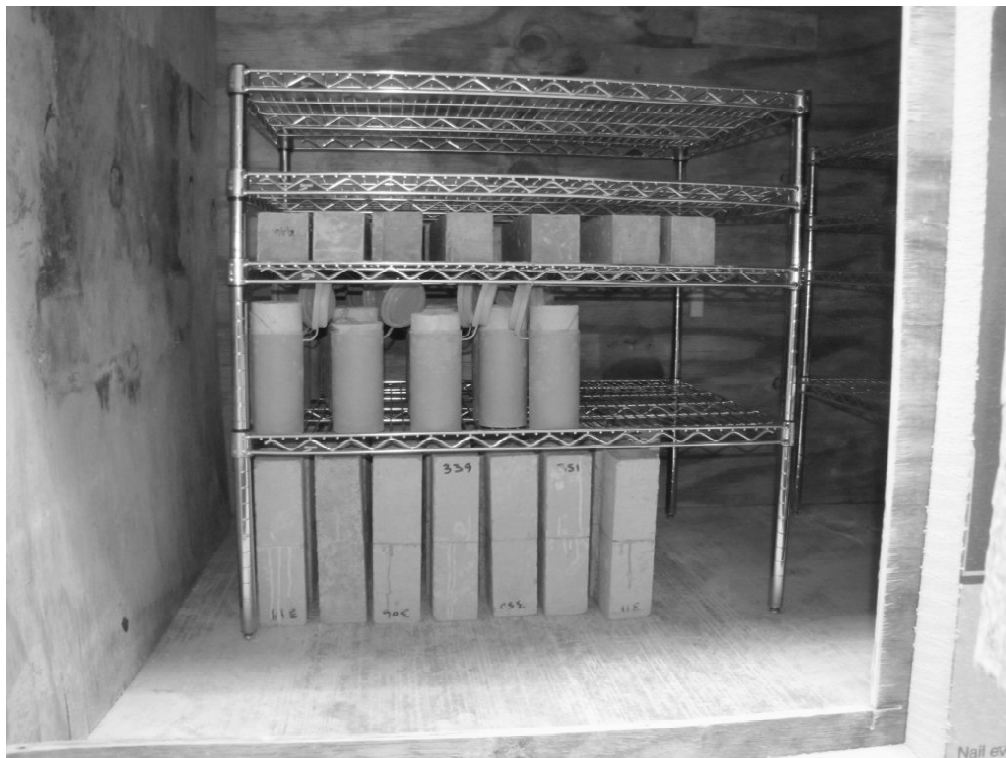
ASTM D6944 Test Method B – Freeze/Thaw was used for this experiment. Two separate chambers, as shown in Figs. 3.1 and 3.2, were used: one chamber was constructed with suitable metal racks and a mechanical heater and insulation in order to maintain the specimens in air at a temperature of 120°F. A separate standard NSF approved chest freezer was used in order to house the specimens in air at a constant temperature of 0°F.

For this research project, a full thermal cycle consists of placing the concrete specimens in the freezer chamber for a minimum of 16 hours followed by moving the specimens to the heating chamber for 8 hours. Five complete cycles constitute a full week after which adhesion testing is performed on all specimens. A minimum of 6 weeks of testing was performed for a total of 30 thermal cycles.





**Fig. 3.1** Heating Chamber for Curing Process and Thermal Cycling

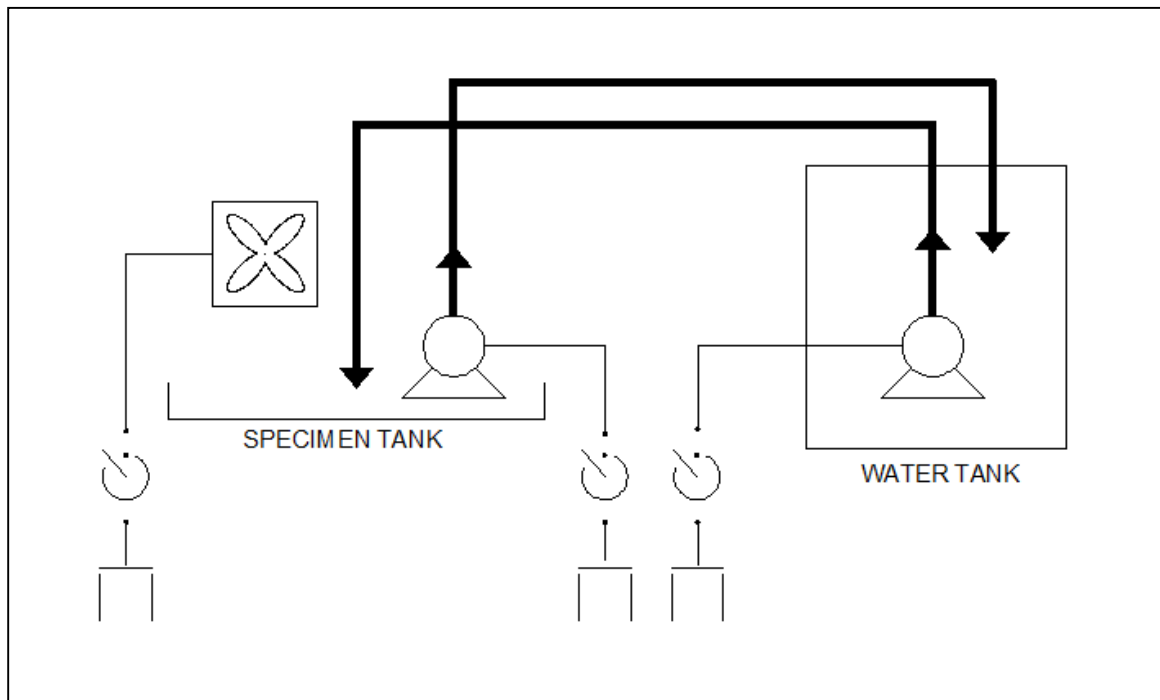


**Fig. 3.2** Metal Shelves Inside Heating Chamber with Specimens Curing

### **3.6 Wet-Dry Cycling**

In order to perform cycles of wetting and drying, an automated testing apparatus was constructed. The apparatus, as shown in Fig. 3.3, consisted of an open air chamber that was used to hold the concrete specimens, a tank filled with water and an industrial fan. The housing chamber for the specimens was lined with plastic sheeting in order to prevent water from escaping. The apparatus was equipped with timers that allowed for the cycling to be completely automated. Two water pumps were installed as part of the apparatus.

The apparatus was designed so that one pump installed in the water holding tank would switch on at a designated time and proceed to pump water from the holding tank into the specimen holding chamber. The float valve on this pump was calibrated to shut off when there was sufficient water in the holding chamber to completely immerse all 17 specimens. After three full hours of water immersion, a separate timer attached to a second pump installed inside the specimen holding chamber would switch on and proceed to pump the water out of the chamber and back into the water holding tank. The float valve on this second pump was also calibrated to shut off when the water was no longer touching any of the 17 specimens.



**Fig. 3.3** Schematic of Wet Dry Testing

An industrial fan located next to the specimen holding chamber was calibrated with a timer to turn on immediately after the second pump had expelled all of the water from the specimen holding chamber. The fan was programmed to operate for a full three hours before shutting off. Table 3.4 outlines the schedule used for the wetting and drying cycling.

Date	Time	Barrel Pump	Tank Pump	Fan	Water	Air	Cycle
Sunday	1:00 PM	ON 1	OFF	OFF	X		BEGIN
	1:10 PM	OFF 1	OFF	OFF	X		1
	4:00 PM	OFF	ON 1	OFF			1
	4:10 PM	OFF	OFF 1	ON 1		X	1
	7:00 PM	ON 2	OFF	OFF 1	X		1 TO 2
	7:10 PM	OFF 2	OFF	OFF	X		2
	10:00 PM	OFF	ON 2	OFF			2
	10:10 PM	OFF	OFF 2	ON 2		X	2
Monday	1:00 AM	ON 3	OFF	OFF 2	X		2 TO 3
	1:10 AM	OFF 3	OFF	OFF	X		3
	4:00 AM	OFF	ON 3	OFF			3
	4:10 AM	OFF	OFF 3	ON 3		X	3
	7:00 AM	ON 4	OFF	OFF 3	X		3 TO 4
	7:10 AM	OFF 4	OFF	OFF	X		4
	10:00 AM	OFF	ON 4	OFF			4
	10:10 AM	OFF	OFF 4	ON 4		X	4
	1:00 PM	ON 5	OFF	OFF 4	X		4 TO 5
	1:10 PM	OFF 5	OFF	OFF	X		5
	4:00 PM	OFF	ON 5	OFF			5
	4:10 PM	OFF	OFF 5	ON 5		X	5
	7:00 PM	ON 6	OFF	OFF 5	X		5 TO 6
	7:10 PM	OFF 6	OFF	OFF	X		6
	10:00 PM	OFF	ON 6	OFF			6
	10:10 PM	OFF	OFF 6	ON 6		X	6
Tuesday	1:00 AM	OFF	OFF	OFF 6			6
	10:00 AM	Attach Dollies					
Wednesday	10:00 AM	Perform Tests					
	1:00 PM	ON 7	OFF	OFF	X		BEGIN
	1:10 PM	OFF 7	OFF	OFF	X		1
	4:00 PM	OFF	ON 7	OFF			1
	4:10 PM	OFF	OFF 7	ON 7		X	1
	7:00 PM	ON 8	OFF	OFF 7	X		1 TO 2
	7:10 PM	OFF 8	OFF	OFF	X		2
	10:00 PM	OFF	ON 8	OFF			2
	10:10 PM	OFF	OFF 8	ON 8		X	2
Thursday	1:00 AM	ON 9	OFF	OFF 8	X		2 TO 3
	1:10 AM	OFF 9	OFF	OFF	X		3
	4:00 AM	OFF	ON 9	OFF			3

	4:10 AM	OFF	OFF 9	ON 9		X	3
	7:00 AM	ON 10	OFF	OFF 9	X		3 TO 4
	7:10 AM	OFF 10	OFF	OFF	X		4
	10:00 AM	OFF	ON 10	OFF			4
	10:10 AM	OFF	OFF 10	ON 10		X	4
	1:00 PM	ON 11	OFF	OFF 10	X		4 TO 5
	1:10 PM	OFF 11	OFF	OFF	X		5
	4:00 PM	OFF	ON 11	OFF			5
	4:10 PM	OFF	OFF 11	ON 11		X	5
	7:00 PM	ON 12	OFF	OFF 11	X		5 TO 6
	7:10 PM	OFF 12	OFF	OFF	X		6
	10:00 PM	OFF	ON 12	OFF			6
	10:10 PM	OFF	OFF 12	ON 12		X	6
Friday	1:00 AM	OFF	OFF	OFF 12			6
	10:00 AM	Attach Dollies					
Sunday	10:00 AM	Perform Tests					
<u>REPEAT TO TOP</u>							

**Table 3.3** Wetting and Drying Testing Schedule

### 3.7 ASTM Testing of High Performance Coatings

Surface applied polymer coatings can be evaluated as protective coatings as well as repair materials. ASTM D6577-06 (2011) [31] Standard Guide for Testing Industrial Protective Coatings governs the appropriate test methods used to evaluate engineering coatings. This guide provides assistance in selecting appropriate tests for evaluating the performance of a coating or coating system on a given substrate exposed to a given type of environment.

An important characteristic of a high performance coating is the adhesive strength of the dry film as it is subjected to environmental degradation. A number of different test methods exist for measuring adhesive properties of protective coatings. The most quantitative method is from ASTM D7234 Test Method for Pull-Off Strength of Coatings

on Concrete Using Portable Adhesion Testers. This test method evaluates the pull-off adhesion strength of a coating on concrete. It measures the greatest perpendicular force (in tension) that a surface area can bear before a dolly is detached. The following section will outline the major principles behind the adhesion testing used for this research.

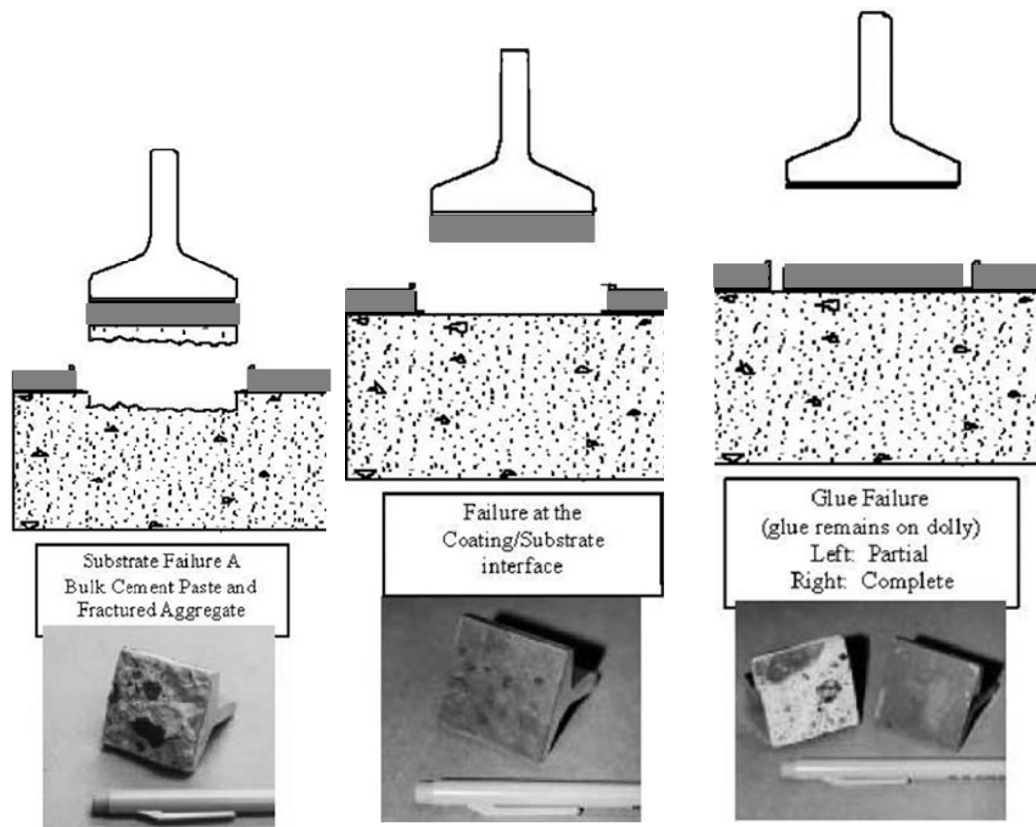
### **3.8 Adhesion Testing**

Bond failure between a polymer material and a concrete substrate can be difficult to categorize. When dealing with a substrate such as concrete with a limited tensile capacity, it is important to note that the bond strength of any composite system is governed by the substrate material itself and, therefore, acts as a maximum value for testing purposes. When testing the pull-off strength of the composite system, it is important to note the failure mechanism as the mode of failure can be considered as important as the adhesion value. These failure mechanisms include a substrate failure or a cohesive failure of the concrete, an adhesive failure occurring between the coating and the concrete substrate, and an adhesive failure between the loading dolly and the manufacturer supplied adhesive.

#### **3.8.1 Potential Failure Modes**

Testing the adhesion properties of an engineered coating undergoing environmental degradation requires a complete understanding of the bond properties of the coating system as well as the effect of environmental conditioning on these properties. Although the interface between the coating and a concrete substrate may not be directly affected by environmental degradation, it is indirectly affected by it. A coating system exhibiting strong bond strength will cause a tensile failure below the surface of

the concrete. This failure mode can be considered the preferred failure mechanism for testing a coating system. The strength value obtained from this failure mode can be attributed to the tensile strength of the concrete substrate at or near its surface. This value can fluctuate depending on the level of substrate failure. Fig. 3.4 below outlines the three primary failure modes within the substrate.



**Fig. 3.4** Primary Failure Modes [32]

A coating exhibiting weak adhesive strength will cause failure in the plane between the coating and the concrete surface. This failure mechanism is not a preferred failure mode for this research especially when adhesion strength values are relatively low. ASTM standards [32] attribute failure from this plane to poor surface preparation of

the concrete substrate prior to applying the coating, a possible contamination of the concrete surface, or even an incompatibility of the coating and concrete substrate.

This failure mode, although categorized as an adhesive failure, may be enhanced by the environmental cycling which could in turn cause a reduction in bond strength. Therefore it can be noted that “the majority of the deterioration of bond capacity associated with environmental conditioning is not in fact bond related but simply reflects the deterioration and failure of the coating material.” However, this implies that a coating material that can withstand the effects of environmental testing will exhibit little to no reduction in adhesive strength. Therefore, a pure coating failure can be attributed to either an initially weak adhesive strength of the material compared to concrete or poor durability characteristics.

An epoxy adhesive failure takes place along the plane between the loading dolly and the manufacturer supplied adhesive. This failure mechanism is also not a preferred failure mode because it provides no information about the adhesive properties of the coating under testing. ASTM D7234 recommends that when this failure mode occurs in more than 20% of the tests, then the results should be disregarded. In this case, it is recommended that more specimens and subsequent tests may need to be performed in order to obtain sufficient results.

### **3.8.2 Pull-Off Adhesion Apparatus**

The adhesion testing apparatus used for this research, as shown in Fig. 3.5, is a PosiTest AT-M (Manual) Pull-Off Adhesion Testing by DeFelsko conforming to ASTM D7234 Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers



[32]. The adhesion testing process requires the use of aluminum dollies that must be adhered to the specimen with an organic epoxy and cured for at least 24 hours to reach sufficient strength. After the dollies are adhered to the substrate, it is recommended to score the coating below the surface of the concrete in order to obtain accurate results. For this research, scoring was performed using a drill press with a diamond tipped core bit just large enough to allow a small amount of space between the dolly and the drill. Coring was performed after adhering the dollies in order to prevent edge damage from microcracks that could affect the results of the testing.



**Fig. 3.5** Adhesion Testing Apparatus

### **3.8.3 Adhesion Testing Procedure**

Following every fifth cycle for the thermal testing the specimens were removed from the testing chamber and placed in an approximately 70°F room in order to reach

room temperature. After every sixth cycle for the wet-dry testing the specimens were left in the wet-dry apparatus. The last cycle of the wetting testing consisted of a drying phase.

Four aluminum dollies were adhered to each of the 17 specimens as per the ASTM recommendations for testing. The aluminum dollies were 14 mm in diameter and scoured with an abrasive sponge prior to adhering in order to enhance their adhesion to the concrete substrate. The dollies were only adhered to the two long sides of the specimens because the long top and bottom sides yielded inconsistent surfaces. After 24 hours, coring was performed around each of the aluminum dollies with a diamond tipped coring drill in order to isolate the dollies from the surrounding concrete substrate and to concentrate stresses to a known cross-sectional area.

Following this, adhesion testing was performed by placing the testing apparatus over the aluminum dollies one by one and manually increasing the pressure at an approximately constant rate of 15-20 psi/sec until failure occurred. The maximum tensile strength of each test was recorded as well as the mode of failure. A minimum of three dollies were tested for each specimen as per ASTM recommendations in order to statistically characterize the results.

## **Chapter 4**

### **Durability Under Wet-Dry Conditions**

#### **4.1 Introduction**

Nearly all building infrastructure, regardless of the climate region, will be exposed to cycles of wet-dry conditioning during its lifespan. Concrete, one of the world's most prevalent building materials, is often directly exposed to these cycles. The durability of concrete is dependent on among other things its permeability and pore size. When concrete has high permeability due to the presence of large pore sizes, water and deicing chemicals can enter the concrete from the surface and eventually corrode the reinforcing steel bars which are critical to the strength of the composite system. Surface cracks from temperature and shrinkage also allow for the intrusion of water under the surface of the concrete. Concrete in marine environments or concrete used in industrial facilities such as wastewater treatment plants, are exposed to water with purifying chemicals that can further contribute to the deterioration of the concrete surface as well as the reinforcing steel bars.

Surface applied concrete protective coatings act to shield the concrete from liquid or moisture intrusion. The ideal coating exhibits the ability to prevent liquid from entering the concrete while also allowing moisture build up from the concrete to escape into the surrounding environment.

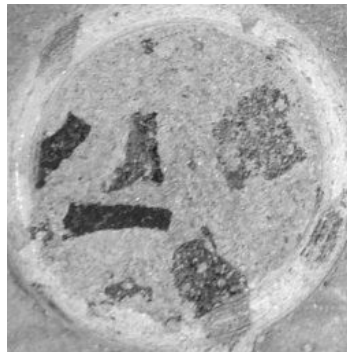
In the following chapter the results of wet-dry testing of the inorganic coating discussed in Chapter 3 will be evaluated and discussed. The goal of this chapter will be to associate the adhesion testing data obtained for each coated sample with the data obtained from the control sample in order to determine the relative durability of the coating systems as well as coating's affect on the durability of the concrete substrate.

## **4.2 Experimental Evaluation**

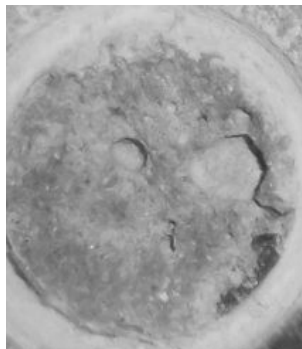
The data from the adhesion testing was evaluated as relative strength values. The adhesive strength values of each coated specimen over the course of the wet-dry cycling were normalized by dividing the average strength for each test by the highest average strength of all the test specimens. These average normalized strength values are being presented as percentages of the highest strength of all of the specimens with 100% being the average normalized strength of the highest strength specimen.

The first evaluation criterion involves a comparison between the average normalized strength values for all 16 specimens. The important parameters considered are the average normalized strength for each specimen as well as change in strength throughout the cycling. The second evaluation criterion was based on the specific mode of failure exhibited by each individual test. As discussed in Chapter 3, the modes of failure that are considered desirable are concrete and partial concrete failures. Examples of concrete related failures are shown below in Figs. 4.1-4.3. Less desirable failure modes take place along the failure plane between the coating and the concrete substrate. An example of a coating related failure is shown below in Fig. 4.4. Another undesirable failure mode is an epoxy type failure that occurs between the aluminum dolly and the

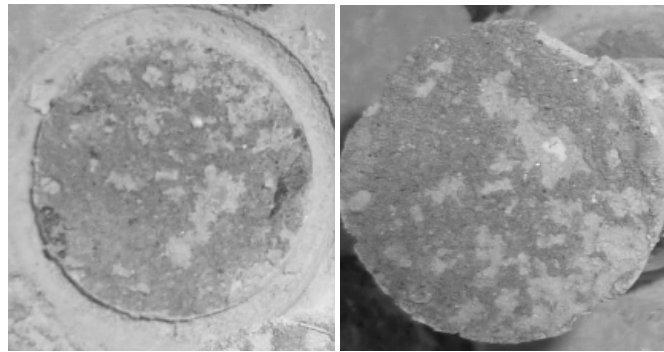
coating system. This failure mode is not indicative of the coating material; therefore, results obtained from epoxy related failures have not been included in the average normalized strength values discussed in these results. An example of an epoxy related failure is shown below in Fig. 4.5. The overall evaluation of each coated specimen will be based on a combination of average normalized adhesive strength as well as mode of failure.



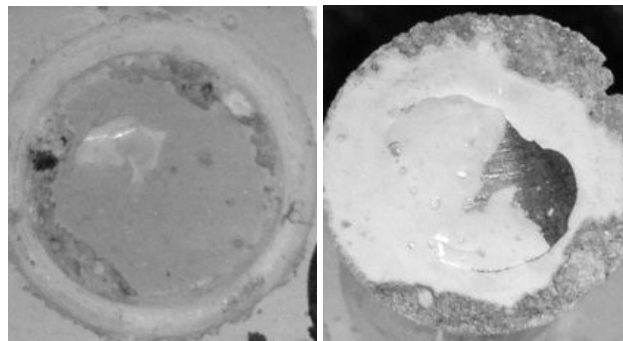
**Fig. 4.1** Example of a Concrete Failure



**Fig. 4.2** Example of a Mortar Failure



**Fig. 4.3** Example of a Coating Failure (Left View is Substrate, Right View is Dolly)



**Fig. 4.4** Example of an Epoxy Failure (Left View is Substrate, Right View is Dolly)

#### **4.3 Adhesion Test Results**

For each specimen, average cyclic values of adhesion strength as well as the standard deviation were computed. Fig. 4.6 – Fig 4.21 display the average strength values evaluated every six cycles throughout the short term testing for all 16 coated samples. The graphs display the coated sample's strength along with a side-by-side comparison of the control specimen.

The control specimen, specimen 413, exhibited a decrease in adhesive strength over the course of the testing. Before the beginning of the cycling, the control demonstrated the highest adhesive strength value at 81%. It then began decreasing as the testing continued. After 30 cycles, the control exhibited its lowest strength values of the cycling at 52%. The control specimen finished the short term testing at 60%. Long term

testing after 138 cycles demonstrated 100% epoxy failures. Overall, the control exhibited a decrease of 12% after the short term testing with a rate of decline of -0.37%, as seen from Fig. 4.5 at the end of the chapter.

Mix 1 was designed for high flexural strength but low shear strength and consisted of specimens 459, 426, 408, and 402. All four specimens exhibited a decrease in adhesive strength throughout the course of the testing. Specimen 426 finished after 138 cycles with 32% strength while experiencing coating failures in 100% of the tests after the 36<sup>th</sup> cycle. Specimen 426 had a decline of 36% at a rate of -0.95%. Specimen 402 had a decrease in strength of about 34% as well as a rate of decline of about -0.8% finishing after 138 cycles with only epoxy failures. Specimen 402 also experienced coating failures in 43% of the tests after the 36<sup>th</sup> cycle. Specimen 408 had a decline of 11% with a rate of decline of about -0.64% finishing after 138 cycles with 34% strength. It also experienced coating failures in 100% of the tests after the 36<sup>th</sup> cycle. Specimen 459 finished experienced a decrease in strength of 23% with a rate of decline of -0.81%. After 138 cycles, specimen 459 demonstrated 100% epoxy failures while experiencing epoxy failures in 70% of the tests.

Mix 2 was designed for moderate flexural and shear strength and consisted of specimens 425, 407, 410, and 432. Specimen 425 had a decrease in strength of about 22% with a rate of decline of -0.8%. Specimen 425 finished after 138 cycles with 41% strength while experiencing coating failures in over 70% of the tests after the 6<sup>th</sup> cycle. Specimen 410 experienced epoxy failures in over 70% of the tests throughout the cycling and was therefore not evaluated for this report. Specimen 407 experienced a decrease in

strength of about 20% with a rate of decline of -0.794%. Specimen 407 finished after 138 cycles with 41% strength while experiencing coating failures in over 70% of the tests throughout the cycling. It declined 14% throughout the testing at a rate of -0.47%. Specimen 432 finished after 138 cycles with 56% strength while experiencing coating failures in over 70% of the tests after the 36<sup>th</sup> cycle. It demonstrated a decrease of 16% throughout the testing at a rate of -0.34%.

Mix 3 was designed for high shear strength and low flexural strength and consisted of specimens 423, 455, 422, and 430. All four specimens exhibited epoxy related failures in at least 88% of the tests after the 6<sup>th</sup> cycle. None of these specimens were evaluated for this report.

Mix 4 was designed for moderate flexural and shear strength using nano-materials and consisted of specimens 453, 456, 414, and 439. None of the specimens in mix 4 experienced a rate of decline over -1%. Specimen 439 exhibited an increase in strength of 31% at a rate of about 0.74% although this can partially be attributed to the low initial strength of only 44%. Specimen 439 finished after 138 cycles with 100% epoxy failures while experiencing 25% coating failures and about 43% epoxy failures throughout the course of the testing. Specimen 414 experienced a decrease in strength of 42% with a rate of decline of -0.91%. Specimen 414 finished after 138 cycles with all epoxy failures while experiencing almost 55% concrete related failures throughout the testing. Specimen 453 had a decrease of 45% with a rate of decline of -0.46%. Specimen 453 finished after 138 cycles with 64% strength while experiencing 100% coating adhesive failures after the 36<sup>th</sup> cycle. Specimen 456 had a decrease in strength of about 25% with a rate of



decline of -0.72%. Specimen 456 finished after 138 cycles with 100% epoxy failures while experiencing 40% coating adhesive failures throughout the course of the testing.

#### **4.4 Discussion of Test Results**

Overall, the wet/dry testing data demonstrated a decrease in normalized adhesive strength following the start of the cycling for nearly all of the coating samples. Specimen 426 experienced a decrease in strength of 36% over the course of the testing. The control specimen, number 413, exhibited a decrease of 12% in strength over the course of the cycling. 27% of the specimens had average normalized adhesive strength values greater than 60% after 36 cycles. Over 55% of the specimens exhibited an average normalized adhesive strength value below 50% after 36 cycles. 44% of the specimens experienced at least 70% epoxy related failures throughout the testing. 19% of the specimens exhibited 100% coating related failures after the 36<sup>th</sup> cycle.

As discussed in Chapter 2, polymeric materials undergo swelling when subjected to cycles of wetting. Swelling causes the polymer to become soft and more ductile, which may be the reason for the high percentage of epoxy adhesive failure modes exhibited throughout the testing. This indicates that the wet/dry cycling also caused a decrease in the adhesive strength of the epoxy resin used for the adhesion testing apparatus causing a subsequent decrease in the adhesive strength of the system. The decrease in adhesive strength of the epoxy resin caused by the wetting process resulted in a large amount of epoxy related failure modes. These types of failure modes are not considered representative of the coating system and therefore were not included in the results presented in this report. The high number of epoxy related failures also made it difficult

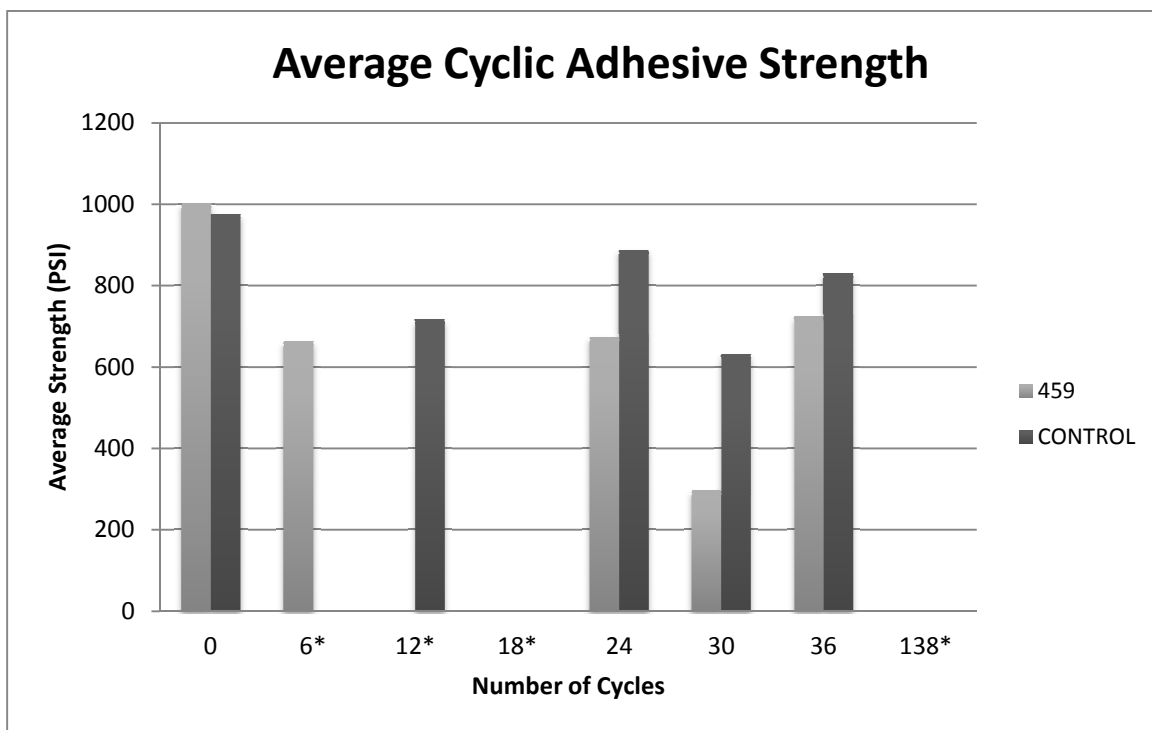
to determine the effect the curing and mixing method had on the performance of the coatings.

The data from the wetting and drying testing was performed using two different methods and were calibrated accordingly once the final data sets were received. Data sets obtained after the 6<sup>th</sup>, 18<sup>th</sup>, 30<sup>th</sup>, and 138<sup>th</sup> cycles were obtained by performing the adhesion testing at a rate of about 10-15 psi/sec. Data sets obtained after the 0<sup>th</sup>, 12<sup>th</sup>, 24<sup>th</sup>, and 36<sup>th</sup> cycles were obtained by performing the adhesion testing at a rate of about 20-25 psi/sec. To account for this discrepancy, the adhesion values from the cycles performed at 10-15 psi/sec were calibrated by increasing the values by 200 psi for the 6<sup>th</sup> cycle, 18<sup>th</sup> cycle, 30<sup>th</sup> cycle, and 138<sup>th</sup> cycle.

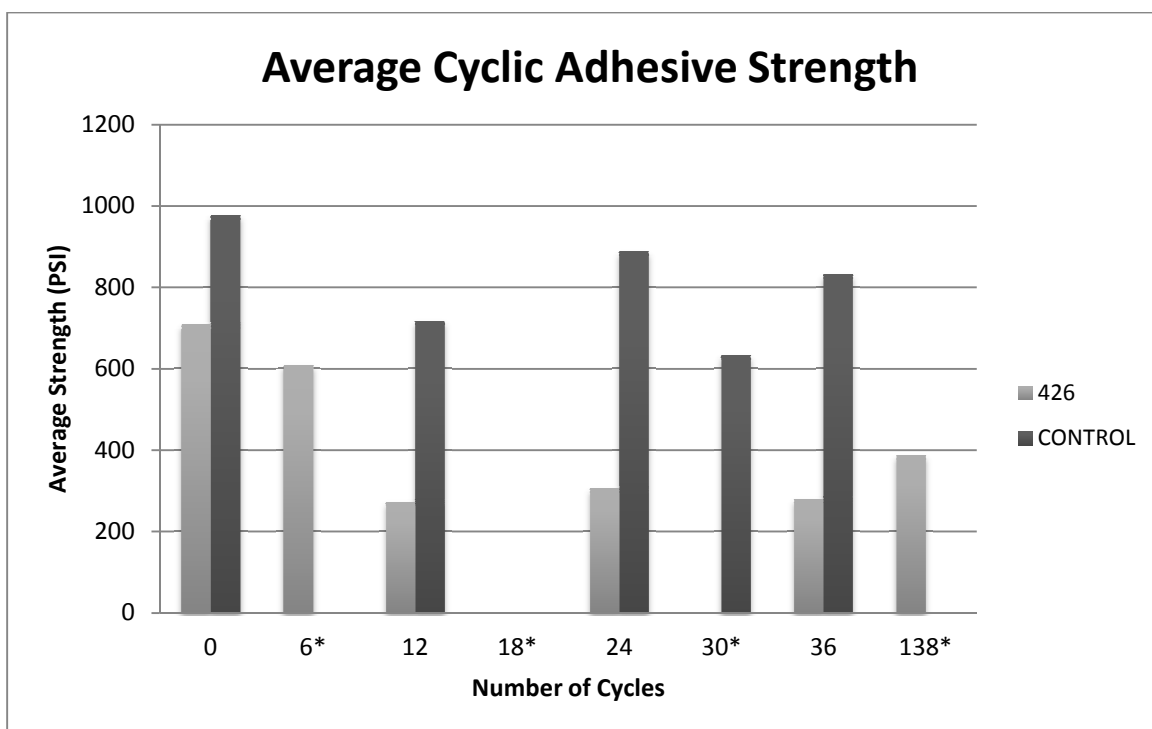
#### **4.5 Summary**

The data displayed in this chapter demonstrate the results of wetting/drying cycling on the adhesive strength of an inorganic concrete coating. The wetting and drying resulted in a reduction in the adhesive strength between the epoxy, the aluminum dolly, and the inorganic coating resulting in epoxy adhesive failures throughout the testing. These epoxy induced strength values were not included in these results. There were also a number of coating or partial coating failures throughout the testing enough to conclude that wetting has a significant effect on the adhesion of the inorganic coatings. None of the specimens in mix 4 experienced a rate of decline in strength over -1%. Specimen 439 finished the short term testing with adhesive strength after 36 cycles of 75% experiencing a rate of increase of 0.74%. Specimen 414 experienced nearly 55% concrete failures throughout. As discussed in Chapter 2, this can be attributed to the use of nano-particles

in Mix 4 which, as previously shown in other studies, helps to significantly reduce the permeability of the composite coating. This decrease in permeability helped prevent the ingress of water resulting in a stronger bond between the coating and the surface of the hardened concrete. The wet/dry testing data was inconclusive with regards to the type of mixer used or the curing temperature for the coatings.

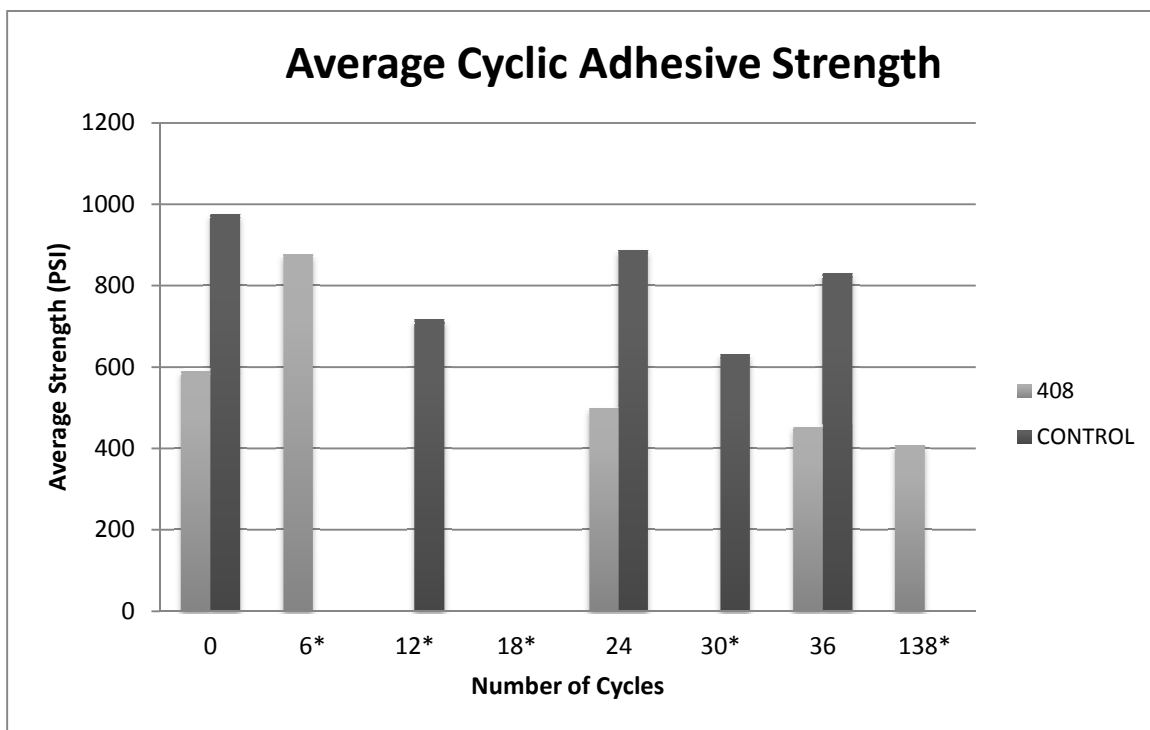


**Fig. 4.5** Average Strength of Mix 1.1 (Specimen 459)\*

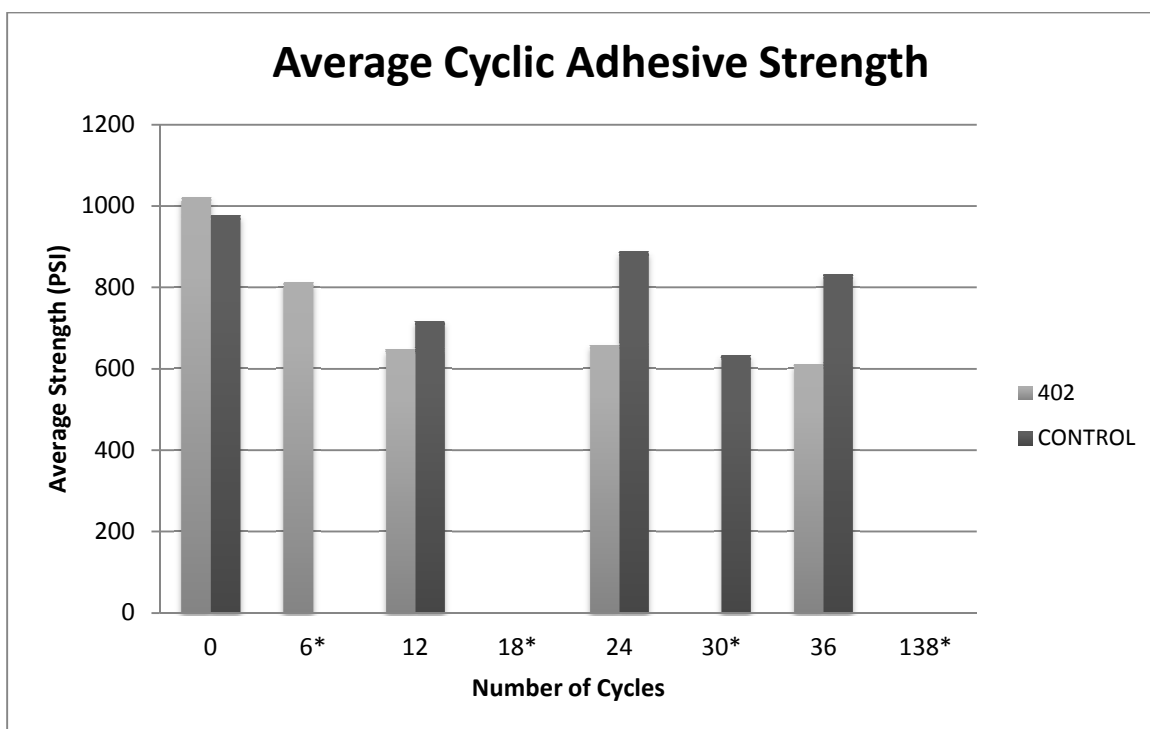


**Fig. 4.6** Average Strength of Mix 1.2 (Specimen 426)

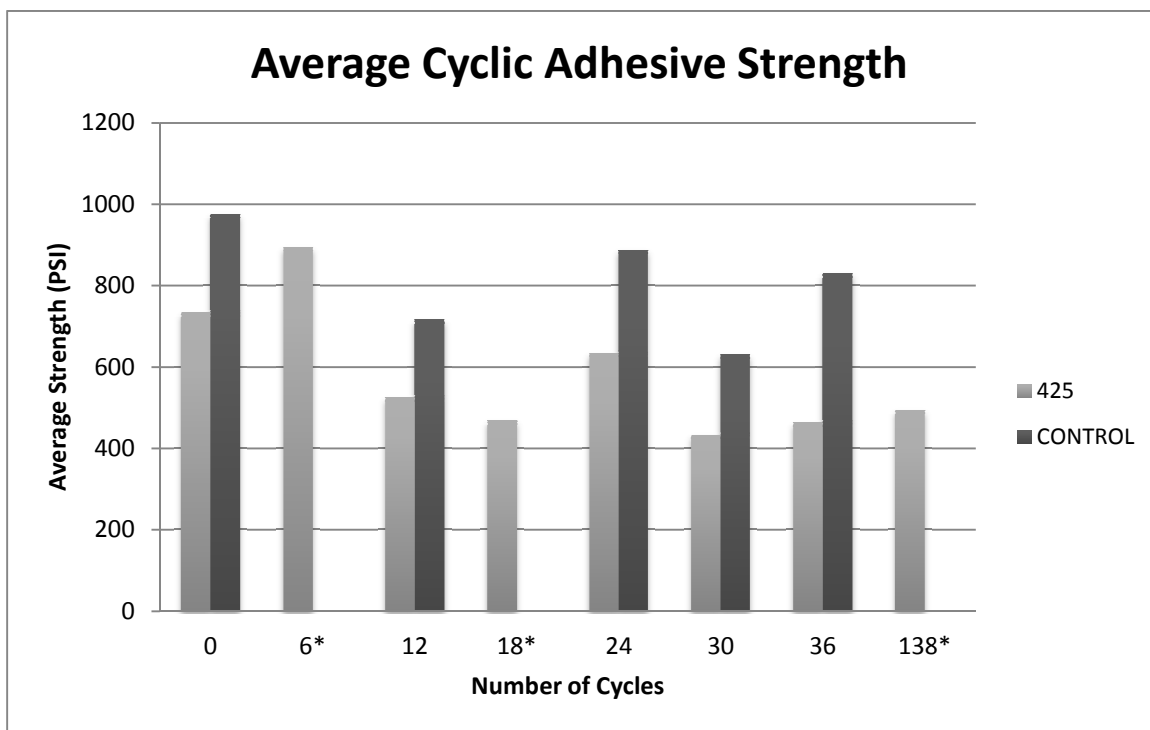
\* Indicates epoxy failures for data set in that cycle (TYP).



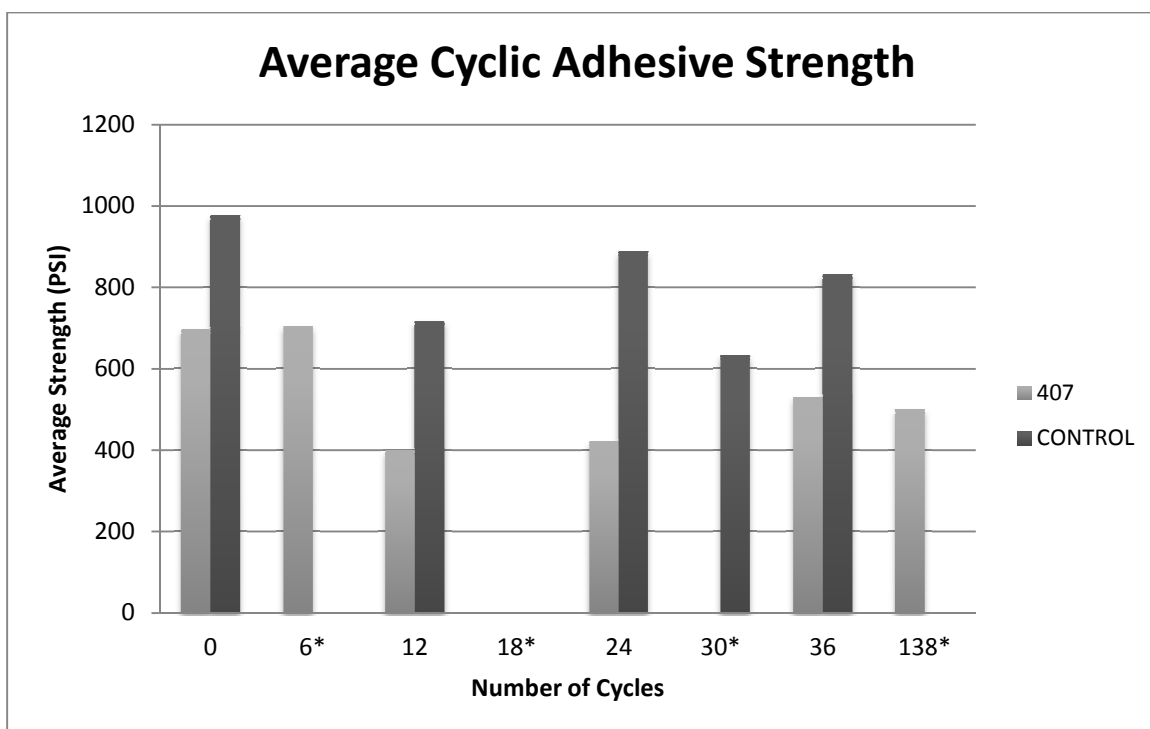
**Fig. 4.7** Average Strength of Mix 1.3 (Specimen 408)



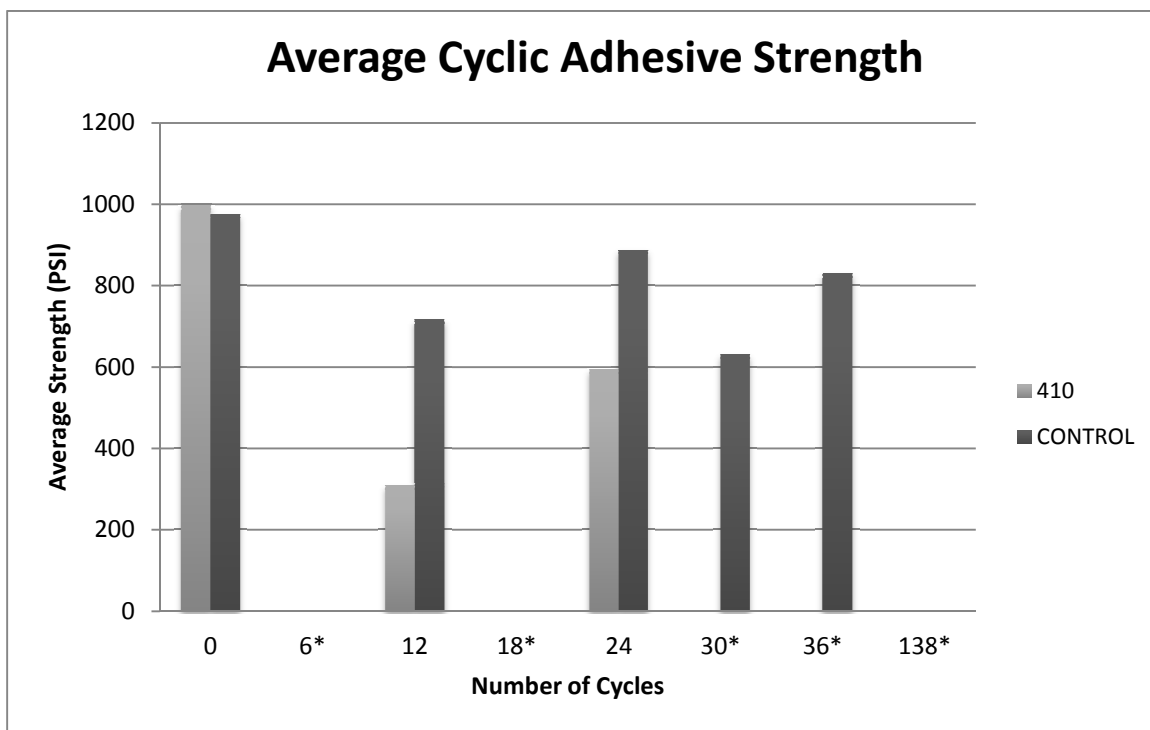
**Fig. 4.8** Average Strength of Mix 1.4 (Specimen 402)



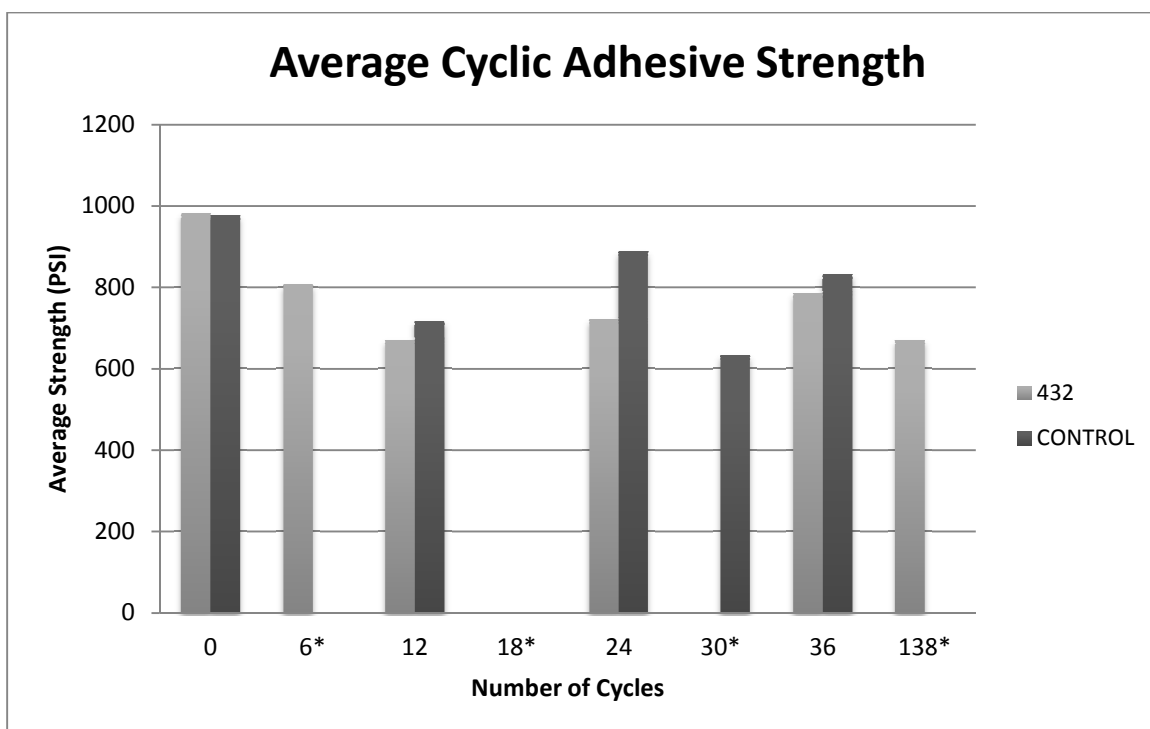
**Fig. 4.9** Average Strength of Mix 2.1 (Specimen 425)



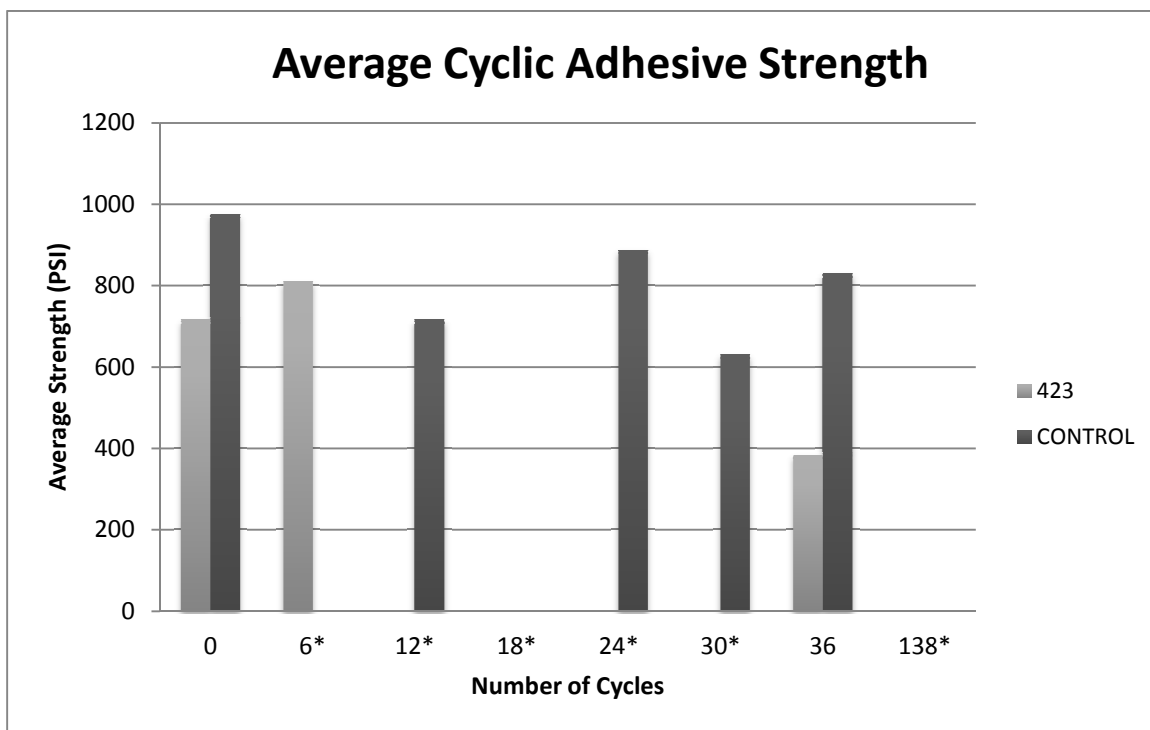
**Fig. 4.10** Average Strength of Mix 2.2 (Specimen 407)



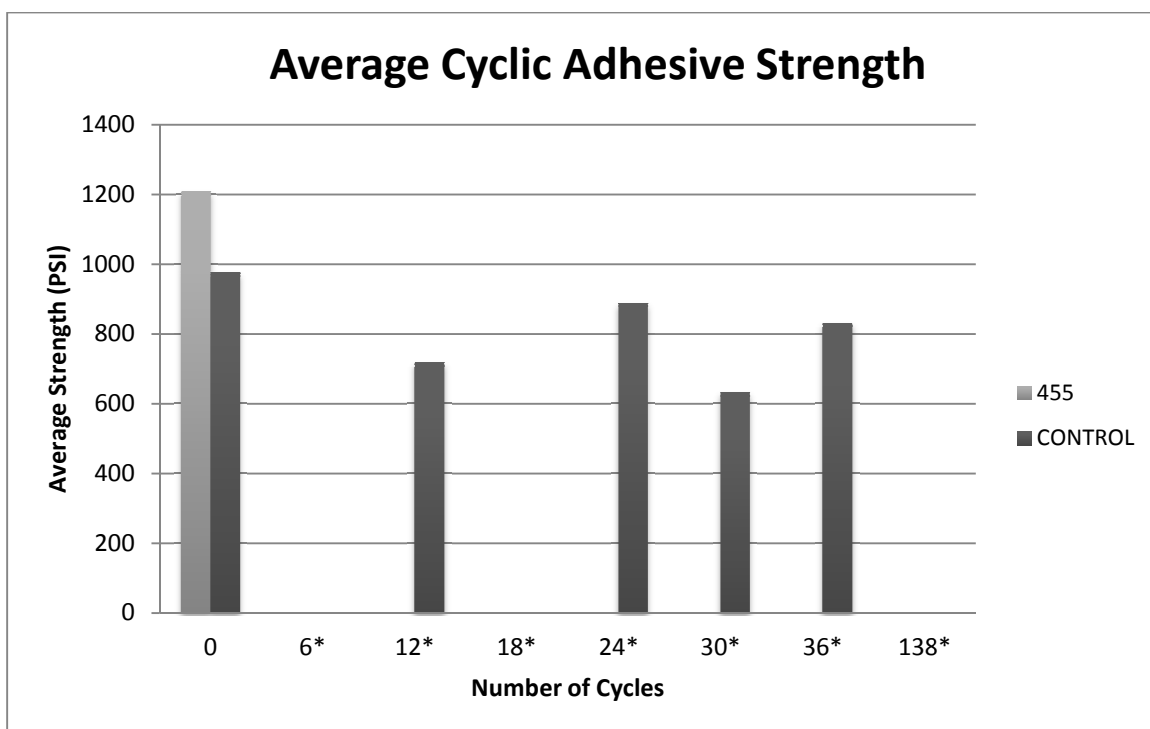
**Fig. 4.11** Average Strength of Mix 2.3 (Specimen 410)



**Fig. 4.12** Average Strength of Mix 2.4 (Specimen 432)

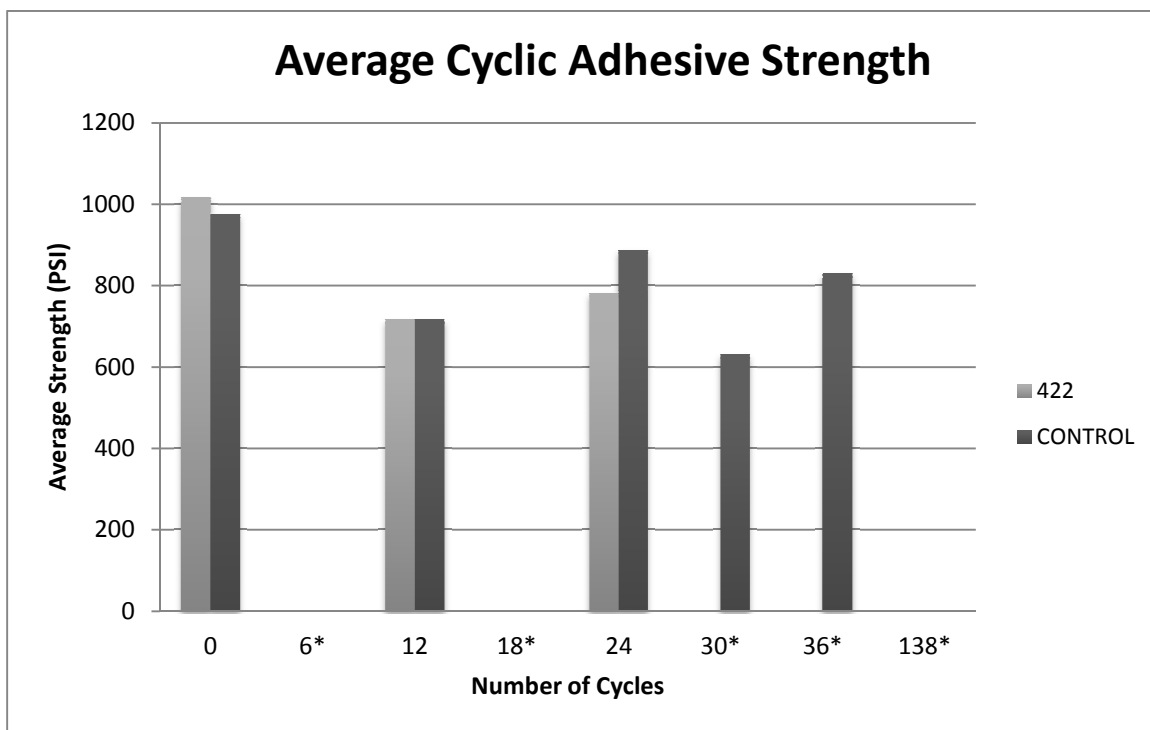


**Fig. 4.13** Average Strength of Mix 3.1 (Specimen 423)

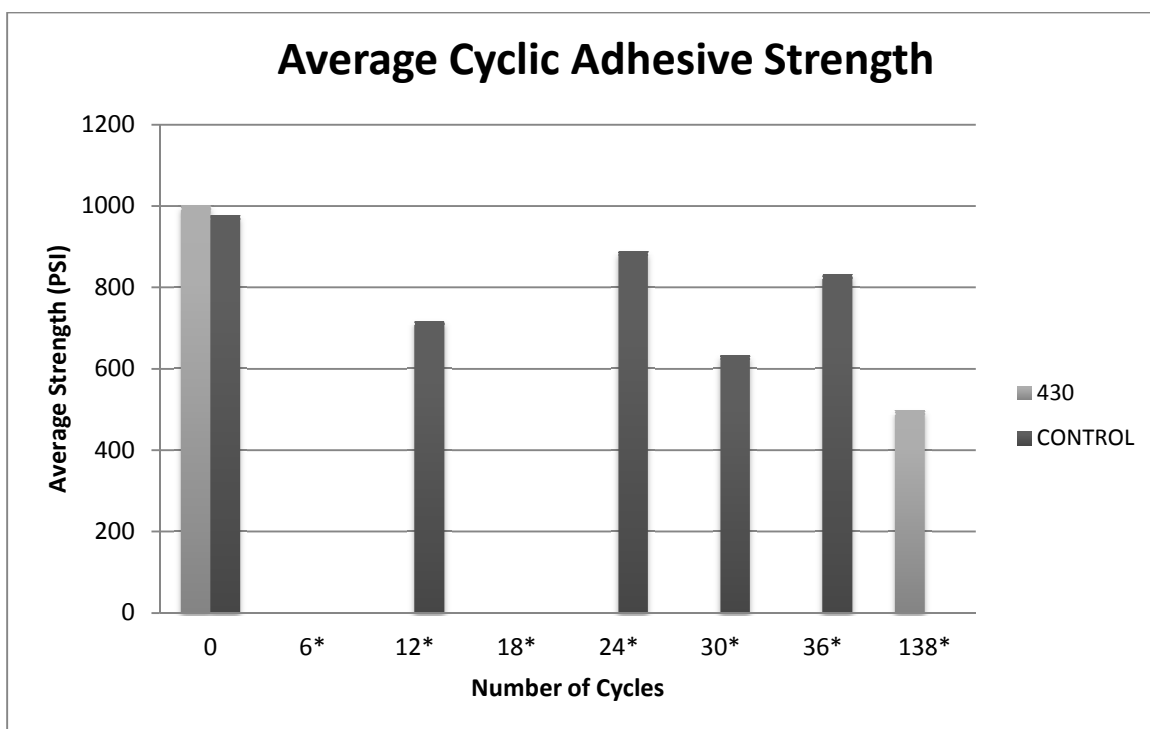


**Fig. 4.14** Average Strength of Mix 3.2 (Specimen 455)

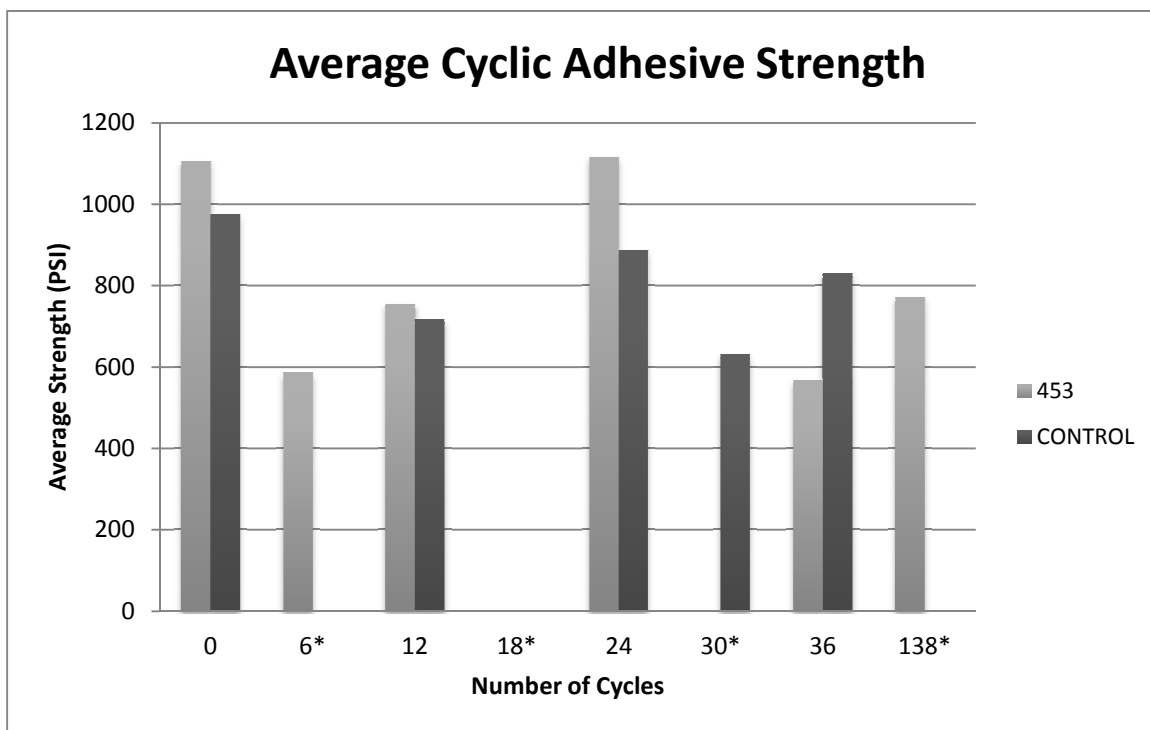




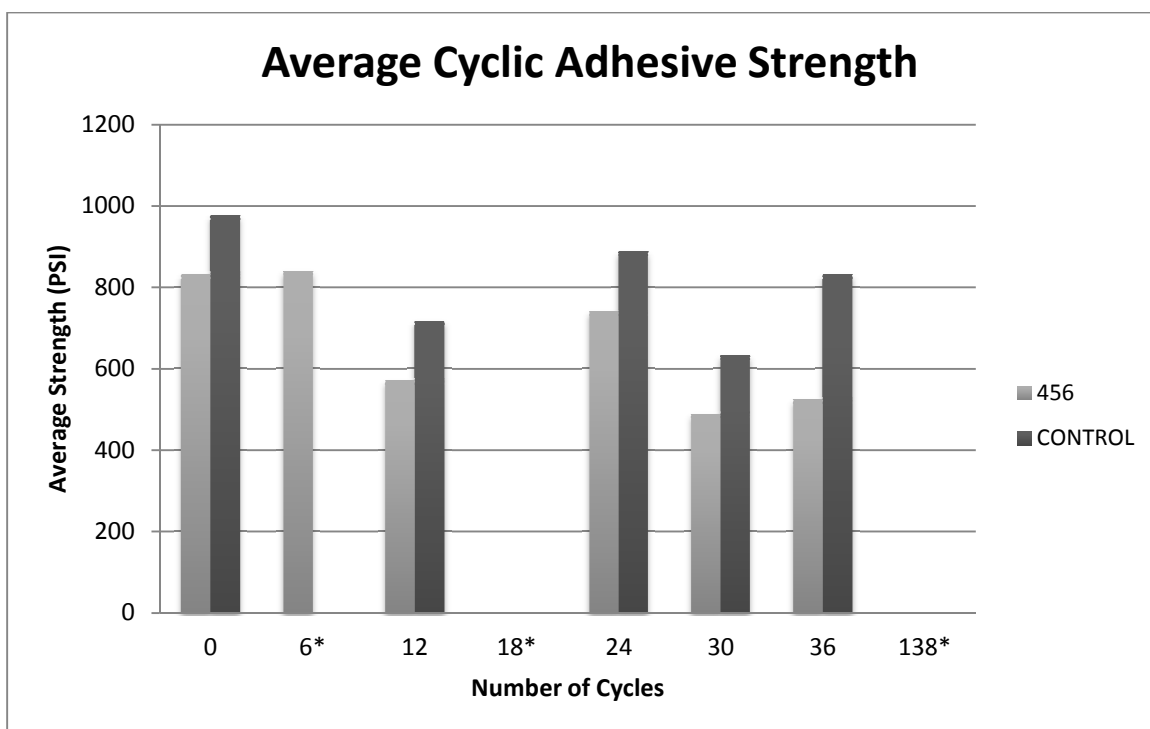
**Fig. 4.15** Average Strength of Mix 3.3 (Specimen 422)



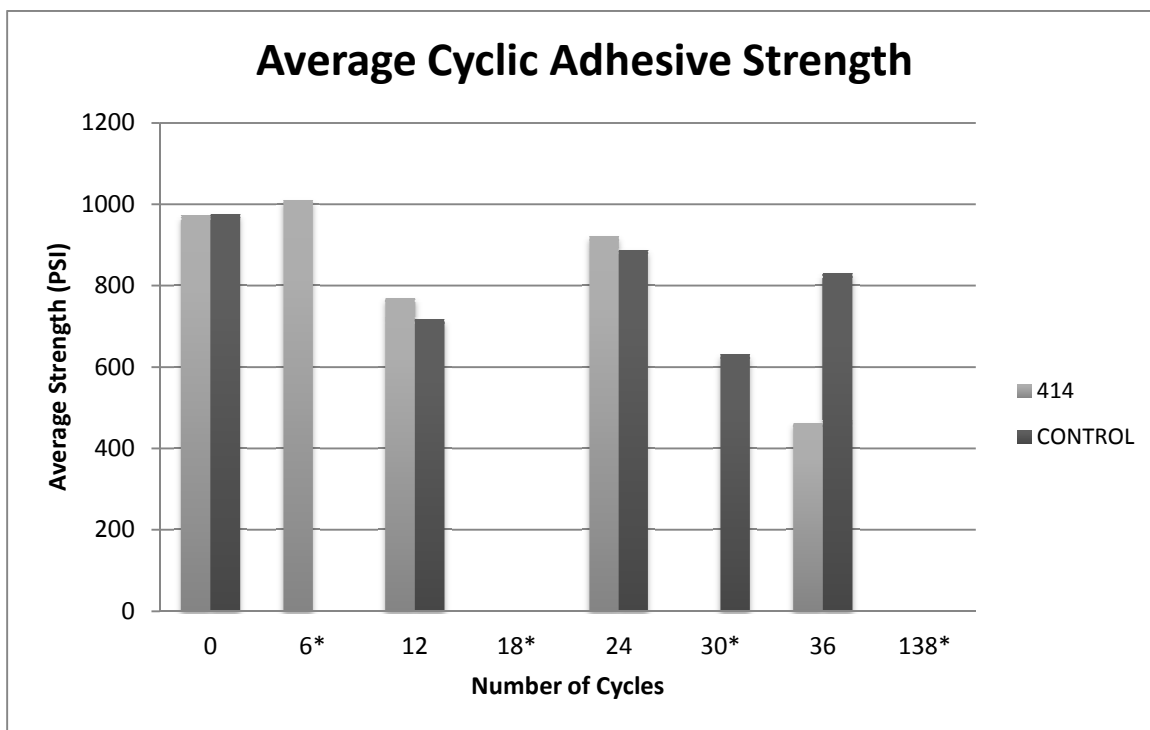
**Fig. 4.16** Average Strength of Mix 3.4 (Specimen 430)



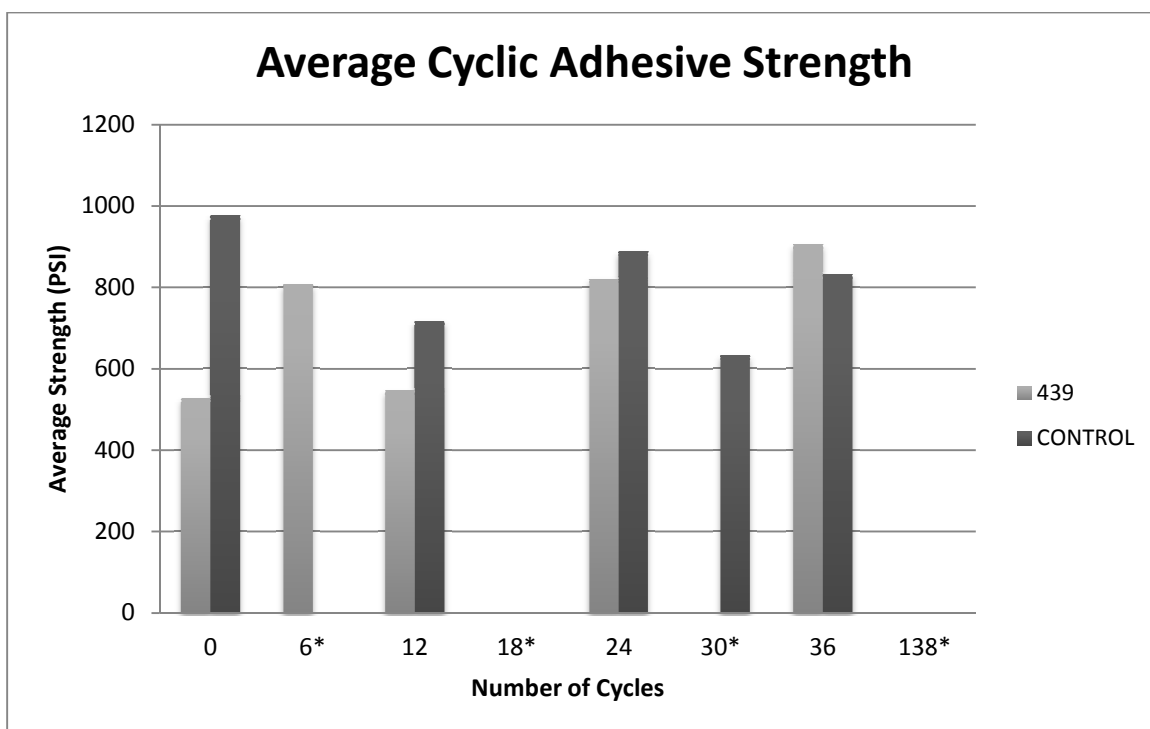
**Fig. 4.17** Average Strength of Mix 4.1 (Specimen 453)



**Fig. 4.18** Average Strength of Mix 4.2 (Specimen 456)



**Fig. 4.19** Average Strength of Mix 4.3 (Specimen 414)



**Fig. 4.20** Average Strength of Mix 4.4 (Specimen 439)

## **Chapter 5**

### **Durability Under Freeze-Thaw Conditions**

#### **5.1 Introduction**

Freeze-thaw conditioning is another cause of environmental degradation similar to wet-dry conditioning. It can occur in combination with wet-dry exposure or independently. Bridge decks, for instance, experience rather severe free-thaw conditioning oftentimes in the presence of water or de-icing chemicals. Concrete is inherently a porous building material, making it susceptible to water and chemical intrusion. When concrete is frozen, water inside the concrete can expand creating micro-cracks that expand into larger macro-cracks. Freeze-thaw cycling can also lead to temperature cracking from the expansion and contraction of the concrete. The goal of a protective coating system for this type of environmental conditioning is to prevent the ingress of water and chemicals by bridging over micro-cracks and even smaller macro-cracks. This means, however, that the coating system itself must be durable enough to withstand the effects of freeze-thaw conditioning without cracking or de-bonding from the substrate.

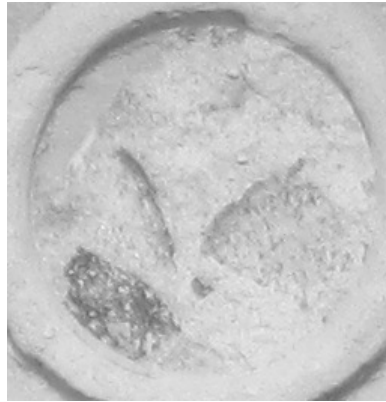
In the following chapter the results of freeze-thaw testing of the inorganic coating discussed in Chapter 3 will be evaluated and discussed. The goal of this chapter will be to associate the adhesion testing data obtained for each coated sample with the data obtained

from the control specimen in order to determine the relative durability of the coating systems as well as its affect on the durability of the concrete substrate.

## **5.2 Experimental Evaluation**

In order to effectively compare the data obtained from each specimen, it was necessary to create a set of parameters by which to compare them. The first parameter was based on the average normalized adhesive strength values of each coated specimen over the course of the thermal cycling. The adhesive strength values of each coated specimen over the course of the cycling were normalized by dividing the average strength for each test by the highest average obtained throughout the testing of all test specimens. These average normalized strength values are being presented as percentages with 100% being the highest average strength throughout the testing.

The second evaluation parameter was based on the specific mode of failure exhibited by each individual tested dolly. As discussed in Chapter 3, there are modes of failure that are considered more desirable than others when dealing with concrete coatings. The modes of failure that are considered desirable are concrete and partial concrete failures. An example of a concrete failure is shown in Fig. 5.1 below. Less desirable failure modes take place along the failure plane between the coating and the concrete substrate. Examples of coating related failures are shown in Figs. 5.2 and 5.3 below. Epoxy related failures, as discussed in Chapter 4, are not indicative of the coating material, and therefore, will not be included in the normalized strength values presented in this report. The overall evaluation of each coated sample will thus be based on a combination of average normalized adhesive strength as well as mode of failure.



**Fig. 5.1** Example of a Concrete Adhesion Failure



**Fig. 5.2** Example of a Coating Adhesion Failure



**Fig. 5.3** Example of a Mortar and Coating Adhesion Failure

### 5.3 Adhesion Test Results

Adhesive strength values for the thermal cycling were evaluated for all 16 concrete specimens. For each specimen, average cyclic values of adhesion strength as well as the standard deviation were computed. Figs. 5.7 - 5.22 depict the average cyclic strength values for all 16 coated specimens. A side-by-side comparison of each sample with the control specimen was also included.

The control specimen, specimen number 404, showed an increase over the course of the thermal cycling. During the first 15 cycles of thermal conditioning, the adhesive strength increased 17% before decreasing as the testing continued. Between 15 cycles and 25 cycles, the adhesive strength decreased about 19%. The last five cycles saw an increase of 10%. Overall, the control specimen increased 8% throughout the cycling with a strength of 79% after 30 cycles, although the overall trend of the data showed a rate of decrease of about -0.078%.

Mix 1 was designed for high flexural strength and low shear strength and consisted of specimens 451, 446, 427, and 405. All four specimens experienced coating failures throughout. Specimen 446 showed a decrease in strength of 11% with a rate of decrease of about -0.57%. Specimen 446 finished after 30 cycles with 61% strength while experiencing 50% coating failures after the 20<sup>th</sup> cycle. Specimen 405 decreased 10% in strength over the course of the testing with a rate of decrease of -0.075%. Specimen 405 had 65% strength after the 30<sup>th</sup> cycle while experiencing 75% coating failures throughout the testing. Specimen 451 displayed an increase of about 18% over the course of the testing although this was attributed to the extremely low initial strength values obtained

in the beginning of the cycling. Specimen 451 increased in strength at a rate of 0.48% while experiencing coating adhesive failures in 83% of the tests after the 5<sup>th</sup> cycle. Specimen 427 exhibited a 10% increase in adhesive strength throughout the course of the testing. Similar to specimen 451, this can be attributed to experiencing low strength values at the start of the testing. Specimen 427 had a rate of increase of 0.131% while experiencing 86% coating failures after the 5<sup>th</sup> cycle.

Mix 2 was designed for moderate flexural strength and moderate shear strength and consisted of specimens 449, 448, 458, and 457. Specimen 458 had a 20% decrease at the end of the cycling with a rate of decline of -0.815 %. Specimen 458 ended the 30<sup>th</sup> cycle with 73% strength while experiencing coating failures in only 31% of the tests throughout. Specimen 457 had a 35% decrease at the end of the testing with a rate of decline of -0.87%. Specimen 457 ended the 30<sup>th</sup> cycle with 51% strength while experiencing 57% coating failures after the 15<sup>th</sup> cycle. Specimen 449 experienced an increase in adhesive strength of 19% throughout the testing although this can be attributed to low strength values at the start of the testing. Specimen 449 finished the testing with 67% strength with a rate of increase of 0.46% while experiencing 91% coating failures throughout. Specimen 448 displayed a 2% increase at the end of the cycling although the trend of the data showed a rate of decrease of -0.31%. Specimen 448 finished the 30<sup>th</sup> cycle with 60% strength while experiencing 55% coating failures throughout the testing.

Mix 3 was designed for low flexural strength and high shear strength and consisted of specimens 454, 436, 437, and 406. All four specimens experienced a large



amount of coating failures throughout the testing. Specimen 454 demonstrated a decrease in strength of 2% at the end of the testing. Specimen 454 had a rate of decrease of about -0.014% finishing after 30 cycles with 68% strength while experiencing 52% coating failures throughout the cycling. Specimen 436 showed a decrease of 25% at the end of the testing with 48% strength after 30 cycles. Specimen 436 showed a rate of decrease of -0.61% while experiencing 50% coating failures after the 20<sup>th</sup> cycle. Specimen 437 had a 16% decrease after 30 cycles with a final strength of 62%. Specimen 437 experienced a rate of decrease of -0.27% while experiencing 63% coating failures until the 25<sup>th</sup> cycle. Specimen 406 had a decrease in strength of 14% finishing after 30 cycles with 59% strength. Specimen 406 demonstrated a rate of decline of -0.60% while experiencing 100% coating failures after the 20<sup>th</sup> cycle.

Mix 4 was designed for moderate flexural and shear strength using nano-materials and consisted of specimens 416, 411, 412, and 424. Specimen 412 experienced an increase in strength of 26% at the end of the cycling while finishing with 86% strength after 30 cycles. Specimen 412 had a rate of increase of 0.79% while demonstrating 100% coating failures after the 30<sup>th</sup> cycle. Specimen 424 demonstrated an increase in strength over the course of the cycling of 12% finishing with 76% strength after 30 cycles. Specimen 424 showed a rate of increase of 0.31% while experiencing less than 20% coating failures throughout the testing. Specimen 416 exhibited a decrease in adhesive strength of 26% throughout the course of the testing finishing with 74% strength after 30 cycles. Specimen 416 had rate of decline of -0.65% while experiencing 87% coating failures after the 25<sup>th</sup> cycle. Specimen 411 displayed a decrease in adhesive strength of

3% throughout the course of the testing ending with 69% strength after 30 cycles.

Specimen 411 showed a rate of decrease of -0.41% while experiencing 75% coating failures throughout the testing.

#### **5.4 Discussion of Test Results**

Performing thermal cycling on cured coatings can be difficult to assess because heating the specimens at 120°C can initially cause post-curing of the inorganic polymer. Many of the coated specimens, as expected, experienced an initial jump in adhesion strength after the first five cycles of the test presumably due to the effects of post-curing on the coating system. Most of the specimens then experienced some form of gradual decline in adhesive strength after this initial spike. The specimens 451, 427, 454, and 449 all exhibited low initial strength values due to the epoxy failure modes observed for these tests before the start of the cycling. These epoxy influenced strength values were not included in the testing results.

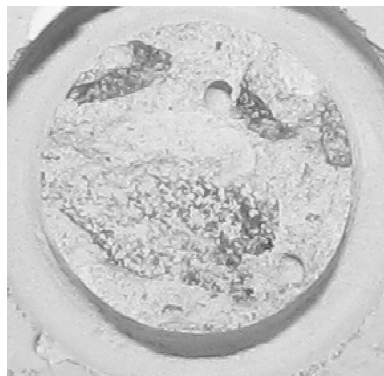
Nearly 69% of the specimens exhibited average normalized adhesive strength values of at least 60% after the 30<sup>th</sup> cycle. Over 31% also exhibited strength values above 70%. However, over 43% of the specimens experienced at least 50% coating related failures throughout the testing. 100% of the specimens in Mix 1 and 75% of the specimens in Mix 3 exhibited at least 50% coating failures after the 20<sup>th</sup> cycle.

The results demonstrated that the mixes made using the high shear mixer resulted in a substantial amount of coating failures. Over 87% of the specimens experienced at least 50% coating failures after the 20<sup>th</sup> cycle as opposed to only 50% of the specimens for the normal shear mixer. The same results were observed for the curing process with

87% of the specimens cured at 120°F experiencing at least 50% coating failures after the 20<sup>th</sup> cycle as opposed to only 50% of the specimens for the 70°F curing. The use of the high shear mixer as well as the 120°F curing appeared to create a more brittle and crack prone coating which in turn resulted in a larger percentage of coating failures.

A number of factors, however, could have lead to the rather inconsistent test data that was gathered from the cycling. Voids along the failure plane, as seen in Figs. 5.4 and 5.5, can lead to a decreased adhesive strength value. A reduction in the surface area of the failure surface will affect its adhesion strength. A failure will occur in the weakest possible plane, and therefore, a failure will most likely occur near the surface where there are voids present.

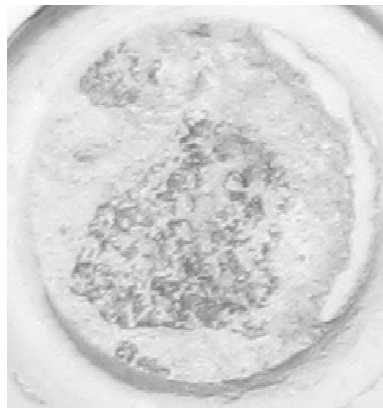
Concrete failure modes may experience lower adhesive strength due to the presence of the interfacial transition zone, or ITZ [33] that can act as a “weak link” in the concrete substrate. The ITZ is a part of the concrete mix that forms around pieces of aggregate and has a higher water to cement ratio than the surrounding bulk paste. This creates a zone of low strength and stiffness that can lead to misleading adhesion testing values. An example of a failure involving the ITZ is shown in Fig. 5.6.



**Fig. 5.4** Example of a Concrete Failure With Voids Detected



**Fig. 5.5** Example of a Coating Failure With Voids Detected



**Fig. 5.6** Example of the Interfacial Transition Zone (ITZ)

## **5.5 Summary**

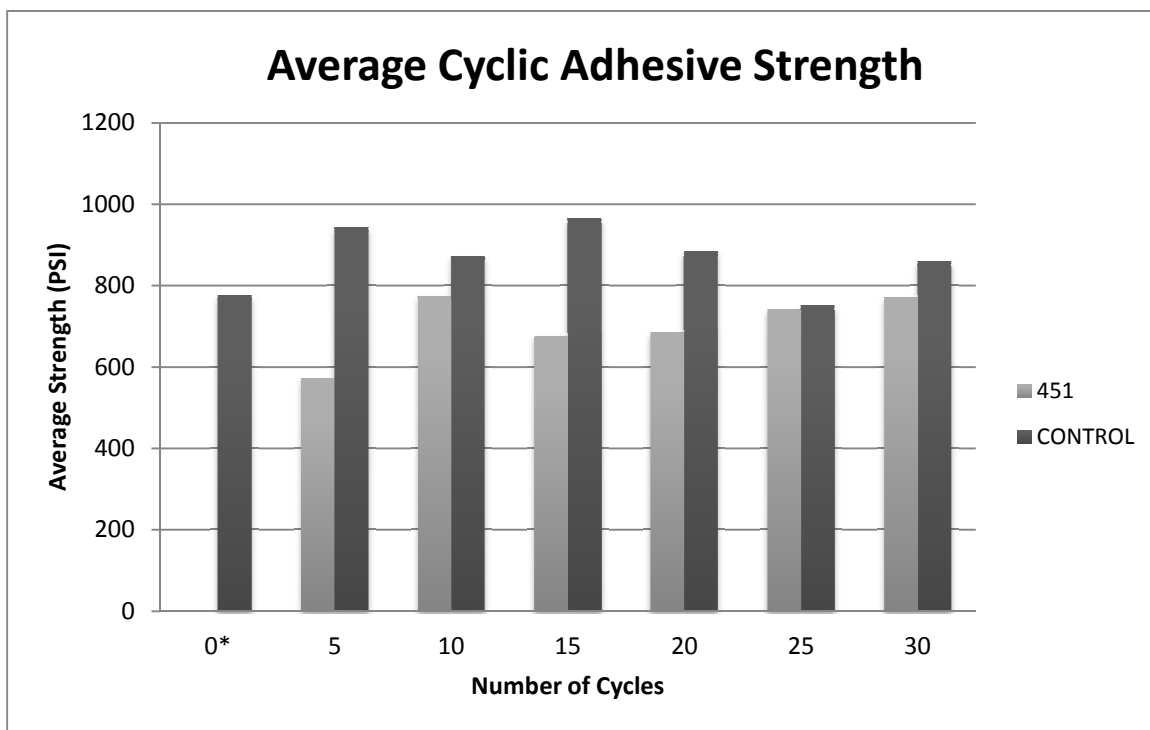
The results presented in this chapter demonstrate the effect of freeze/thaw cycling on the adhesive strength of the inorganic polymer coating on a concrete substrate. With the exception of the initial testing, there were very few epoxy related failures throughout the 30 cycles; however, there were a number of coating adhesive failures indicating that some mixes were less suitable for the environmental exposure.

The results show that Mix 4, which was designed for moderate flexural and shear strength, had three out of the four specimens displaying average normalized adhesive

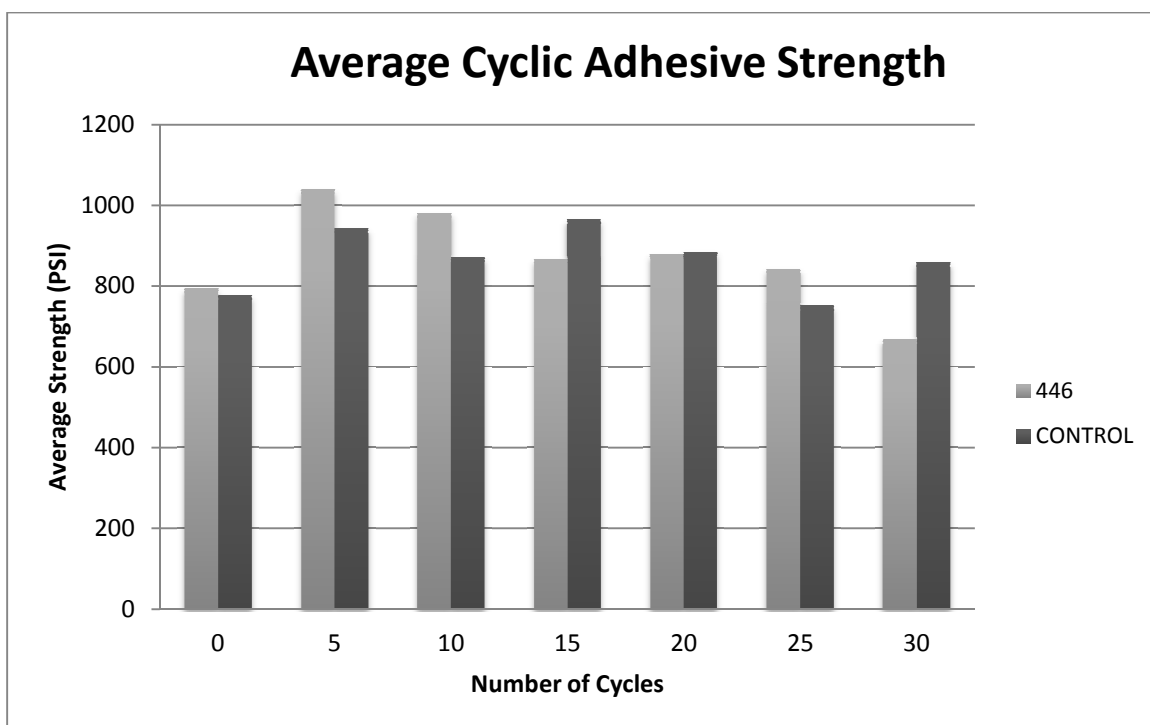
strength values above 70%. Specimen 412 from Mix 4 experienced a rate of increase of 0.79% and coating failures were observed only after the 30<sup>th</sup> cycle.

The use of nano-materials for mix 4 as opposed to the use of micro-materials for the three other mixes was paramount to the durability of the coating. As indicated in Chapter 2, the size of the particle plays a major role in the permeability of the composite material. Nano-particles, which are orders of magnitude smaller in size than micro-materials, create a less permeable barrier to protect the concrete substrate. The size of the particle also helps with the bond strength of the coating since there can be larger number of particles in contact with the concrete substrate which in turn increases the adhesive strength of the composite system.

The use of the normal speed mixer as well as the 70°F curing process resulted in more desirable concrete failure modes than the high speed mixer and 120°F curing process.

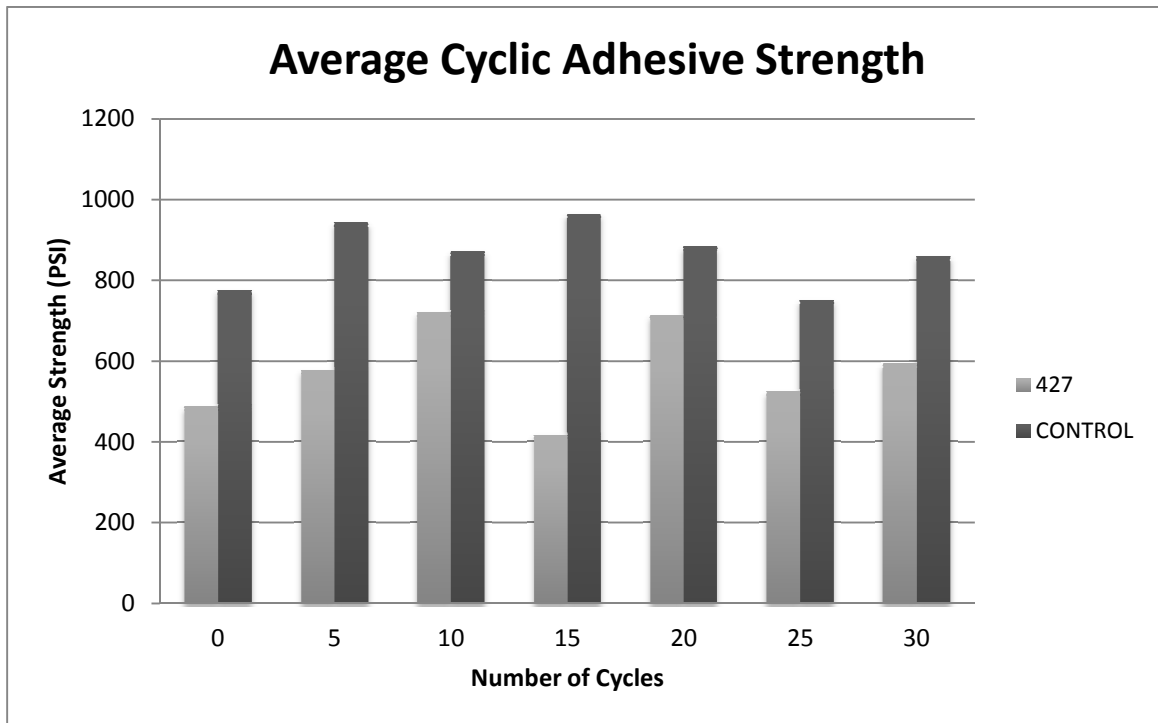


**Fig. 5.7** Average Strength of Mix 1.1 (Specimen 451)\*

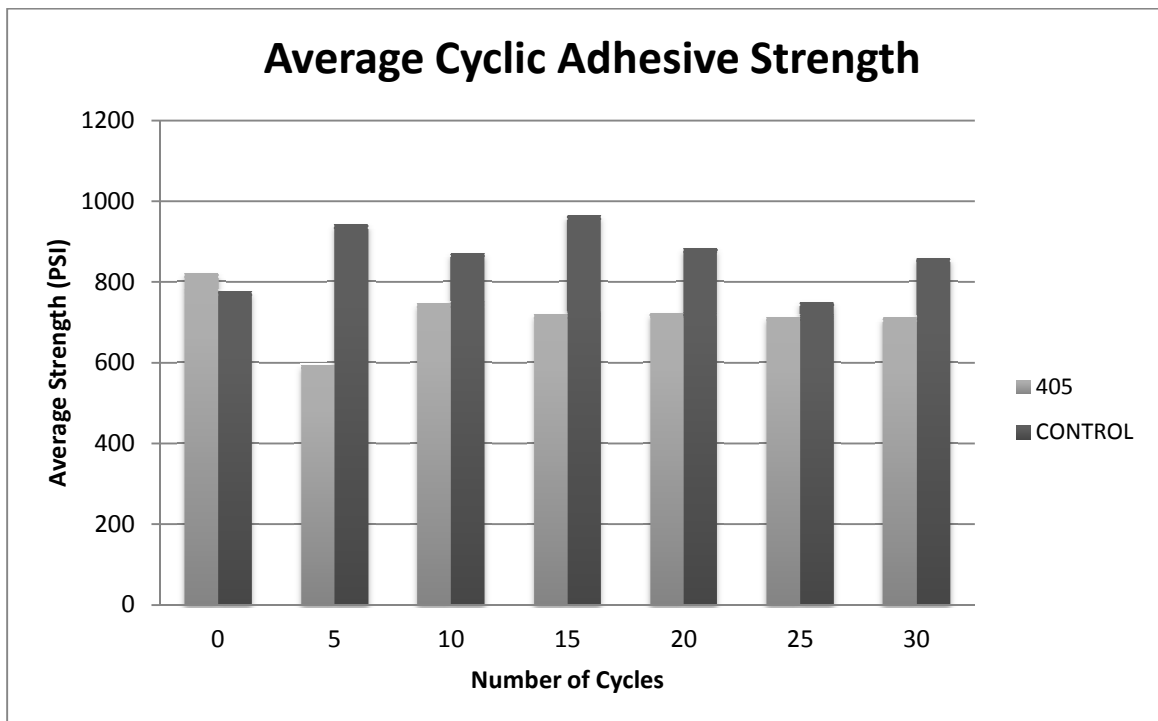


**Fig. 5.8** Average Strength of Mix 1.2 (Specimen 446)

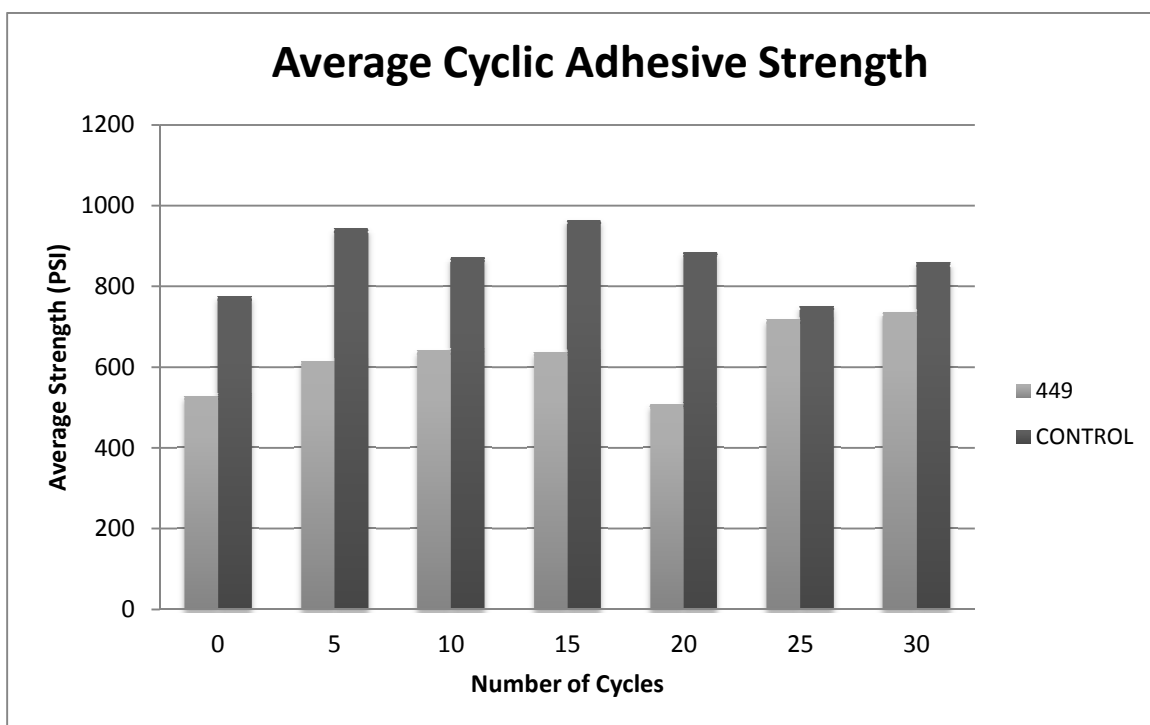
\* Indicates epoxy failures for data set in that cycle (TYP).



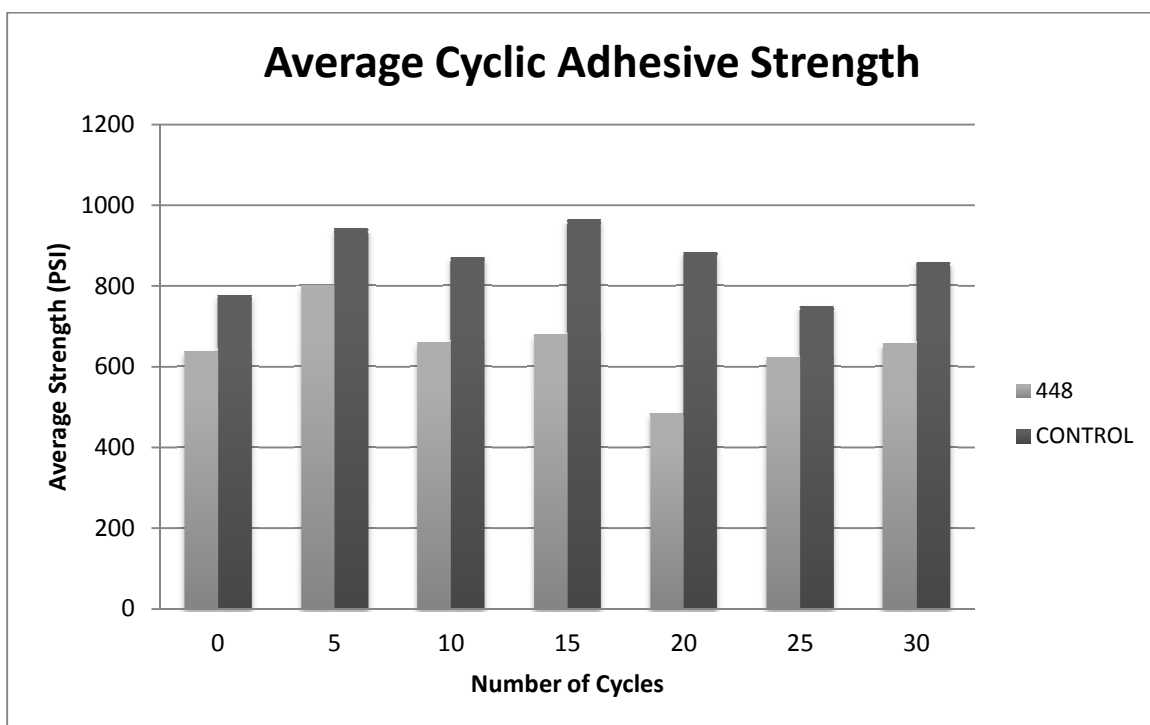
**Fig. 5.9** Average Strength of Mix 1.3 (Specimen 427)



**Fig. 5.10** Average Strength of Mix 1.4 (Specimen 405)

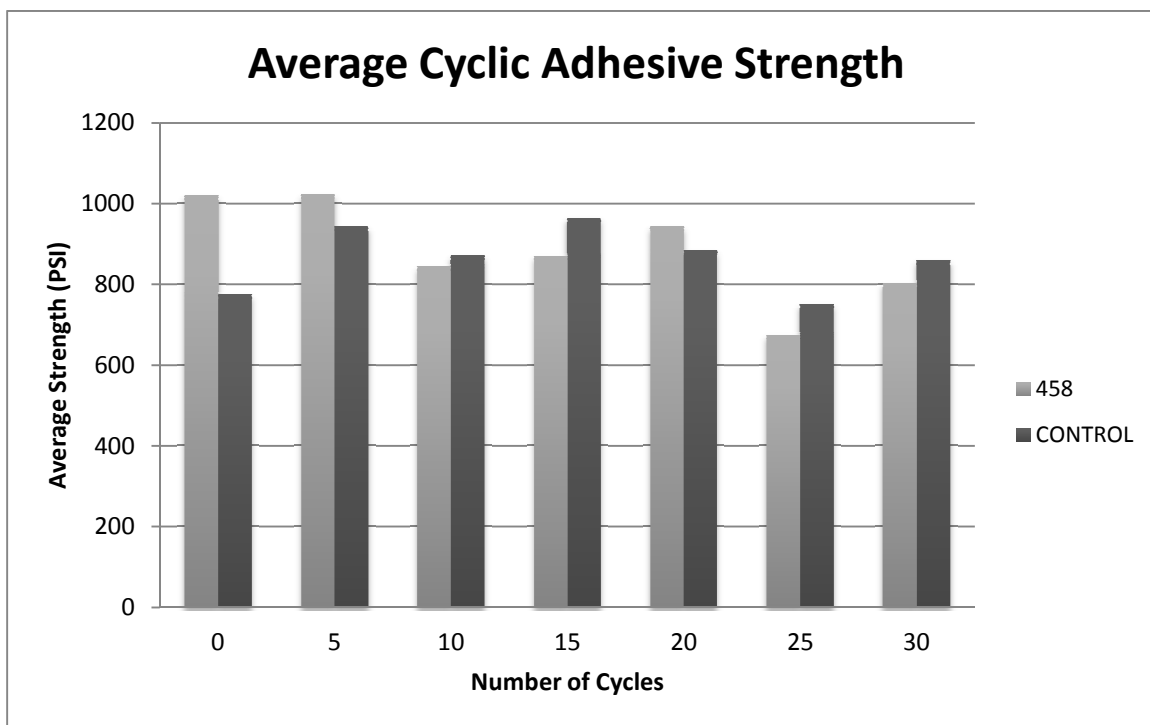


**Fig. 5.11** Average Strength of Mix 2.1 (Specimen 449)

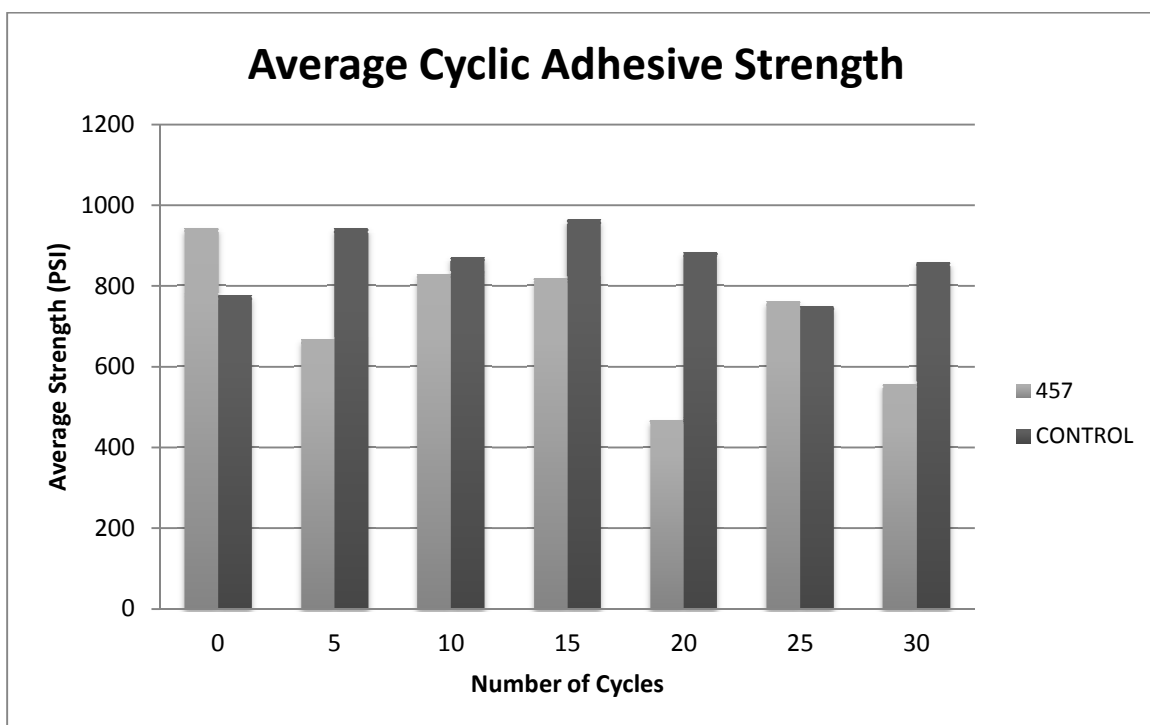


**Fig. 5.12** Average Strength of Mix 2.2 (Specimen 448)

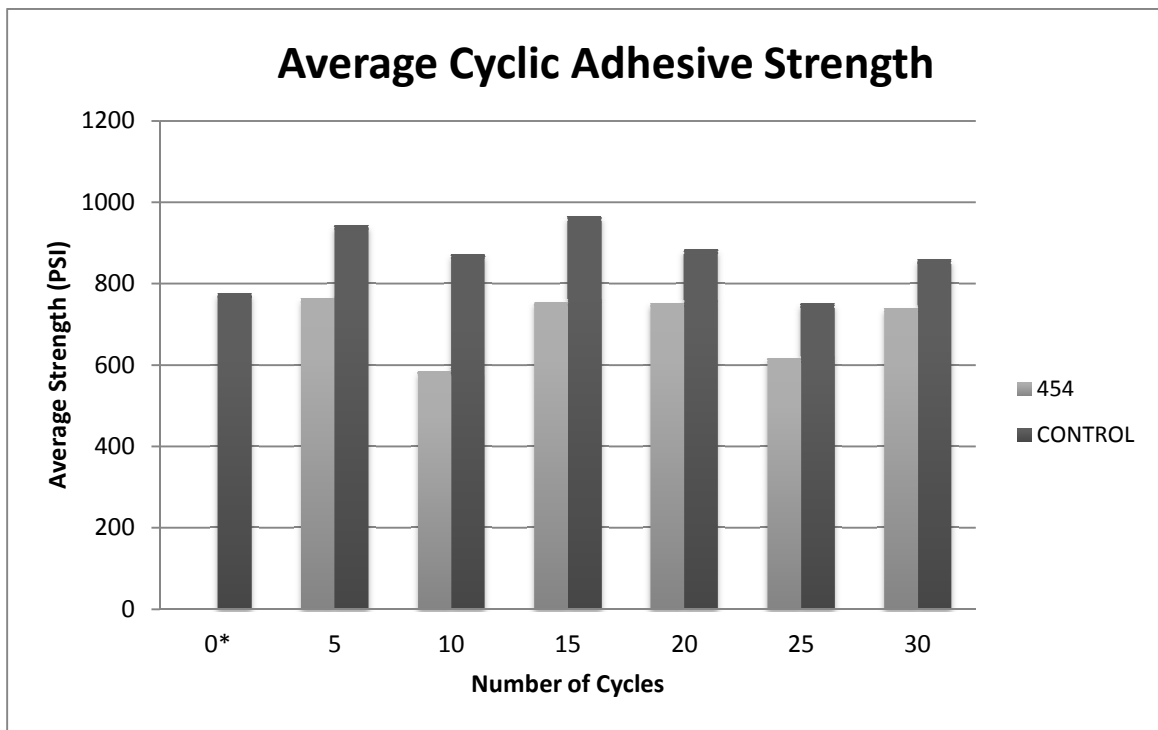




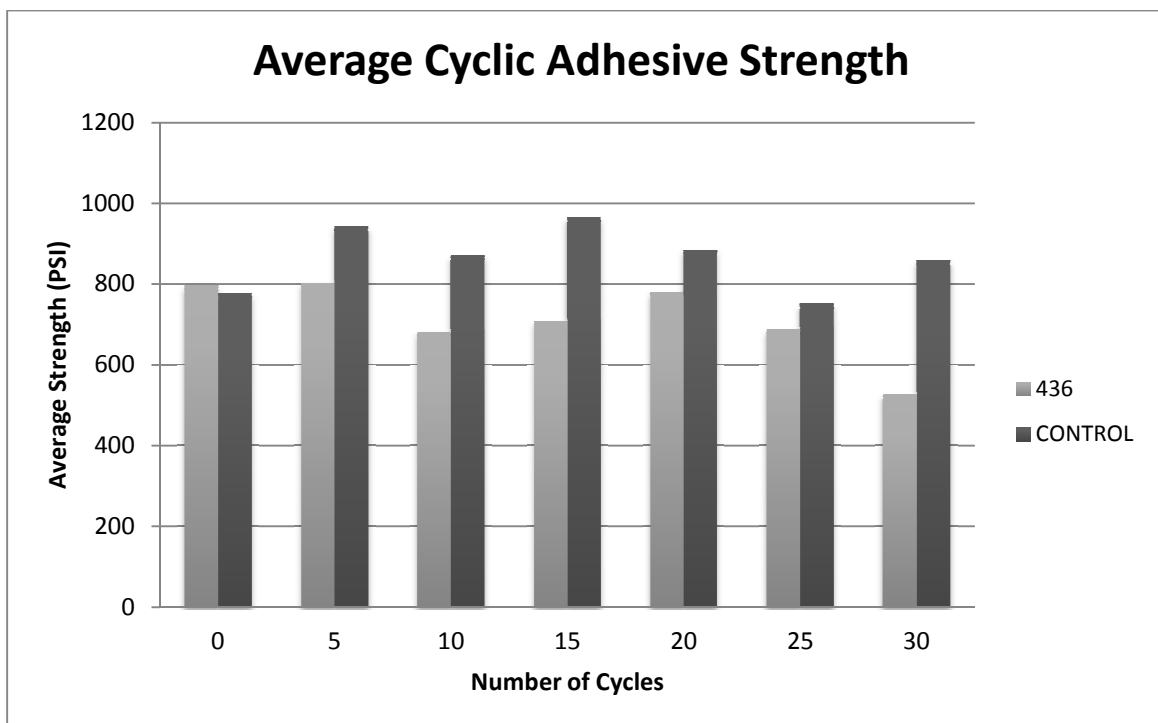
**Fig. 5.13** Average Strength of Mix 2.3 (Specimen 458)



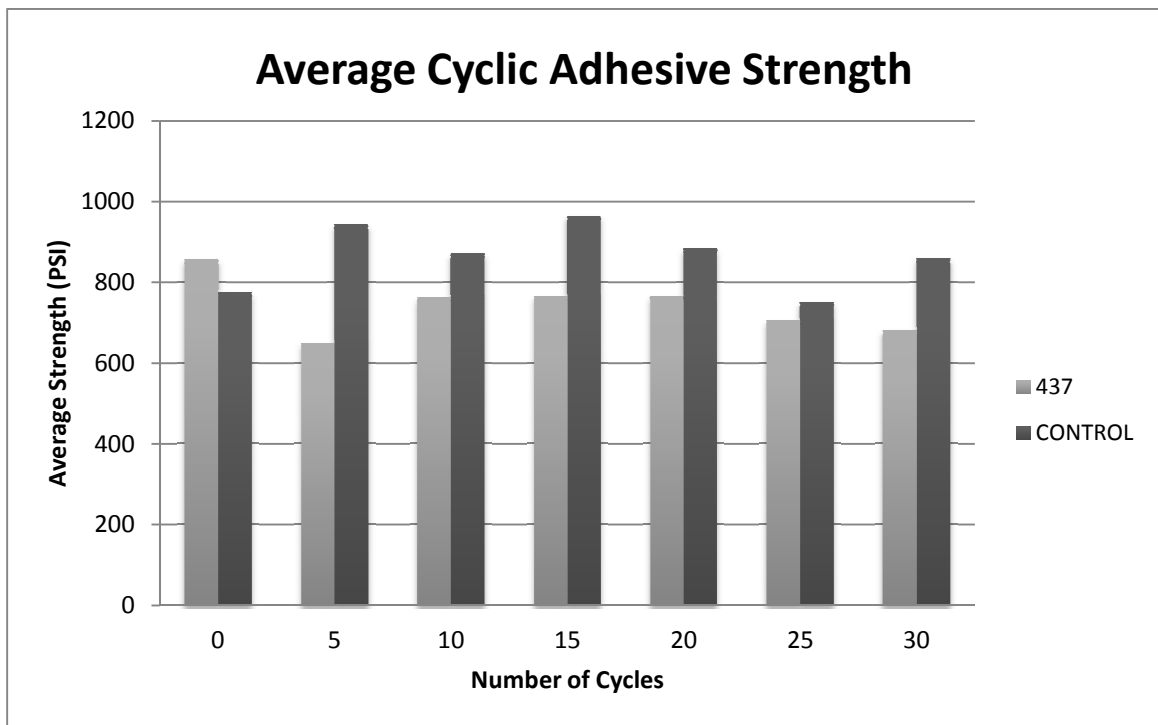
**Fig. 5.14** Average Strength of Mix 2.4 (Specimen 457)



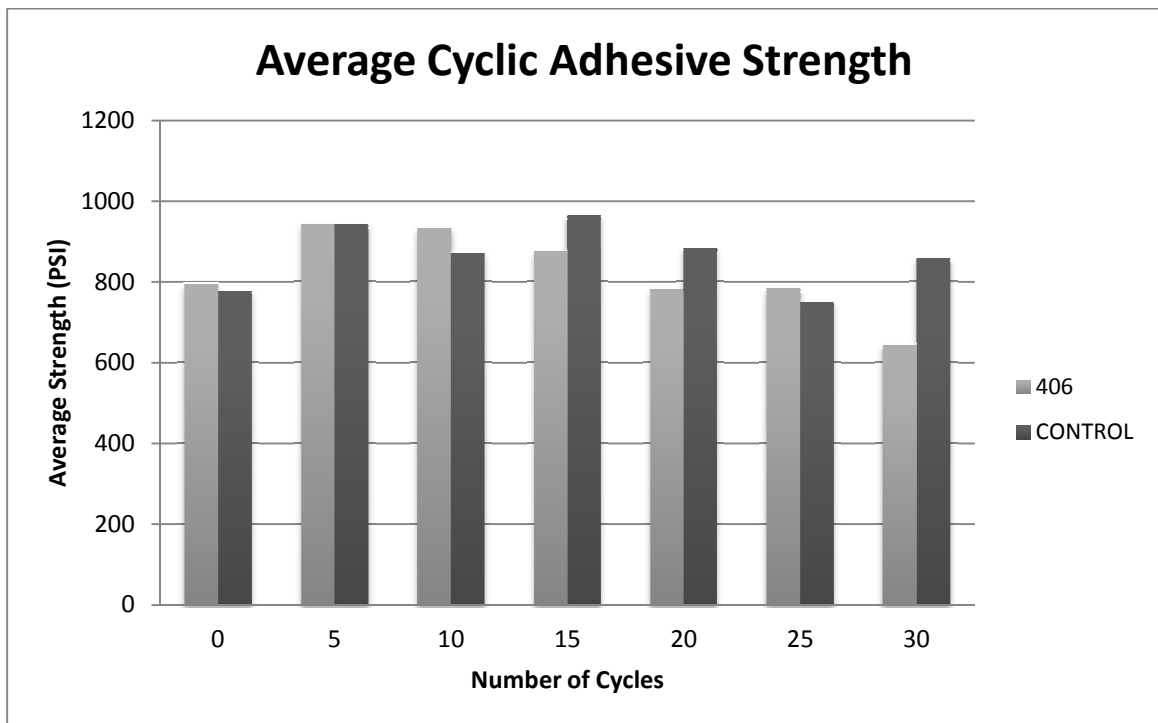
**Fig. 5.15** Average Strength of Mix 3.1 (Specimen 454)



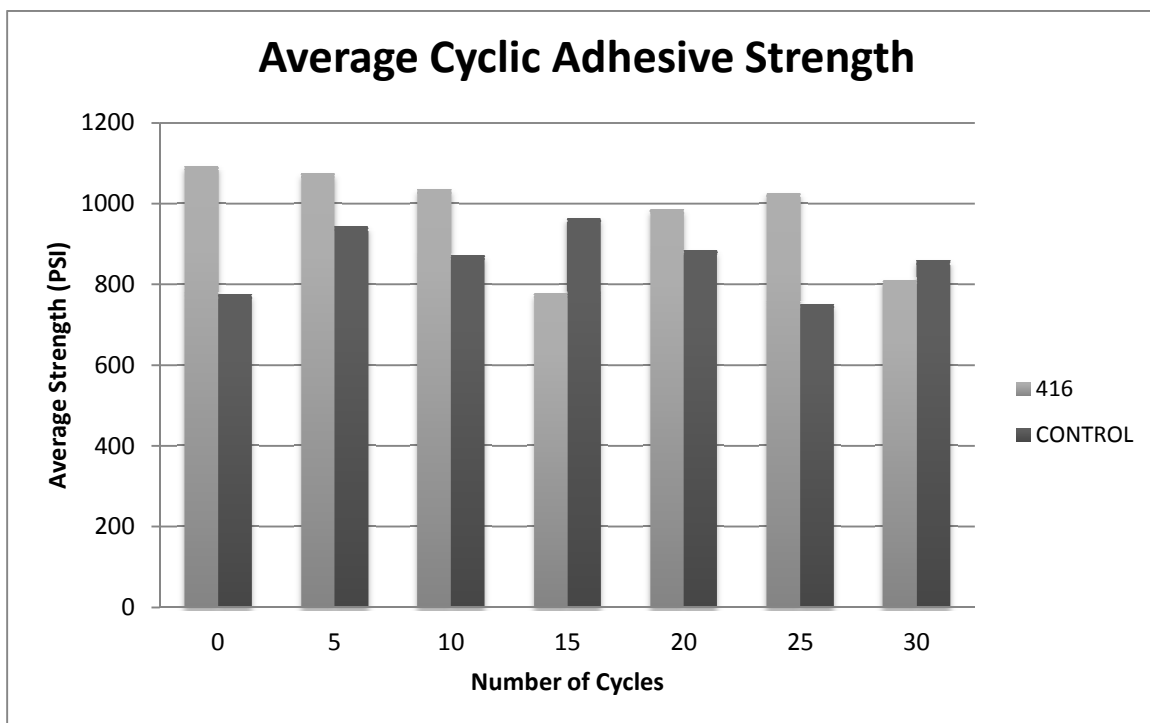
**Fig. 5.16** Average Strength of Mix 3.2 (Specimen 436)



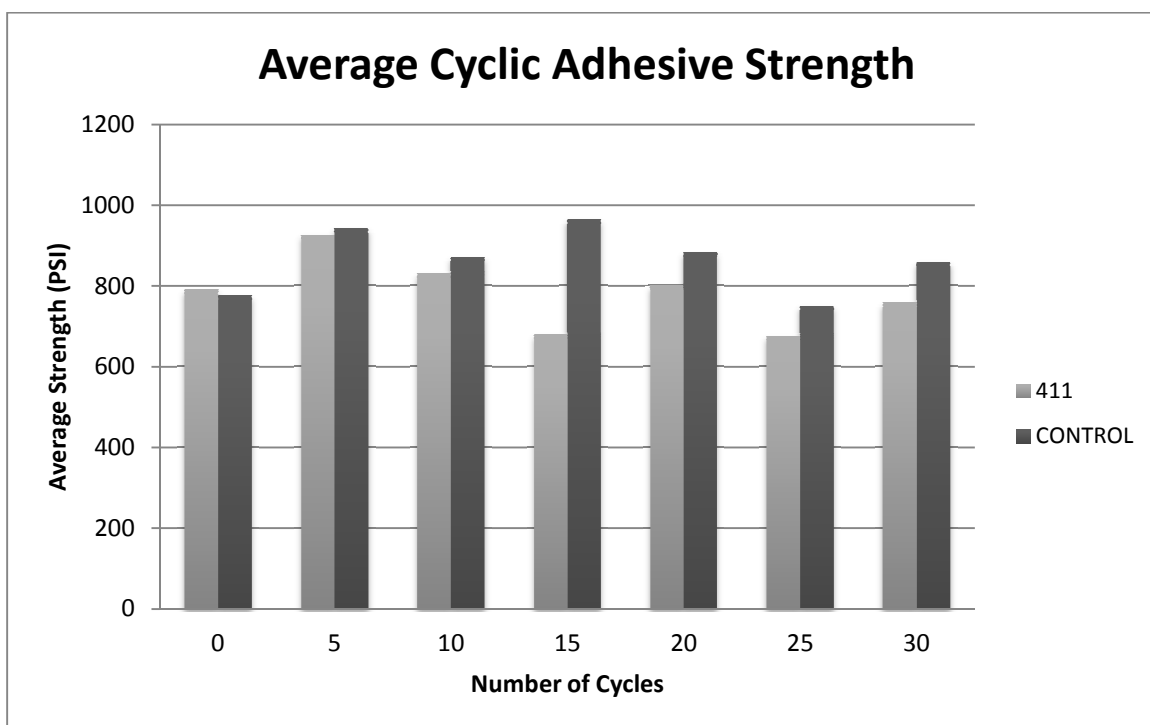
**Fig. 5.17** Average Strength of Mix 3.3 (Specimen 437)



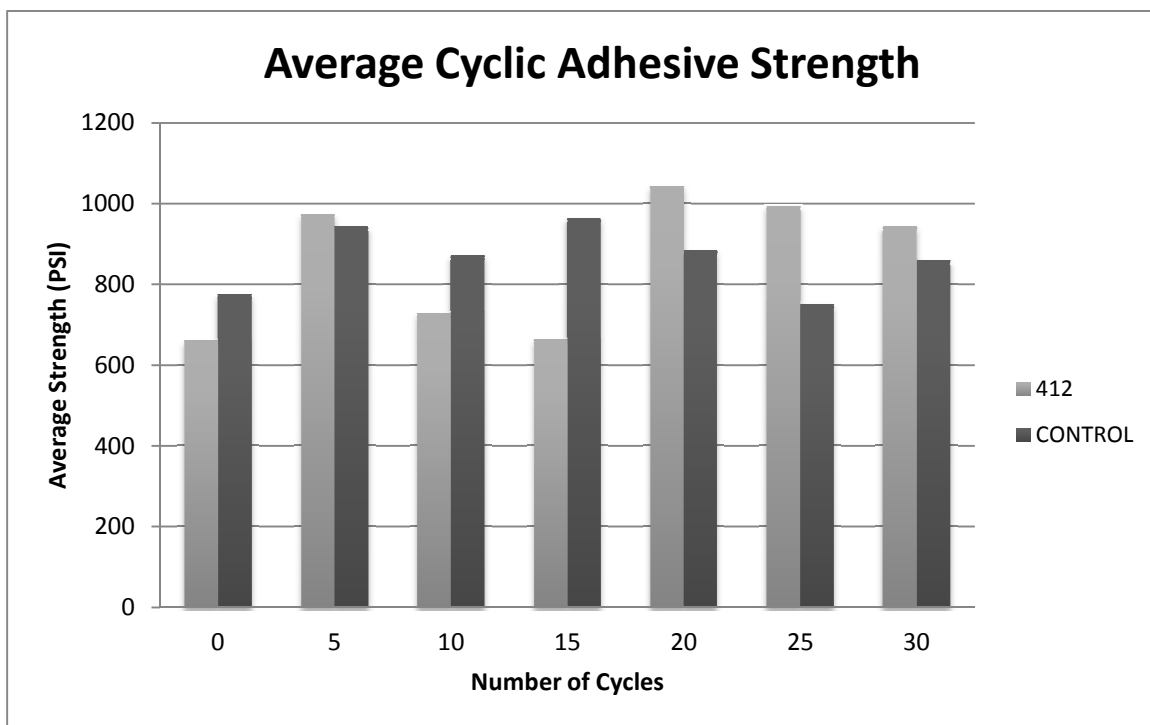
**Fig. 5.18** Average Strength of Mix 3.4 (Specimen 406)



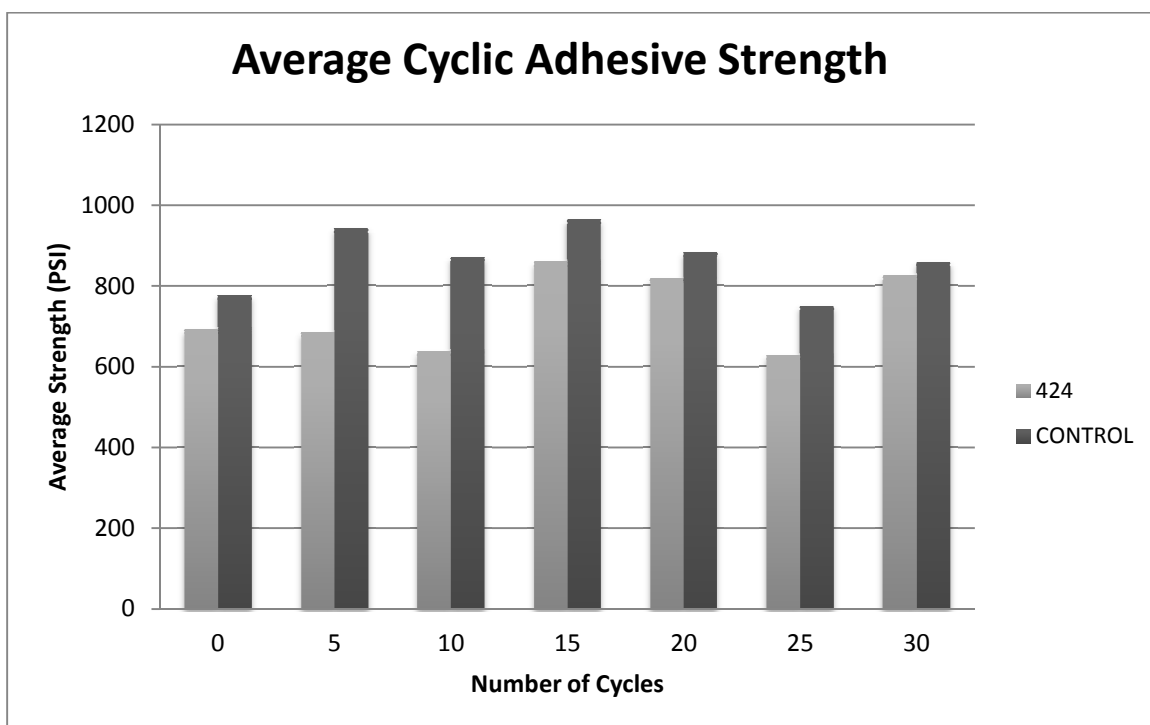
**Fig. 5.19** Average Strength of Mix 4.1 (Specimen 416)



**Fig. 5.20** Average Strength of Mix 4.2 (Specimen 411)



**Fig. 5.21** Average Strength of Mix 4.3 (Specimen 412)



**Fig. 5.22** Average Strength of Mix 4.4 (Specimen 424)

## **Chapter 6**

### **Conclusions**

This thesis presented the results of durability testing performed on an inorganic polymer protective coating that has previously been studied as a repair material. The results discussed in chapters 4 and 5 will help broaden the literature on this composite material as well as help to expand its potential commercial applications.

A review of current literature on composite polymers has led to the following conclusions:

- It is important to quantify the bond strength of composite polymer coatings under certain environmental conditions in order to determine the potential commercial applications for the material.
- Organic polymer coatings are ideal for substrates used in environments that do not require a permeable membrane for the release of vapor pressure, whereas, inorganic polymer matrices can be used in nearly any application.
- Alumino-silicate based inorganic matrices have shown considerable promise as a high performance coating that creates a fire resistant but permeable protective barrier that can be used on a number of different substrates including concrete and masonry.

- Composite coatings created using nano-particles are capable of increasing the durability of the composite coating by reducing the permeability of the hardened concrete.

Freeze/thaw and wet/dry durability testing were performed on four different mixes of the inorganic polymer matrix developed for repair of concrete components. The mixes consisted of a high flexural strength/low shear strength matrix, a moderate flexural/shear strength matrix, a low flexural strength/high shear strength matrix, as well as a moderate flexural/shear strength matrix comprised of nano-materials instead of micro-materials. The samples were tested for adhesion strength using a portable adhesion testing apparatus and the results are summarized in Table 6.1. The durability testing lead to the following conclusions:

Wet/Dry cycling:

- The control (specimen 413) had a high of 977 psi and a low of 632 psi.
- Mix 1.1 (specimen 459) had a high of 1003 psi and a low of 296 psi.
- Mix 1.2 (specimen 426) had a high of 709 psi and a low of 272 psi.
- Mix 1.3 (specimen 408) had a high of 878 psi and a low of 408 psi.
- Mix 1.4 (specimen 402) had a high of 1023 psi and a low of 611 psi.
- Mix 2.1 (specimen 425) had a high of 895 psi and a low of 433 psi.
- Mix 2.2 (specimen 407) had a high of 705 psi and a low of 399 psi.
- Mix 2.3 (specimen 410) had a high of 998 psi and a low of 309 psi.
- Mix 2.4 (specimen 432) had a high of 983 psi and a low of 670 psi.
- Mix 3.1 (specimen 423) had a high of 810 psi and a low of 384 psi.

- Mix 3.2 (specimen 455) had a high of 1208 psi and a low of 1208 psi.
- Mix 3.3 (specimen 422) had a high of 1017 psi and a low of 718 psi.
- Mix 3.4 (specimen 430) had a high of 1001 psi and a low of 498 psi.
- Mix 4.1 (specimen 453) had a high of 1116 psi and a low of 567 psi.
- Mix 4.2 (specimen 456) had a high of 841 psi and a low of 488 psi.
- Mix 4.3 (specimen 414) had a high of 1010 psi and a low of 463 psi.
- Mix 4.4 (specimen 439) had a high of 906 psi and a low of 527 psi.
- The use of nano-materials for mix 4 helped provide superior adhesive strength as opposed to the micro materials used in mix 2.
- The size of the nano-materials compared to the size of the micro-materials created a less permeable barrier as well as creating a stronger bond as more particles become in contact with the concrete substrate.
- High shear mixer and higher curing temperature did not provide better results for wet/dry testing.

#### Freeze/Thaw cycling:

- The control (specimen 404) had a high of 965 psi and a low of 751 psi.
- Mix 1.1 (specimen 451) had a high of 775 psi and a low of 571 psi.
- Mix 1.2 (specimen 446) had a high of 1040 psi and a low of 669 psi.
- Mix 1.3 (specimen 427) had a high of 722 psi and a low of 417 psi.
- Mix 1.4 (specimen 405) had a high of 822 psi and a low of 594 psi.
- Mix 2.1 (specimen 449) had a high of 737 psi and a low of 508 psi.
- Mix 2.2 (specimen 448) had a high of 801 psi and a low of 485 psi.



- Mix 2.3 (specimen 458) had a high of 1022 psi and a low of 674 psi.
- Mix 2.4 (specimen 457) had a high of 943 psi and a low of 468 psi.
- Mix 3.1 (specimen 454) had a high of 764 psi and a low of 585 psi.
- Mix 3.2 (specimen 436) had a high of 803 psi and a low of 528 psi.
- Mix 3.3 (specimen 437) had a high of 857 psi and a low of 649 psi.
- Mix 3.4 (specimen 406) had a high of 944 psi and a low of 644 psi.
- Mix 4.1 (specimen 416) had a high of 1093 psi and a low of 777 psi.
- Mix 4.2 (specimen 411) had a high of 927 psi and a low of 676 psi.
- Mix 4.3 (specimen 412) had a high of 1045 psi and a low of 661 psi.
- Mix 4.4 (specimen 424) had a high of 860 psi and a low of 629 psi.
- Similar to the conclusions drawn from the wet/dry cycling, it was seen that the use of nano materials for mix 4 helped provide superior adhesion strength compared to the other three mixes.
- The specimens that were formulated with the normal shear mixer as well as the 70°F curing performed better and are therefore recommended instead of the high shear mixer and 120°F curing.

Suggestions for future research:

The wetting and drying testing resulted in primarily epoxy related failures. Future research is needed to determine the effects of wet/dry cycling on these coatings using a different testing method that is not affected by the wetting part of the cycling. Further durability testing under 100% humidity conditions as well as testing under exposure to

deicing chemicals would provide a complete profile of the durability of the inorganic matrix. Future research on the use of nano-particles in this inorganic matrix would also be beneficial.

Thermal Cycling Beams	Wetting and Drying Beams	Normalized Adhesive Strength At End of Short Term Testing		Notes
		Thermal Cycling	Wetting and Drying	
Mix 1.1: Blendtec 70° Curing		71% - (83% coating failures after 5 <sup>th</sup> cycle)	60% - (70% epoxy failures throughout)	High flexural resistance / low shear resistance
451	459			
Mix 1.2: Blendtec 120° Curing		61% - (50% coating failures after 20 <sup>th</sup> cycle)	23% - (100% coating failures after 36 <sup>th</sup> cycle)	High flexural resistance / low shear resistance
446	426			
Mix 1.3: Ninja 70° Curing		54% - (86% coating failures after 5 <sup>th</sup> cycle)	37% - (100% coating failures after 36 <sup>th</sup> cycle)	High flexural resistance / low shear resistance
427	408			
Mix 1.4: Ninja 120° Curing		65% - (75% coating failures throughout)	51% - (43% coating failures after 36 <sup>th</sup> cycle)	High flexural resistance / low shear resistance
405	402			
Mix 2.1: Blendtec 70° Curing		67% - (91% coating failures throughout)	38% - (70% coating failures after 6 <sup>th</sup> cycle)	Moderate flexural resistance / moderate shear resistance
449	425			
Mix 2.2: Blendtec 120° Curing		60% - (55% coating failures throughout)	44% - (70% coating failures throughout)	Moderate flexural resistance / moderate shear resistance
448	407			
Mix 2.3: Ninja 70° Curing		73% - (Coating failures in less than 31% of tests)	(70% epoxy failures throughout)	Moderate flexural resistance / moderate shear resistance
458	410			
Mix 2.4: Ninja 120° Curing		51% - (57% coating failures after 15 <sup>th</sup> cycle)	65% - (70% coating failures after 36 <sup>th</sup> cycle)	Moderate flexural resistance / moderate shear resistance
457	432			
Mix 3.1: Blendtec 70° Curing		68% - (52% coating failures throughout)	(92% epoxy failures after 6 <sup>th</sup> cycle)	Low flexural resistance / high shear resistance
454	423			
Mix 3.2: Blendtec 120° Curing		48% - (50% coating failures after 20 <sup>th</sup> cycle)	(100% epoxy failures after 6 <sup>th</sup> cycle)	Low flexural resistance / high shear resistance
436	455			
Mix 3.3: Ninja 70° Curing		62% - (63% coating failures until 25 <sup>th</sup> cycle)	(88% epoxy failures after 6 <sup>th</sup> cycle)	Low flexural resistance / high shear resistance
437	422			
Mix 3.4: Ninja 120° Curing		59% - (100% coating failures after 20 <sup>th</sup> cycle)	(96% epoxy failures after 6 <sup>th</sup> cycle)	Low flexural resistance / high shear resistance
406	430			
Mix 4.1: Blendtec 70° Curing		74% - (87% coating failures after 25 <sup>th</sup> cycle)	47% - (100% coating failures after 36 <sup>th</sup> cycle)	Moderate flexural resistance / moderate shear resistance (nano materials)
416	453			
Mix 4.2: Blendtec 120° Curing		69% - (75% coating failures)	43% - (40% coating failures)	Moderate flexural resistance / moderate

411	456	throughout)	throughout)	shear resistance (nano materials)
Mix 4.3: Ninja 70° Curing		86% - (100% coating failures after 30 <sup>th</sup> cycle)	38% - (42% epoxy failures throughout)	Moderate flexural resistance / moderate shear resistance (nano materials)
412	414			
Mix 4.4: Ninja 120° Curing		76% - (Coating failures in less than 20% of tests)	75% - (43% epoxy failures throughout)	Moderate flexural resistance / moderate shear resistance (nano materials)
424	439			
Control		79% - (100% concrete failures throughout)	69% - (75% epoxy failures throughout)	No coating applied.
404	413			

**Table 6.1** Summary of Testing Results

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