NANOPOROUS THIN-FILM ADDITIVES TO IMPROVE PRECAST CONCRETE CONSTRUCTION OF TRANSPORTATION FACILITIES

CFIRE Project 03-13
December 2011

National Center for Freight & Infrastructure Research & Education
College of Engineering
Department of Civil and Environmental Engineering
University of Wisconsin, Madison

Authors: Jessica M. S. Silva, Marc A. Anderson, Michael G. Oliva, and M. Isabel Tejedor
University of Wisconsin-Madison
1415 Engineering Dr., Madison, WI 53706

Principal Investigator: Marc A. Anderson;
Professor, Department of Civil and Environmental Engineering

EXHIBIT B

Technical Report Documentation Page

1. Report No. CFIRE 03-13	2. Government Accession No	3. Recipient's Catalog No **CFDA 20.701**
4. Title and Subtitle Nanoporous Thin Film Additives to Improve Precast Concrete Construction of Transportation Facilities		5. Report Date **December 2011** 6. Performing Organization Code
7. Authors Silva, J. M. S., Anderson, M., Oliva, M., Tejedor, M. I.		8. Performing Organization Report No. CFIRE 03-13
9. Performing Organization Name and Address National Center for Freight and Infrastructure Research and Education (CFIRE) University of Wisconsin-Madison 1415 Engineering Drive, 2205 EH Madison, WI 53706		10. Work Unit No. (TRAIS) 11. Contract or Grant No. DTRT06-G-0020
12. Sponsoring Organization Name and Address Research and Innovative Technology Administration United States Department of Transportation 1200 New Jersey Avenue, SE Washington, DC 20590		13. Type of Report and Period Covered **Final Report (Oct. 2009-Dec. 2011)** 14. Sponsoring Agency Code
15. Supplementary Notes Matching funds ($12,000) from Spancrete. Project completed for USDOT RITA by CFIRE.		

16. Abstract

The national transportation network contains a significant number of highway and railway bridges. This research is intended to transform the use of precast/pre-stressed materials in the transportation infrastructure. Specifically it examines how nanoporous thin-films create a new jointing mechanism between precast concrete bridge components. Mechanical testing was conducted from which it was determined that these thin-films can increase the bonding strength of grout in joints. Preliminary durability testing was performed and showed these thin-films help strengthen the jointed area. Lastly, Scanning Electron Microscopy was used to help better define the mechanism by which the thin-films improved the bonding strength of grout to hardened concrete.

17. Key Words Grout bonding, bonding agents, bonding strength, bonding durability, silica thin-films	18. Distribution Statement **No restrictions. This report is available through the Transportation Research Information Services of the National Transportation Library.**		
19. Security Classification (of this report) **Unclassified**	19. Security Classification (of this page) **Unclassified**	20. No. of Pages **36**	21. Price **-0-**

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Executive Summary

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The national transportation network contains a significant number of highway and railway bridges. This research is intended to transform the use of precast/pre-stressed materials in this type of transportation infrastructure. Specifically we will examine how nanoporous thin-films improve the jointing mechanism between precast concrete components.

The principal aim of this project is to show the benefit and practicality of using nanoporous thin-film coatings to improve: 1) the adhesion between grouts and already hardened concrete surfaces in joints between precast pieces and 2) the performance of pre-stressed concrete components by decreasing pre-stress loss due to shrinkage and creep, and by minimizing the cement content without compromising mechanical properties.

Background

Rapid bridge construction has received increased emphasis over the past 15 years. A portion of this focus has multiplied the use of precast members in the construction process. These members are created in controlled environments making them a very consistent product. Their use also expedites the construction process. Despite these positive advantages, a weakness of precast systems occurs in the joints between members where grouting is frequently used. Previous research for other applications has indicated that nanoporous thin-films can improve bonding between aggregate surfaces and mortar. This finding prompted an examination of nanoporous thin-films for the specific application of bonding between hardened precast concrete surfaces and grout. An enhancement of bonding strength by using these thin-films has also lead to the hypothesis that nanoporous thin-films could promote high early strength concrete that does not have increased creep and shrinkage.

This research was conducted at the University of Wisconsin-Madison with funds from the Center for Freight and Infrastructure Research and Education (CFIRE). Additionally, the obvious benefactor of this research is the precast concrete industry. Spancrete Inc., a private company and a major precast manufacturer in the Wisconsin area, has contributed in kind to this project.

Process

This research was conducted to determine if the nanoporous thin-films could improve the bonding of grout to hardened precast concrete surfaces. The research was conducted over a 2 year period and included various testing procedures. For example, means of applying thin-films, bonding strength, alterations in the microstructure, and durability with respect to freeze-thaw were measured in this study. Where possible, standard ASTM procedures were followed so the data could be compared to other existing industry data.

The testing and data analysis was completed in UW-Madison laboratories. As this research explores the use of this material for a new function, procedures for the application of the thin-films were tested first. Secondly, mechanical testing was conducted in grouted joints as well as microstructural examination using scanning electron microscopy with energy dispersive spectroscopy capabilities (SEM/EDS). Data from these tests were used to evaluate the strength and characteristics of the bond. Lastly, durability testing was conducted to verify that this new joining method could survive subjection to freeze-thaw environments.

Findings and Conclusions

The ability of the nanoporous thin-films to increase the bonding strength between hardened precast concrete and grout was proved through the mechanical testing of joints. Preliminary durability testing was performed and showed these thin-films help strengthen the jointed area. Lastly, microscopy analysis was used to determine the mechanism by which the thin-films improved the bonding strength of grout to hardened concrete. Tests were also conducted to show that these nanoporous nanoparticulate thin-films could be used to develop high early strength in concrete with greatly reduced cement content that does not have increased creep and shrinkage. However, it was discovered that this improvement can only be achieved when carbonate based aggregate materials are *not* utilized. Unfortunately most precast products in Wisconsin are manufactured with carbonate aggregates.

In order to qualify the results of the study on joints, comparisons were made to typical jointing methods currently in practice. It was shown that the thin-films can increase the strength of the bond in comparison to normal grouting procedures. Also, comparisons of the performance of the thin-films on non-sandblasted joints to normal joints with sandblasted surfaces proved that the thin-film could outperform the enhancements sandblasting can make to improve joint strength. Skipping this sand blasting step could reduce costs of the overall precast production process. Practical application methods were also considered in the analysis of these thin-films. This material is very easy to apply and fast to use. The material is best applied just before grouting without any drying time. It also does not need to be mixed or combined with any other product before use. The material can be used in a similar manner as that of current materials so new regulations or policy implications are not a factor.

Recommendations for Further Action

The results of this research could be put in place as is, but further modifications to the nanoparticle solutions (sols) used to create the thin-film material could be sought in order to reduce the cost of the material. Also, further testing on other materials currently in use could be conducted to prove the benefits of these thin-films compared to a large range of present materials. The study included slant shear measurements which combine compression and shear forces. In order to more explicitly show the improved strength, direct shear testing could be conducted. Also, direct tension testing should be conducted to provide a measure of joint resistance to volume change effects. Lastly, additional testing with igneous aggregates to determine if the new coatings in concrete mixes could affect shrinkage and creep while possibly utilizing reduced cement content mix designs could be conducted. To prove the use of the product in-situ, the material could be tested on a trafficked bridge joint.

In order to implement the use of this product, a manufacturing company will have to produce the sol material for this application. A partnership between the product licenser, UR-Water, and a production company would need to be made and production started so the material can be produced, packaged, and sold to distributors. The use of the material could be explained in the product guidelines that would be distributed as the material is purchased. The benefits of the material could be described in a newsletter or publication and distributed to potential users.

Acknowledgements

The authors would like to acknowledge the financial support from the National Center for Freight and Infrastructure Research and Education (CFIRE) and Spancrete for their in kind support. The concrete mixing and mechanical testing was carried out in the Structures and Materials Laboratory, and the microstructural analysis was performed in the Material Science Center at the University of Wisconsin-Madison.

Abstract

The national transportation network contains a significant number of highway and railway bridges. This research is intended to transform the use of precast/pre-stressed materials in the transportation infrastructure. Specifically it examines how nanoporous thin-films create a new jointing mechanism between precast concrete bridge components. Mechanical testing was conducted from which it was determined that these thin-films can increase the bonding strength of grout in joints. Preliminary durability testing was performed and showed these thin-films help strengthen the jointed area. Lastly, Scanning Electron Microscopy was used to help better define the mechanism by which the thin-films improved the bonding strength of grout to hardened concrete.

1. Objective

The principal aim of this project is to show the benefit and practicality of using nanoporous thin-film coatings to improve: 1) the adhesion between grouts and already hardened concrete surfaces in joints between precast pieces, and also 2) the performance of concrete used for precast prestressed members by decreasing pre-stress loss due to shrinkage and creep, and by minimizing the cement content required without compromising mechanical properties of the concrete.

2. Scope of Study

This study was used to determine if nanoporous thin-film oxide coatings could be 1) used to improve adhesion in precast concrete and between grouts and hardened precast concrete surfaces in joints and 2) reduce creep and shrinkage. The evaluation of the use of thin-films to improve joints using grout included identifying the best application, quantity, and technique. The study also included mechanical testing for the joint strength of normal grouted joints, joints prepared with current commercial bonding agents, and joints with the thin-film coating. Long term durability was measured by freeze-thaw testing for normal grouted joints and joints prepared with the thin-film. In order to determine the chemical-mechanical mechanisms occurring in the joints, an analysis of the microstructure of the bond between the grout and the hardened concrete surface was conducted.

An initial investigation focused on the second objective and involved covering concrete aggregates with thin-film coatings before mixing the concrete. The first tests proved that limestone aggregates did not achieve the improved strength as originally expected from other studies conducted with igneous aggregates. Since Wisconsin precasters use limestone aggregates, these efforts were aborted before extensive full scale testing was conducted and the scope of the study was narrowed to the second objective.

3. Introduction

The national transportation network contains a myriad of highway and railway bridges. The constant development of new methods to fix, repair, and maintain these important structures rapidly and with better durability has always been a priority objective. However, the importance of these activities is growing at an exponential rate due to the critical condition of our infrastructure. The aim of this research program is transformational and is intended to greatly change the use of precast/pre-stressed materials in the transportation infrastructure upon which freight movements rely. This research has the potential to change the way precast concrete is assembled. Specifically, we examine the use of nanoporous nanoparticulate thin-film oxide coatings that should lead to an improved jointing mechanism between precast concrete components.

In 1998, as a consequence of the need to produce better performing, lower maintenance, and more sustainable highways and bridges, a group of Wisconsin University and state engineers started working together under the Federal Highway Administration's Innovative Bridge Research and Construction program to develop and implement new construction techniques for bridges. *(1)* The result was the construction of four bridges using innovative techniques and materials for construction. One example was the use of a precast panel instead of the more traditional cast in place method for bridge deck construction. This construction technique is significantly faster, allowing for shorter restriction of lanes during construction. Furthermore, the precast concrete is a higher quality material that can result in a superior bridge system. These advantages partly result from the fact that precast materials are produced in a highly controlled production process, thus resulting in a material with superior performance in terms of durability as compared to more traditional cast in place concrete.

Unfortunately, in spite of the advantages obtained by using precast construction techniques, there are still problems and limitations related to precast concrete units. The most important weakness lies in the deterioration of the connections between precast concrete components under repeated truck loading such as between pavement or deck panels. *(2)* Similar joints are used between precast bridge deck panels, flanged bridge girders, and precast bridge abutment units. They all tend to be weaker and more vulnerable than the high strength precast components.

Field experience with bridge, highway, and airport paving panels has indicated that among all the different joint designs the female-female type has provided better performance. A typical female-female grouted joint for precast bridge deck slabs is shown in Figure 1, but identical joints of different size are used between pavement panels, flanged girders, abutment panels, and many other precast components. These grouted joints often create a pre-cracked condition due to low bond adherence of the grout with the hardened concrete and grout shrinkage. The principal causes of joint failure in bridge decks under repeated loads from traffic is poor bonding strength between fresh grout and already hardened concrete, and shrinkage of the grout. *(3)* The initial joint failure leads to the development of leaking, freeze-thaw pockets, and continued degradation. The present solution for avoiding these problems implies complicated post-tensioning of the joints or the use of intensive and costly maintenance operations.

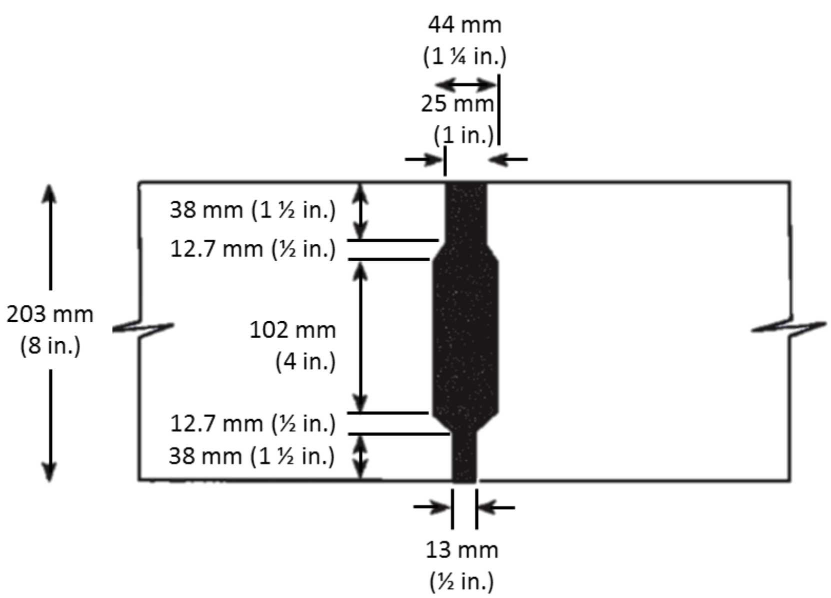


Figure 1. Configuration of female-female joint [3]

Even though precast concrete components represent high quality final products, major opportunities exist for improving their performance as well. High strength components often demand excessively high early strength to allow rapid pre-stress application, often achieved by high cement contents. These mixes may then exhibit higher shrinkage and creep than is desirable in pretensioned components. Both mechanisms produce significant changes in the original length of the pieces that may ultimately lead to loss of tension in pre-stressing tendons, loss of capacity, and the potential development of larger than expected deformations. *(4)* By obtaining better performance, while reducing cement content, the precasting industry can provide an innovative construction product that is sustainable and durable for the freight transportation infrastructure.

This research is directed toward developing improved connections between precast concrete components used in transportation infrastructure construction. By using nanoporous thin-film oxide coating technologies, the performance and the durability of joining materials can be enhanced. These nanoporous films should significantly increase the longevity of infrastructures employed for freight movement and also reduce disruptive times for repair or rehabilitation.

The principal aim of this project is to show the benefit and practicality of using these thin-film coatings to improve the adhesion between grouts and already hardened concrete surfaces in joints between precast pieces. The project included determining the optimal materials and best application method for improved bonding. Bond strength testing was performed and an analysis of the microstructure of the bond between the grout and hardened concrete surface was conducted using a scanning electron microscope with energy dispersive spectroscopy capabilities (SEM/EDS). The study also included a long-term durability test of these joints by using freeze-thaw methods.

4. Project Description

The primary aim of the research in this project was to show the benefit and practicality of nanoporous thin-films for improving connections between precast concrete components. The bond strength between grout and hardened concrete in joints should be improved by coating the concrete surface with the nanoporous thin-film. This enhanced adhesion should improve mechanical properties and durability, while potentially eliminating a sandblasting process for preparing the hardened concrete surface. This improved bond strength may also be beneficial in other applications where grout is applied to hardened concrete such as retro-fitting dowels held with grout at highway pavement joints.

Initially the project had a second aim of decreasing shrinkage, achieving high early strength for pre-stress applications, and minimizing the cement content without compromising mechanical properties in concrete used for precast members. However, early testing proved that this objective could not be obtained by coating limestone aggregates, as used in Wisconsin, and the objective was abandoned.

To achieve the objectives the following tasks are defined (Tasks 5&6 proved elusive):

- Task 1: Obtain materials from precast plants and prepare test specimens.
- Task 2: Use thin-film coatings to improve adhesion between grouts and hardened concrete surfaces as proved by laboratory testing.
- Task 3: Evaluate long term durability of these joints through freeze-thaw durability tests.
- Task 4: Analyze the microstructure of the bond between grouts and hardened concrete surfaces.
- Task 5: Evaluate the potential of nanoporous thin-films applied as aggregate coatings for mitigating shrinkage in precast concrete mixtures.
- Task 6: With the success of task 5, reduce cement content in precast panels.

5. Materials

Concrete materials were obtained from a Wisconsin precaster, Spancrete Inc., and local manufacturers. Preliminary characterization studies on the materials were immediately performed. Aggregate characterization included gradation according to ASTM C33, absorption according to ASTM C127 and ASTM C128 for coarse and fine aggregates respectively, and lastly, air percentage according to ASTM C231. The aggregate material characterization summary is listed in Table 1. The cement used in the study was ordinary Type I Portland cement, and the slag was a grade 120.

Table 1. Aggregate material description used throughout study.

	Coarse Aggregate	Fine Aggregate
Description	3/4 inch washed dolomite limestone	combination of dolomite and quartz
Absorption	1.08%	1.10%
Gradation	conforms to ASTM C33	conforms to ASTM C33

The primary aim of this study is to determine if the nanoporous films can be used to improve the bonding of grout to hardened precast concrete surfaces. For this, nanoparticle solutions were created, and grout materials were obtained. The synthesis of silica nanoparticles in basic solution were produced in accordance with Sheng and Chu's methodologies. *(5)* This material is a solution of particles (sol), but if the solution becomes dehydrated or the particles which are suspended are forced together, they will aggregate. Upon aggregation, these nanoparticles form silica polymer chains and become a gel eventually yielding a solid material.

Knowledge of the time required for the nanoparticle sol to form a gel and solid is important to this research as it will help to define the proper application time necessary to improve bonding. Axelsson preformed a study to determine the mechanical properties of silica sol in the gelation stages of the material. The silica sol was studied for its application in tunnel systems as a treatment for cracking in concrete. Often small cracks can form in concrete where the use of fresh concrete for patching is impossible due to the restricted size of the opening. In these cases, silica sol can be injected and left to aggregate, consequently filling the crack. The material was found to become stronger as it dries. *(6)* During the mechanical testing portion of this project, application time will be varied in order to determine the time frame by which the largest increases in strength can be obtained.

Grout mixes were purchased from a local construction material supply store. Several types of grout were obtained to determine if the makeup of the material would affect the efficacy of the nanoporous thin-film. The materials were tested for repeatability in results. A medium range construction grade non-shrinkage grout and a high performance non-shrinkage grout were selected from five tested for use in the majority of the tests in this study. The various grouts tested are described in Table 2. Bonding agents were also obtained to determine if the nanoporous thin-film could outperform existing commercially available materials. The agents used in this study are listed in

Table 3. The current examination was a pilot study and full factorial testing protocol could not be carried out with all materials.

Table 2. Reported data from manufacturer of various grout materials used in study.

Material (company)	Description (all non-shrink)	Reported Compressive Strength (at 28 days)		Compressive Testing Standard Used	Bonding Strength at 28 days using ASTM C882 Modified	
		Plastic	Flowable		Plastic	Flowable
NS Grout (Euclid)	non-metalic grout		8500 psi (59 MPa)			
Sika 211 SCC Plus	one-component cementitious, silica fume and polymer-modified, self-consolidating	7000 psi (48.3 MPa)		ASTM C39	2500 psi (17.2 MPa) 28 days	1500 psi (10.3 MPa) 7 days
Sika 212	high performance cement based grout	7500 psi (51.7 MPa)	6200 psi (42.7 MPa)	CRD C-621	2000psi (13.1 MPa)	1900 psi (13.1 MPa)
CG-86 (Euclid)	construction grade grout	7500 psi (51.7 MPa)	7000 psi (48.3 MPa)	ASTM C109-93	2300 psi (15.8 MPa)	2500 psi (17.2 MPa)
588 10K (WR Meadows)	Portland cement based precision grout	11000 psi (75.8 MPa)	9800 psi (67.6 MPa)			

Table 3. Reported data from manufacturer of bonding agents used in study.

Material	Description	Mix Ratio	Compressive Strength 7 days		Bonding Strength		Bond Strength Standard Used
			unmodified	modified	unmodified	modified	
Tammsweld (Euclid Chem)	Latex Bonding Agent	do not dilute				4600psi	ASTM C1042
AKKRO-7T (Euclid Chem)	Admixture (acrylic polymer)	0.45 admixture to cement	2345 psi (sand-cement)	3410 psi (sand-cement)	56 psi	486 psi	ASTM C321

Use of the above bonding agents was tested with ASTM C882. However, the application and use of the agents did not render a higher bonding strength than the control specimens, and was therefore not continued. Comparisons between bonding agents and the nanoporous thin-films were conducted using a literature review.

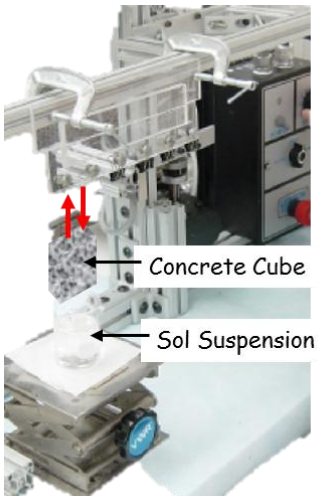
6. Methods

6.1. Nanoporous thin film application methods

The use of nanoporous thin-films as concrete bonding enhancers is novel. In order to determine the best method of applying these films to the hardened concrete, several methods were tested. Methods included slip casting, painting, spraying, and dip coating. Application methods are depicted in Table 4.

- Slip casting consists of soaking the concrete in the solution of nanoparticles (sol) and allowing capillary suction to draw the material into the pores of the material, thereby depositing a thin-film upon drying which penetrates the surface of the material.
- The painting application is simplistic and includes using a brush to apply a generous layer of the sol onto the surface of the concrete to be bonded to the grout. This was conducted both on a dry surface and a damp surface of concrete.
- Spraying the material included using an atomizer spray bottle to dispense the sol onto the surface.
- Dip coating is a common application method because of its predictable thickness and ease of use. This method consists of dipping the material into the sol and removing it at a controlled rate. Due to the nature of the material this method produces a film similar to slip casting.

Table 4. Description of sol application techniques.

Description of Coating Techniques		
Dip Coating		
Cast pieces		*Aggregates*
		
Dip specimen into sol material, extracting at a controlled rate to regulate the thickness of the coating.		
Spraying	Painting	Slip casting
		
Spray a layer of sol material onto the surface to be bonded.	Paint a layer of sol material onto the surface to be bonded.	Soak specimen in sol material allowing the sol to absorb up into the pores of the substrate. Upon drying, the complete surface has a coating and sol has absorbed into the concrete.

6.2. Bond testing

The improvement of adhesion between newly applied grouts and hardened concrete surfaces is one of the key aspects of improving durability and thereby increasing the life of these joints. Previous microstructure analyses of interfacial transition zones (ITZ) between aggregates and cement paste revealed that these coatings significantly decrease the porosity in the ITZ and therefore potentially decrease diffusion of undesirable ionic species. *(7)* Therefore, we hypothesized that these nanoporous thin-film coatings could result in a dramatic improvement in the durability of joints in precast concrete construction. In order to accomplish this goal, tests on 8”x8”x8” cubes joined by a 2” thick layer of grout were originally intended to simulate an actual grouted joint.

In the initial examination of thin-film application methods, however, small two-inch cubic mortar specimens were cast and grouted together with a thin grout layer after applying the sol material to the hardened cube. The bond strength was tested by shearing the two pieces apart at the grout joint as depicted in Figure 2. The application method producing the highest bond strength was painting. Because of its effectiveness and simplicity, painting was used in the remainder of the tests.

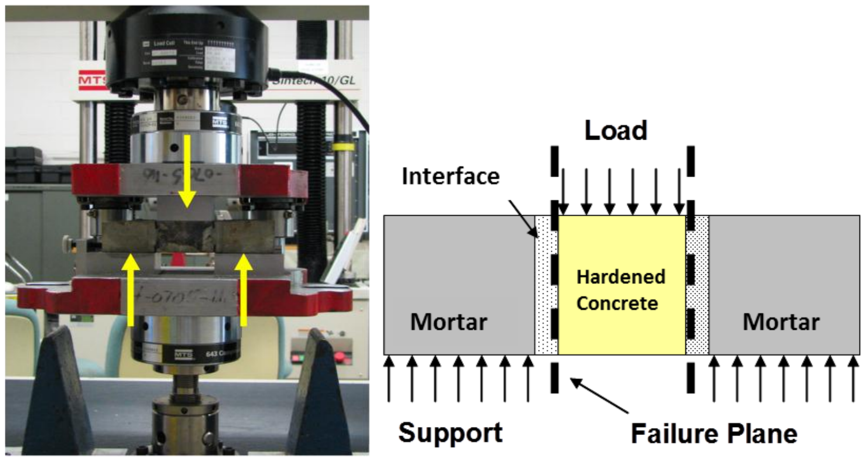


Figure 2. Testing apparatus, applied loads, and schematic view of the shear test.

Once the application method was evaluated, the remaining bond tests were conducted utilizing the ASTM C882 method rather than the 8” x 8” x 8” cubes originally envisioned. The variables tested were the nature and thickness of the nanoporous thin-film, and the surface preparation. The nanoporous thin-films can be applied in layers to increase the overall thickness of the film. Bonding tests were conducted utilizing 1, 2, and 3 layers of the thin-film.

This research also tested the possibility of eliminating the costly sandblasting of the precast concrete surface at the joints. It is expected that the high strength bond developed by the use of these nanoporous thin-films could allow the sandblasting process to be eliminated. Joints with and without sandblasted surfaces were tested.

The ASTM C882 method outlines the use of cylinders for testing grouted joints. The cylinders used for the testing measured 3 inches in diameter and 6 inches in height. The specimens were made in batches, and mixing conformed to ASTM C31. A local precast manufacturer’s concrete mix design was mimicked with the exception that this study utilized a slightly higher water cement ratio because no water reducers were used. Fresh air content and slump was measured according to ASTM C231 and ASTM C143 respectively. The mix design and fresh properties are reported in Table 5.

Table 5. Cylinder mix design used for joint bond testing.

cement	slag	coarse aggregate	fine aggregate	w/c ratio
1	0.14	1.9	1.8	0.42
			corrected fresh air content	6% ± 1%
			aggregate air content	1%
			average slump	4 in ± 1 in

Once the cylinders were cast, they were allowed to cure for 20 days at room temperature (approximately 73°F) at 100 % RH. At that point the cylinders were cut using a masonry saw and jig to comply with the shape designated in ASTM C882. The dimensions are depicted in Figure 3 along with an assembled specimen. The cut surfaces were then sand blasted to roughen the surface or left as is. The sand blasted specimens were blown off with compressed air to remove remnant sand and lose debris. Specimens were then placed in water for a maximum of 24 hours after which time a thin layer of grout (1/16 inch) was applied. The assembled cylinders were tested under a vertical load of 7500 lbs/min. This is a modification to the ASTM C882 method that calls for a loading of 15000 lbs/min so the strengths reported in this study are likely lower than with an unmodified testing protocol unless indicated.

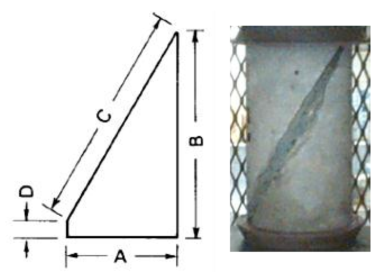


Figure 3. Dimensions of the cut cylinder are as follows: A-diameter 3.000in (75±2mm); B-height 5.598in (140±2mm); C-slant height 6.000in (150±2mm); D-base height 0.402in (10±2mm). To the right of the schematic is an image of a capped assembled cylinder.

Strength data was collected to determine if the nanoporous thin-film improved the bond, the ideal thickness of the thin-film, if the thin-film could improve bonding as much as sand blasting, and if the thin-film could improve bonding to levels of a high performance grout or commercial bonding agents. If the nanoporous thin-film could increase strength on a non-sand blasted surface to the level of a sand blasted surface, potentially this process of blasting could be skipped saving time and money. If the nanoporous thin-film could increase the strength of a bond utilizing construction grade grout to that of high performance grout, this could also save expense without compromising quality.

To determine if these potential benefits are viable, the following specimens were cast in four replicates using construction grade non-shrink grout:

- no bonding agent (control) with sand blasting,
- control without sand blasting (sawn surface),
- nanoporous thin-film with sand blasting,
- and nanoporous thin-film without sand blasting (sawn surface).

A second set of specimens was cast in an identical manner utilizing high performance non-shrink grout. As all specimens were not able to be cast in one batch, control specimens were cast and compared with each batch.

6.3. Durability Testing by Freeze-Thaw Method

Durability testing was conducted by the use of freeze-thaw cycling. Beams were cast and after 90 days, cut into 4 pieces. The pieces were bonded together after abrading the surface by sand blasting, dampening with water or sol by painting, and applying grout. The grout was allowed to cure for 7 days after which, the beam was cycled from 50°F to 10 °F following the cycling pattern of ASTM C666. The testing chamber cycled the specimens four times per day. Specimens were checked for cracks periodically. The ASTM C666 standard states that modulus of elasticity, length, and weight changes should be monitored. However, since this test was aimed at determining the durability of the grouted interfaces, these procedures were not conducted as there was no way to separate grout degradation from beam degradation. After 515 cycles without fracture, the beams began to degrade so the specimens were broken using flexure testing following ASTM C78.

6.4. Aggregate Interface Testing and Reduced Cement Content

The introduction of highly pozzolanic coatings inside concrete over the surface of the aggregates provides the opportunity to improve one of the most vulnerable areas of the concrete, namely the interface between cement and aggregate (interfacial transition zone – ITZ). *(8)* This region can be improved with the use of additives and particularly nanoporous thin films. Previously reported in Muñoz *et.al.,* it has been shown that this region can become stronger due to densification and improved microstructure. *(7)* This improved ITZ is thought to have impact on dry shrinkage and possibly creep issues found in the precast industry. Our preliminary work had demonstrated that concrete made with igneous coarse aggregate coated with highly pozzolanic materials, such as silica, showed decreased curing times as well as shrinkage. It should be noted that this variation in the drying shrinkage was achieved using a low quantity of the nanoporous thin films; the nanosilica/cement ratio was 0.003. It is expected that higher amounts of nanosilica will render significant differences in drying shrinkage compared to control samples. Less shrinkage would be a desirable attribute for precast prestressed concrete. If less shrinkage was achieved along with higher early strengths, testing to determine if reduced cement contents were feasible would be conducted. The reduced cement content testing was not conducted because the thin-films did not behave on carbonate aggregates as expected from earlier testing on igneous aggregates.

Based on preliminary results on dry shrinkage and creep testing using igneous aggregates, this study aimed at determining the potential of our nanoporous thin-films for mitigating these undesirable characteristics in precast pre-stressed concrete components.

Figure 4 should be examined for the preliminary shrinkage results when using igneous aggregates. The change in the interface region is unlikely to affect creep, but proof of little change is essential to ensure that no additional pre-stress loss will develop when the material is used in production of precast components.

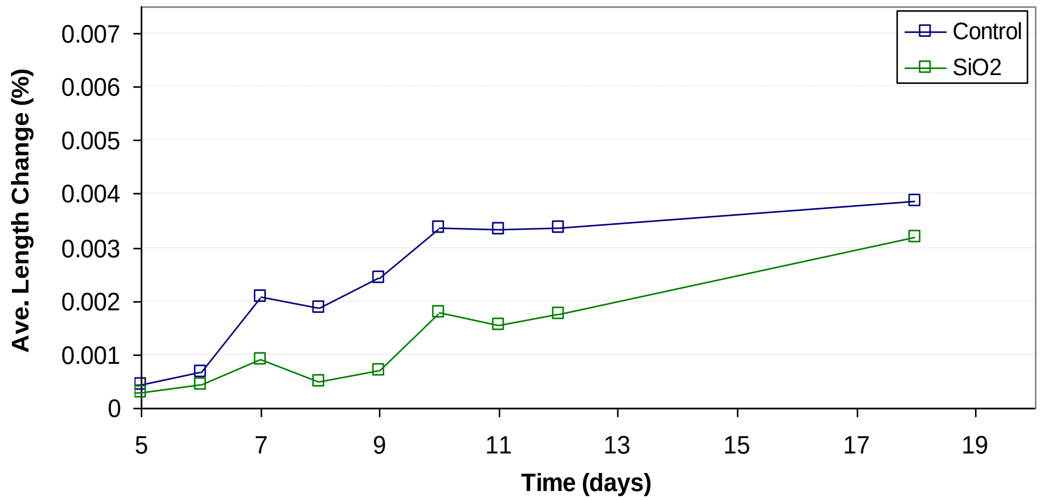


Figure 4. Initial variation in drying shrinkage for 6x12 cylinders. Control with no coated aggregates and SiO_2 with silica coated coarse (igneous) aggregates.

The first step of the current process was to determine if the nanoporous thin films affected the ITZ when using limestone aggregates. Our preliminary work indicated that the deposition of nanoporous coatings over the surface of an igneous rock significantly improved adhesion with fresh mortar. In particular, thin-films having pozzolanic activity such as silica and alumina oxide increased the shear strength between rock and cement paste up to 50% as compared with titania-coated and non-coated rock even after 14 days of curing. In preliminary studies no adverse reactions between the alumina and hydration products had been detected. These results can be seen in Figure 5

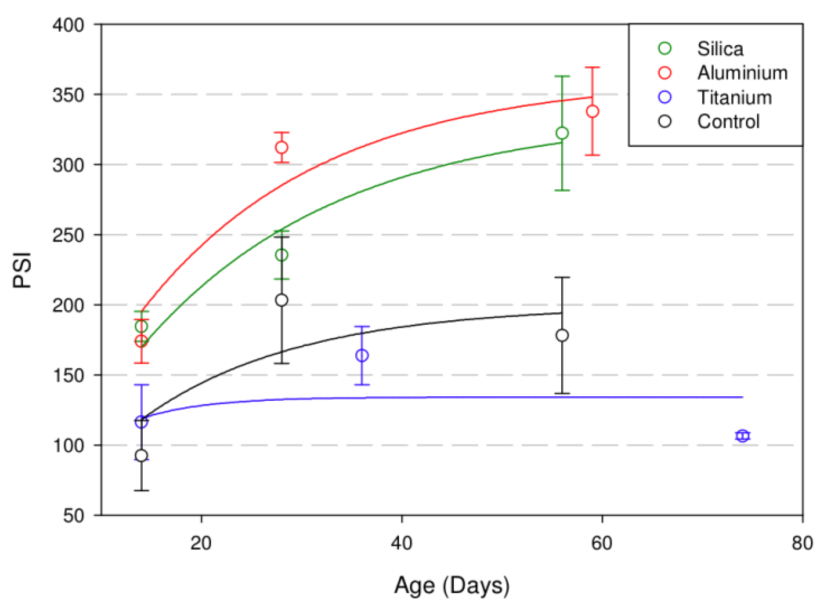


Figure 5. Shear strength trends for specimens coated with nanoparticles (using igneous aggregate).

For this portion of the study, the limestone coarse aggregate and sand grains larger than #16 sieve (1.18 mm) obtained from a local precast manufacturer were coated with the nanoporous thin films. After 24 hours of drying, cylinders were cast measuring four inches in diameter by eight inches in height in accordance with ASTM C31. The same mix design that was utilized for the bond testing (

Table 5) was used for this test. After curing for 1, 3, or 7 days, the specimens were tested in compression according to ASTM C39. The compression test results indicated that the nanoporous thin films did not improve the bond strength of the ITZ when using limestone aggregates unlike when using igneous aggregates. See Figure 6 depicting the results of the ITZ tests.

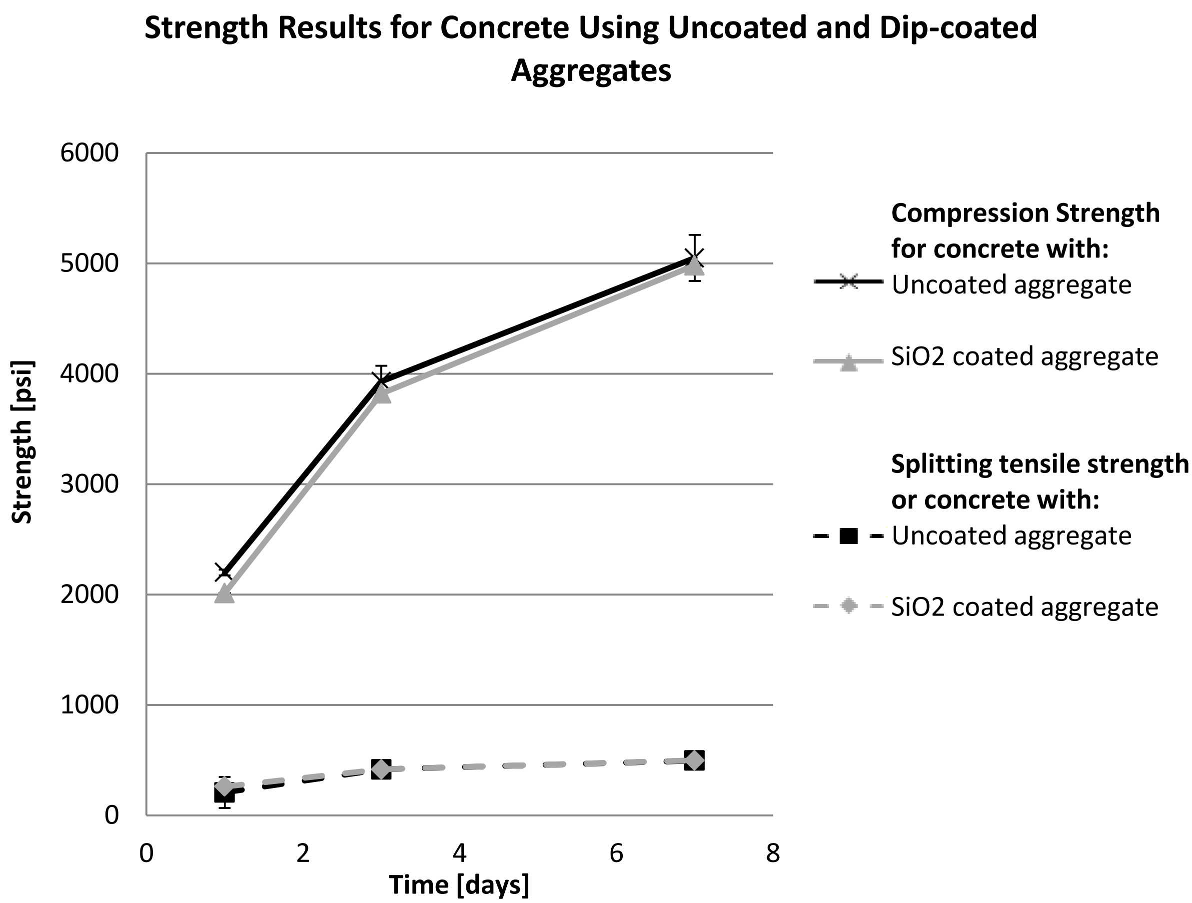


Figure 6. Compressive and splitting tensile strength results for concrete incorporating uncoated and thin-film coated limestone aggregates.

Likely these results are due to a chemical change in the aggregate surface. Igneous aggregates have a less porous surface so the sol will create a film on the surface which can react with the paste material that is in contact during the mixing and curing process. On the contrary, limestone and dolomite aggregates are porous allowing the sol to soak into the surface. These aggregates contain calcium, magnesium, and carbonates which transform when exposed to the silica nanoparticle solutions. The sol reacts with the surface of the particles and forms calcium magnesium silicates. This reaction prohibits a proper film from being deposited as is created on igneous aggregates. The newly formed aggregate surface renders these thin-films ineffective for limestone aggregates. Whereas on the surface of the igneous aggregates, the sol does not penetrate, but rather a thin-film of SiO_2 is formed on the surface ready to react with the cement paste during mixing. Limestone is the material used in the local precast industries. With no improvement, creep and shrinkage testing was aborted. However, it is possible that a process using igneous aggregates could benefit from coated aggregates.

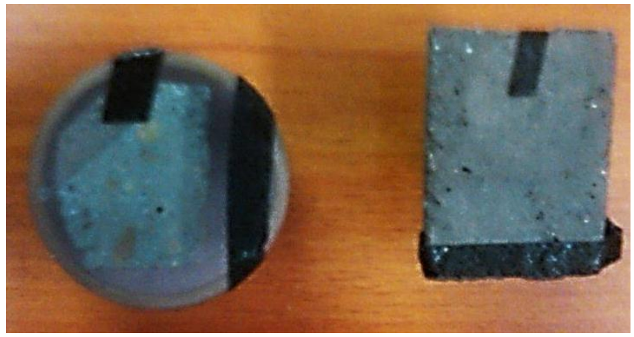
After the compression results were collected and analyzed, creep and shrinkage test were to be conducted using both control specimens and treated materials. The drying shrinkage and the creep were to be evaluated during the first year. The ability to develop rapid high early strength, to allow early pre-stressing, was to be measured. Due to the results found during the compression testing, however, further examination of thin-film coatings on limestone aggregates was abandoned and creep and shrinkage tests were not preformed. Also, testing to determine if a reduction in cement content was a viable option was not conducted. Since the compression testing utilizing coated aggregates did not show strength enhancements, it was determined the thin-films were unlikely a desirable additional aggregate treatment. As with the bond strength, it is possible that concrete made using igneous aggregates could also be studied for improved shrinkage and creep properties based on previous testing.

6.5. Interface Analysis (SEM)

This portion of the study examined the microstructural properties and chemical composition of the interface between the grout and the hardened mortar. The examination utilized a scanning electron microscope with energy dispersive spectroscopy (SEM/EDS) capabilities to discern the controlling microstructural properties responsible for the anticipated superior performance of these coatings. SEM analysis was conducted on the concrete to identify which coating applications could be best and what changes the coating caused when grout was applied to the affected surface.

The following procedure was used to prepare the SEM specimens for surface analysis which examine the morphology and general surface of the sheared interface. Specimens were obtained after the bond tests were concluded. A piece of the bond test specimen incorporating the grout and concrete interface was cut from the broken concrete cylinders using a tile saw cooled by propylene glycol to avoid altering the specimen by further hydration. The cut sample was attached to an aluminum peg using double sided carbon tape. At this point, each specimen was coated with a thin layer of gold for 80 seconds at 20mV using a Denton Vacuum Desk II sputter coater/etch unit. The samples were examined using a LEO 1530 Scanning Electron Microscope (SEM) in the back scattering electron (BSE) mode using an acceleration voltage of 15 kV and working distance of 9 mm.

The grouted specimens were also used in a similar manner for a profile analysis by means of polished specimens. These specimens were used to analyze the alterations that may have occurred over the depth of the interface. Again, after the cylinders were broken, a piece of the grouted surface and the concrete surface was cut, in this case at a location where the bond was still intact. The cut specimens were then processed in a similar manner to that of Muñoz *et al* and Balachandran. *(9, 10)* After cutting, the cut specimens were placed in acetone for 5 minutes then dried. Subsequently they were vacuum-impregnated with Buehler 2 part epoxy. The impregnated specimens were cut again using a Buehler lapidary saw. Afterwards, they were lapped and polished on a Buehler Ecomet 4 Polisher using a series of lapping paper at 330, 400, 600, and 1000 grit and finished with 0.05μm micropolish. At this point, each specimen was coated with a thin layer of gold in the same manner as the unpolished specimens, and each of the samples were examined using the same SEM system. An example of each of the



prepared samples can be seen in Figure 7.

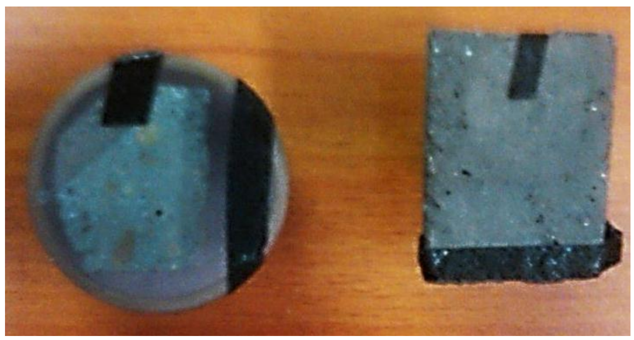


Figure 7. SEM samples for interface examination. Left - polished specimen for profile analysis; Right - unpolished specimen for surface analysis.

7. Results and Discussion

7.1. Nanoporous thin film application methods

The various methods of applying sol to form the nanoporous thin-film on the surface of the hardened concrete were tested by shearing coated, hardened concrete from grout as described in 6.2. From the analysis it was seen that the painting application method produced the greatest bond strength in the coated regions in comparison to the other methods of applications. Painting would also seem to be the most viable for industrial applications. This method was employed for the remainder of the tests. Current practices could also incorporate a roller application method. It is expected that this method would work similarly to painting, but testing would need to be conducted to verify this hypothesis.

7.2. Grout Bond Testing

The grout bond testing determined if the thin-films improved the grout bonding with the use of both construction grade and high-performance grout, the optimal thickness of the nano material, and the ideal surface preparation. A time of application parameter was also tested. It is important to note that application of a layer in this case is defined as applying the sol material and letting it dry. Applying two layers would require that the first layer be completely dried before application of the second layer. The results reported earlier in the report indicated that multiple layers are ineffective so this parameter is used to determine how long one layer should dry before the grout is applied. To determine this parameter, the sol material was applied and let to dry for the following three allotments of time: (1) 15 minutes (still visually damp), (2) 30 minutes (mostly dry but not completely) and remoistened with sol just before grouting, and (3) completely dry after one hour. Note, remoistening the sol slows the gelation process resonsible for forming the thin-film so this method is in effect applying one layer. It was determined that the material performs best when still moist from initial aplication or remoistened with sol just before grouting. Either case is similar to wetting the surface before applying the grout, often recommended by grout manufacturers. In this case, rather than using water to be sure the surface is moist just before grout application, sol is used to moisten. If the sol dried completely, it appears its bonding abilities are diminished. This is reasonable because the sol forms bonds as it turns from a sol to a gel to a thin-film. It seems the material is most successful when it is allowed to form initial bonds with the hardened concrete surface and fresh grout simultaneously rather than first to the hardened concrete and then to the fresh grout after the film has dried.

The graphs below show how the bonding utilizing thin-films was improved when compared to a control within the same batch. All results shown are an average of four tests. The standard deviations of the tests are well within the acceptable range of 365psi.

- From the bond test results it was found that the nano material performs best as a thin layer (1 application). Figure 8 shows that 1 layer of the material performs best.
- Figure 9 shows that the material improves bonding over the control specimen in both construction grade grout and high-performance grout.
- Lastly, Figure 10 shows how well the material performs without sandblasting (sawn surface) in comparison to sand blasting as a surface preparation step.

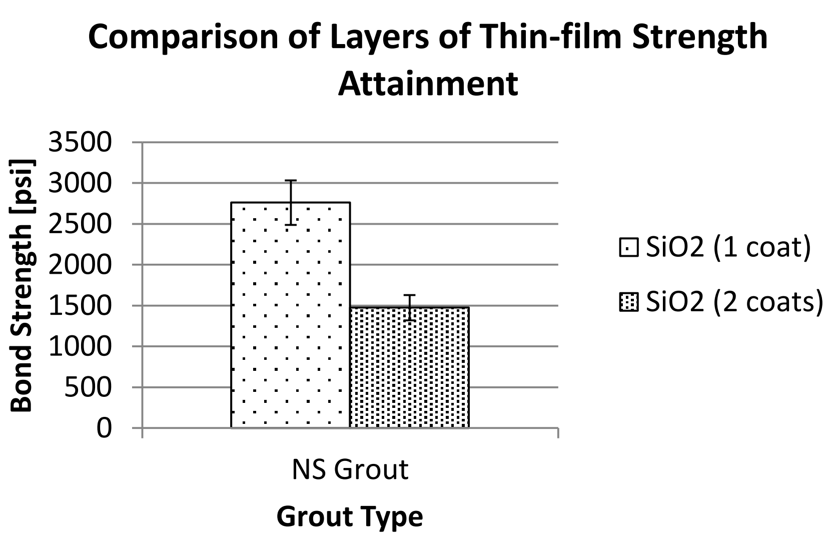


Figure 8. Strength comparison of thin-film application layers.

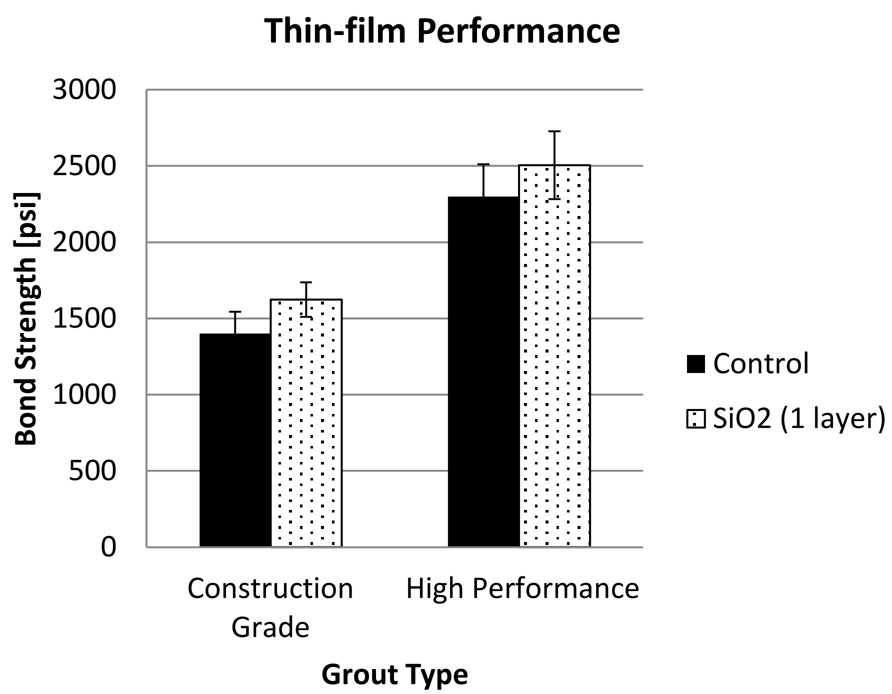


Figure 9. Effect of thin-film coating on hardened concrete, grout-joined surface using construction grade and high-performance grout.

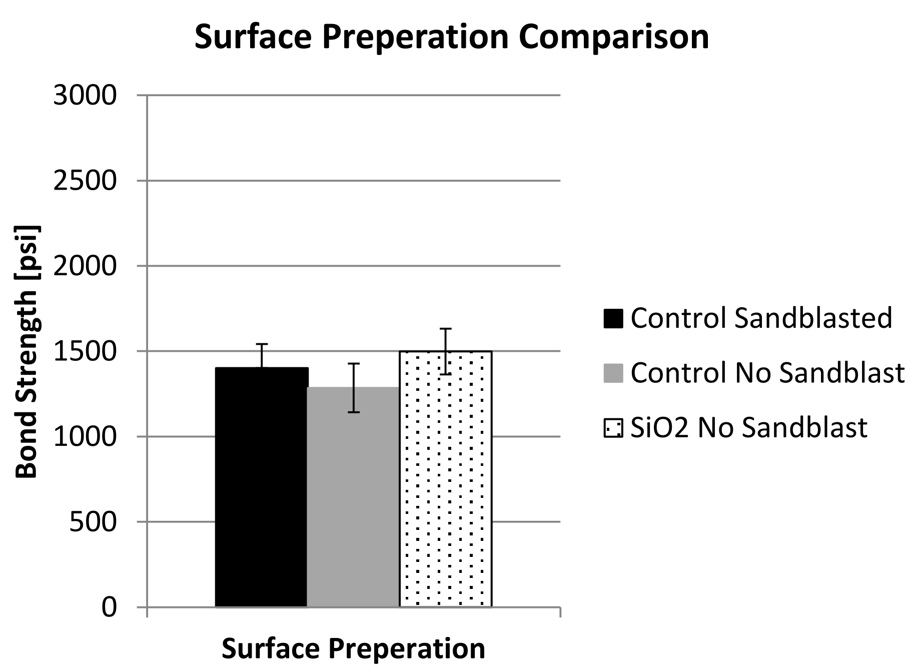


Figure 10. Effect of thin-film coating on hardened concrete, grout-joined surface when sand-blasting is not performed as a preparation step. (Control is normal grout with no surface coating. No sand blasting has a sawn surface texture.)

In addition to the ASTM C882 tests conducted, a review of current products was conducted to further compare our materials to general grout systems. Table 6 lists a few grout materials and their reported strength values. In order to make an equal comparison, additional specimens with thin films were assembled and tested using a vertical load of 15080 lbs/min, a rate within the specified range of ASTM C882. It is clear from the comparison that the thin-film coating had a high bond strength.

Table 6. Comparison of currently available grout products to thin-films from this research.

Material	Description	Reported Bonding Strength at 28 days using ASTM C882	
		Plastic	Flowable
211 SCC Plus (Sika)	one-component cementitious, silica fume and polymer-modified	2500 psi* (17.2 MPa)	
212 (Sika)	high performance cement based grout	2000psi* (13.1 MPa)	1900 psi* (13.1 MPa)
Quick 2500 (Sika)	cementitious rapid-hardening grout (hardened concrete to hardened concrete)	2700 psi (18.6 Mpa) 7 days	
CG-86 (Euclid Chem)	construction grade non-shrink grout	2300 psi* (15.8 MPa)	2500 psi* (17.2 MPa)
NS Grout (Euclid Chem)	Non-shrink, non-metalic grout	3349 psi** (23.1 MPa)	
NS Grout (Euclid Chem)	**with thin-film layer**	3664 psi** (25.3 MPa)	

*ASTM C882 was modified **Reported from this research

Other bonding agents might perform as well as this thin-film material. There are various bonding agents used in industry, the majority of which fall into two categories, latex based or epoxy based materials. Latex bonding agents can either be mixed into a mortar material or applied topically while epoxy bonding agents are more often applied topically and typically more expensive than latex agents. A topical Latex based bonding agent must produce a bonding strength of 1250 psi according to ASTM C1059 while epoxy bonding agents must produce strengths of 1790 psi.

Latex bonding agent joint strength is found following ASTM C1042, a testing method similar to ASTM C882, but the specimen is cured for 14 days rather than 7 as in the C882 test. Also, the C1042 test requires that the cylinders are composed of identically shaped triangular concrete halves, but the bonded half is made entirely from fresh grout material.

In order to compare bond strengths, our ASTM C882 strength results at 7 and 28 days were interpolated for a 14 day value. This comparison testing utilized a loading rate of 15080 lbs/min, within the ASTM loading recommendations. Also, the standard specifies the hardened concrete surfaces are to be sandblasted before grout is applied. Based on the requirements currently in place, the thin-film is a viable bonding enhancer as it can produce similar strength improvements to current market products as seen from the comparisons made in Table 7. Please note the degree of improvement when utilizing the thin-films should be made in comparison to the reported control as the bonding surfaces have been prepared in the same manner.

Table 7. Comparison of currently available bonding agent products to thin-films from this research

Material	Description	Bonding Strength	Bond Strength Standard Used	Notes
		Latex Bonding Agents Reported Technical Data		
Quikrete Concrete Bonding Adhesive	adhesive latex	> 1250 psi	ASTM C1059 compliant	
WR Meadows Intralok	latex admixture	> 1250 psi	ASTM C1059 compliant	
Euclid Chemical AKKRO-7T	latex admixture	486 psi	ASTM C321	
Euclid Chemical Tammsweld	latex	4600 psi	ASTM C1042	
Larsen Products Corp. Weld-Crete	latex	> 1250 psi	ASTM C1059 compliant	
		Epoxy Bonding Agents Reported Technical Data		
Sika Sikadur 32, Hi-Mod	epoxy	2200 psi	ASTM C882	14 day moist cure
Euclid Chemical Corr-Bond	3 component epoxy	1950 psi	ASTM C882	12 hours
Euclid Chemical Duralcrete	2 part epoxy	2385 psi	ASTM C882	14 day moist cure
		Materials from this Study		
Thin-film *		3788 psi	ASTM C882	interpolated 14 day dry cure
Control *		2718 psi	ASTM C882	interpolated 14 day dry cure

*Reported from this research using ASTM C882 specifications including load rate.
Note: ASTM C1059 requires 2150 psi; ASTM C882 requires 1790 psi +/- 365 psi

7.3. Durability Testing

The durability testing showed that the grouted surfaces were improved with nanoporous thin-films when subjected to freeze-thaw cycling. After cycling a simple flexure test was performed in order to determine if the thin-film improved the remaining bond between grout and hardened concrete.

Figure 11 below depicts the joints and broken surfaces of the specimens. In specimen “a”, every grouted surface has a thin-film layer as highlighted on the schematic in yellow. Specimen “b” combines both uncoated and coated surfaces. The coated surfaces are again highlighted in yellow. Specimen “c” does not have any thin-film surfaces. The location of the flexural break is depicted with a black dotted line on each of the 3 specimens. From the flexure testing that was performed on the beams (as no specimens failed in the joint due to freeze-thaw alone), it was determined that the bonds which included a thin-film performed better than the bonds without the film. The thin-film bonded specimens never broke where a thin-film was applied. All fractures occurred in either the concrete or in the uncoated grout-surface joint as seen in Figure 10.

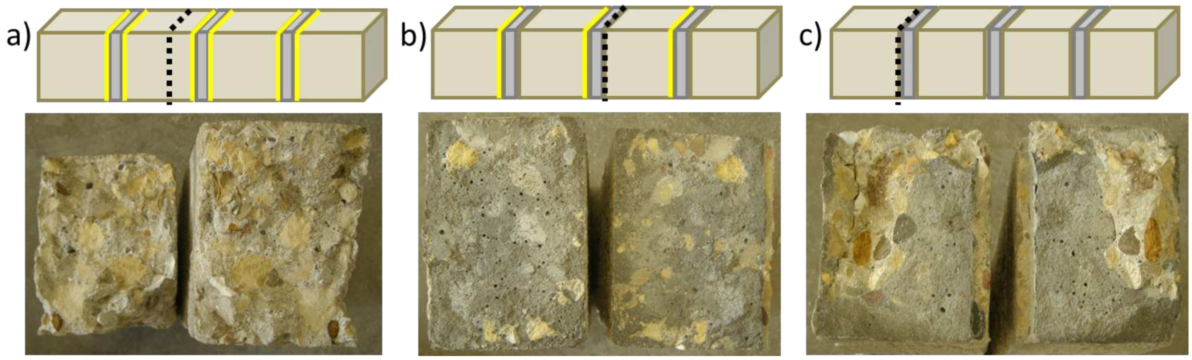


Figure 11. Depiction of the broken surfaces after flexure testing of freeze-thaw cycled specimens. A schematic representation of the assembled beams is shown above the broken surfaces. The surfaces with thin-films are highlighted in yellow and the fissure is shown as a broken black line.

7.4. Interface Microstructural Analysis (SEM)

An SEM analysis was conducted to discern any major morphological differences in the thin-film coated specimens from the control specimens. An EDS analysis was completed to determine if there were any microstructural differences in the joints.

From the SEM analysis it was evident the concrete that contained a thin-film showed more deformation on the sheared surface. It is possible, when the specimens without a thin-film were sheared, the grout "popped" off. However, in the specimens utilizing a thin-film, adhesion was stronger so the shear forces were greater causing more final deformation of the concrete side. This can be seen at low magnification in Figure 12.

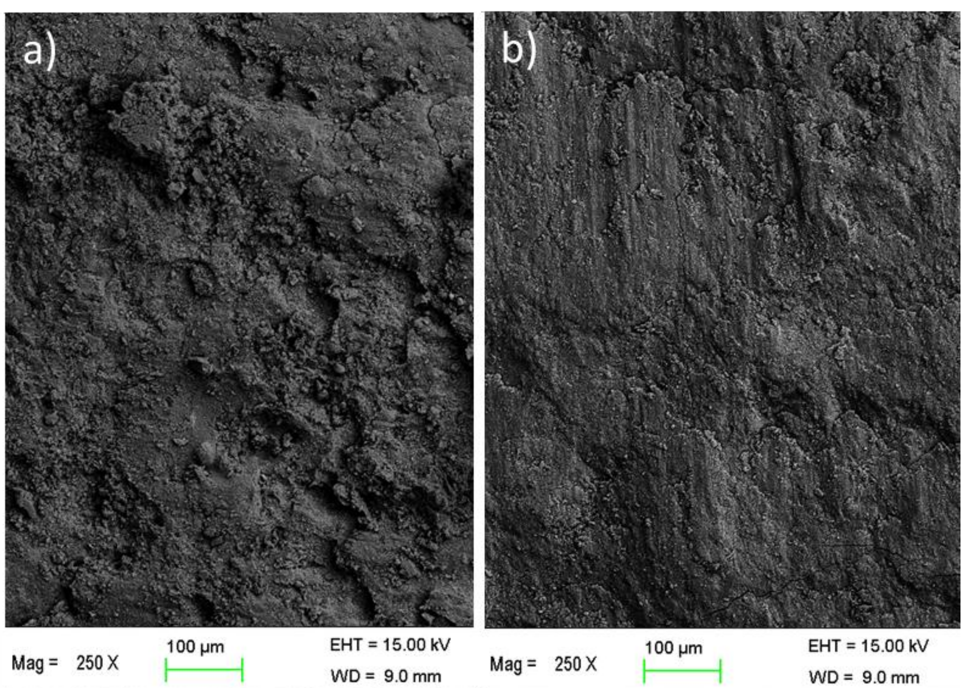


Figure 12. SEM analysis of the surface morphology from the concrete side of the sheared grout-concrete bond. a) control specimen concrete side b) thin-film specimen concrete side deformed.

An EDS analysis was also conducted to determine the chemical character of the microstructure at the joint. A minimum of eight scans were taken of the general bulk matrix of concrete in each of the control and the thin-film specimens. The bulk EDS analysis showed that the thin-film coated specimens had CSH everywhere whereas the control had some areas of aluminum and some areas of aluminum and sulfur. A representative scan of this analysis is shown in Figure 13. It is possible that the thin-film promoted the growth of CSH over the growth of any ettringite or other type of crystal due to the high content of silica found within the thin-film material. An analysis of the grout side for the sheared specimen was also conducted using SEM/EDS. The grout sides of both the thin-film specimen and the control specimen look similar. The thin-film seems to impact the hardened concrete surface more than the grout material during the bonding process.

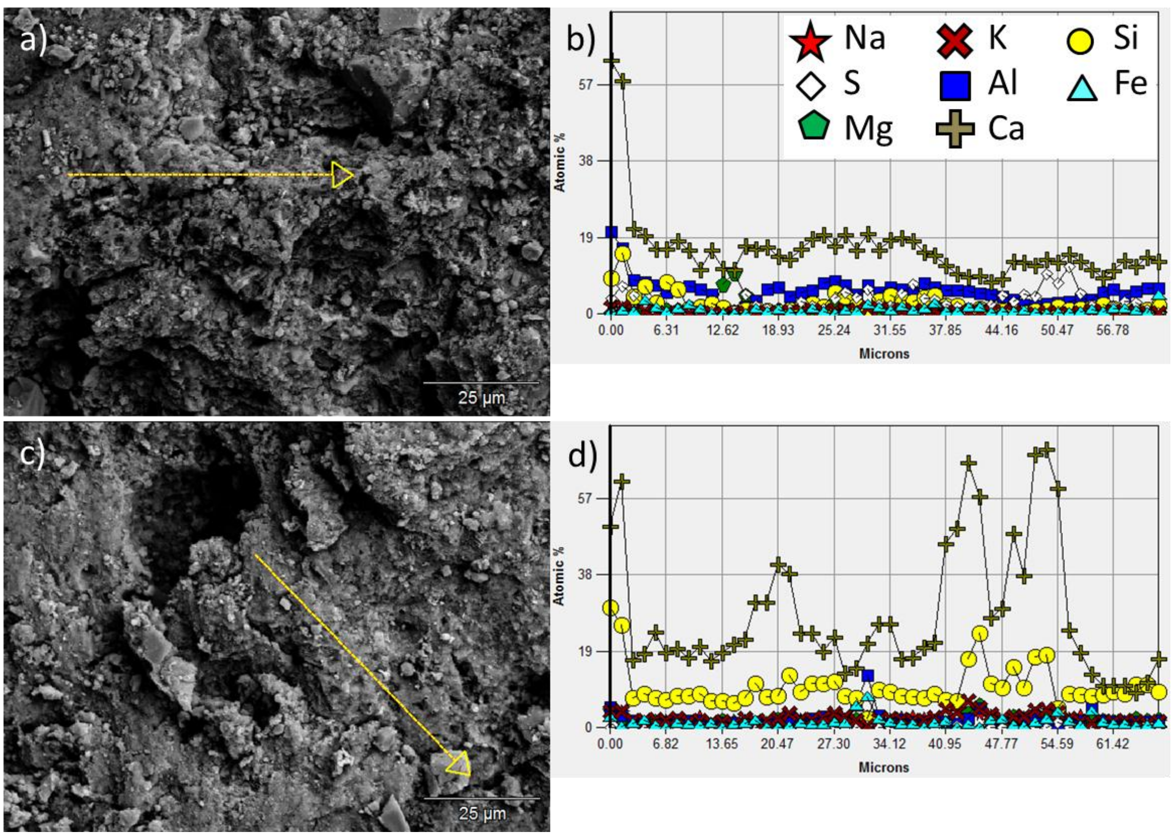


Figure 13. Images are of the concrete side of the grout bonded surface after shearing the bond. The graphs to the right of the image show the EDS analysis of the scan taken along the yellow line in the image with the Atomic % on the vertical axis and the distance on the horizontal axis. "a" and "b" show the scan from the control specimen, and "c" and "d" show the thin-film coated specimen.

Further investigation of the alterations occurring at this bonding interface was performed through a profile analysis. An examination of the concrete into the grout material was obtained to further investigate the penetration of the thin-film and modifications it induced in the concrete matrix. This investigation helps to identify whether the thin-film is promoting greater hydration and creation of CSH than the control as this would likely be the reason for the increased strength.

The profile analysis investigated to what depth the thin-films altered both the hardened concrete surface and grout. Analysis was conducted on specimens which had not been sand blasted so penetration could be more easily measured. Specimens were composed of the hardened concrete and grout that was still intact for both samples containing a thin-film layer and samples without this layer. Scans were taken from the grout into the hardened concrete surface. The bonded area was identified visually in each scan. An example of a low magnification image of the hardened concrete to grout interface is seen in Figure 14. The distances were normalized so any elevated levels of chemicals could be linked to the distance from the bonded region. Also, by normalizing the distances, averages of the chemical analysis could be produced.

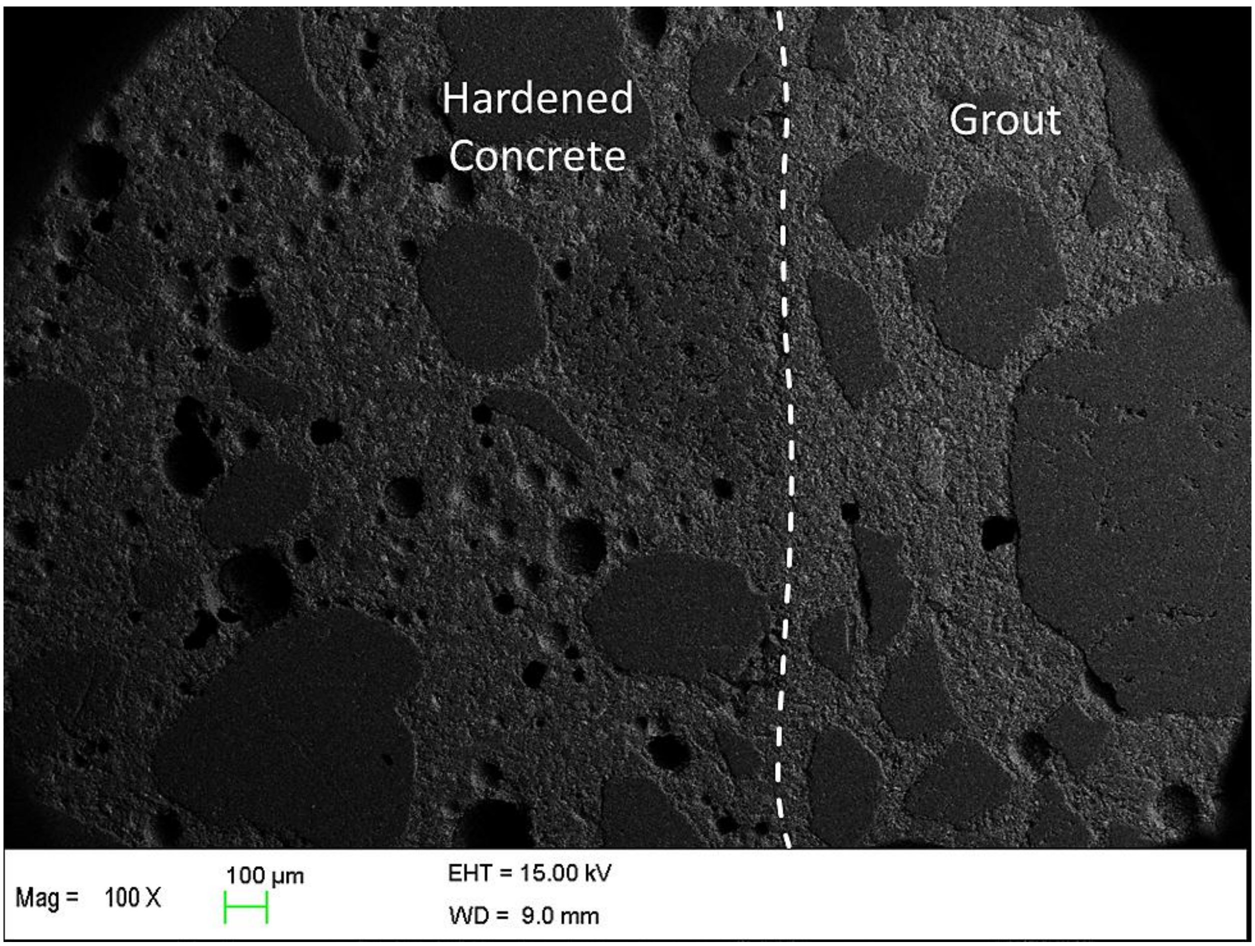


Figure 14. Low magnification image of hardened concrete to grout profile analysis.

From the analysis, penetration of the thin-film could not be identified as the area containing the thin-film matched the control surface. In other words, high levels of silica were not detected in the bonded region. Although the thin-film layer could be in the range of 50µm, a measureable distance in SEM, the silica likely absorbed and reacted with the concrete and grout so a physical layer was not readily seen. With anticipated expectations of this possibility, microstructural alterations of the concrete were investigated. However, noticeable variations were not detected. EDS was employed to determine if chemical alterations occurred. Possible higher levels of calcium silica hydrate (CSH) were sought, but chemical analysis did not display any spikes in the region of the affected area. Figure 15 shows the predicted EDS analysis of the bonded region. Figure 16 shows an example of the actual scans conducted in the bonded region. Figure 17 shows the averaged chemical analysis for the control and thin-film bond.

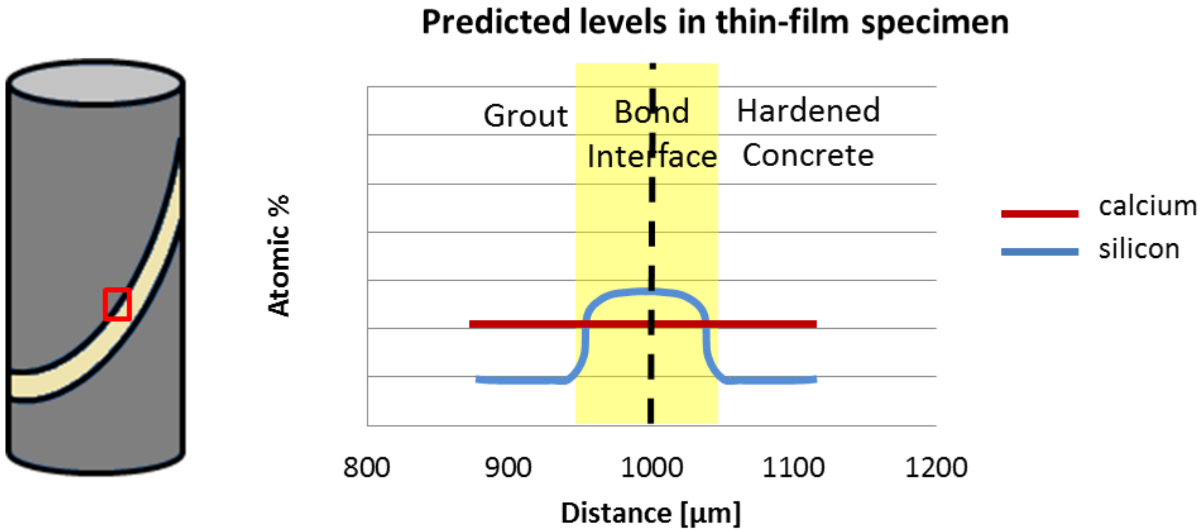


Figure 15. Predicted analysis of the hardened concrete to grout interface for specimens containing thin-films.

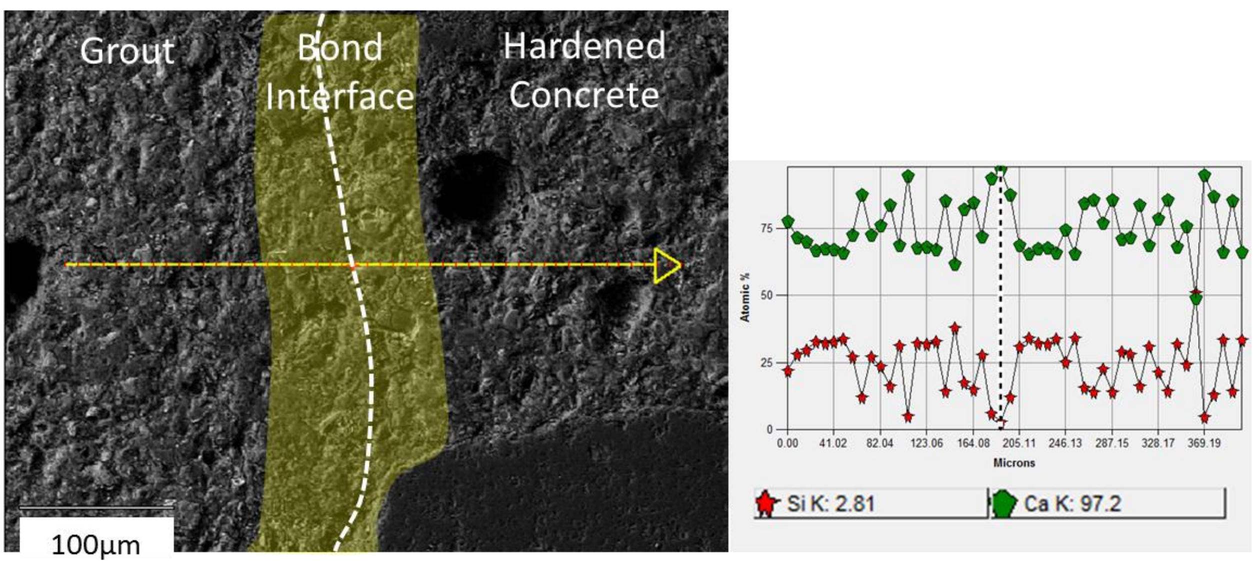


Figure 16. SEM analysis of interface between hardened concrete and grout containing a thin-film.

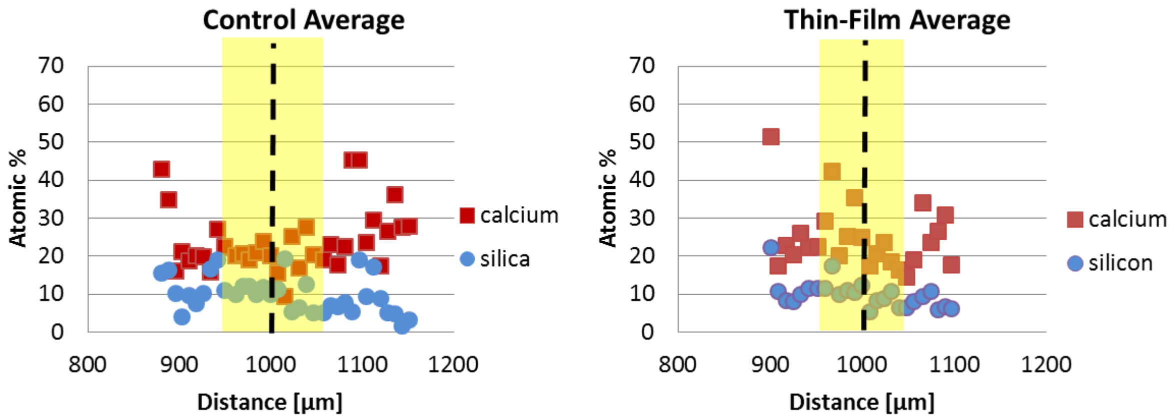


Figure 17. Comparison of control (left) and thin-film (right) averaged EDS analysis of hardened concrete to grout bond.

It can be seen from Figure 17 that the average levels calcium and silica are indistinguishable from one another when comparing the control and thin-film specimens. This analysis shows the thin-films are likely performing as a bonding enhancer unlike typical bonding agents. Likely the thin-film is consumed in the hydration process of the grout. Possibly the thin-film is helping the grout form a normal bonding microstructure, just stronger. It is not glue per say, but rather a material which promotes the growth of normal bonding microstructure components. This is a positive result because there is not a fear of deteriorating the material or introducing any foreign species into the matrix of the concrete. The thin-films are helping to promote growth of normal concrete microstructural features, an expected result as the film itself is a pozzolanic material.

7.5. Cost Analysis

The thin-films are applied to hardened concrete surfaces by means of a water-thin sol. This material has an approximate coverage rate of 230 ft^2/gal. The material has an approximate density of 40 grams/liter of solid material. It could be sold for an estimated cost of $100/gal, and less if produced in large batches. Given the strength and coverage rate of this material, it is comparable to the current materials. It has a cost close to epoxy systems, but a much higher coverage rate than typical epoxy systems, almost 3 times higher. The coverage area is close to the rate of a latex agent, but has strength enhancing capabilities comparable to epoxy systems. An additional cost saving strategy could be to eliminate the sand-blasting process as the material can produce strengths above a sand blasted surface when applied to a non-sand blasted surface. However it is recommended to use the thin-films in conjunction with sand blasting for maximum strength.

8. Conclusions

In conclusion, the thin-films increased the bonding strength of the hardened concrete to fresh grout. Table 8 summarizes the strength improvements seen in this study. Furthermore, the SEM analysis indicates that the thin-film has altered the hardened concrete surface. The research also showed improvements in durability testing with the use of the thin-films.

The thin-films are simply applied to the hardened concrete similar to how one would apply a bonding agent and wet the hardened surface before grout application. The thin-films seem to improve bonding the greatest degree when applied in conjunction with construction grade grout materials. This is the more likely area to use a bond enhancing step as high performance grouts are more costly so additional bonding steps are less desirable. If the industry can use a low cost thin-film which can improve performance it can utilize general construction grout materials more often.

This study also showed that the thin-film could increase bonding strength as much as sand blasting. This could reduce a processing step and save the money used in sand blasting. Reported data using similar testing methods showed that by sandblasting and exposing aggregates, the bonding strength can be improved by an average of 26%. *(11)* In this study, application of a thin-film to a smooth, hardened concrete surface increased the bonding strength by 17% of the comparable smooth, sawn control, and 7% over the sand blasted surface. Removing the sand blasting process could be a cost savings potential, however, it is recommended to use the thin-films in conjunction with sand blasting for maximum strength. In general it was seen that the thin-film material can perform within the ASTM C882 or C1059 specifications for epoxy or latex bonding agents respectively.

Table 8. Percent improvements in bonding strength with the use of thin-films.

grout type	surface preparation	strength increase with thin-film compared to control
construction grade	sand blasted	16%
high performance	sand blasted	9%
construction grade	not sand blasted	17%

9. Recommendations

Use these thin-film materials to improve the bonding strength of hardened concrete to fresh grout. Apply the sol using a paint brush, and apply fresh grout while the surface is still damp with sol. It is recommended the sol be applied, allowed to absorb into the hardened concrete surface so that the concrete interface is at a surface saturated moist state, and then fresh grout applied. If the sol material on the hardened concrete is allowed to dry, dampen the surface with the sol in low quantities so the surface remains damp. Do not reapply a thick layer to the hardened concrete surface as this tends to form a weaker bond. Then follow normal curing procedures. The bond will be stronger than bonds formed without the thin-films.

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University of Wisconsin-Madison
Department of Civil and Environmental Engineering
1410 Engineering Drive, Room 270
Madison, WI 53706
Phone: 608-263-3175
Fax: 608-263-2512
cfire.wistrans.org