Acceptance Tests for Surface Characteristics of Steel Strands in Prestressed Concrete

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NATIONAL COOPERATIVE HIGHWAY RESEARCH PROGRAM

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Acceptance Tests for Surface Characteristics of Steel Strands in Prestressed Concrete

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FOREWORD

By Edward T. Harrigan
Staff Officer
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This report provides practical tests to identify and measure residues on the surface of steel pre-stressing strands and to establish thresholds for residue types found to affect the strength of the strand's bond to concrete. Key products presented here are four test methods suitable for use in a quality assurance program for the manufacture of steel pre-stressing strand. The report will be of particular interest to bridge engineers in state highway agencies and industry and to suppliers of steel pre-stressing strand.

In the 1980s, tests conducted on uncoated and epoxy-coated steel pre-stressing strands found that the measured transfer and development lengths of the uncoated strands were in excess of lengths computed using the equation in the AASHTO standard specifications.

These results raised concerns that the AASHTO equation was not conservative enough for modern strands with larger diameter and higher ultimate tensile strength. As a result, a 60% increase in the development length over the length computed using the AASHTO equation was implemented.

As part of the investigation of possible causes for the discrepancy between measured and computed development lengths, attention was drawn to variations in the surface condition of strands used throughout the United States. The presence of surface residues—e.g., rust, lubricants used in manufacturing processes, or corrosion inhibitors—suggested a possible source for the wide scatter that was observed in bond test data.

There is a need for (1) practical tests to quantify the surface condition of steel pre-stressing strands and (2) quality assurance criteria based on these tests to control the effect of strand surface condition on bond strength. Under NCHRP Project 10-62, "Acceptance Tests for Surface Characteristics of Steel Strands in Pre-Stressed Concrete," Wiss, Janney, Elstner Associates, Inc. was assigned the tasks of (1) identifying the common types of residues on steel pre-stressing strands, (2) developing practical tests to measure deleterious strand residues and estimate their effect on the performance of the strand-concrete bond, and (3) preparing a practical quality assurance procedure for strand acceptance.

The research team designed and conducted a program of laboratory experiments to accomplish these tasks. Residues commonly found on pre-stressing strands after manufacture and as a result of the strand exposure to the elements during storage and transport were identified. A wide variety of chemical and physical test methods for measuring the levels of such residues were evaluated for accuracy, speed, and ease of use. The results of promising methods were correlated to the results of transfer length testing and limited mechanical pull out testing to establish performance thresholds.

Four test methods showed satisfactory correlation with bond in concrete or mortar or both and are recommended for inclusion in a quality assurance program for steel prestressing strand:

1. Weight Loss on Ignition,
2. Contact Angle Measurement After Lime Dip,
3. Change in Corrosion Potential, and
4. Organic Residue Extraction with Fourier Transform Infrared Analysis.

Further, the following combinations of the methods above provided better correlation with bond in concrete or mortar or both than the methods used individually:

1. Weight Loss on Ignition combined with Contact Angle Measurement After Lime Dip and Change in Corrosion Potential,
2. Contact Angle Measurement After Lime Dip combined with Change in Corrosion Potential, and
3. Contact Angle Measurement After Lime Dip combined with Organic Residue Extraction (100% stearate only).

This report presents the main text of the contractor's final report of the project and two appendices: (1) Evaluation of Mechanical and Chemical Test Methods (Appendix B) and (2) Specifications for Standard Surface Test Methods (Appendix C). The following three appendices are available upon request to NCHRP:

1. Appendix A: Review of Strand Bond Literature;
2. Appendix D: Supplemental Investigations of Strand Bond; and
3. Appendix E: Bibliography of Strand Bond.

CONTENTS

SUMMARY

Acceptance Tests for Surface Characteristics of Steel Strands in Prestressed Concrete

The predictable transfer of prestressing force from strand to concrete is essential for the reliable performance of prestressed concrete. Residual films of lubricant and other contaminants remaining on the strand surface after manufacture have been shown to reduce the bond between the concrete and steel. A set of quality control (QC) procedures have been developed for use by strand manufacturers or their customers as part of a routine QC program to enable rapid assessment of factors that affect bond quality.

The development of the QC test methods was based on an experimental program that evaluated a number of proposed methods for testing the physical and chemical properties of the strand surface. These methods were designed to be performed more quickly and easily than mechanical pull-out testing. This program included limited mechanical pull-out testing and extensive surface and chemical testing. These tests, as well as transfer length tests, were conducted on a range of strand sources to establish correlations between the proposed QC tests methods and bond quality. Although concrete pull-out testing appears to correlate best with transfer length, the evaluation of correlations between the proposed QC test methods and bond quality was based on available mortar pull-out test results for the strand sources.

The four test methods that showed the best correlation with bond in concrete, mortar, or both, and that are recommended for inclusion in future QC programs are as follows:

- Weight Loss on Ignition (LOI) (QC-I),
- Contact Angle Measurement after Lime Dip (QC-I),
- Change in Corrosion Potential (QC-I), and
- Organic Residue Extraction with Fourier Transform Infrared (FTIR) Spectroscopy Analysis (QC-II).

The first three methods have been designated Level I QC tests (QC-I), while the fourth is a Level II QC test (QC-II). The Level I tests are quicker and less complex than Level II tests.

Regression analyses on combinations of these methods also were performed to evaluate their ability to predict bond. The three combinations that showed the best correlation, based on the adjusted coefficient of determination (R^2 *adj.*), were as follows:

- Weight Loss on Ignition (LOI) & Contact Angle Measurement after Lime Dip & Change in Corrosion Potential;
- Contact Angle Measurement after Lime Dip & Change in Corrosion Potential; and
- Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only).

The adjusted coefficients of determination for each of these combinations were higher than the coefficients of determination for the single-predictor regression models.

Acceptance thresholds for two of these individual QC tests and all of the combinations were developed. These thresholds were based on: (1) prediction intervals for the regression calculated from the available data and (2) the minimum criterion for mortar pull-out stress adopted by NASPA. Thresholds for multiple-predictor regressions are not determined using the same procedure used for single-predictor regressions. Instead, the lower bound on the prediction interval must be calculated for each combination of test results. A computational tool in the form of a Microsoft Excel™ spreadsheet had to be developed for this purpose.

It is recommended that the three Level I QC tests be adopted as part of a routine QC program for strand producers. To supplement the quarterly mortar pull-out testing program currently being performed by producers supplying the domestic market, this testing should be conducted on a weekly basis for each size of strand produced. Regular QC testing would decrease the likelihood that poor bonding strand would reach the market. When lots of strand are produced that exhibit suspicious behavior, this should then prompt additional testing using the Level II organic residue extraction test and mechanical pull-out testing.

The determination of thresholds for two of the individual QC tests (Contact Angle Measurement after Lime Dip and Change in Corrosion Potential of Strand) was possible based on the relationships between the QC test and the mortar pull-out test results for this sample set, and these thresholds are conservative. The available data were not sufficient to allow threshold determination for the other two individual methods with the same constraints. The threshold determination process is governed by the prediction intervals, which are determined by the uncertainty in the regression results.

Although a significant amount of work and scientific rigor has gone into the development of the thresholds, they should not be considered absolute. Additional data collected should be used to increase the statistical confidence in the bond-QC test result relationships and may allow the development of less restrictive thresholds in the future. One potential source of additional information is NASPA's quarterly pull-out testing program, which began in 2007. Conducting the recommended QC tests on the strand samples would provide valuable information for further refining the regression relationships, even if all the samples tested demonstrated adequate or better bond properties.

Another possible means for implementing these test methods is the development of process-specific regression models and thresholds. The dataset for this study included strand sources manufactured with a number of different pretreatment and lubricant processes. Limiting the data included in the regression analysis to data collected from a single manufacturing process, such as might be done by an individual strand producer, is expected to significantly improve the correlation of the QC test methods, since the QC test results would be influenced mainly by variations in lubricant and pretreatment concentration with chemical composition remaining relatively constant. This improved correlation would also permit less restrictive thresholds.

CHAPTER 1

Introduction

The transfer of prestressing force from prestressed concrete (PC) strand to concrete over a predictable length is essential for the reliable performance of prestressed concrete. It also is essential that lubricants be used in the wiredrawing process to manufacture PC strand so that the process is cost effective and does not damage the wire. However, residual films of lubricant and other contaminants remaining on the strand surface after manufacture are known to be highly effective in preventing the cementitious bond developed between the concrete and steel. Residual films on wire can be difficult to remove since some residual films, including those resulting from calcium stearate-based lubricants, are water insoluble.

The residual film that persists on strand is influenced by many factors, including the condition of the raw rod stock, the pretreatment and lubrication materials and procedures, and the production system, particularly the die condition and line speed. Therefore, to produce strand that reliably bonds with concrete in prestressed elements, the manufacturing process must be carefully controlled, and the appropriate surface treatments must be selected throughout the wire drawing and stranding processes. Finally, a set of testing procedures to be used as part of a routine quality control (QC) program is needed to rapidly assess factors that are known to affect bond properties.

Background to Strand Bond Uncertainty

Tests conducted in the early 1980s on uncoated and epoxy-coated steel strands found that the measured transfer and development lengths of the uncoated strands were in excess of lengths computed using the equation found in the AASHTO standard specifications.

Publication of these results led to concerns by FHWA and others that the AASHTO equations for transfer and development lengths were not conservative enough for modern strands with larger diameters and higher ultimate tensile strengths. As a result, an increase of the development length by 60% over the length computed using the AASHTO equation was mandated in 1988 (U.S. DOT 1988).

Numerous test programs conducted by industry, academia, and state agencies in response to this mandate, including a major study by FHWA (Lane 1998), provided data to reevaluate the transfer and development length performance of modern strands. Beam test data summarized by FHWA in 1998 led to the formulation of new proposed equations for both transfer and development lengths for uncoated strands.

Following completion of these tests, the industry became aware of variations in the surface condition of strands produced in the United States. The presence of surface residues arising from varying manufacturing processes suggested a possible source for the wide scatter that was observed in the test data; however, the conclusions were not definitive. The North American prestressing strand producers created an organization called the North American Strand Producers Association (NASPA) to study the problem and recommend solutions to its members. It was primarily concluded that calcium stearate, a non-water soluble metallic soap used as a lubricant in the initial wiredrawing process, was not being consistently or adequately removed during the stress relieving and strand rinsing operations. Therefore, manufacturers were advised to use an alternative lubricating soap, namely sodium stearate, which is water soluble. Other changes, not shared with those outside the strand production industry, may also have been instituted.

In spite of the efforts made by NASPA to date, there are still occasional incidents where strand bond problems occur. From the late 1980s to the present, the research team has investigated and is aware of other investigations involving cases of strand slippage problems. Accordingly, rapid QC tests that could be performed frequently were judged to be needed to assess the acceptability of strand surfaces meant to be bonded to concrete. It was the goal of this research project to develop such methods.

A more complete description of the history of strand bond-related research is given in Appendix A.

Manufacture and Surface Condition of Prestressing Strand

The production of prestressing strand, as observed at one manufacturing facility, includes the following basic components (Figure 1):

- Coils of nominal 1/2-in. AISI C1080 steel rod stock are cleaned and pretreated. This can be achieved using a variety of methods; in one example, the raw steel coils are cleaned by pickling (dipped in acid, then rinsed with water) and then phosphate treated (coils are submerged in a zinc phosphate solution, then rinsed in water and dried).
- Rods from several coils are butt welded end to end, and then fed into the wiredrawing machine.
- The wire drawing machine consists of a series of eight successively smaller dies that draw down the rod stock to wire with a diameter of about one-third the strand diameter. For 1/2-in. diameter strand, the center, or "king" wire, has a diameter of 0.174 in., approximately 5% greater than that of the six outer wires. Integral with each die is a box containing wiredrawing lubricant that the wire passes through before entering the die. (This arrangement allows for different lubricants to be used with different dies, and it is not uncommon to use a different lubricant for the first ["ripper"] die than for subsequent dies.) Both the die and the capstan, which pulls the wire through the die, are water cooled since the performance of the lubricant and properties of the wire are very sensitive to temperature. At the end of the machine, the individual wire is spooled.
- The spools of wire are installed in a skip strander. In this machine, the six outer wires are helically wound around the king wire to form the seven-wire strand.
- The strand is drawn under tension through an induction furnace. This stage imparts the stress relieving and low-relaxation properties to the strand. The plant visited has a box installed at the end of the induction furnace that contains equipment to wash and cool the strand.
- A venting hood is situated between the furnace and the final cooling bath to draw off any vapors created by treatment in the induction furnace.
- The strand is cooled in a spray chamber with recirculated water. This process may remove some additional wiredrawing lubricants if they are sufficiently water soluble.
- The strand is spooled, packaged, warehoused, and shipped to customers.

Pretreatment and Lubrication

The character and quantity of the residual film on the prestressing strand is governed by the pretreatment of the rod, lubricants used during manufacture, and postdrawing processes including stress relieving. The purpose of the pretreatment, which typically is conducted on the spooled rod stock, is to provide a foundation for the drawing lubricants. The drawing lubricants are applied to minimize friction, which dictates the amount of energy required for drawing, and to prolong the life of the dies.

The cleaning and pretreatment performed on the rod stock are critical and influence all remaining steps in the production process. This is because the surface quality of the resultant steel that must be subsequently drawn through the dies governs the lubricant selection and effectiveness.

For strand production, the most common coating applied to rod stock during the pretreatment process is zinc phosphate, which serves as a carrier for the lubricants applied during the wiredrawing process. The phosphating process often consists of some of the following steps: (1) mechanical cleaning, (2) pickling—cleaning in acid, (3) rinsing—using neutralizing lime solution or water to remove all chloride if hydrochloric acid solution (HCl) is used for pickling, (4) activating prerinse—a dip in a solution or suspension of titanium phosphate to produce a thinner zinc phosphate layer, and (5) zinc phosphating in a solution that may also contain orthophosphoric acids and sodium nitrate (Wire Industry 1992, Liberti 1994).

The cleaning (i.e., descaling) of the wire may be done mechanically or chemically. Mechanical methods of descaling include reverse bending, belt sanding, or shot blasting. Chemical descaling usually involves a soak in an acid solution and is typically more effective than mechanical methods. As a result, acid baths are most common in strand plants, since high carbon steel is difficult to clean using other methods.

Zinc phosphating deposits a thin layer of zinc phosphate crystals on clean wire to act as substrate for dry lubricating soaps and to provide a barrier to metal-to-metal contact. The best results are achieved with a dense layer of fine crystals. The phosphating process is influenced by temperature and acidity of the phosphate bath, since these factors influence the phosphate solubilities. A longer immersion time will produce a heavier coating (Liberti 1994). It has been observed that zinc phosphate/lubricant coated wires have better corrosion performance suggesting that a residual film may be left on the wire produced with such a pretreatment (Rutledge 1974). Phosphate coatings are themselves difficult to remove (Wire Industry 1992).

Borax and lime may also be used for pretreatment, either alone or in combination with zinc phosphate. A fundamental difference between phosphate coatings and other coatings

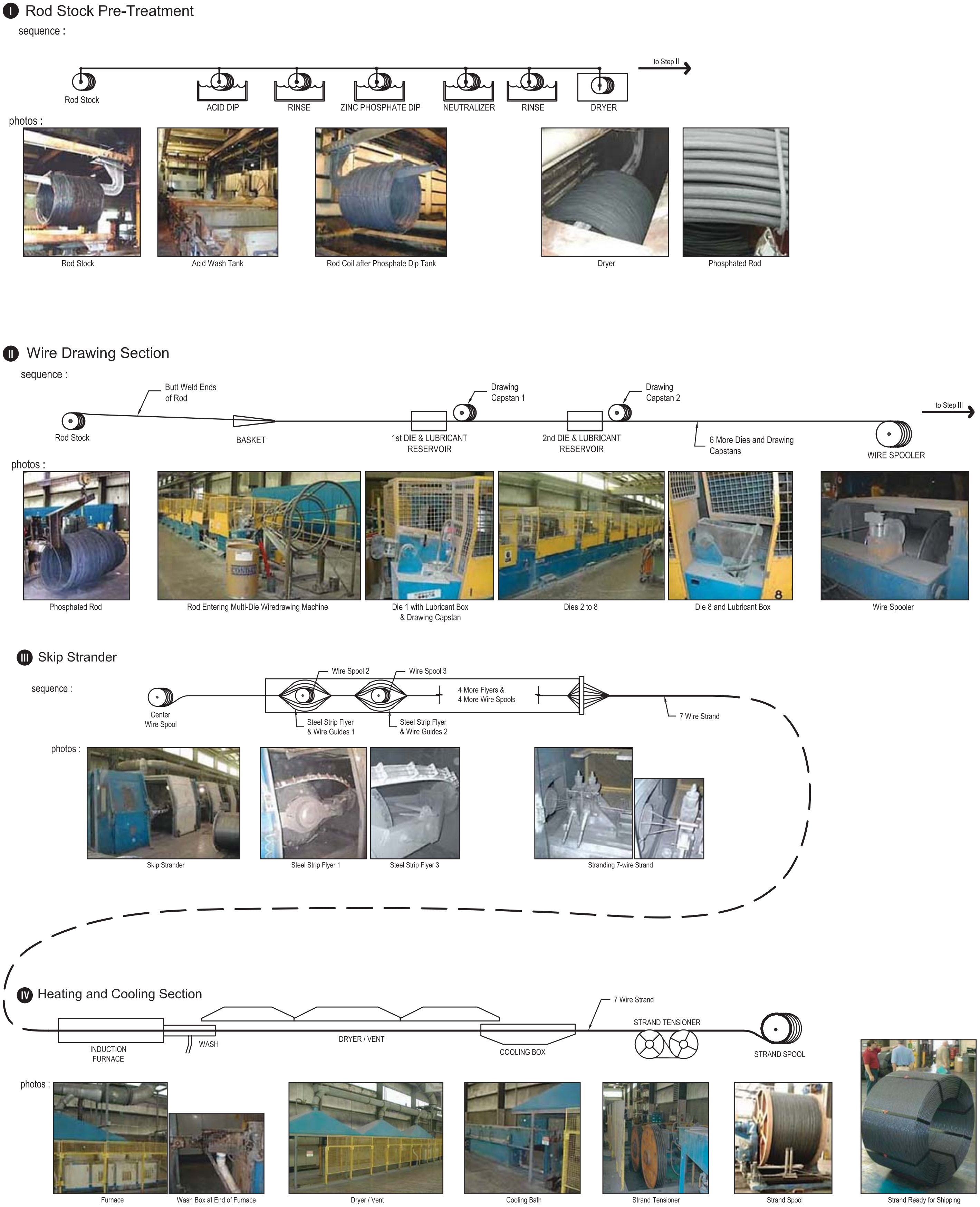

Figure 1. Schematic of strand manufacturing process.

such as borax is that the phosphate reacts with the steel surface to provide the foundation for lubricant, but the borax does not. Borax is more likely to be removed during processing than phosphate coatings but is less effective at aiding lubrication (Hajare 1998). Therefore, although a non-phosphate-based process is used in at least one North American manufacturer's plant for pollution control reasons, that approach is not common. The most common combination of pretreatments is to follow the zinc phosphate treatment with a bath in hot borax solution prior to drying. The alkalinity of borax serves to neutralize acid from the pickling process not removed by washing. However, borax has some disadvantages including its highly hygroscopic nature (i.e., it absorbs water) (Wire Industry 1992). It should also be noted that both zinc and borax can retard Portland cement hydration.

Following the pretreatment processes, lubricant is applied to the wire at each die during the drawing process. Dry lubricants are used exclusively by strand manufacturers in the United States in the wiredrawing process for strand. The lubricity agent in such lubricants is typically a chemical compound of a metallic element (calcium, sodium, aluminum, potassium, barium, or combinations of these) plus a fatty acid (such as stearic acid). The dry lubricants may also contain borates. It is commonly felt that calcium-based dry lubricants provide the lubricating properties needed for wiredrawing more cheaply and effectively than any other material.

Powdered wiredrawing lubricants are usually classified by their solubility in water. Insoluble lubricants are usually calcium based (e.g., calcium stearates; partially soluble lubricants are usually mixtures of sodium stearates and calcium stearates; and soluble lubricants are typically sodium stearates). Within each classification, additives are used to modify the properties of the lubricant to a considerable extent. Thickeners or fillers are usually unreactive, fine powders blended into the lubricant base to increase its viscosity, or resistance to flow under pressure at a given temperature. Although lime (possibly limestone dust in quantities of 30% to 70%) is the most popular thickener additive in general wiredrawing lubricants, the choice of thickener depends on the application and the end use of the wire; for example, coatings that must be easily cleaned should contain soda ash, borax, or other soluble material. Calcium and sodium sulfate compounds are also potential fillers. Extreme pressure additives are used in dry lubricants to reduce friction and increase die life. Molybdenum disulfide is the most popular of such agents; however, it is relatively expensive and may leave a very slippery and difficult-to-clean surface on finished wire. Graphite, sulfur, chlorine, and phosphates are also possible additives (Gzesh and Colvin 1999).

One of the key properties that determines which lubricants are most suitable in a given operation is the softening point (related to melting point) since this governs how the lubricant is applied to the wire and how quickly it is removed during drawing. If the lubricant is too soft at operating temperatures, it may come off before the drawing is complete. If too hard, the lubricant film is not applied uniformly and scratching or feathering (flaking) will result (Gzesh and Colvin 1999). The temperatures generated at the strand surface are determined by the plant configuration, including the drawing speed, die geometry, and area reduction in each die. As a result, the desirable lubricant properties vary from plant to plant and even from die to die. The softening point is determined by the alkali and fatty acids (such as stearic acid or tallow acids) on which the lubricant is based. The viscosity of the lubricant, which affects the thickness of the coating applied to the steel, is determined by the fat content and filler materials. To minimize the amount of residual film, the ideal lubricant system would be one that, at the plant die operating temperatures, provides a coating of just sufficient thickness to facilitate drawing, but which would be nearly all removed from the wire by the dies.

As previously stated, the two most common lubricants are sodium and calcium stearate-based materials. These lubricants are compounds made from sodium hydroxide or calcium hydroxide and a fatty acid (stearic acid) in combination with additives to impart special properties to the lubricant. These materials are soaps (Ivory soap, for instance, is 99% sodium stearate) and are supplied in dry form. Calcium stearate typically has a lower softening point than sodium stearate (Gzesh and Colvin 1999).

Calcium stearate may have been more appealing to strand manufacturers at one time because its lower softening point permits calcium stearate to produce a more effective coating on the wire early in the drawing sequence, when the rate of draw is slower, and the wire is at lower temperatures. In addition, it is typically cheaper than sodium stearate. As a result, it is not uncommon for calcium stearate to be used for the first one to three drafts (dies) and then for sodium stearate to be used for the remainder (Wire Industry 1991). The drawback in the use of calcium stearate-based lubricants is that they are more difficult to remove from the drawn wire, since they are water insoluble. In fact, calcium stearate lubricants may be chosen in certain applications (such as nails or coat hangers) because they leave a residue film that makes certain subsequent wire processing procedures easier (Platt 1991). On the other hand, sodium soaps or lubricants are "generally used when subsequent operations demand wire that may be readily cleaned" (Wire Association 1965, p. 285).

The strategy of using calcium stearate in only the first die(s) does not necessarily limit the residual film of the final product. This is because 80% of the lubricant needed is applied in the ripper box (the first die). Subsequent applications of lubricant retard loss, but typically do not significantly add to the residual lubricant (Gzesh and Colvin 1999, Wire Association 1965).

The effectiveness of lubricants is also influenced by the pretreatment selected for use during cleaning. For example, insoluble lubricants (calcium and aluminum) are most compatible with both borax and zinc phosphate coatings. Soluble lubricants (sodium and potassium) react with borax pretreatments to such a degree that the film is weaker and adequate lubrication may not be provided (Dove et al. 1990).

When the strand bond problems first began to surface in the United States in the early 1990s, one of the causes was thought to be the use of calcium stearates. As a result, North American strand producers reportedly stopped using calcium stearates, at least in the second and subsequent dies. However, it has been reported that European strand producers still use them.

Residual Film

Residual films are always present after wire drawing (Wire Association 1965). Prior to about 20 years ago, residual films and possibly other organic residues on prestressing strand that may have been detrimental to bond with the concrete were burned off during the stress-relieving operation (Preston and Sollenberger 1967). However, as noted in a 1982 article (Quick 1982, p.104-105), the replacement of open flame furnaces with far more efficient induction furnaces greatly improved line speed, but residues were no longer being burned off during stress relieving operations. Although the newer induction coils were effective in heating the strand and altering the physical characteristics of the steel, the short duration heating does not burn-off surface contaminate like convection heating had done. “Contaminants, such as the efficient wiredrawing lubricant calcium stearate, which do not sublime at stress-relieving temperatures in an induction furnace and are insoluble in water, are of particular concern . . . Induction heating only promoted surface flow of this contaminant, resulting in a glazed surface appearance which tended to seal other surface contaminants (i.e., zinc phosphate).” In addition, convection heating, unlike induction heating, is a combustion-based process and that “may have aided in oxidizing impurities on strand surfaces” (Rose and Russell 1997, p. 57).

The link between residual films and poor bond was verified when scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS) analyses conducted on strand tested in structural bond tests confirmed the presence of “copious amounts of surface process chemical . . . on the outer wires of uncleaned strand which failed bond development tests” (Quick 1982, p. 107). The link between lower amounts of lubricant and increased bond strength has also been demonstrated recently by others (Maehata and Ioka 2006).

Additional evidence of an increase in residual film on prestressing strand was found in the early 1980s, when bright prestressing strand exhibited approximately six times the chloride ion corrosion threshold of black reinforcing bar in an FHWA sponsored study (Pfeifer 1986). It is believed that the unexpected corrosion protection was due to the presence of residual rod treatments and wiredrawing lubricants, namely zinc phosphate and calcium stearate, on the strand as manufactured. The corrosion performance of strand that had been subsequently “ultrasonically cleaned” by the manufacturer was indistinguishable from that of the “as manufactured” strand, suggesting that the drawing lubricants were not removed by the cleaning. Similar corrosion behavior was noted in 1984 by another researcher, who reportedly cleaned the strand with xylene prior to testing (Stark 1984).

Quantifying the amount of residual film present on prestressing strand is typically performed through gravimetric methods that consist of weighing a segment of strand before and after stripping with sodium hydroxide (Wire Association 1965) or some solvent. In several investigations of suspected strand bond problems, the research team has employed a method involving a solvent (acid/chloroform) extraction of residue from the strand surface and the cement paste in contact with the strand.

The removal of residual films on drawn wire is not a trivial process. The cleaning mechanisms applicable to wire reviewed in a recent article included: detergency (displacement of soil by active agents with greater affinity for the substrate surface), mechanical removal (external physical action), chemical reaction (conversion of soil from an insoluble form to a soluble form), and dissolution (soil dissolved with solvent cleaner) (Colvin and Carlone 1998). For strand production, a post-drawing cleaning operation employing the first three mechanisms listed above is not typically performed because of the high line speeds involved. However, individual wires may be dipped in water before stranding or the stand may be rinsed with water to cool and clean the strand, if soluble lubricants have been used.

When insoluable lubricants are present, cleaning is more difficult. In tests of cleaning solutions, a sodium hydroxide solution was able to remove all but 10 of 300 to 400 mg/ft^2 of an insoluble stearate lubricant residue originally on the tested wire. However, cleaning effectiveness is enhanced by increased solution temperature and higher rinse volumes and temperatures, all of which require additional effort to produce (Colvin and Carlone 1998). Multi-pass immersions in sodium hydroxide solutions have been used in the production of other wire products where surface cleanliness is critical, such as aluminum-clad steel wire, but these methods are not ideal because of the hazardous nature of the caustic solutions and the large volume of waste solution that is generated (Chow 2001). As alternatives, in-line methods incorporating neutral salt water electrolysis and high-frequency focused ultrasound have been proposed (Quick 1982, Chow 2001). However, because of cost, these methods have not found widespread acceptance in strand production.

Research Objectives

The original objectives of this study were to: (1) identify the common types of strand residues, determine their impact on bond characteristics and strand performance, and recommend methods for their reduction; (2) develop quality control and assurance methods for assessing the level of deleterious residues and recommend thresholds for strand acceptance; and (3) develop a performance-based test procedure and a minimum specification requirement for strand acceptance based on bond behavior. At the direction of the supervisory panel, this third objective was modified during the execution of this project to include the adoption of a pre-existing performance-based test procedure.

Organization of Document

This final report summarizes the findings of this study and discusses their potential application. The following appendices have been written to provide more background and additional details of this work. These include

Appendix A -	Review of Strand Bond Literature
Appendix B -	Evaluation of Mechanical and Chemical Test Methods
Appendix C -	Specifications for Standard Surface Test Methods
Appendix D -	Supplemental Investigations of Strand Bond
Appendix E -	Bibliography of Strand Bond

CHAPTER 2

Research Approach

Overview

The work plan developed to achieve these objectives was divided into the following three phases.

The initial phase involved gathering information about strand manufacturing and potential test methods from the prestressed concrete, strand manufacturing, and wiredrawing lubricant industries, as well as from the available literature. Based on this information, a number of chemical and surface test methods and performance-based (i.e., mechanical) test methods that were considered to have potential for use in a quality control program were proposed for evaluation.

In the second phase of this work, the proposed surface and chemical test methods were conducted on a limited number of available sources of strand with variable bond properties: (1) to evaluate the ability of these methods to predict bond performance and (2) to assess their suitability for routine quality control operations. In addition, performance-based tests were conducted on some of the same strand sources. Those test methods that showed good correlation with bond performance were selected for further study, while those that did not were abandoned. A parallel set of investigations, termed *supplemental investigations*, was conducted to learn more about the relationship between bond and residual lubricants.

In the third and final phase, the promising surface and chemical test methods were performed on a different group of strand sources to validate their correlation with bond performance. At the direction of the supervisory panel, bond performance was quantified by another researcher using the pre-existing performance-based test procedure. Testing was also conducted to support the development of a precision statement. Finally, statistical analysis was performed to identify minimum acceptance thresholds for the surface and chemical test methods that would predict adequate bond performance as defined by the pre-existing performance-based test procedure.

Industry Survey

Letters and accompanying questionnaires were sent out to obtain additional information about strand production. One letter was sent to strand producers and the other was sent to drawing lubricant manufacturers. No one returned the questionnaire. Instead, the research team received a few phone calls, primarily from strand producers who expressed concern that this research would interfere with their business.

Several lubricant producers were cooperative when contacted by phone. They shared some information about their products and their use in strand production.

To facilitate discussion, a meeting was held with members of the project team, NASPA, and Bruce Russell, co-principal investigator of NCHRP Project 12-60: Transfer, Development, and Splice Length for Strand/Reinforcement in High-Strength Concrete, and a consultant to NASPA. This meeting was held at WJE's Northbrook, IL, headquarters on 7 January 2004. The objective of this meeting was to present the strand producers with an opportunity to give feedback on the proposed work plan for this project, which had been provided in advance for their review, and to provide the project team the opportunity to gain some insight into industry practice and the applicability of the proposed testing methods. In addition, it was hoped that this meeting would lay the groundwork for future interactions regarding this project.

Quality Control Program Development

The purpose of a QC program is to assess, by routine monitoring and testing, whether a particular level of quality is maintained during production. In this context of strand bond, the desired QC program would evaluate the surface condition of strand so that steps can be taken in a timely manner, if needed, to ensure that the bond between strand and concrete products is reliable and structurally adequate.

In the past, the quality of bond has been evaluated using mechanical methods, such as pull-out or transfer length tests. However, such methods are expensive, time consuming, and conducted infrequently; currently, routine pull-out tests are conducted quarterly by strand manufacturers. A main goal of this project was to develop fast, accurate, reproducible, simple to conduct, and inexpensive QC test methods for detecting and measuring the level of deleterious residues on strand that could be performed frequently (i.e., weekly). A number of methods were proposed involving testing surface and chemical properties of the strand that could be linked to strand bond. All tests were intended as part of a routine testing program that could be conducted by strand manufacturers, precasters, or other interested parties.

Quality Control Program Overview

The individual tests that were proposed required a varied range of time, expertise, and equipment. Therefore, it was envisioned that they would be applied as part of a two-tiered QC program, in which the value of the test is proportionate to the test complexity, with the following distinct components:

- Level I QC component (QC-I) and
- Level II QC component (QC-II).

The Level I QC component consists of relatively quick, simple, and inexpensive tests that can be conducted by strand manufacturing personnel. These tests would be performed on a daily basis. Each test would take less than one-half hour to perform.

The Level II QC component consists of tests that require more in-depth training and advanced equipment, and could be performed by testing laboratories on behalf of strand manufacturers. These tests would be performed at longer intervals, with changes in processes, or as dictated by the Level I QC test results.

Evaluation of Proposed Surface and Chemical Test Methods

Several chemical and surface tests were proposed to predict poor bonding characteristics for strand. The purpose of the experimental program conducted in this research program was to determine if these proposed tests would be applicable for use in a QC program. To do this, several rounds of experimentation were needed.

Screening Testing

The first round of experiments consisted of "screening" experiments. The objective for the screening experimentation was to eliminate those tests that were not helpful for predicting bond performance. Thus, the first step of the analysis in this round of testing was to estimate the correlation between each surface or chemical test and bond performance, and the second step was to identify those methods where some degree of correlation was indicated. For each source of strand, bond performance was measured in terms of pull-out stresses, transfer lengths, or both. Accordingly, for the purpose of this analysis, the bond performance was treated as the independent variable. The best experimental design for estimating a correlation is to place the design points as far apart as possible in terms of the independent variable. Thus, the optimal statistical design is to run each test on strands that show a range of bonding performance. For the screening experiments high, medium, and low bonding sources were desired. However, efforts to obtain a very low bonding strand were not successful. Although reports of low bonding strand incidents continue to surface in the precast concrete industry, "unused" samples of such strand remained elusive. Therefore, the screening tests on new strands were run on what are essentially high and intermediate bond strands.

Correlation Testing

The second round of experiments was performed for confirmation and calibration purposes. This round involved running additional tests using those methods that showed promise in the screening experiments. These selected tests were conducted on five new strand sources. This complete dataset was then used to assess the correlation between the QC tests and bond performance, and to determine if the tests were able to accurately identify good and bad strand. It was also used as a basis for discussing pass/fail criteria for acceptable bond performance.

Precision Testing

A third round of testing was conducted to determine the precision (i.e., repeatability) of those methods, showing good correlation with bond strength. This was used to develop precision statements included in the published test methods.

Basis for Evaluation—Transfer Length and Pull-Out Tests

Transfer length is the most reliable and realistic measure of bond performance. During the screening testing, the evaluation of correlations between the pull-out tests and bond performance were based on performance as measured with transfer length tests conducted on the same sources of strand.

Pull-out testing was conducted as part of the screening studies using three materials as the test matrix: a concrete, a Portland cement mortar, and a gypsum plaster mortar. Based on comparisons with transfer length tests conducted in this study and described in Appendix B, the concrete pull-out test showed the best correlation with bond quality. The surface and chemical test methods were evaluated in the Screening Round based on the results of pull-out tests from concrete, again, on strand samples from the same source. However, the evaluation of correlation of test results to bond in the correlation round of testing was based on results from a mortar pull-out test program associated with NCHRP Project 12-60. The principle investigator from that project supplied the strand samples for this portion of the study. No pull-out testing was conducted in the Correlation Round of the experimental program.

Strand Samples

To assess the effectiveness of the mechanical and surface chemistry-based testing procedures, it was essential that samples representing the range of possible performance be evaluated. Since neither precasters nor strand suppliers were enthusiastic about associating themselves with poor-bonding strand, obtaining samples of strand from the lower end of the performance spectrum was difficult.

The strand sources included in testing for this program are listed in Table 1. This table also includes a result from concrete pull-out tests or mortar pull-out tests (the bond stress at the observed first slip or after 0.1-in. slip at the non-loaded end of the strand). Each pull-out stress is the average of the pull-out stresses from at least six individual pieces of strand. The bond stresses are calculated from the measured loads based on the actual surface area and the embedment length of the strand.

These strand sources fall into three groupings: historic, recently manufactured, and OSU (Oklahoma State University) strand.

Historic Strand—This study initially identified samples of strand for testing from prior unpublished tests conducted at Kansas State University (KSU) by Bob Peterman and at StressCon Corporation, Inc. by Don Logan that cover a wide range of pull-out behavior. These are referred to as "historic" strand and were manufactured between 1997 and 2004. Figure 2 is a plot of first slip bond stress or bond stress at 0.1-in. end slip versus maximum bond stress from the data available from historic concrete pull-out tests.

When suggested minimum pull-out loads for acceptable bonding performance (suggested by Logan, based on a limited number of flexural beam tests conducted in the mid-1990s and his engineering judgment) are converted to bond stresses, they are 425 and 955 psi for the first slip and maximum stresses, respectively. These thresholds have been reproduced in Figure 2.

Recently Manufactured Strand—Figure 2 also shows the concrete pull-out performance of recently manufactured samples identified during this project. These recently manufactured

Table 1. Strand sources.

Strand Source ID	Strand Geometry				Mortar Pull-Out Testing			Concrete Pull-Out Testing (LBPT)		
	Size (in.)	Measured Diameter (in.)	Pitch (in.)	Lay (Handedness)	Location	Date	0.1-in. Slip Stress (psi)	Location	Date	0.1-in. Slip Stress (psi)
Historic Strand										
KSU-F	1/2 Special	0.524	7 5/8	Left	--	--	--	KSU	Mar 2004	241
KSU-H	1/2 Special	0.523	7 1/2	Left	--	--	--	KSU	Mar 2004	209
SC-F	1/2	0.503	8	Left	--	--	--	SC	May 1997	223
SC-H	1/2 Special	0.530	7 1/4	Left	--	--	--	SC	Nov 2002	472
SC-IS	1/2	0.501	7	Left	--	--	--	SC	Mar 2003	682
101	6/10	0.601	8 1/2	Left	--	--	--	SC	Oct 2004	241
Recently Manufactured Strand										
102	1/2	0.501	7 1/2	Left	KSU	Jun 2005	315	KSU	Jun 2005	441
103	1/2	0.503	8	Left	KSU	Jun 2005	397	KSU	Jun 2005	944
151	1/2 Special	0.517	7 1/2	Left	KSU	Jun 2005	273	KSU	Jun 2005	541
153	6/10	0.588	9	Right	--	--	--	KSU/SC	Jun / Aug 2006	142/406
OSU Strand										
349	1/2	0.505	8 3/4	Left	OSU	Jun 2004	156	--	--	--
548	1/2	0.500	7 5/8	Left	OSU	Jan-Feb 2004	623	--	--	--
697	1/2	0.503	7 1/4	Left	OSU	May 2004	606	--	--	--
717	1/2	0.500	8	Left	OSU	Feb 2004	206	--	--	--
478 *	1/2	0.499	7 5/8	Left	OSU	May-June 2004	409	--	--	--
960 *	1/2	0.500	7 1/2	Left	OSU	May-June 2004	409	--	--	--

* Samples designated 478 and 960 were from same source.
KSU = Kansas State University; OSU = Oklahoma State University; SC = StressCon Corporation, Inc.

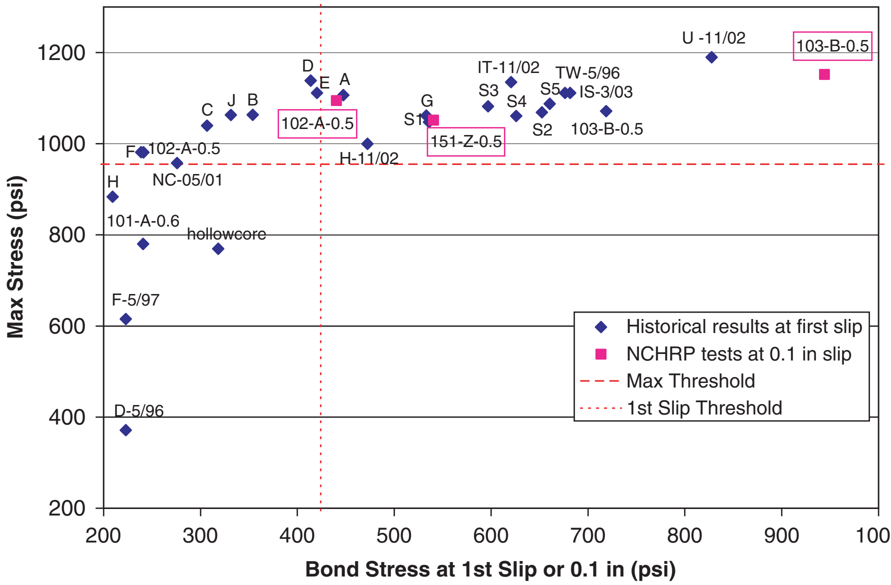

Figure 2. Correlation between maximum stress and first observed or 0.1-in. slip stress for historic and recently manufactured strand.

samples were obtained in large quantities for the purpose of this research and were used in the screening experiments. Notice that the greatest variation in the recently manufactured strand is not in terms of maximum stress but in terms of stress at 0.1-in. slip.

The recently manufactured strand sources (102, 103, and 151) were selected because initial testing indicated that they represented a range of first-slip pull-out performance. None of these strands had significantly low maximum load pull-out performance. Source 103 is the strand used by StressCon Corporation, Inc. in their ordinary production of precast/prestressed concrete and has a proven record of good bond from pull-out tests, flexure beam tests, transfer length tests, and end slips observed in hollow core precast/prestressed concrete members.

The bond stress at 0.1-in. slip of Source 102, measured in concrete pull-out testing performed as part of this project, is slightly above Logan's 425 psi threshold. Of the 31 results from historic and other sources presented on this plot, 13 are to the left of Source 102. Only one of these (D-5/96) was available in enough quantity to enable additional testing, and the condition of this strand was variable.

OSU Strand—The sample sources used for the Correlation Round of testing were selected by Bruce Russell of Oklahoma State University (OSU). These sources of strand had been tested in work performed by Russell for NCHRP Project 12-60: Transfer, Development, and Splice Length for Strand/Reinforcement in High-Strength Concrete, the Oklahoma Department of Transportation, and the NASPA (also known as the Committee of the American Wire Products Association [AWPA]). Two of the six strand sources provided by Russell were actually the same strand source, a fact that was not known by the research team before the testing was completed. This was intended to test the repeatability of the surface and chemical test methods. Complete mortar pull-out and some transfer length test results were provided in tabular form by Russell after the chemical and surface testing had been completed.

Transfer Length Testing

The transfer length test is not proposed as a QC test method, but was conducted as a basis for evaluating the proposed QC test methods, since the transfer length quantifies bond performance under the most realistic conditions. Transfer length is defined as the distance over which the effective prestressing force is transferred to the concrete element. In other words, this is the distance from the end of the strand where no stress is applied to the concrete to the point where the maximum amount of stress has been transferred into the concrete. The test for transfer length involves casting a prism of concrete around a stressed strand or strands and then measuring the strain profile along the length of the prism after the stress is released. The transfer length is defined as the length over which the measured strain in the prism increases from zero at the ends of the prism to the edge of the strain plateau region in the middle of the prism. The end slip (i.e., the distance that the end of the strand moves relative to its original position) is proportional to transfer length and also was measured. The strain profiles of the transfer length prisms were monitored over time, starting with the initial reading immediately after release; additional measurements were taken at 28 days, 6 months, and 18-22 months.

The data obtained during the transfer testing are not reported as a length, but instead as an average bond stress over the transfer length. This was done to enable comparisons of stress transfer behavior between the tested strand sources, since this approach eliminates complications from strands of varying sizes and varying initial stress conditions. The average bond stress over the transfer length, U_t, is calculated as

$$U_t = \frac{f_{se} A_{ps}}{C_p L_t} \quad \text{(Eq. 1)}$$

where f_{se} is the effective prestress after transfer, A_{ps} is the cross-sectional area of the strand, C_p is the circumferential perimeter of the strand ($4/3\ \pi\ d_b$) and L_t is the transfer length. Average bond stress is thus dependent both on effective prestress as well as transfer length for a given strand geometry. For the purpose of this calculation, f_{se} was taken as the difference between the stresses in the strand before release and the elastic losses only. The elastic loss was determined based on the strain measured immediately after release in the central region of the test prism over which the strain is approximately constant, assuming no relaxation losses in the strand.

Proposed Quality Control Test Methods

The test methods that were proposed and conducted as part of the screening and correlation test program are summarized in Tables 2 and 3. These tables also list the QC levels for these tests, if applicable. These test methods consist of (1) surface and chemical testing, (2) pull-out testing, and (3) transfer length testing. Since insufficient lengths of strand were available for pull-out and transfer length testing during the Screening Round from the historic sources, these tests were conducted only on the recently manufactured strand. The surface and chemical testing program has been conducted on both the historic strand samples and on the recently manufactured samples.

Surface and Chemical Testing

The surface and chemical test methods that were attempted are described briefly. More complete descriptions are given in Appendix B.

Contact Angle Measurement

The contact angle is a measure of surface tension (wettability). It was anticipated that the presence of drawing lubricants would affect this property. The contact angle is measured on the projected shadow of a small droplet of distilled water applied to the strand surface. Measurements were taken with the strand: (1) in an as-received condition, (2) after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution, and (3) after an ignition process. The calcium hydroxide exposure (also called a lime dip) will convert sodium soaps (e.g., sodium stearates) to insoluble calcium salts. For example, water-soluble sodium stearate (a soap or wetting agent) is converted to a film of insoluble calcium stearate (a wax-like, water repellent that increases the surface energy of the strand). This conversion reaction was chosen to simulate the reaction of concrete with surface residues of soaps and is intended to produce a condition where the effect of similar calcium stearate compounds on the contact angle are compared, even if the original residue did not result from a calcium stearate-based lubricant. The ignition process was performed on samples to volatilize organic compounds expected to be present in the drawing lubricants.

Examination under Ultraviolet Light

Certain lubricant additives (e.g., hydrocarbon oils, fluorescein additives, and some inorganic deposits) will fluoresce under ultraviolet (UV) radiation. An examination under UV light was conducted using a range of light sources, the most promising of which was a 366-nm wavelength.

Testing pH

Testing of the pH of the surface was attempted with each of the strand sources to see if alkalinity of a solution generated by placing drops of water on the residue could be linked to bond. Testing of the pH of the surface was conducted using indicator papers, indicator solutions, and a pH meter.

Loss on Ignition

The weight loss on ignition (LOI) represents the weight of compounds that can be volatilized or burned off the strand surface at high temperature. This property was measured with the expectation that the weight lost would consist mainly of the organic component of residues, such as drawing lubricants.

Loss in Hot Alkali Bath

The weight loss after hot alkali bath (LAB) represents the weight of compounds that can be washed off the strand surface in a hot sodium hydroxide solution. As with the LOI test, this property was measured with the expectation that the weight lost would consist mainly of drawing lubricants.

Change in Corrosion Potential

Past studies of the corrosion resistance of prestressing strand in concrete have suggested that strand with a coating of residue does not corrode as readily as a clean strand. To assess the

Table 2. Test methods conducted during screening and correlation testing programs.

Test Method	Condition/Type of Test	QC Level	Property Measured	Objective
Contact Angle Measurement	As received After $Ca(OH)_2$ dip After ignition	I	Surface energy of strand	Detect presence of materials that reduce water surface tension (Na-based soaps) or increase steel surface energy (Ca-based salts)
Examination under UV Light	-	I	Presence of fluorescing materials	Identify lubricant additives such as hydrocarbon oils, some inorganic deposits, or possibly fluorescing-based tracers that may fluoresce under UV light
pH Testing	Universal indicator Indicator solutions pH meter High-res. indicator	I	pH of surface	Detect presence of pretreatment lubricant residues containing alkaline salts or alkalies
Weight Loss on Ignition (LOI)	-	I	Weight of material burned off strand	Determine amount of material that can be oxidized on the strand surface at 415°C, expected to be largely organic
Weight Loss in Alkali Bath	Method 1 Method 2	I	Weight washed off strand	Determine amount of material that can be washed off the strand surface after soak in a NaOH solution
Change in Corrosion Potential	As received After $Ca(OH)_2$ dip After ignition	I	Average change of potential	Assess the potential for corrosion by comparing the corrosion potential to a reference cell monitored versus time
Surface Roughness	-	I	Roughness parameters R_a, R_z, P_c	Quantify surface profile
Corrosion Rate	As received After $Ca(OH)_2$ dip After ignition	II	Corrosion current	Determine the shift in potential of a metal sample from a stable corrosion potential due to an external current
Organic Residue Extraction	Warm water/acid-chloroform wash Hot water/acid-chloroform wash	II	Weight of extracted organic residue	Determine amount of individual components of strand manufacturing lubricants from a warm/hot water wash procedure then an acid/solvent-wash procedure
Atomic Absorption (AA) Spectroscopy	Sodium Calcium Potassium Boron Zinc Phosphate	II	Concentrations of inorganic components of extraction residue	Quantify inorganic elements (sodium, calcium, potassium, zinc, and boron) in residue
Pull Out from Large Concrete Block	-	II	Maximum bond stress and stress at 0.1 in. displacement (or first slip)	Mechanically measure stresses required to break bond with concrete
Pull Out from Portland Cement Mortar	-	II	Maximum bond stress and stress at 0.1 in. displacement (or first slip)	Mechanically measure stresses required to break bond with mortar
Pull Out from Hydrocal-Based Mortar	-	II	Maximum bond stress and stress at 0.1 in. displacement (or first slip)	Mechanically measure stresses required to break bond with Hydrocal-based mortar
Transfer Length	-	Analytical	Length over which the prestress is transferred to a concrete beam	Directly measure bond performance in prestressed concrete beam

Table 3. Coefficient of determination (R^2) from linear regression with average bond stress over transfer length.

Test Method	QC Level	Coefficient of Determination (R^2) from Regression with Average Bond Stress over Transfer Length
Concrete Pull Out	II	0.98
Mortar Pull Out	II	0.85
Hyrdocal Mortar Pull Out	II	0.36

potential for corrosion, the strand samples were placed in a solution of deionized water, and the corrosion potential measured with a reference cell (saturated calomel reference electrode), was monitored versus time. This corrosion potential is determined by the amount of ferrous ions in solution surrounding the sample, and a greater drop in this potential is indicative of a greater tendency to corrode. Measurements were taken with the strand in an as-received condition, after immersing the strand sample in a saturated $Ca(OH)_2$ solution, and after an ignition process.

Surface Roughness

Microscopic examinations of sectioned portions of wire taken from strand have indicated that an observable difference in the surface roughness of the good- and poor-bonding strand sources exists. Based on images captured using a scanning electron microscope, the depth of the roughened surface features is typically 3 μm (0.0001 in.) or less. Trials with a portable profilometer suitable for a QC setting were conducted to determine whether these physical measurements could accurately represent the surface roughness and to investigate the correlation with bond performance.

This system works by measuring the deflection of a diamond probe, with a 2-μm tip radius, as it is dragged 2 mm across the surface of the sample.

Corrosion Rate

To further explore the interaction between strand bond and corrosion, the instantaneous rate of corrosion of samples of strand in a salt solution was measured with a polarization resistance technique. The polarization resistance technique measures the corrosion current, which quantifies the rate at which the electrochemical corrosion reaction is occurring. This is a much faster test than the test for change in corrosion potential, but requires specialized equipment (a potentiostat). Measurements were taken with the strand in an as-received condition, after immersing the strand sample in a saturated $Ca(OH)_2$ solution and after an ignition process.

Organic Residue Extraction

The tests for identification and quantification of organic drawing-compound residues were based on solvent extraction procedures, together with gravimetric and Fourier transform infrared spectroscopical (FTIR) analyses. Essentially, the amount of material extracted from a defined length of strand was determined by weighing the extraction residue on an analytical balance. The material in the extraction residue was then identified by FTIR analysis of the residue. The FTIR spectrum obtained is like a fingerprint of the material.

The extraction procedure used is a modification of a procedure found in ASTM C114 for organic materials in cement. Multiple extractions were used to differentiate between various forms of drawing-compound residue. The strand was first washed with warm or hot water to remove water-soluble materials, such as sodium stearate. Then, the strand was exposed to hydrochloric acid and chloroform to extract water-insoluble residues such as calcium stearate and stearic acid.

At the conclusion of the Screening Round, it was observed that the water temperature had little effect in most cases, but that the residue concentrations measured with the warm water method seemed to generally correlate better with bond tests. Therefore, only a warm-water wash was used in the Correlation Round. In addition, to minimize the effort spent on performing the time-consuming chloroform organic extraction, the wash solutions from the warm water and acid-chloroform washes were combined, and a single separation was performed. Therefore, only one FTIR scan and residue weight determination was made per piece of strand in this round of testing. However, this is considered essentially equivalent to the combination of sequential warm water and acid-chloroform washes performed in the Screening Round. Since quantifying the water-soluble materials was still of interest, because it might provide insight into possible cleaning methods, separate warm water washes were performed on additional pieces of strand. This wash solution was acidified and saved for elemental analysis.

Atomic Absorption and Colorimetric Analysis

To identify the chemical composition of residual inorganic components of pretreatment chemicals and drawing compounds, chemical analyses of the acidified water extract and acid/solvent extract solutions, which had been obtained during the organic residue extraction procedure and had been separated from the chloroform, were performed. Either zinc phosphate or borax (sodium borate) is often applied to the wire before the drawing process begins to help drawing lubricants stick to the surface of the rod stock. Most common drawing lubricants are expected to include stearate salts, particularly sodium and calcium stearates. The elemental concentrations of sodium, potassium, calcium, and zinc were determined by atomic absorption spectroscopy. The solutions were also scanned for detectable quantities of aluminum during the Screening Round. Colorimetric analyses of the wash solutions for boron and phosphate (PO_4^{3-} as total phosphate) were performed using visible light spectroscopy.

Pull-Out Testing

The original project scope included the development of a performance-based test method for use in evaluating strand bond. As a result, in the initial phases of this study, efforts were made to develop a procedure for quantifying bond using a pull-out test conducted on untensioned strand embedded in some material. Two types of pull-out tests have been commonly used to evaluate strand bond. The first method involves pulling untensioned strand out of a block of concrete. The second method involves pulling untensioned strand out of a steel cylinder filled with mortar.

In its current form, the concrete pull-out test resembles a method developed by Moustafa (1974). The method was primarily developed to judge the capacity of strand to be used as lifting loops to handle product during shipping and erection. The test developed by Moustafa was modified by Logan (1997) to judge the bond quality of strand in pretensioned applications. Further developments of the method have occurred and are the basis for the testing reported herein.

In its current form, the mortar pull-out test method resembles a method originally developed for the Post Tensioning Institute in 1994 (Hyett et al. 1994, Post-Tensioning Institute 1996). The method was primarily developed to judge the bond quality of prestressing strand used in rock anchors. The method became the basis of *ASTM A981-97 (2002) Standard Test Method for Evaluating Bond Strength for 15.2 mm (0.6 in.) Diameter Prestressing Steel Strand, Grade 270, Uncoated, Used in Prestressed Ground Anchors* (ASTM 2002). Later, this method was modified by Russell and Paulsgrove (1999) for NASPA and became known as the NASPA test. The NASPA test has been modified slightly by this research project to make it less sensitive to the test apparatus.

One of the goals of this project was to try to eliminate variables by using standardized and universally available embedment media referred to in the NCHRP Project 10-62 Request for Proposal (RFP) as a "surrogate homogeneous material." Accordingly, a third type of pull-out test was attempted: pulling untensioned strand out of a steel pipe filled with a modified gypsum plaster (Hydrocal).

The three types of pull-out tests were performed on the three sources of strand at KSU in March and May of 2005. Each of these test procedures and their results are described briefly below. More complete descriptions are given in Appendix B.

In each of these methods, the load applied to pull out the strand and the movement (or slip) of the non-loaded (free) end of the strand were monitored throughout testing. To allow comparison of data among strand of different sizes, the bond stress has been calculated from the measured loads based on the nominal surface area (equal to $^{4}/_{3}\,\pi\, d_b\, l$, where d_b is the nominal strand diameter and l is the embedment length) of the embedded section of the strands. Two characterizations of performance are determined during strand bond pull-out tests. The first characterizes the early part of the bond stress-slip relationship, while the second is based on the maximum stress measured throughout the test. In concrete pull-out tests performed on historic strand, the early performance was characterized in terms of the stress at which movement is first visually observed at the loaded end of the strand, called the stress at "first observed slip" or "first slip." For the tests conducted as part of this experimental program, the stress selected to characterize the early part of the bond stress-slip relationship is the bond stress at 0.1-in. slip, measured at the non-loaded end of the strand. This 0.1-in. slip criterion was adopted to give a more precisely defined location on the stress-slip curve.

Large Concrete Block Pull-Out Test

The large concrete block pull-out test (LBPT) involves pulling six untensioned strands bonded over 18 in. from a large (2 ft × 2 ft × 2 ft 8 in.) block of concrete. This concrete was produced from a conventional mix design used by a precaster, and is produced with a coarse aggregate with Mohs hardness greater than 6.0. The strength of the concrete at the time of the test is 3500 to 5900 psi. The test is conducted in load-rate control with a load rate of 20 kips per minute. The bond stress at 0.1-in. slip and the average maximum bond stress for each strand tested are reported and averaged. In addition, for the concrete pull-out tests conducted as part of this study, the load at which slip at the loaded end was first observed visually also was recorded. This "observed first slip" was determined because it relates back to historic pullout data recorded by Logan (1997) and others.

Mortar Pull-Out Test

In the mortar pull-out test, each prestressing strand is embedded in 5-in. diameter by 18-in. long steel cylinders filled with mortar (Portland cement, sand, and water). The top 2 in. of the embedded portion of the strand is debonded, leaving 16 in. of strand in contact with the mortar. Six cylinders are tested for each source of strand. The mortar is produced with a Type III cement-to-sand ratio equal to 2:1 by weight, and the required mortar strength at time of the test is 3500 to 5000 psi. The bond stress at 0.1-in. slip and the average maximum bond stress for each strand tested are reported and averaged.

The test was conducted in load-rate control with a load rate of 5 kips per minute, which was reportedly similar to the rates achieved during testing conducted by previous researchers, such as in the NASPA-funded work, which was performed under ram displacement-rate control. The mortar pull-out test was conducted with load-rate control instead of displacement-rate control because load rate, which may influence the results of the physical tests on structural materials, is independent of the stiffness of the testing frame. It is desirable that the test method be universally applicable, and so should not be influenced by the load frame.

Hydrocal Pull-Out Test

A range of Hydrocal-based mixtures was evaluated as possible surrogate materials for use in a pull-out test. The final mixture used in this testing contained Hydrocal White (a material similar to plaster of Paris made by United States Gypsum), Ottawa graded sand (ASTM C778), calcium hydroxide flakes, USG Retarder for Lime-Based Plasters, and water. This formulation of Hydrocal is almost pure plaster of Paris and was chosen because it is produced at only one manufacturing facility from consistent raw materials. Also, like cement, it is a calcium compound (plaster of Paris is hemihydrated calcium sulfate). Calcium hydroxide flakes were added to simulate the alkalinity of concrete, and the Hydrocal was combined with sand and plaster retarder to limit the heat production generated during the rapid plaster hydration.

In this test, each prestressing strand is embedded 12 in. in a 3-in. diameter steel cylinder filled with the Hydrocal mortar (gypsum/lime plaster, sand, and water). Six cylinders are tested for each source of strand. The required mortar strength at time of the test is 3000 to 4000 psi. The test is conducted similarly to the mortar pull-out test. The bond stress at 0.1-in. slip and the average maximum bond stress for each strand tested are reported and averaged.

Interpretation of Historic and Recent Concrete Pull-Out Test Results

The "first observable slip" and "0.1-in. slip" are measured in different ways, yet are considered to be close in value to one another. This was verified during the current test program, which showed that the first observed slip at the live end of the strand occurred at generally the same time at which 0.1-in. of end slip was measured in the pull-out test. This similarity is significant because the stress at 0.1-in. end slip was not determined during the historic concrete pull-out tests conducted on the historic samples that were included in the screening testing program. However, because of the similarity, when evaluating the correlation between bond performance and the screening test results, both the "first observable slip" and "0.1-in. slip" characterizations of bond are used together.

Mortar Pull-Out Testing for Correlation Round of Evaluation

As mentioned, the sample sources used for the Correlation Round of testing were selected by Bruce Russell of OSU. Mortar pull-out data were provided for each of the sources. The reported NASPA pull-out forces represent the average load at 0.1-in. slip for multiple (5 to 12) specimens, all tested on the same day with the same batch of mortar. Per the protocol outlined by Chandran (2006), the mortar pull-out force was measured on strands embedded in 5-in. diameter by 18-in. long cylinders (with 16 in. of strand in direct contact with mortar). These mortar pull-out tests were conducted under displacement-rate control, with an additional criterion for load rate. For comparison with mortar pull-out test results for the Screening Round of testing, the loads at 0.1-in. slip provided from OSU were converted to average bond stresses at 0.1-in. slip.

Statistical Evaluation of Results

A large experimental program was conducted to support the evaluation of the various proposed test methods for strand bond that were intended for use as part of a QC program. These were classified as performance-based (i.e., mechanical) tests and surface and chemical tests. The correlation between bond and the methods that fall under each of these classifications was evaluated differently based on the strand sources that were collected for testing and the data quantifying bond performance that were available. The evaluation of correlations between the various pull-out testing methods and the bond were based on bond performance as measured with transfer length tests. The correlations between the surface and chemical test methods were evaluated based on the results of pull-out tests from concrete in the Screening Round and based on the results of pull-out tests from mortar in the Correlation Round.

Statistical analyses have been performed with two objectives: (1) to determine and quantify the relationship between the chemical and surface test results and bond performance, and (2) to allow the determination of acceptance thresholds for the chemical and surface test results that can predict, with a given level of confidence, that adequate bond performance can be achieved. The first objective was achieved based on standard linear regression techniques, while the second objective requires the determination of prediction intervals. A more extensive discussion of both of these analysis methods is given in Appendix B.

Regression

To provide a quantitative measure of the goodness-of-fit to aid in the evaluation of these methods, a linear regression has been performed, and the coefficient of determination (R^2) was determined for the relationship between each proposed test method and the bond quality measure. No physical basis for a linear relationship between these measures of bond is known; however, the linear relationship was assumed as the simplest model relating the parameters. The R^2 is a measure of the adequacy of a regression model (i.e., it describes the amount of variability in the data explained by the regression model). The closer the R^2 is to 100%, the more completely the model describes the relationship between the test method results and the basis for evaluation.

To further evaluate the validity of these methods, the significance of the linear models developed based on these data was evaluated by the calculation of P-values for the coefficients (slope) from the linear models. The coefficient from the linear model is judged to be significant when there is a sufficiently high confidence that it is not equal to zero. If this is the case, the relationship represented by the model is statistically significant and the results of the surface tests are meaningful in the prediction of the pull-out test result. A 95% confidence level is commonly used to evaluate significance. The level of confidence of significance on the coefficient is given by $(1 - \text{P-value}) \times 100\%$, so a $\text{P-value} < 0.05$ implies that the confidence interval does not include zero with higher than 95% confidence.

For the contact angle and organic residue extraction test methods, coefficients of determination have been calculated using only data from sources identified in the FTIR analyses as carrying only stearate-based lubricants. This was done to eliminate the potentially confounding influences of non-stearate-based lubricants. Analyzing these data in this manner has a practical motivation, since such models could be useful in a production setting where the lubricant in use is known to be stearate-based only.

In addition to the regression with a single predictor, regression analyses were also performed based on selected combinations of the surface and chemical test results to see if the pull-out performance could be better predicted using more than one predictor variable. When regression was performed with multiple predictors, the R^2 *adjusted* was calculated and used to interpret how well the model fits the data. The R^2 *adjusted* is the most appropriate measure of goodness-of-fit for multiple-predictor regression. Since adding predictors makes the model more flexible and thus better able to fit the data, the R^2 *adjusted* measure includes a penalty for additional predictors in the model.

Prediction Intervals

The models generated by the regression analysis allow for the prediction of the pull-out stress based on results obtained with the surface and chemical QC test methods. However, the prediction formulas give the average estimated pull-out stress, but do not account for variation that is bound to occur in the QC test results or uncertainty in the regression model. Instead, what is needed to interpret and practically apply a given QC test result is the computation of a lower bound on the interval that, with a given confidence, includes the pull-out stress for a strand sample with that QC test result. This type of interval is known as a one-sided prediction interval and is a standard part of regression theory and practice.

The prediction interval concept is a necessary part of the development of acceptance/rejection thresholds for the recommended QC test methods, since, to conservatively ensure that a specified pull-out bond stress is achieved, the threshold on the QC test must be chosen as the value where the prediction interval lower bound is equal to the pull-out stress threshold. The regression model gives an estimate of the average pull-out stress if the pull-out test was actually conducted repeatedly on the same source of strand. For a given measurement of the predictor, half of the actual pull-out test results would be expected to fall above this average and half would fall below. The distribution of individual pull-out observations about that average pull-out stress is the basis for the prediction interval, which is calculated based on the variability in the data used for the regression.

This concept is demonstrated graphically in Figure 3, which shows the prediction interval lower bound plotted along with the regression line, and data for the mortar pull-out plotted versus the change in corrosion potential. If a specified threshold on mortar pull-out is defined as 0.313 ksi, the threshold on the corrosion potential is the value where the pull-out threshold and the curve delineating the lower bound of the prediction interval intersect, shown by the red lines in the plot. In this case, the threshold would be approximately −0.175 V.

If a multiple-predictor regression model is used for prediction of the pull-out stress, the prediction interval is still needed. Determining the prediction interval for models based on multiple predictors is possible; however, it is more complicated and can not be shown graphically. The predicted pull-out stress is not uniquely determined by a single combination of predictors, but can be found based on numerous combinations of those predictors. However, the prediction interval for the pull-out stress will be different depending on the specific combination of predictors used. That means that when multiple regression is used to improve the predictive ability of the model, a single threshold can not be defined. Instead, for a specific set of predictors, a new prediction interval must be calculated based on the set of data used to develop the regression model. The lower bound of the newly calculated prediction interval must then be compared with the specified pull-out threshold.

Computational Tool

To facilitate the implementation of the prediction interval concept, a computational tool in the form of a Microsoft

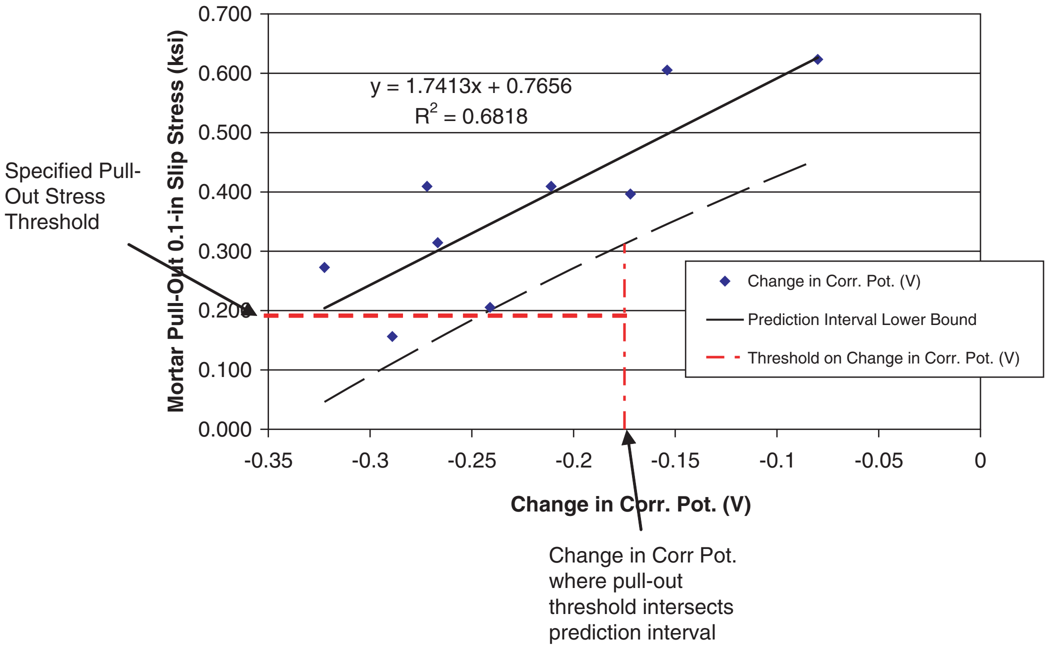

Figure 3. Threshold determination using the prediction interval.

Excel worksheet was developed that demonstrates the calculation of prediction intervals for single- and multiple-predictor regressions. This tool employs the calculation outlined in Appendix B.

Selection of Confidence Level

For the threshold determinations performed based on the data collected in this study, the confidence level was taken as 90%. This means that for a given surface and chemical test result, 10% of the pull-out results would be expected to fall below that prediction interval. This confidence level is lower than the 95% confidence interval that is most commonly used as the basis for probabilistic design in structural engineering analysis. Using a confidence level as high as 95% will result in very conservative thresholds for the surface and chemical tests, so a 90% confidence level was used instead.

Precision

A final round of testing using the QC methods that correlated well with bond performance was conducted to provide the basis for a precision statement to be included in the test methods. To determine the precision (i.e., the repeatability) of the methods, the selected tests were repeated up to six times on samples of strand obtained from the same source. This testing was conducted on a single source identified as a middle-range performer in that particular test during the correlation testing. The results of this testing were developed according to ASTM practice and are reported based on the standard deviations measured among the test results. A determination of bias in the testing methods is not possible at this time since a known reference sample can not be selected in a universally acceptable manner.

Supplemental Investigations of Strand Bond

In addition to testing performed to evaluate the proposed QC testing methods, supplemental investigations were conducted to provide insight into the causes of poor bond. These investigations aided in the development and interpretation of the results of the QC tests. The supplemental investigations included studies of: (1) surface roughness and distribution of lubricant residue, (2) the concrete/strand interface, and (3) local variation of strand diameter. The studies of surface roughness included metallographic studies of strand from poor and good bonding sources, scanning electron microscopical studies of the strand surface, and study of surface roughness by electrochemical impedance spectroscopy. Investigation of the concrete/strand interface included a study of the cement hydration at the strand interface, and a petrographic and chemical investigation of the strand/concrete interface in transfer length prisms in which poor bond was observed.

CHAPTER 3

Findings and Applications

Findings of Industry Survey

Information regarding strand manufacture and the appropriate test methods was solicited directly from manufacturers of lubricants used to manufacture PC strand and from PC strand manufacturers.

Lubricant Manufacturers

Some specific information gained from representatives of the lubricant manufacturing industry included the following:

- A wide range of lubricants is used in U.S. strand manufacturing operations. One supplier suggests at least six of their products are used throughout the country in strand production.
- Lubricants are often a blend of multiple soaps, including sodium and calcium stearates, and can include fillers such as lime.
- The type (formulation) of lubricant recommended by the lubricant manufacturer is dependent on the type of metal pretreatments and the condition of the metal surface.
- Rod or wire can be either mechanically or chemically cleaned (acid pickled). Mechanical descaling can be performed using shot blasting, reverse bending, or wire brushing. Chemical cleaning was considered superior to mechanical descaling by lubricant manufacturers. Subsequent pretreatments include borax and lime.
- Wire that is only marginally cleaned (retained scale) requires a lubricant with a heavy residue.
- Products are formulated with different melting points. Twenty or more different sodium soaps are available, as well as different sources of fatty acid, such as stearic acid.
- One-to-three different products can be used in line for manufacturing prestressing strand.
- Calcium salts of fatty acids are often used in the first set of dies. Some plants use calcium soaps in three die boxes and sodium stearate in the rest. Other plants use sodium soaps in all die boxes.
- High-carbon wire and high-speed lines result in greater heat and require lubricant formulation with a higher softening point and a high fat content. (High fat content equals 60% for calcium-based lubricants and 75% for sodium-based lubricants.) Some formulations use a blend of fats (hydrotallow and stearic acid) to achieve certain melting (softening) points.
- In general, calcium soaps melt at a lower temperature than do sodium soaps.
- Additives in lubricants can include lime, sodium carbonate, sodium phosphate, sodium sulfate, antioxidants, and borax.

Strand Manufacturers

Some specific information gained from representatives of the strand manufacturing industry included the following:

- Induction furnaces are typically used during the stabilization process after the wires have been stranded. The strand surface temperature is typically measured with in-line infrared sensors and is in the range of 700-785°F as the strand leaves the furnace. The strand remains at approximately this temperature for 2-10 sec, until the steel is quenched in a water bath. Although there may be some limited washing action as the strand passes through the bath water, which is circulated through filters and a heat exchanger, no other cleaning methods were reported. Additional cleaning methods used by some plants may be proprietary.
- Problems in the quality of raw materials used in the strand production process are identified when a significant change in drawing performance, such as reduced die life or an increase in energy demand, is observed. It is common for adjustments in the drawing process to be necessary since lubrication performance is highly temperature-dependent

and modified behavior may be caused by ambient humidity and temperature differences. Such variations are typically accommodated by modifying line speed.

- No QC testing is conducted on lubricants used in strand production.
- Until recently, the post-production strand QC testing at strand production facilities generally consisted of periodic relaxation strength and modulus of elasticity testing, with no specific tests performed to verify bond performance. However, in 2007, a 2-year quarterly program of NASPA bond tests (pull-out tests from mortar) was initiated, and initially included the participation of 10 NASPA members supplying strand to the domestic U.S. market. One producer has since stopped supplying to the domestic market and has dropped out. Currently, this program consists of tests conducted on lengths of strand randomly sampled from the first 500 ft. of a 1500-ft. pack of strand supplied for testing. This testing is performed under the supervision of Bruce Russell of OSU, who has been contracted to perform this work. Three NASPA bond tests, consisting of six strands each are performed. Only 0.5-in. diameter strands have been included in this effort.
- Strand producers demonstrated interest in the chemistry-based surface characterization methods and expressed a willingness to use such methods in their plants, if their efficacy can be proven.

Findings of Supplemental Investigations

Detailed results of supplemental investigations are presented in Appendix D. The significant findings are summarized here.

The supplemental investigations focused on the strand and strand/concrete interface, in an attempt to gain a greater understanding of some factors that may influence bond. Studies of surface roughness and the relationship between surface roughness and lubricant residue were carried out using metallographic methods, scanning electron microscopy with energy dispersive X-ray spectrometry, and electrochemical impedance spectroscopy (EIS). The effect of lubricant residue on cement hydration was evaluated with petrographic and chemical investigations of the concrete immediately adjacent to the strand.

Possible relationships between surface-roughness characteristics (together with the concentration of residual lubricant on the strand) and bond strengths were assessed. The relationship between surface roughness and bond strength is complicated by the presence of residual lubricant, as demonstrated by scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) analysis; higher concentrations of residual lubricant appear to be associated with greater surface roughness, with the lubricant occurring predominately within depressions on the surface. Based on metallographic studies, a strong correlation between surface roughness and bond strength was not apparent, but only a very limited number of strand cross-sections were evaluated because of the labor-intensive nature of this technique. However, EIS studies were used to measure the capacitance per nominal unit area, a property controlled by the surface area of the strand on a microscopic scale. This microscopic surface area is linked to the microstructural roughness. A strong correlation between bond strength and a parameter equal to the ratio of the lubricant concentration and this capacitance per nominal unit area was found. Since the lubricant concentration is also based on the nominal surface area, this parameter is a measure of the relative concentration of lubricant residue per actual microscopic surface area.

Petrographic studies have shown that there is a difference in cement hydration at the interface of strand compared to the surrounding concrete that appears to be a direct result of the presence of residual lubricant. Cement hydration studies comparing the interfacial features of as-received strand with strand that had been cleaned and stripped of its residual lubricant demonstrate that the cement particles at the strand interface of the as-received strand appears to be less hydrated than the surrounding cement, while the interface of the cleaned strand is similar to the bulk cement. This reduction in cement hydration at the interface was also seen in studies of the transfer zone of a concrete sample containing strand that exhibited poor bond. The residue on the strand appears to have the ability to affect hydration of the cement immediately adjacent to the strand.

These supplemental investigations, although not directly involved in formulating the QC tests, provided a greater understanding of concrete, lubricant, and strand interaction. The presence of residual lubricant on the surface of the strand is a result of the amount of lubricant used, heating procedures, washing procedures, and the microscopic surface roughness. Residues of chemical surface treatments and/or lubricants appear to have an impact on cement hydration adjacent to the strand, possibly also reducing the strength of the bond.

Findings of Evaluation of Test Methods

Detailed results of the testing program are presented in Appendix B. The conclusions of the testing program are summarized here.

Mechanical Testing

As mentioned, the transfer length test is considered the most realistic measure of bond performance and was used as the basis for evaluating the ability of the pull-out test methods to accurately measure bond characteristics. The coefficients of determination calculated based on the regression between these methods and the average bond stress over the transfer length are given in Table 3. The concrete pull-out test results correlated better with bond quality than the other pull-out test methods that were evaluated. Pull-out testing from mortar also showed promise as a means to evaluate bond, and the existing correlation is deemed sufficient to justify further study. This limited program can not be considered a definitive evaluation of these methods; just three strand sources were evaluated. Nevertheless, this conclusion that pull-out from concrete is the superior test is contrary to that of other studies of pull-out test methods, including that sponsored by NASPA, which have concluded that the mortar pull-out test is superior at assessing bond performance (Russell and Paulsgrove 1999, Russell 2001, and Russell 2006). This correlation, evaluated during the Screening Round of testing, was not explored further in the Correlation Round.

Chemical and Surface Testing

Transfer length was not measured on the historic strand and only partial transfer length test results were available for the OSU strand. Consequently, the evaluation of the effectiveness of the chemical and surface QC test methods in predicting bond performance was determined by comparing the QC test results against performance measured in pull-out tests. The coefficients of determination for these QC methods are given in Table 4. The P-values for selected methods are given in Table 5.

Table 4. Coefficient of determination (R^2) from linear regression with concrete and mortar pull-out at 0.1-in. and 1st slip.

Test Method		QC Level	Coefficient of Determination (R^2) from Regression with Mechanical Test	
			Concrete Pull-Out (0.1-in. and 1st Slip)	Mortar Pull-Out (0.1-in. Slip)
Contact Angle (°)	As received	I	0.04	0.35
	After $Ca(OH)_2$ dip	I	**0.61**	**0.57**
	After $Ca(OH)_2$ dip—stearate only†	I	**0.44**	**0.84**
	After ignition	I	N.A.	0.00
pH		I	0.97	0.18
Loss on Ignition		I	**0.86**	**0.16**
Loss on Alkali Bath		I	0.17	0.76*
Change in Corrosion Potential after 6 h	As received	I	**0.72**	**0.68**
	After $Ca(OH)_2$ dip	I	0.80	1.00*
	After ignition	I	N.A.	0.00
Surface Roughness, R_a		I	0.93	0.16
Corrosion Rate	As received	II	0.67	0.09
	After $Ca(OH)_2$ dip	II	1.00	0.00
	After ignition	II	N.A.	0.18
Organic Residue Extraction	Total	II	**0.81**	**0.12**
	Total—stearate only†	II	**0.88**	**0.63**
Sodium	Warm water	II	0.34	0.31
	Total	II	0.12	0.02
Potassium	Warm water	II	0.39	0.00
	Total	II	0.28	0.14
Calcium	Warm water	II	0.17	0.06
	Total	II	0.22	0.07
Zinc	Warm water	II	0.21	0.05
	Total	II	0.25	0.21
Boron	Warm water	II	0.30	0.10
	Total	II	0.28	0.11
Phosphate	Total	II	N.A.	0.17
Combined Index for B, Ca, & Org. Res.	Scaled for combination	II	0.78	0.28

R^2 values presented in bold are for those methods recommended for use in a QC program.
* Test method not included in Correlation Round. Regression based on three sources.
†Only those sources identified as containing primarily stearate-based compounds by FTIR analysis are considered.

Table 5. P-value from linear regression with concrete and mortar pull-out at 0.1-in. and 1st slip.

Test Method		QC Level	P-Value from Regression with Mechanical Test	
			Concrete Pull-Out (0.1-in. and 1st Slip)	**Mortar Pull-Out (0.1-in. Slip)**
Contact Angle (°)	After $Ca(OH)_2$ dip	I	**0.039**	**0.019**
	After $Ca(OH)_2$ dip—stearate only†	I	**0.262**	**0.029**
Loss on Ignition		I	**0.003**	**0.285**
Change in Corrosion Potential after 6 h	As received	I	**0.356**	**0.006**
Organic Residue Extraction	Total	II	**0.002**	**0.353**
	Total—stearate only†	II	**0.006**	**0.110**

†Only those sources identified as containing primarily stearate-based compounds by FTIR analysis are considered.

At the initiation of this study, the surface and chemical methods were divided into Level I and II QC tests, as based on the required effort and complexity of each test. These correlations are discussed separately here, since the level of correlation required to justify the use of each test method is different for each QC level. As can be seen in Table 4, a number of the surface and chemical test methods that showed good correlation with concrete pull-out test results did not correlate as well with the mortar pull-out test results. This may be indicative of the inadequacy of the surface and chemical methods, but may also be related to inaccuracies or inconsistencies in the pull-out test methods.

Level I Quality Control Tests

The objective of the Level I QC test methods is to quickly and easily determine if strand properties that have been correlated with questionable bond are present. The minimum correlation required for these tests to be useful is somewhat lower than for the Level II QC tests.

- Contact angle—Contact angle correlated with bond only after the strand sample was subjected to exposure to a saturated calcium hydroxide solution. This correlation is higher for those sources judged to carry only stearate-based lubricants, when performance assessed with mortar pull-out is considered. Nevertheless, the P-values calculated when comparing this test against mortar and concrete pull-out testing are low (0.039 and 0.019, respectively), suggesting that the relationships between both pull-out test methods and this surface test are statistically significant. It is likely that this high correlation after the calcium hydroxide solution exposure occurs because the resulting residues are similar compounds (the stearates having converted mostly to calcium stearate) that influence the surface tension in proportion to their concentration on the strand surface. Greater concentrations of residue make the strand surface more hydrophobic and increase the contact angle. It is recommended that this method be included as part of a future QC program.
- Examination under UV light—A limited quantity of fluorescing material was observed, and no correlation to bond was found. This method should be abandoned.
- Testing pH—The pH test was successful in finding a correlation with bond as measured by concrete-based pull-out testing on a limited dataset, but it was unsuccessful at finding a similar correlation based on mortar pull-out test results. It also appeared that this test was only effective for differentiating strands produced with a borax pretreatment. Therefore, this method may only be applicable for strand produced with borax pretreatments. More study is needed before a recommendation regarding the adaptation of this method can be made.
- Loss on ignition—A good correlation was found between the weight LOI and bond performance measured in concrete pull-out tests. Further statistical analysis suggests that there is greater than 99% confidence that the relationship between concrete pull-out and this test method is significant. This correlation and significance was not found based on mortar pull-out test results. Nevertheless, this is one of the easiest tests to perform and is recommended for future QC testing, although not alone. Some other measure of bond performance should be included along with LOI in a QC program.
- Loss in alkali bath—Multiple cleaning procedures using sodium hydroxide solutions were attempted, but no correlation was observed between the weight loss and bond in concrete. Although a higher correlation was found with the mortar test, this higher correlation is only based on three sources. Interestingly, this is the only method suggested by the Wire Association International manual for measuring surface residues on wire. It is recommended that this test be abandoned.

- Change in corrosion potential—The drop in corrosion potential showed a good correlation with bond in both the Screening and Correlation Rounds of evaluation. The P-value (0.006) calculated when comparing this test against mortar pull-out testing suggests that the relationship between mortar pull-out and this test method is statistically significant. Testing after exposure to a saturated calcium hydroxide solution showed better correlation than testing in the as-received condition in the Screening Round; however, this higher correlation is only based on results from three sources, and it was judged that this additional conditioning effort is not worthwhile. It is hypothesized that the increased tendency for corrosion measured on poor bonding strand is a consequence of greater surface roughness measured at the microscopic scale. This microscopic roughness occurs at too fine a scale to affect bond through mechanical interlock, but makes the strand more likely to accumulate lubricant residue, which leads to poor bonding behavior. It is recommended that this method, conducted on strands in the as-received condition, be included as part of a future QC program.
- Surface roughness—The surface roughness parameter, R_a, correlated well with bond in concrete based on only three sources, but not with bond in mortar. Since an increased roughness was associated with poor bond, it appears that correlation to bond is not a direct effect, but is related to the tendency of the wire surfaces to retain residue. The profilometer used to measure this property is convenient for use in a QC setting, but does not appear to be sensitive enough to measure the roughness at the scale needed, nor does it test a sufficiently large surface of the strand for the test result to be representative of a property that can be tied to bond performance. Therefore, it is recommended this method be abandoned.

Level II Quality Control Tests

The objective of Level II QC testing is to provide a more conclusive prediction of bond performance than possible with Level I QC tests. These tests require either more advanced methods or more complicated equipment. The minimum correlation required for these tests is higher than that required for Level I QC tests.

- Corrosion rate—A strong correlation was measured between corrosion rate and pull-out bond stress in concrete. However, the correlation between corrosion rate and pull-out bond stress in mortar was relatively weak. Given this lack of consistent correlation, the uncertainty about the mechanisms involved in establishing the initial good correlation, and the complexity and equipment-dependent nature of this test, it is not recommended for inclusion in a future QC program.
- Organic residue extraction—The concentration of the organic residue correlated well with the bond performance in concrete, but only moderately with bond in mortar. Nevertheless, the P-value calculated when comparing this test against mortar pull-out testing for all samples was less than 0.01. This test is time consuming to perform, but gives the best direct measure of the type and quantity of drawing lubricants left on the strand surface during the manufacturing process. Of all the methods proposed, this method evaluates the property of the strand tied most obviously to bond quality. The presence of organic lubricants on the surface of the strand can only be expected to reduce bond performance. Therefore, it is recommended that this method be included as part of a future QC program. FTIR spectroscopy should be performed on the organic residues that result to ensure that residues being evaluated are consistent. This is necessary because the effect of residues with different chemistries is unlikely to be proportionally similar (e.g., a stearate-based lubricant residue will likely effect bond differently than a non-stearate-based lubricant residue). FTIR analyses will also identify contamination of the samples from other organic materials, such as oils, greases, or from release agents. The correlation between mortar pull-out stress and residue concentration was much higher when those sources carrying only stearate-based lubricants were included in the correlation analysis.
- Elemental analysis—Atomic absorption and visual light spectroscopy were used to determine the surface concentration of various elements. The concentrations of sodium and boron showed signs of a correlation with the mechanical properties measured in pull-out tests in both concrete and mortar. The concentrations of zinc, however, did not. The elemental analysis gives some insight into the type of pretreatment and lubricant in use and was useful for the purposes of this study. A combined index calculated from the normalized concentrations of boron, calcium, and organic residue (as explained in Appendix B) showed good correlation to pull-out bond in concrete. This correlation was not found with mortar pull-out results. Given the cost and equipment-dependent nature of the atomic absorption testing, it is not recommended for inclusion in a future QC program.

In summary, the following methods are recommended for inclusion in future QC programs:

- Weight Loss on Ignition (LOI),
- Contact Angle Measurement after Lime Dip,
- Change in Corrosion Potential, and
- Organic Residue Extraction with FTIR Analysis.

Test Methods and Precision Testing

The recommended QC methods have been written in AASHTO/ASTM standard method format in Appendix C, where they are titled:

1. Test Method for the Determination of the Surface Tension of Steel Strand by Contact Angle Measurement,
2. Test Method for Weight Loss on Ignition (LOI) of Steel Strand,
3. Test Method for Change in Corrosion Potential of Steel Strand, and
4. Test Method for Identification and Quantification of Residue on Steel Strand by Extraction, Gravimetric, and Spectroscopical Analyses.

Testing was conducted to provide the basis for a precision statement accompanying the proposed test methods developed for identifying strand bond performance.

The results for the four recommended test methods are given in Table 6. Note that each repeat test included testing of three strand sections. The precision and bias statements to be added to the standard test methods take the form illustrated in the following example:

> ***Test Method for Weight Loss on Ignition (LOI) of Strand***
>
> **Single-Operator Precision**—The single-operator standard deviation was found to be 0.014 mg/cm². Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.041 mg/cm². (These numbers represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670* [ASTM 2003].)
>
> **Bias**—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Development of Thresholds

For the recommended surface and chemical test methods to be useful in a QC setting, thresholds for acceptable bond behavior are needed. The usefulness of acceptance/rejection thresholds for the surface and chemical test results is dependent on the correlation of these results with minimum acceptable bond strengths established by physical test methods. The validity of thresholds developed in this way is also dependent on the validity of the physical test methods (such as pull-out tests) used as the basis for measuring bond performance. At the direction of the NCHRP supervisory panel, the transfer length testing originally planned for this test program as a basis for developing thresholds for the surface and chemical test results was not conducted. Instead, the thresholds for the chemical and surface test methods were based on the acceptance limits for the mortar pull-out tests proposed by Russell and adopted by NASPA.

The bond strength thresholds proposed by Russell are stated in terms of the force at 0.1-in. slip measured by the NASPA mortar pull-out test procedure. They are based on a set of development length tests conducted in parallel with the development of the NASPA strand bond test (Russell 2001, Russell 2006). The thresholds were derived using development length tests on four strand sources, (in what is referred to as the NASPA Round III study [Russell 2001]), and they are defined in terms of acceptance criteria for the average force at 0.1-in. slip from six pull-outs with a lower criterion for any single measurement of the six pull-outs. The Round III report proposed thresholds of 7,300 and 5,500 lbs, for the minimum permissible average and single test result, respectively for 1/2-in. diameter strand (Russell 2001). These minimum thresholds have since been increased to 10,500 and 9,000 lbs, but without additional testing (Russell 2006). For 0.6-in. diameter strand, the suggested thresholds are 12,600 and 10,800 lbs for the minimum permissible average and single test result, respectively (Russell 2006). No threshold has been suggested for other sizes of strand.

Despite the somewhat limited scope of the development process used to establish these NASPA test thresholds, the threshold determination effort for the surface and chemical testing conducted in this study was performed assuming that these thresholds were well-defined lower bounds for good bonding behavior. As has been done throughout this study, the thresholds were converted to bond stresses calculated as the force divided by the nominal surface area to support comparisons among all of the tested strands. When converted to a bond stress, the minimum threshold on the average of six tests of 10,500 lbs is equal to 0.313 ksi. This value was used as the basis for the threshold analysis.

Table 6. Precision test results for recommended QC tests.

Test Method	Organic Residue Extraction (mg/cm^2)	Weight Loss on Ignition (LOI) (mg/cm^2)	Average Contact Angle after Lime Dip (°)	Change in Corrosion Potential (V) after 6 h
Number of Repeats	6	6	6	5
Average of Results	0.069	0.091	73	-0.334
Standard Deviation of Results	0.013	0.014	4	0.047

Table 7. Regression coefficients for single-predictor models.

Predictor	Constant (x_0)	Coefficient (β)	Coefficient of Determination (R^2)	Threshold Corresponding to Mortar Pull-Out Stress of 0.313 ksi
Weight Loss on Ignition (mg/cm^2)	0.445	-1.403	0.16	Not Possible
Contact Angle after Lime Dip (°)	1.393	-0.012	0.57	73
Change in Corrosion Potential after 6 h (V)—as Received	0.766	1.741	0.68	-0.175
Extracted Organic Residue (mg/cm^2)	0.453	-1.752	0.12	Not Possible
Extracted Organic Residue (mg/cm^2)—Stearate Only	0.436	-1.943	0.63	Not Possible

Thresholds Based on Regression with Single Predictor

The initial efforts made to define thresholds for each of these recommended QC methods were based on single-predictor linear regressions and are described below. The results are summarized in Table 7.

Weight Loss on Ignition (LOI)—The prediction interval for LOI with a one-sided confidence level of 90% is shown in Figure 4. As can be seen in this figure, the prediction interval does not exceed 0.313 ksi anywhere over the range of test results observed in this study. For that reason, no threshold can be determined.

Contact Angle Measurement after Lime Dip—The prediction interval for Contact Angle after Lime Dip with a one-sided confidence level of 90% is shown in Figure 5. As can be seen in this figure, this prediction interval exceeds 0.313 ksi when the contact angle is less than 73°. Therefore, based on the data and the NASPA-defined threshold on mortar pull-out stress at 0.1-in. slip, a Contact Angle after Lime Dip of 73° or lower is recommended to give a good (90%) confidence of adequate bond. This test must be run on recently manufactured strand with no surface weathering or rust (i.e., bright strand).

Change in Corrosion Potential—The prediction interval for Change in Corrosion Potential with a one-sided confidence level of 90% is shown in Figure 6. As can be seen in this figure, this prediction interval exceeds 0.313 ksi when the Change in Corrosion Potential is less negative than –0.175 V. Therefore, based on the data and the NASPA-defined threshold on mortar pull-out at 0.1-in. slip stress, a Change in Corrosion Potential of –0.175 V or more (i.e., less negative) is recommended to give a good confidence of adequate bond.

Organic Residue Extraction—The prediction interval for organic residue extraction with a one-sided confidence level of 90% is shown in Figure 7. As can be seen in this figure, the prediction interval does not exceed 0.313 ksi anywhere over the range of test results observed in this study. For that reason, no threshold can be determined. A similar analysis was attempted considering only those sources with organic residue that the FTIR analyses indicated was primarily stearate. This was done to eliminate potentially confounding influences of

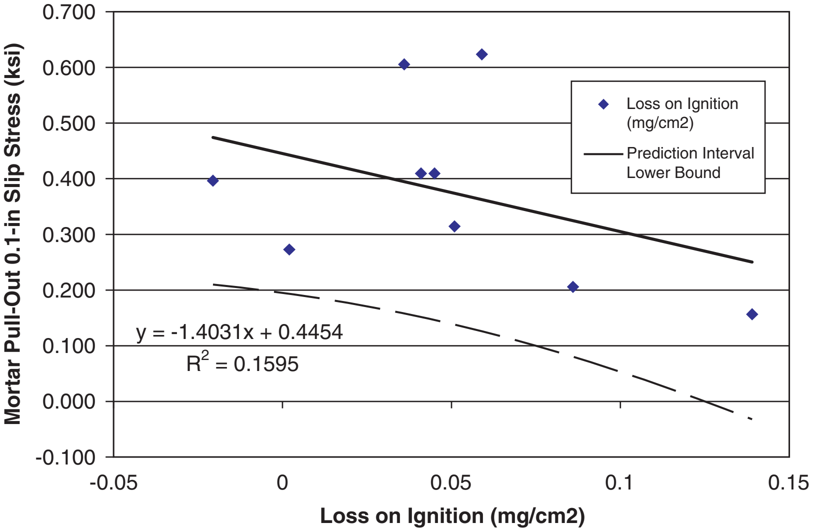

Figure 4. Prediction interval (confidence level = 90%) for Loss on Ignition. Threshold not possible.

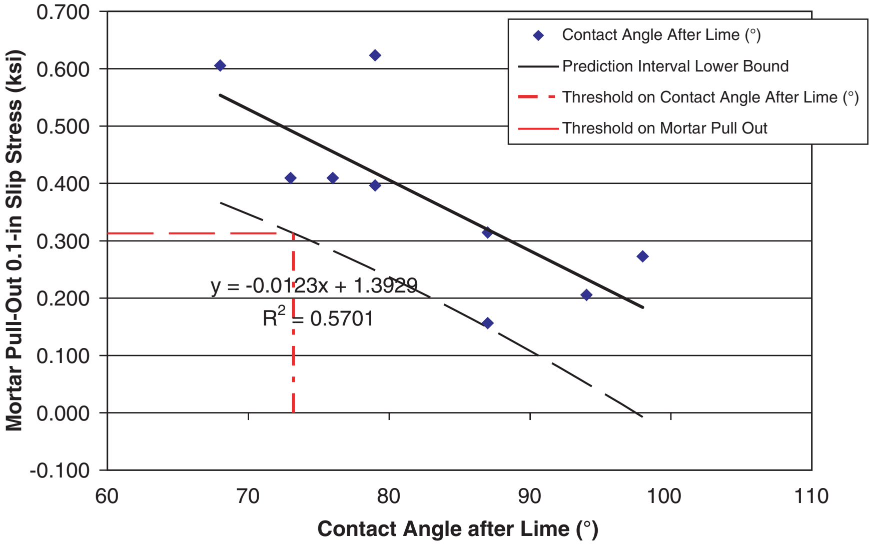

Figure 5. Prediction interval (confidence level = 90%) for Contact Angle after Lime Dip.

non-stearate-based lubricants and other surface contaminants. The prediction interval for this stearate residue with a one-sided confidence level of 90% is shown in Figure 8. As can be seen in this figure, the R^2 is higher, but the prediction interval still does not exceed 0.313 ksi anywhere over the range of test results observed in this study, and no threshold can be determined.

Thresholds Based on Regression with Multiple Predictors

An attempt also was made to determine if combinations of test results (e.g., a combination of contact angle and organic residue extraction test results) correlated with bond performance. Although numerous linear combinations were examined, the three combinations that showed the best correlation, based on the adjusted coefficient of determination (R^2 *adj.*), were as follows:

- Contact Angle Measurement after Lime Dip & Change in Corrosion Potential,
- Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only), and
- Weight Loss on Ignition (LOI) & Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Note that for multiple-predictor regression, a larger number of variables will increase the R^2. Therefore, the adjusted R^2 statistic, which accounts for the number of degrees of freedom in the dataset, was calculated as a means to compensate for this potentially misleading effect. The regression coefficients

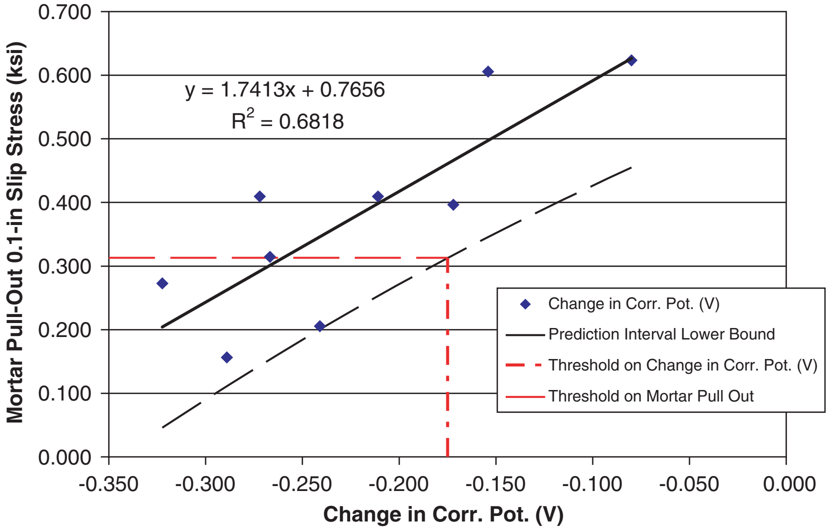

Figure 6. Prediction interval (confidence level = 90%) for Change in Corrosion Potential.

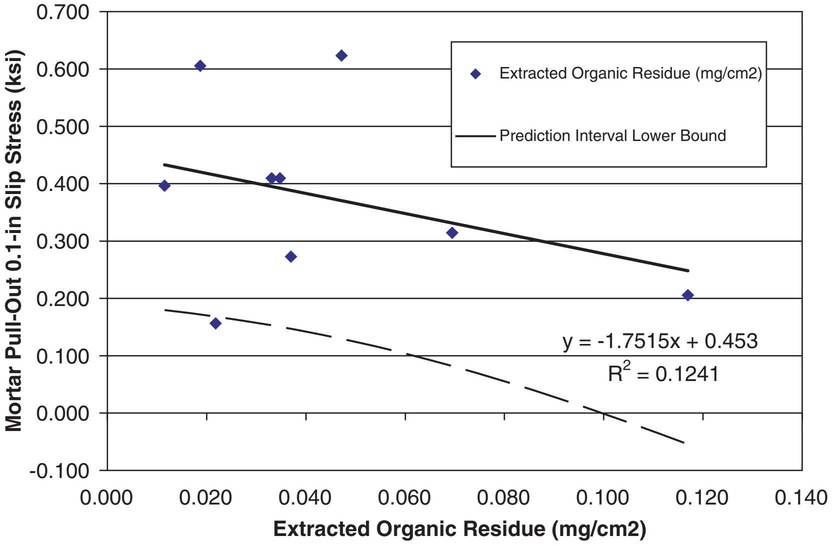

Figure 7. Prediction interval (confidence level = 90%) for Organic Residue. Threshold not possible.

and the R^2 *adj.* for these three models are given in Tables 8 to 10. The R^2 *adj.* values for these combinations were high and equal to 0.73, 0.98, and 0.76, respectively.

The regression indicated that the last combination of predictors listed above (Contact Angle Measurement after Lime Dip & Organic Residue Extraction) was a good predictor of bond and was performed based only on those strand sources that the FTIR analysis of the organic residue identified as being stearate only. This limited the number of data points used to develop the regression model to five, but was done as a means of eliminating potentially confounding influences of non-stearate-based lubricants on the results obtained by the contact angle and organic residue extraction measurement methods. Given the high level of correlation with the multiple regression approach, this model may be particularly useful in a production setting where the lubricant in use is known.

The prediction interval can not be shown in a two-dimensional plot as was done with the single-variable models. This is because multiple combinations of variables can give the same output. For this reason, a separate prediction interval must be calculated for each combination of variables. To give a sense of how these multiple regression models might be used, tables have been prepared showing the predicted pull out, the

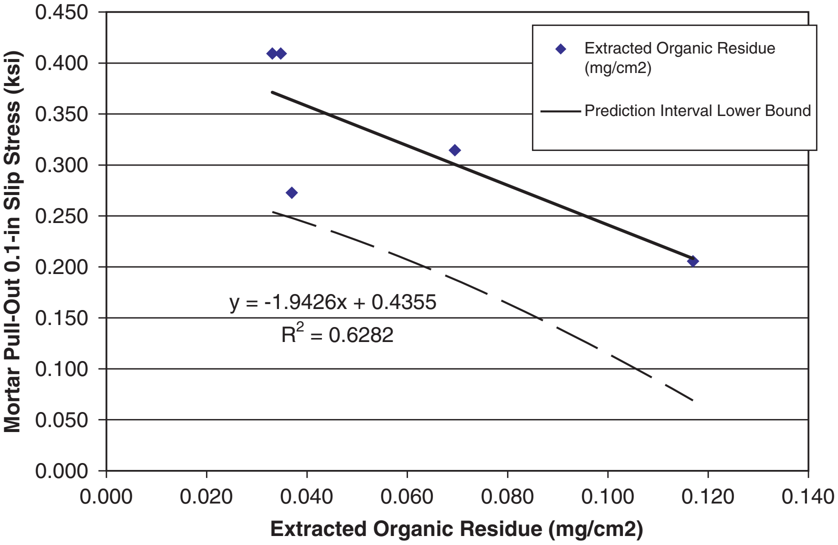

Figure 8. Prediction interval (confidence level = 90%) for Organic Residue when FTIR analysis indicates organic residue is primarily stearate. Threshold not possible.

Table 8. Regression coefficients for model based on Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Predictor	Coefficient
Constant	1.209
Contact Angle after Lime Dip (°)	-0.007
Change in Corrosion Potential after 6 h (V)—as Received	1.233
Adjusted Coefficient of Determination (R^2 *adj.*)	**0.73**

Table 9. Regression coefficients for model based on Contact Angle Measurement after Lime Dip & Organic Residue Extraction (stearate-based residues).

Predictor	Coefficient
Constant	0.864
Contact Angle after Lime Dip (°)	-0.006
Extracted Organic Residue (mg/cm^2)	-1.093
Adjusted Coefficient of Determination (R^2 *adj.*)	**0.98**

Table 10. Regression coefficients for model based on Weight Loss on Ignition (LOI), Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Predictor	Coefficient
Constant	1.203
Weight Loss on Ignition (mg/cm^2)	-0.846
Contact Angle after Lime Dip (°)	-0.006
Change in Corrosion Potential after 6 h (V)—as Received	1.178
Adjusted Coefficient of Determination (R^2 *adj.*)	**0.76**

lower bound on the prediction interval, and the comparison of the lower bound and the actual pull-out test result with the specified mortar pull-out threshold of 0.313 ksi, for two of the three multiple regression models. These are shown as Table 11, which was developed for the model based on the combination of Contact Angle Measurement after Lime Dip & Change in Corrosion Potential, and as Table 12, which was developed for the model based on the combination of Contact Angle Measurement after Lime Dip & Organic Residue Extraction for those residues determined to be stearate only.

Using Table 11 as the example, the first row shows the results of these two individual QC tests obtained for Source 349. Based on the regression model, the predicted mortar pull-out stress at 0.1-in. slip is 0.264 ksi. The lower bound on the prediction interval for that combination of the two test results must be calculated specifically using those values and is 0.131 ksi. Since 0.131 ksi is less than the mortar pull-out threshold of 0.313 ksi, this source fails to meet the minimum threshold for the combined Contact Angle Measurement after Lime Dip & Change in Corrosion Potential performance. For Source 548, the lower bound on the prediction interval calculated for the specific combination of test results measured for that source is 0.420 ksi, and this source "passes" since this value exceeds the 0.313 ksi threshold. The last column shows whether that strand would be expected to pass based on the actual pull-out test result. The conclusions reached based on the prediction interval and the actual pull-out test results are consistent for Sources 349 and 548. However, this is not always the case; the evaluation process based on the prediction interval is by definition conservative, and some sources will be judged as failing that may not fail in the actual pull-out test.

A similar example is shown in Table 10, which was developed for the model based on the combination of Contact Angle after Lime Dip & Organic Residue Extraction (for stearate-based residues).

Interpretation of Elemental Analyses Relative to Manufacturing Processes

The atomic absorption and colorimetric analyses of wash solutions from strands from various sources indicated varied

Table 11. Evaluation of prediction interval for model based on Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Strand Source ID	Contact Angle after Lime Dip (°)	Change in Corrosion Potential (V)	Mortar Pull-Out 0.1-in Slip Stress (ksi)			Pass/Fail* Based on Prediction Interval from QC Tests	Pass/Fail* Based on Pull-Out Test Result
			Experimentally Determined in Pull-Out Test	Value Predicted by Regression for QC Results	Lower Bound of Prediction Interval		
349	87	-0.289	0.156	0.264	0.131	Fails	Fails
548	79	-0.080	0.623	0.576	0.420	Passes	Passes
697	68	-0.154	0.606	0.559	0.417	Passes	Passes
717	94	-0.241	0.206	0.276	0.136	Fails	Fails
478	73	-0.272	0.409	0.38	0.232	Fails	Passes
960	76	-0.211	0.409	0.435	0.303	Fails	Passes
102	87	-0.266	0.315	0.291	0.161	Fails	Passes
103	79	-0.172	0.397	0.463	0.331	Passes	Passes
151	98	-0.322	0.273	0.149	0.003	Fails	Fails

* Threshold for passing is 0.313 ksi.

Table 12. Evaluation of prediction interval for model based on Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only).

Strand Source ID	Contact Angle after Lime Dip (°)	Extracted Organic Residue (mg/cm^2)	Mortar Pull-Out 0.1-in Slip Stress (ksi)			Pass/Fail* Based on Prediction Interval from QC Tests	Pass/Fail* Based on Pull-Out Test Result
			Experimentally Determined in Pull-Out Test	Value Predicted by Regression for QC Results	Lower Bound of Prediction Interval		
717	94	0.117	0.206	0.211	0.176	Fails	Fails
478	73	0.033	0.409	0.420	0.388	Passes	Passes
960	76	0.035	0.409	0.401	0.371	Passes	Passes
102	87	0.069	0.315	0.303	0.274	Fails	Passes
151	98	0.037	0.273	0.276	0.240	Fails	Fails

* Threshold for passing is 0.313 ksi.

levels of sodium, calcium, potassium, zinc, boron, and phosphate on the strand samples. The presence and concentration of these elements is largely governed by the specific pretreatment process and wiredrawing lubricants used in the manufacturing of strand from each specific source, although other sources of some of these elements may also be involved. The pretreatments commonly used in strand production are zinc phosphate and sodium borate, also known as borax. Lubricants typically consist primarily of either sodium stearate, calcium stearate, or some other fatty acid blend.

Tables 13 and 14 show each strand source ranked in ascending order of concrete or mortar pull-out bond performance together with the prominent elements removed during the extraction process, and the presumed pretreatment and

Table 13. Compounds likely used in manufacture of each source—historic and recently manufactured strands.

Strand	Prominent Elements	Presumed Pretreatment	Presumed Lubricants	Concrete Pull-out Bond Stress (ksi)
153	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid	-
KSU-H	Na, K, B	borax	Na/K stearate	0.209
SC-F	Ca, Zn	zinc phosphate	calcium stearate	0.223
101	Na, K, B	borax	Na/K stearate	0.241
KSU-F	Na, K, B	borax	Na/K stearate	0.241
102	Na, Ca, B	borax	Na/Ca stearate	0.441
SC-H	Na, K, Zn	zinc phosphate	sodium stearate	0.472
151	Na, B	borax	calcium stearate	0.541
SC-IS	Na, Zn	zinc phosphate	sodium salt of fatty acid	0.682
103	Na, Zn	zinc phosphate	sodium salt of fatty acid	0.944

Table 14. Compounds likely used in manufacture of each source—OSU strands.

Strand	Prominent Elements	Presumed Pretreatment	Presumed Lubricants	Mortar Pull-Out Bond Stress (ksi)
349	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid and resin	0.156
717	Na, Ca, Zn, P	zinc phosphate	Na/Ca stearate	0.206
478	Na, K, Zn, P	zinc phosphate	Na/K stearate	0.409
960	Na, K, Zn, P	zinc phosphate	Na/K stearate	0.409
697	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid and resin	0.606
548	Na, K, Zn, P	zinc phosphate	Na/K salt of fatty acid	0.623

lubricants used based on the prominent elements. Knowledge about the types of pretreatments and drawing lubricants can be an important part of the interpretation of the QC test results.

These results of the elemental analyses suggest that the wires in the sampled strands were pretreated using one of two methods during the manufacturing process. Those strands that carried high amounts of boron typically carried low amounts of zinc and phosphate. As expected, the lubricants appeared to be either calcium or sodium/potassium stearates, but a number of strands showed evidence of both. This may result from different types of lubricants being used in separate dies in the drawing process.

There is a greater amount of sodium, potassium, and calcium than would be expected from the stearate compounds alone (based on the concentration of the organic residues extracted). Therefore, other sources of these elements are contributing to the values measured here. Possible sources include chemicals in the pretreatment processes described above, detergents used for cleaning, or fillers, such as lime, used in some drawing lubricants.

Interpretation and Applications

Development of Quality Control Program for Strand Bond

A number of QC test methods for predicting strand bond performance have been developed and evaluated in this testing program. The value of these tests was judged based on the correlation observed between these methods and mechanical testing methods. Three pull-out test procedures, differing mainly by the embedment material, were also examined during this program. Although pull-out testing from concrete appears to correlate best with transfer length, the most reliable and realistic measure of bond performance, the Correlation Round of this test program had to be based on available mortar pull-out results provided from the NCHRP 12-60 Program.

The following four test methods showed the best correlation with pull-out bond and are recommended for inclusion in future QC programs:

- Weight LOI (QC-I),
- Contact Angle Measurement after Lime Dip (QC-I),
- Change in Corrosion Potential (QC-I), and
- Organic Residue Extraction with FTIR Analysis (QC-II).

The QC tests have been divided into two categories, depending on the complexity and time required to conduct the tests: Level I (QC-I) and Level II (QC-II) tests. The QC level is shown in the bulleted list above.

A main objective of this study was to develop test methods that were more easily performed at more frequent intervals than mechanical pull-out tests, which are time consuming, especially for a prestressing strand producer. The three recommended Level I QC tests are all easier to conduct than pull-out tests and, although they require some training and the acquisition of some specialized equipment, could be conducted by strand producers or precasters. If a QC lab was set up, it is envisioned that performing all three of these tests on a given sample of strand would require less than four hours of an appropriately trained QC inspector's time. If more than one sample is tested, the amount of time required per sample would be much less since much of the effort would be duplicative. Although, as discussed further in the next section, the definition of thresholds on all four of these tests was not straightforward, all of these methods showed a correlation to bond performance in concrete, mortar, or both and would have value in a QC program as an indicator of bond quality.

Recently, strand manufacturers in the United States have begun conducting pull-out testing from a single, specially prepared spool of $^1/_2$-in. strand on a quarterly basis. Although this is obviously better than no testing, it currently represents a small portion of the strand produced annually by each supplier. Therefore, it is suggested that the recommended Level I QC methods could be conducted by strand producers on a weekly basis for each size of strand produced. As a frame of reference, a requirement of weekly testing is much less onerous than the QC program requirements for at least one other reinforcing steel product—during production of epoxy-coated reinforcing steel at many manufacturing facilities, a number of QC tests, such as checks of blast cleaning effectiveness and coating flexibility, are conducted more frequently than every four hours of production. It is also not uncommon for precasters to test concrete properties (including slump, air content, and strength) more frequently than once per day.

Regular QC testing should greatly decrease the likelihood that poor bonding strand would reach the market, and this type of testing would be a valuable supplement to the quarterly testing of only a single size of strand currently being performed. When lots of strand are produced that exhibit suspicious behavior identified by these test methods, this could then prompt additional testing using the Level II organic residue extraction test and mechanical pull-out testing.

It is also noted that routine QC analyses of new batches of the drawing lubricants are not routinely conducted. Instead, problems with lubricant are generally only noted while the wiredrawing process is ongoing. Although the development of such a test program was beyond the scope of this research, greater QC as part of the lubricant acquisition process also would add to the confidence in bond quality.

Thresholds

Thresholds for two of these QC tests (Contact Angle Measurement after Lime Dip and Change in Corrosion Potential) have been developed. This was done based on prediction intervals for the regression calculated from the available data, a minimum criterion on the mortar pull-out stress adopted by NASPA, and a selected confidence level. The available data, consisting of the mortar pull-out results and QC test results for the included strand sources, were not sufficient to allow threshold determination for the other two methods with the same constraints.

The thresholds that were possible were calculated in a conservative manner to ensure adequate bond performance. However, the 90% confidence prediction interval thresholds on the change in corrosion potential and contact angle test would suggest that of the nine samples (two of which came from the source) included in the program, only two and three of the nine samples would be judged to be acceptable based on these test methods, respectively. Although any conservative approach for predicting a response based on an empirically developed relationship should be expected to underestimate that response, this is in contrast to the six of nine that would be judged acceptable based on the pull-out test itself. The inability to develop thresholds for two QC test methods and the strongly conservative nature of the thresholds that were developed has resulted from the large prediction intervals calculated for these relationships.

Regression with multiple predictors also has been performed to determine if results of selected QC methods could be combined to better predict bond. The following three combinations showed the best correlation, based on the adjusted coefficient of determination (R^2 *adj.*):

- Weight LOI & Contact Angle Measurement after Lime Dip & Change in Corrosion Potential,
- Contact Angle Measurement after Lime Dip & Change in Corrosion Potential, and
- Contact Angle Measurement after Lime Dip & Organic Residue Extraction (for stearate based residues).

The adjusted coefficients of determination for each of these combinations were higher than the coefficients of determination for the single-predictor regression models.

Thresholds for multiple-predictor regressions can not be determined using the same procedure used for single-predictor regressions. Instead, the lower bound on the prediction interval must be calculated for each combination of test results. One of the models (for the combination of Contact Angle Measurement after Lime Dip & Organic Residue Extraction) for stearate-based residues accurately predicted performance. However, the two models that included all nine of the strand samples both predicted that three of these strand samples would be judged to be acceptable, in contrast to the six of nine that would be judged acceptable based on the pull-out test itself. Although these multiple-predictor regression models do appear to be more effective than the individual QC tests, the strongly conservative nature of the conclusions regarding acceptable performance is related to the large prediction intervals.

There are a number of possible reasons, as follows, that the prediction intervals are not smaller:

- The QC test methods are inadequate—It is possible that the QC methods do not measure a property of the strand that is sufficiently strongly linked to bond performance. It is also possible that the QC tests measure only a part of what determines bond and that other factors exist that are equally or more important. This may mean that, while an individual QC test result is not sufficient to determine strand bond performance by itself, the QC result must be combined with the result from another test.
- The QC test methods or the mortar pull-out test method were susceptible to large scatter—All four of the recommended test methods produced regression models that predicted average results for the range of QC test results obtained that spanned the mortar pull-out threshold. However, the difference between the predicted average result and the lower bound on the prediction interval is strongly influenced by the scatter about the best-fit line. Recall that the line fit shown on the regression plots is the average result (i.e., half of the pull-out test results will be above this line and half will be below). A large amount of variation in the data used in the regression analysis will lead to a large prediction interval. Therefore, despite the fact that the method may test a property strongly linked to bond, if that property is difficult to measure with good precision, definition of threshold may be difficult. It should also be noted that this large scatter may be the result of significant local variations in the bond properties of the strand. It has been suggested by other researchers that the concentration of lubricant residue is highly variable even within a single spool and that significant differences may exist in strand separated by as little as 20 ft. Although some attempt was made to track the proximity of strand samples used in each of the various test methods in the Screening Round, this was not possible in the Correlation Round, and any such variation has become inseparably combined with variations in the test methods.
- The sampled sources were too closely grouped in terms of bond performance—In any regression analysis, greater

separation of the ranges of dependent variables (QC test result) and independent variable (pull-out response) will result in greater confidence in the model that is developed. Even for the same scatter on the individual test results, if samples with a wider range of performance were used, a smaller prediction interval would result.

- A limited number of data points were available for the regression analysis—A greater number of data points would increase the confidence in the regression model estimates' ability to accurately predict performance. This would reduce the prediction interval.

Future of Quality Control Program and Thresholds

The discussion of the QC test methods and QC test program given here has focused on the sampled sources used in this test program. A number of thresholds have been proposed based on the relationships between the QC test and the mortar pull-out test results for this sample set. Although a significant amount of work and scientific rigor has gone into their development, these thresholds should not be considered absolute and immutable.

Because of the issues discussed and the finite nature of this test program, the number of samples included in the regression analyses was limited. However, it is suggested that the threshold development process could be an ongoing process. If the recommended QC methods were conducted on the samples selected for inclusion in the quarterly mortar pull-out test program currently underway by NASPA plants, that would provide nine additional data points for inclusion in the regression dataset each quarter. These data could be included in future regression analysis and used to refine the existing thresholds or perhaps allow the definition of future thresholds for those methods for which determination was not possible at this time. Even if these sources proved to be all of similar bond quality, the additional data would likely serve to improve confidence in the regression model.

The proposed thresholds can be applied to new sources of strand, but this should be done with some caution. Strand produced with a different pretreatment process or with a lubricant with significantly different chemistry may not respond similarly to strand produced with a borax or zinc phosphate pretreatment and largely calcium or sodium stearate lubricants. A main objective behind the inclusion of the FTIR test in the organic residue extraction test method is to confirm that the organic component of the lubricant at least is consistent. It is likely that the existing thresholds will reject a larger number of sources than the mortar pull-out test alone.

If the type(s) of pretreatment and lubricants used in the wiredrawing process are known, method-specific correlations and thresholds could be determined. For example, if only a borax pretreatment and stearate-based drawing compounds were used to manufacture the strand in a certain plant, the effects of other wiredrawing compounds on the QC test results would not be present, and it is likely that a more consistent response would be achieved (resulting in higher R^2 and smaller prediction intervals). Based on the same predefined mortar pull-out threshold, different QC test thresholds could be developed for that particular manufacturing process. This would require each manufacturer to maintain a record of QC test and pull-out test results from strand they produced. With the computational tool developed as part of this program, regression analysis could be conducted. A QC program developed on this basis will be the most effective use of the recommended QC test methods.

Computational Tool

As mentioned, a computational tool in the form of a Microsoft Excel-based spreadsheet has been developed. This Excel workbook was designed to predict whether prestressing strand will exhibit adequate bond properties based on results from surface and chemical QC test methods developed as part of this project. The workbook performs this prediction according to the procedures outlined in Appendix B of this report.

This is done by calculating the prediction intervals for single- and multiple-predictor regressions and for determining the threshold on the QC test that corresponds to a predefined threshold using the mechanical test method. This tool is designed to: (1) develop a regression model to predict mortar pull-out stress from inputted surface and chemical QC test and mechanical (mortar) pull-out test results, (2) establish the lower bound of the prediction interval for the regression model for a desired level of confidence, (3) compare the lower bound of this prediction interval with a predefined threshold for the mortar pull-out stress with the chosen level of confidence, and (4) determine a pass/fail threshold for the QC test, if possible. There are individual worksheets for each of the recommended QC tests and for the recommended combinations of these tests. The user has the ability to modify the following inputs: desired level of confidence (default: 90%), threshold for acceptability (default: 0.313 ksi), and the QC test result for a new strand source.

The acceptability of a given source is determined by comparing the lower bound on the prediction interval to a predefined pass/fail threshold for the mortar pull-out test. Based on this comparison, a judgment is made as to whether the source exhibiting the QC test results is expected to exceed that threshold with the chosen confidence level. Indication is then given if the source passes (lower bound on prediction interval above threshold) or fails (lower bound on prediction interval below threshold). For the combined, multiple-predictor regressions, the prediction interval is different for each set of input QC results.

A universally applicable set of thresholds on the QC test results can not be generated, and the prediction interval cannot be simply plotted. Instead, the prediction interval must be calculated separately for each combination of QC test results. This tool accomplishes that calculation task. The multiple-predictor analysis is interpreted in terms of the pass or fail statements.

CHAPTER 4

Conclusions and Recommendations

The predictable transfer of prestressing force from strand to concrete is essential for the reliable performance of prestressed concrete. Residual films of lubricant and other contaminants remaining on the strand surface after manufacture have been shown to reduce the bond between the concrete and steel. A set of QC procedures has been developed for use by strand manufactures or their customers as part of a routine QC program to enable rapid detection of potential bond problems related to strand residues.

An experimental program was conducted to evaluate a number of test methods proposed for this purpose. This included limited mechanical testing (pull-out testing from concrete, from Portland cement mortar, and from gypsum plaster-based mortar) and extensive surface and chemical testing (contact angle, examination under UV light, pH, LOI, loss in alkali bath, change in corrosion potential, corrosion rate, surface roughness, organic residue extraction/FTIR analysis, and elemental analysis). These tests, as well as transfer length tests, have been conducted on a range of strand sources to establish correlations between the proposed QC tests methods and bond quality.

Although pull-out testing from concrete appears to correlate best with transfer length, the most reliable and realistic measure of bond performance, the Correlation Round of this test program was based on available mortar pull-out results provided by Russell of OSU from the NCHRP 12-60 Program.

The following four test methods showed the best correlation with bond in concrete, mortar or both, and are recommended for inclusion in future QC programs:

- Weight LOI (QC-I),
- Contact Angle Measurement after Lime Dip (QC-I),
- Change in Corrosion Potential (QC-I), and
- Organic Residue Extraction with FTIR Analysis (QC-II).

The quality control tests have been divided into two categories, depending on the complexity and time required to conduct the tests: Level I (QC-I) and Level II (QC-II) tests. The QC level is shown in the bulleted list above.

Regression with multiple predictors has also been performed to see if results of these methods could be combined to better predict bond. The three combinations that showed the best correlation, based on the adjusted coefficient of determination (R^2 adj.), were

- Weight Loss on Ignition (LOI) & Contact Angle Measurement after Lime Dip & Change in Corrosion Potential (R^2 *adj.* = 0.76),
- Contact Angle Measurement after Lime Dip & Change in Corrosion Potential (R^2 *adj.* = 0.73), and
- Contact Angle Measurement after Lime Dip & Organic Residue Extraction (when organic residue is primarily stearate, R^2 *adj.* = 0.98).

The adjusted coefficients of determination for each of these combinations were higher than the coefficients of determination for the single-predictor regression models.

Thresholds for two of these individual QC tests and all of the combinations have been developed based on prediction intervals for the regression calculated from the available data and a minimum criterion on the mortar pull-out stress test adopted by NASPA. Thresholds for multiple-predictor regressions are not determined using the same procedure used for single-predictor regressions. Instead, the lower bound on the prediction interval must be calculated for each combination of test results. A computational tool in the form of a Microsoft Excel spreadsheet has been developed for this purpose, and is called the NCHRP No. 10-62 Prediction Interval Calculation.xls.

It is suggested that the three recommended Level I QC tests be adopted as part of a routine QC program for strand producers. To supplement the quarterly mortar pull-out testing program currently underway, this test should be conducted on a weekly basis for each size of strand produced.

Regular QC testing should decrease the likelihood that poor bonding strand would reach the market. Lots of strand exhibiting unacceptable behavior identified by these test methods should then be tested further using the Level II organic residue extraction test and mechanical pull-out testing.

The determination of thresholds for two of the individual QC tests (Contact Angle Measurement after Lime Dip and Change in Corrosion Potential of Strand) was possible based on the relationships between the QC test and the mortar pull-out test results for this sample set; however, these thresholds are conservative. The available data were not sufficient to allow threshold determination for the other two individual methods with the same constraints. The threshold determination process is governed by the prediction intervals, which are determined by the uncertainty in the regression results. Sources of uncertainty, which ideally would be minimized, include inability of the test methods to predict bond, scatter in both the QC and mortar pull-out test results, close grouping of sources in terms of bond performance, and a limited number of data points for the regression analysis.

Although a significant amount of work and scientific rigor has gone into the development of the thresholds, they should not be considered absolute. Additional data could possibly be used to reduce the uncertainty alluded to above and may allow a reduction in the thresholds. Specifically, if the QC tests were conducted on the samples included in the quarterly pull-out testing program currently being conducted by NASPA, this information would be valuable to further refine the regression relationships.

Another possible means for implementing these test methods is the development of process-specific regression models and thresholds. The dataset for this study included strand sources manufactured with a number of different pretreatment and lubricant processes. Limiting the data included in the regression analysis to a single production process, such as might be done at an individual strand manufacturing facility, would likely significantly improve the correlation of the QC test methods, since the QC test results would be influenced mainly by variations in concentration of a specific lubricant and pretreatment and not the simultaneous variations of a variety of lubricant and pretreatment chemistries and concentrations. A better correlation would also allow the development of less restrictive thresholds.

Future Work

Although a variety of researchers has made conclusions about the most appropriate mechanical test methods for evaluating strand bond, the limited results of this research program suggested that concrete pull-out test results correlated better with transfer length test results than with mortar pull-out test results. Although the mortar pull-out test demonstrated good correlation, it is not clear that this is the best test for measuring the strand bond quality. Further work is needed to resolve the questions related to performance-based (i.e., mechanical) tests of strand bond that were left unanswered by the elimination of further development of mechanical tests from this research program. It is recommended that a multi-laboratory test program be conducted following the concepts outlined by ASTM E1169-07 *Standard Practice for Conducting Ruggedness Tests* (ASTM 2007) and conducted at qualified laboratories that are not already involved in the historically contentious discussions regarding strand bond issues.

Although a small task if an effective test method can be defined, further work is also needed to ascertain the extent and significance of local variations in the strand performance within a single spool. During the program outlined in this document, the relationship between test results and proximity on the strand could not be tracked. Nevertheless, such a variation has direct impact on the evaluation of any test method, since such variation would become combined with variations related to the test method itself.

As stated, additional work is needed to refine the thresholds for bond acceptability that will be used to establish alert (pass/fail) thresholds for the QC test results. The incorporation of additional data into the regression analysis would improve the confidence in the validity and usefulness of the QC test methods and may also allow less restrictive thresholds to be defined.

The threshold on the mortar pull-out test result adopted by NASPA is based on development length testing of four strand sources. For the sake of the QC threshold determination conducted during this program, it has been assumed that this threshold is a well-defined absolute. However, although additional work to refine this threshold would require a significant effort, such an effort would be valuable and would provide greater confidence in the performance of strand.

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APPENDIX A

Review of Strand Bond Literature

Appendix A to the contractor's final report for NCHRP Project 10-62 is not published herein. For information, contact NCHRP.

APPENDIX B

Evaluation of Mechanical and Chemical Test Methods

CONTENTS

Introduction

A number of test methods were proposed for use as part of a quality control (QC) program to evaluate bond of prestressed concrete strand. These were classified as performance-based (i.e., mechanical) tests and surface and chemical tests.

The mechanical methods included:

- Pull out from concrete,
- Pull out from Portland cement mortar,
- Pull out from gypsum plaster-based mortar.

The surface and chemical methods included:

- Contact angle,
- Examination under UV light,
- pH,
- Loss on ignition,
- Loss in alkali bath,
- Change in corrosion potential,
- Corrosion rate,
- Surface roughness,
- Organic residue extraction/Fourier transform infrared (FTIR) spectroscopical analysis,
- Elemental analysis,
  - AA,
  - Visible light spectroscopy.

These tests, as well as transfer length tests, have been performed as part of this research program, which is reviewed in detail in this appendix. The purpose of this program was to determine if any of these proposed tests are applicable for use in a QC program. The QC tests have been divided into two categories, depending on the complexity and time required to conduct the tests: Level I and Level II QC tests. A summary of the tests methods, their QC Level, and the test objectives are given in Tables 2 and 3 of the Project Report.

Overview of Test Program

To evaluate these test methods, several rounds of experimentation were conducted: screening, correlation, and precision.

Screening Testing

The first round of experiments consisted of "screening" experiments. The objective for the screening experimentation was to eliminate those tests that are not helpful for predicting bond performance. Thus, the first step in this round of testing was to estimate the correlation between each surface or chemical test and bond performance; the second step was to identify those methods where some degree of correlation was indicated. For each source of strand, bond performance was measured in terms of pull-out stresses, transfer lengths, or both. Accordingly, for the purpose of this analysis, the bond performance was treated as the independent variable. The best experimental design for estimating a correlation is to place the design points as far apart as possible in terms of the independent variable. Thus, the optimal statistical design is to run each test on strands that show a range of bonding performance. For the screening experiments high, medium, and low bonding sources were desired. However, efforts to obtain a very low bonding strand were not successful. Although reports of low-bonding-strand incidents continue to surface in the precast concrete industry, "unused" samples of such strand remained elusive. Therefore, the screening tests on new strands were run on what are essentially high bond and intermediate bond strands. Some strand from a project in India with apparently very poor bond properties was tested. However, the surface chemicals on this strand did not appear to be the same as for North American manufactured strand, and therefore, the test results could not be directly compared to the rest of the test program.

Correlation Testing

The second round of experiments was performed for confirmation and calibration purposes. This round involved running additional tests using those methods that showed promise in the screening experiments. These selected tests were conducted on five new strand samples. This complete data set was then used to assess the correlation between the QC tests and bond performance, and to determine if the tests were able to accurately identify good and bad strand. It was also used as a basis for discussing pass/fail criteria for acceptable bond performance.

Precision Testing

A third round of testing was conducted to determine the precision, that is, repeatability, of those methods showing good correlation with bond strength. This was intended to form the basis of precision statements to be included in published test methods.

Basis for Evaluation—Transfer Length and Pull-Out Tests

Transfer length is the most reliable and realistic measure of bond performance. During the screening testing, the evaluation of correlations between the pull-out testing and the bond performance were based on performance as measured with transfer length tests conducted on the same source of strands.

Pull-out testing was conducted as part of the screening studies using three materials as the test matrix: a concrete, a Portland cement mortar, and a gypsum plaster mortar. Based on comparisons with transfer length tests conducted in this study and described in this document, the concrete pull-out test showed the best correlation with bond quality. The surface and chemical test methods were evaluated in the Screening Round based on the results of pull-out tests from concrete, again on strand samples from the same source. However, the evaluation of correlation of test results to bond in the Correlation Round of testing was based on results from a mortar pull-out test program associated with NCHRP Project 12-60 *Transfer, Development, and Splice Length for Strand/Reinforcement in High-Strength Concrete.* The Principal Investigator from this project supplied the strand samples for this portion of the study. No concrete pull-out testing was conducted in the Correlation Round of the experimental program.

Strand Samples

To assess the effectiveness of the mechanical and surface chemistry-based testing procedures, it was essential that samples representing the range of possible performance be evaluated. Since neither precasters nor strand suppliers were enthusiastic about associating themselves with poor-bonding strand, obtaining samples of strand from the lower end of the performance spectrum was difficult.

The strand sources included in testing for this program are listed in Table B-1. This table also includes the first slip or 0.1-in. slip stress measured in concrete pull-out tests or in mortar pull-out tests, depending on what was available. Each pull-out stress is the average of the pull-out stresses from at least six individual pieces of strand. The bond stresses are calculated based on the actual surface area and the embedment length of the strand.

These strand sources fall into three groupings: historic, recently manufactured, and OSU (Oklahoma State University).

Historic Strand—This study initially identified samples of strand for testing from prior tests conducted at Kansas State University (KSU) by Bob Peterman and at Stresscon by Don Logan that cover a wide range of pull-out behavior. These are referred to as "historic" strand and were manufactured between 1997 and 2004. Figure B-1 is a plot of first slip stress or stress at 0.1-in. end slip versus maximum stress from the data available from historic concrete pull-out tests.

When suggested minimum pull-out loads for acceptable bonding performance (suggested by Don Logan of Stresscon, based on a limited number of flexural beam tests conducted in the mid-1990s and his engineering judgment) are converted to bond stresses, they are 425 and 955 psi for the first slip and maximum stresses, respectively. These thresholds have been reproduced in Figure B-1.

Recently manufactured strand—Figure B-1 also shows the concrete pull-out performance of recently manufactured

Table B-1. Strand sources.

Strand Source ID	Strand Geometry				Mortar Pull-Out Testing			Concrete Pull-Out Testing (LBPT)		
	Size (in.)	Measured Diameter (in.)	Pitch (in.)	Lay (Handed-ness)	Location	Date	0.1-in. Slip Stress (psi)	Location	Date	0.1-in. Slip Stress (psi)
Historic Strand										
KSU-F	1/2 Special	0.524	7 5/8	Left	--	--	--	KSU	Mar 2004	241
KSU-H	1/2 Special	0.523	7 1/2	Left	--	--	--	KSU	Mar 2004	209
SC-F	1/2	0.503	8	Left	--	--	--	SC	May 1997	223
SC-H	1/2 Special	0.530	7 1/4	Left	--	--	--	SC	Nov 2002	472
SC-IS	1/2	0.501	7	Left	--	--	--	SC	Mar 2003	682
101	6/10	0.601	8 1/2	Left	--	--	--	SC	Oct 2004	241
Recently Manufactured Strand										
102	1/2	0.501	7 1/2	Left	KSU	Jun 2005	315	KSU	Jun 2005	441
103	1/2	0.503	8	Left	KSU	Jun 2005	397	KSU	Jun 2005	944
151	1/2 Special	0.517	7 1/2	Left	KSU	Jun 2005	273	KSU	Jun 2005	541
153	6/10	0.588	9	Right	--	--	--	KSU/SC	Jun/Aug 2006	142/406
OSU Strand										
349	1/2	0.505	8 3/4	Left	OSU	Jun 2004	156	--	--	--
548	1/2	0.500	7 5/8	Left	OSU	Jan-Feb 2004	623	--	--	--
697	1/2	0.503	7 1/4	Left	OSU	May 2004	606	--	--	--
717	1/2	0.500	8	Left	OSU	Feb 2004	206	--	--	--
478 *	1/2	0.499	7 5/8	Left	OSU	May-June 2004	409	--	--	--
960 *	1/2	0.500	7 1/2	Left	OSU	May-June 2004	409	--	--	--

* Samples designated 478 and 960 were from same source.

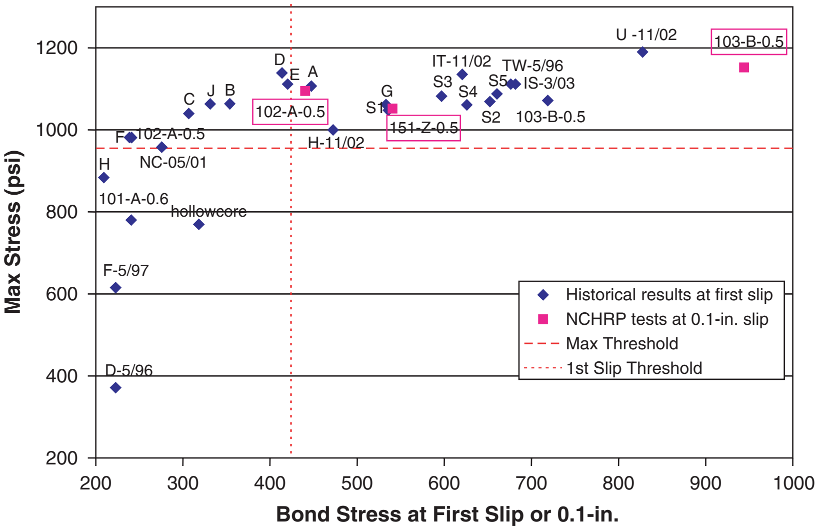


Figure B-1. Correlation between maximum stress and first observed or 0.1-in. slip stress for historic and recently manufactured strand.

samples identified during this project. These recently manufactured samples were obtained in large quantities for the purpose of this research and were used in the screening experiments. Notice that the greatest variation in the recently manufactured strand is not in terms of maximum stress but in terms of the early bond stress.

The recently manufactured strand sources (102, 103, and 151) were selected because initial testing indicated that they represented a range of first slip pull-out performance. None of these strands had significantly low maximum load pull-out performance.

The bond stress at 0.1-in. slip of Source 102, measured in concrete pull-out testing performed as part of this project, is slightly above Logan's 425 psi threshold. Of the 31 sources presented on this plot, 13 are to the left of Source 102. Only one of these (D-5/96) was available in enough quantity to enable additional testing, and the condition of this strand is variable.

As stated, the three recently manufactured sources of strand obtained for testing were identified as 102, 103, and 151. Source 103 is the strand used by Stresscon in their ordinary production of precast/prestressed concrete. It is a source with a proven record of good bond from pull-out tests, flexure beam tests, transfer length tests, and end slips observed in hollow core precast/prestressed concrete members. Source 102 is from a lot of strand delivered to a Midwest precast concrete producer at the same time as another lot from the same strand manufacturer who experienced excessive slip in hollow core products produced by the precaster. After this precaster experienced the slip problems, he contracted with Peterman of KSU to perform concrete pull-out tests. Tests were run on this strand with the results shown in Figure B-2. Later, it was reported that this same strand manufacturer was routinely running Portland cement mortar pull-out tests. Source 151 is the same strand manufacturer as 102, but from a precaster in the eastern United States who had not reported any strand slip problems. As received, the spool of strand from the east coast precaster had small areas of rust on the outer windings of the spool. These outer windings were removed, and the tested strand was taken from an inner winding on the same spool. The 102 and 103 strands are both 0.5-in. diameter. The 151 strand is 0.52-in. diameter.

One additional recently manufactured strand was received after the completion of the Screening Round of evaluation. This strand was supplied from India and was numbered 153. This strand was tested in portions of the correlation program, but was not included in the correlation analysis, since it was learned that the manufacturing processes used in its production were markedly different from those used to produce strand in the United States.

OSU strand—The sample sources used for the Correlation Round of testing were selected by Bruce Russell of OSU. These sources of strand had been tested in work performed by Russell for the NCHRP Project No. 12-60 *Transfer, Development, and Splice Length for Strand/Reinforcement in High-Strength Concrete*, the Oklahoma Department of Transportation, and NASPA, also known as the Committee of the American Wire Products Association. Two of the six strand sources provided by Russell were actually the same strand source, a fact that was not known before the testing was completed. This was intended to test the repeatability of the surface and chemical test methods. Transfer length and mortar pull-out test results were provided in tabular form by Russell after the chemical and surface testing had been completed. These

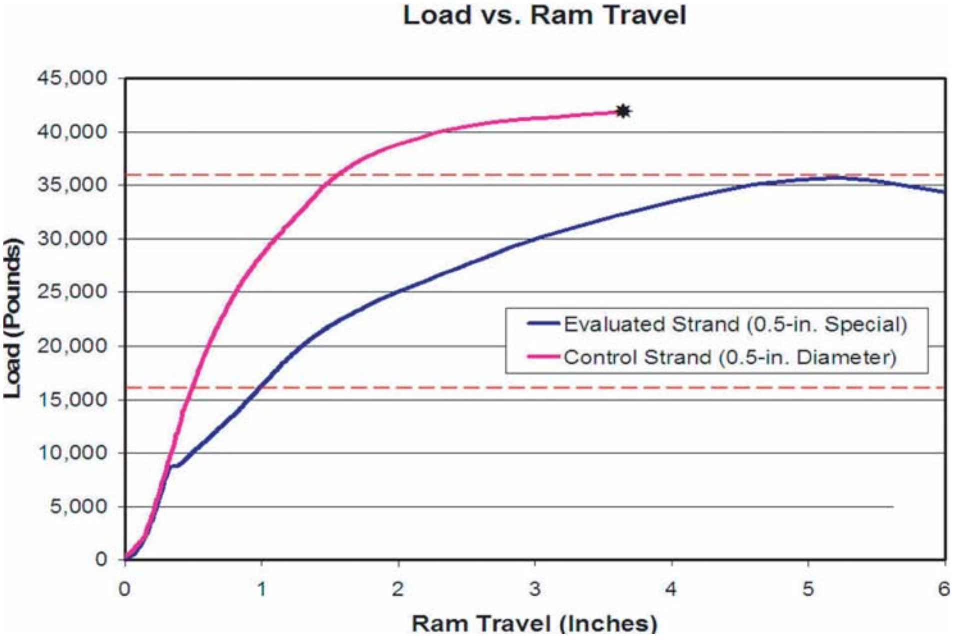


Figure B-2. Large concrete block pull-out behavior of strands tested at KSU that demonstrated excessive slip in a hollow-core product. In this figure, behavior is compared against that of a well-bonding control.

tables are reproduced in Table B-2 and Table B-3. Transfer length testing had been conducted on only two sources, while mortar pull-out testing had been conducted on all five sources. Although Russell did not provide a description of his test methodology, it is believed that the data were obtained as described in the recently published Master of Science thesis, "Assessing the Bond Quality of Prestressing Strands Using NASP Bond Test" (Chandran 2006). Russell did not supply the strand tested in Project 12-60 with the lowest bond.

Mechanical Test Methods and Results

The original project scope included the development of a performance-based test method for use in evaluating strand bond. The Screening Round of the experimental program included mechanical testing of strand sources using transfer length, pull-out from concrete (large concrete block pull-out test [LBPT]), pull out from Portland cement mortar, and pull out from gypsum plaster-based mortar tests. Each test is presented separately in this section. The number of samples of each strand source tested by each mechanical test method is shown in Table B-4 and B-5.

Transfer Length Testing

Transfer length testing was conducted in three rounds, with its procedure changing slightly for each subsequent round of testing.

During the Trial Round, two 4 in. × 4 in. × 16 ft-prisms were cast for strand designations 102 and 151. The size of the prisms was based on two considerations: (1) the cross-sectional area of the prism was sized to obtain a concrete compressive stress of approximately 2000 psi after release of prestress; (2) its length was designed to obtain four transfer lengths per specimen—two from the initial release of prestress and two from saw-cutting the specimen at its mid-point. The prism had to be long enough to assure that the transfer lengths from the four ends would not overlap.

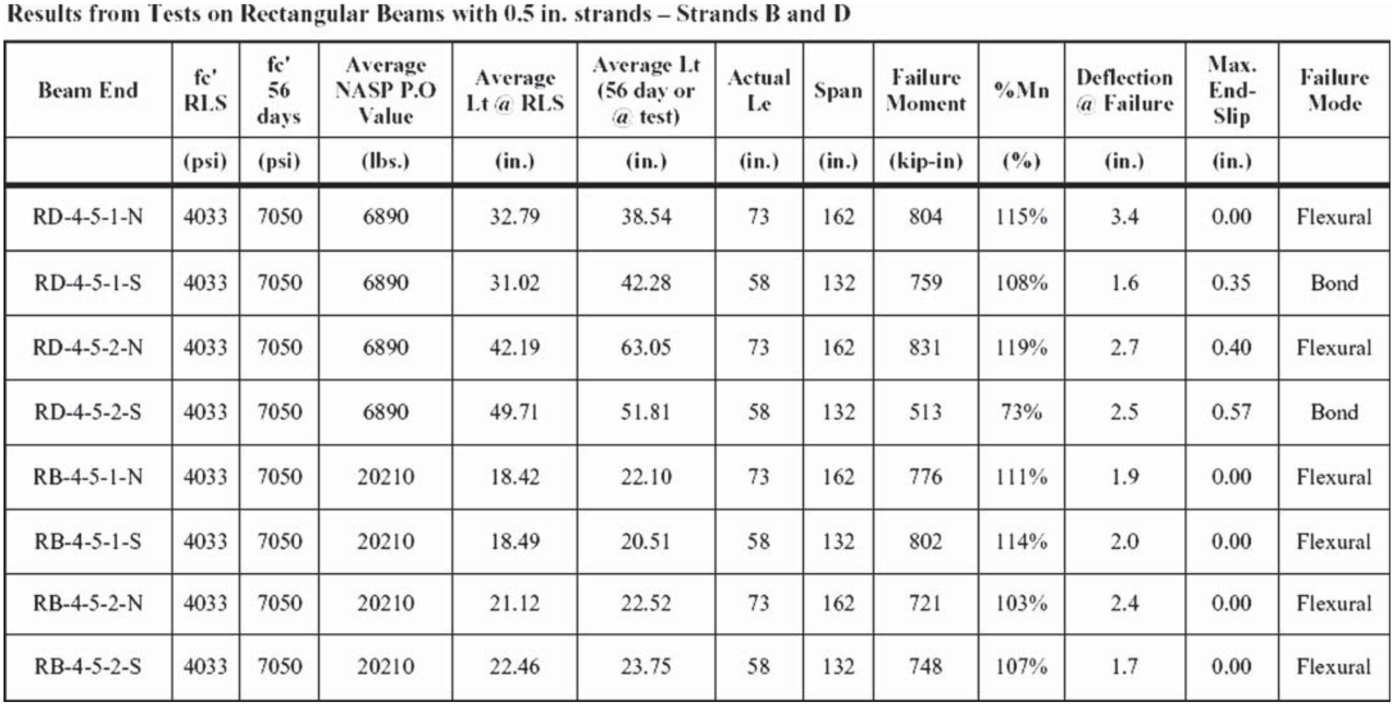
Figure B-3 shows a schematic of the transfer length specimen, as well as the concrete mixture proportions used for the Trial Round and for Round 1. The cement was a Type III with a blaine fineness of 564 m^2/kg. The coarse aggregate was a siliceous gravel meeting ASTM C33 #67 gradation requirements, and the fine aggregate had a fineness modulus of 2.89. The mixture proportions were determined essentially according to the procedure given in ACI 211.1-91, Standard Practice for Selecting Proportions for Normal, Heavyweight, and Mass Concrete.

Figure B-4 schematically shows the transfer length specimen mounted in its stressing frame. Figure B-5 shows one such frame ready for casting, with prestressing strand tensioned in the form. After the prisms were cast and the formwork was stripped, Whittemore points were attached to both sides of the prisms as shown in Figure B-6. The strain in the beam relative to the position along the beam was measured based on readings taken with a Whittemore gauge (Figure B-7). This device measures the change in location of Whittemore

Table B-2. NASPA (mortar) pull-out and transfer length test data accompanying strand from OSU.

NCHRP 10-62 Designation	Strand Diameter (in.)	NCHRP 12-60	NASP Pull-Out Force (lbs) - OSU			Measured End Slips (in.) (Beams with f'ci = 4 ksi)			Transfer Lengths from End Slips (in.) (Beams with f'ci = 4 ksi)		Transfer Lengths from DEMEC (in.)	
		OSU (ID)	Batch No.	Mortar Strength (psi)	@ 0.10 in. slip	Strand (End/Side)	@ Release	@ Test	@ Release	@ Test	Beam End	@ Release
548	0.5	A	11N	4730	20,710							
			15N	4815	21,190							
717	0.5	D	8N	4765	6,870		RD4-5-1	RD4-5-1	RD4-5-1	RD4-5-1		RD4-5-1
				4484	6,910	NW	0.126	0.140	33.9	38.0	North	25.57
						NE	0.113	0.139	31.7	39.2		
						SW	0.110	0.158	29.9	42.9	South	24.79
						SE	0.114	0.148	32.1	41.7		
							RD4-5-2	RD4-5-2	RD4-5-2	RD4-5-2		
						NW	0.175	0.278	47.5	75.1		
						NE	0.131	0.181	36.9	51.0		
						SW	0.180	0.182	49.0	49.4		
						SE	0.179	0.193	50.4	54.2		
349	0.5	E	39N	4303	5,240							
478 (C1) and 960 (C2)	0.5	C	29N	4723	14,130							
			31N	4927	13,300							
697	0.5	B	30N	4723	19,330		RB4-5-1	RB4-5-1	RB4-5-1	RB4-5-1		RB4-5-1
			31N	4927	21,090	NW	0.071	0.080	19.8	22.3	North	24.18
						NE	0.062	0.079	17.1	21.9		
						SW	0.066	0.078	18.7	21.9	South	27.11
						SE	0.066	0.069	18.3	19.1		
							RB4-5-2	RB4-5-2	RB4-5-2	RB4-5-2		
						NW	0.065	0.075	18.1	20.9		
						NE	0.087	0.087	24.1	24.1		
						SW	0.080	0.084	22.5	23.4		
						SE	0.081	0.087	22.5	24.1		

Table B-3. Transfer length and beam test data accompanying strand from OSU.



Results from Tests on Rectangular Beams with 0.5 in. strands – Strands B and D

Beam End	fc' RLS	fc' 56 days	Average NASP P.O Value	Average Lt @ RLS	Average Lt (56 day or @ test)	Actual Le	Span	Failure Moment	%Mn	Deflection @ Failure	Max. End-Slip	Failure Mode
	(psi)	(psi)	(lbs.)	(in.)	(in.)	(in.)	(in.)	(kip-in)	(%)	(in.)	(in.)	
RD-4-5-1-N	4033	7050	6890	32.79	38.54	73	162	804	115%	3.4	0.00	Flexural
RD-4-5-1-S	4033	7050	6890	31.02	42.28	58	132	759	108%	1.6	0.35	Bond
RD-4-5-2-N	4033	7050	6890	42.19	63.05	73	162	831	119%	2.7	0.40	Flexural
RD-4-5-2-S	4033	7050	6890	49.71	51.81	58	132	513	73%	2.5	0.57	Bond
RB-4-5-1-N	4033	7050	20210	18.42	22.10	73	162	776	111%	1.9	0.00	Flexural
RB-4-5-1-S	4033	7050	20210	18.49	20.51	58	132	802	114%	2.0	0.00	Flexural
RB-4-5-2-N	4033	7050	20210	21.12	22.52	73	162	721	103%	2.4	0.00	Flexural
RB-4-5-2-S	4033	7050	20210	22.46	23.75	58	132	748	107%	1.7	0.00	Flexural

Table B-4. Number of samples tested for each mechanical and surface and chemical test method.

Strand Source ID	Transfer Length	Pullout Bond Stress			Contact Angle			pH		
		Concrete	Mortar	Hydrocal	As-Rec'd	After $Ca(OH)_2$	After Ignition	pH Meter	Duotest	pH-Fix 7.5-9.5
Historic Strand										
KSU-F	--	6	--	--	1	1	--	--	--	--
KSU-H	--	6	--	--	2	2	--	--	--	--
SC-F	--	6	--	--	3	3	--	--	--	--
SC-H	--	6	--	--	3	3	--	--	--	--
SC-IS	--	6	--	--	3	3	--	--	--	--
101	--	6	--	--	3	3	--	--	--	--
Recently Manufactured Strand										
102	6	6	--	6	3	3	--	2	2	2
103	6	6	--	6	3	3	--	2	2	2
151	6	6	--	6	3	3	--	2	2	2
153	--	6	--	--	--	3	--	--	6	--
OSU Strand Samples										
349	--	--	6	--	--	3	3	--	6	--
548	--	--	18	--	--	3	3	--	6	--
697	--	--	11	--	--	3	3	--	6	--
717	--	--	6	--	--	3	3	--	6	--
478 *	--	--	12	--	--	3	3	--	6	--
960 *	--	--	12	--	--	3	3	--	6	--

* Samples designated 478 and 960 were from same source.

Table B-5. Number of samples tested for each mechanical and surface and chemical test method.

Strand Source ID	LOI	LAB		Change in Corrosion Potential			Surf. Rough.	Corrosion Rate			Org. Ext. Res.	AA and Color. Analysis	
		Method 1	Method 2	As-Rec'd	After $Ca(OH)_2$	After Ignition		As-Rec'd	After $Ca(OH)_2$	After Ignition		Water	Acid
Historic Strand													
KSU-F	1	2	--	--	--	--	--	1	--	--	2	2^A	2^A
KSU-H	2	2	--	--	--	--	--	2	--	--	2	2^A	2^A
SC-F	2	2	--	--	--	--	--	-	--	--	3	3^A	3^A
SC-H	2	2	--	--	--	--	--	2	--	--	3	3^A	3^A
SC-IS	2	2	--	--	--	--	--	2	--	--	3	2^A	2^A
101	2	2	--	2	--	--	--	2	--	--	3	3^{AB}	3^A
Recently Manufactured Strand													
102	2	2	2	3	1	--	6	2	1	--	3	3^{AB}	3^A
103	2	2	2	3	1	--	6	2	1	--	3	3^{AB}	3^A
151	2	2	2	3	1	--	6	2	1	--	3	3^{AB}	3^A
153	--	--	--	3	--	3	12	--	3	3	3	3	3
OSU Strand Samples													
349	3	--	--	4	--	3	12	--	3	3	3	3	3
548	3	--	--	3	--	3	12	--	3	3	3	3	3
697	3	--	--	3	--	3	12	--	3	3	3	3	3
717	3	--	--	3	--	3	12	--	3	3	3	3	3
478 *	3	--	--	3	--	3	12	--	3	3	3	3	3
960 *	3	--	--	3	--	3	12	--	3	3	3	3	3

* Samples designated 478 and 960 were from same source.

[A] Acid wash performed on strand subsequent to water wash.

[B] Both warm- and hot-water washes performed.

points to 1/10,000th of an inch. The Whittemore points are cylindrical buttons that contain a central indentation into which styli on the gauge are inserted.

After the initial readings were recorded, the strand was released gently, and surface strains were measured. These two trial tests resulted in transfer lengths of 20 to 28 in. for each source. It was determined that the concrete strength may have been too high and the release method too gradual to provide a true measure of transfer length.

In Round 1, two prisms were cast for each of the three sources of recently manufactured strand (102, 103, and 151); a total of six stressing frames similar to those needed for the Trial Round were used. After casting, in preparation for measuring the strand end slip, a small notch was made at the exposed ends of the strand. The distances from this small cut to the end of the prism were measured with calipers, in digital photos, and with a steel scale (see Figure B-8). This position was later re-measured with all three of these methods after release and at later times to determine the end slip of the strand.

Initially, the release was achieved by quickly cutting the strand with an acetylene torch similar to what is commonly done in precast concrete plants. However, this resulted in splitting and fracturing at the end region of the concrete for three specimens (Figure B-9). For the remaining three specimens, release was achieved by gradually heating up the strand until it failed in tension due to its reduced tensile strength at high temperatures. Even using this method, one of the three remaining prisms split at its end. The strength of the concrete at the time of prestress transfer was 4060 psi. The strength of the concrete at the time of center was 4810 psi.

The strain versus position was determined after the strand was released. The prisms were then cut in half (Figure B-10), and the strain in the prisms was measured along with the end slip (measured at the far ends of the prisms) and strand suck-in (measured at the saw-cut ends).

To examine the effect of time on transfer length, end slip, and suck-in, these measurements were repeated 28 days, 6 months, and 22 months after release. Strand suck-in measurements were abandoned after the 28-day data collection because of their inefficacy. During the initial saw-cutting process, when the saw initially nicked the strand, the individual wires fractured sequentially, leaving an uneven surface from which accurate measurement of the strand displacement proved difficult (see Figure B-11).

A typical plot of strain versus position on the prisms is given in Figure B-12. This plot shows several sets of strain

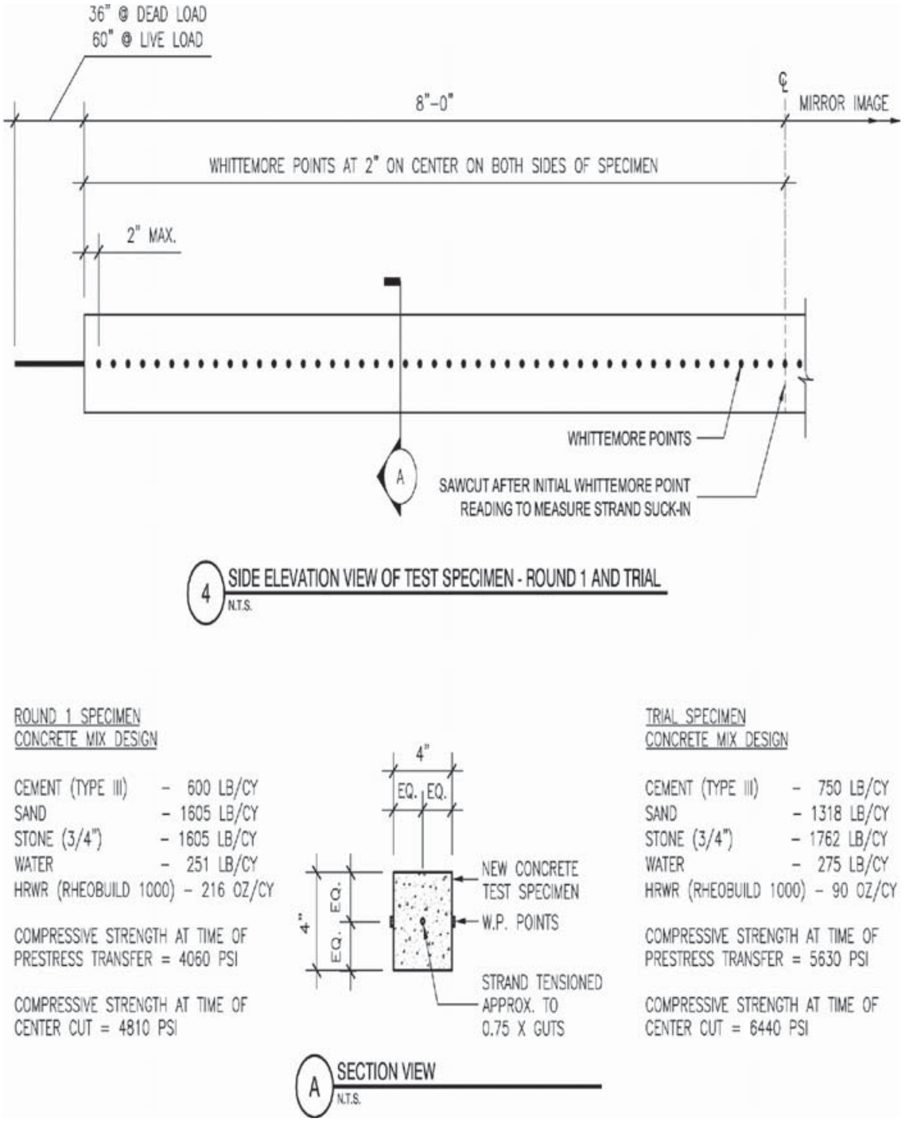


Figure B-3. Transfer length specimen used in the Trial Test and Round 1 Test.

data collected over a series of months as detailed above. Additionally, this plot shows lines drawn at 100% of the plateau strain for each measurement. The transfer length measured in this test is defined as the intersection of the strain on this plateau and the linear extension of the slope from the transfer region.

Because of the problems experienced with this first set of transfer length specimens, a second set was fabricated and tested (Round 2). In Round 2, the use of saw cutting to measure strand suck-in was omitted due to the previously mentioned ineffectiveness of this method. End slip measurements for this round of testing were continued using the scale and photos. End slips were also measured using a depth gauge. As a final change, in Round 2, the cross-sectional dimensions of the prisms were increased to prevent splitting on rapid release of prestress. Rapid release was not abandoned, as was done by necessity during Round 1, because releasing the strand in this way realistically imitates what is done at a typical precaster's yard. To prevent end-splitting, specimen cross-sections were increased to 4.5 in. × 4.5 in., which reduced the compressive stress on the concrete to 1500 psi. The Round 2 specimen and stressing frame are presented schematically in Figure B-13 and Figure B-14, respectively. Figure B-13 also lists the concrete mixture proportions for Round 2. The strength of the concrete at the time of prestress transfer was 4380 psi.

Because the center saw cut was eliminated, it was possible to cast two 8-ft long specimens end-to-end within the fabrication frame. A space was left between the specimens using a stiff standoff, exposing a portion of the strand. The strand in this segment between the two specimens was torch cut after the initial release (see Figure B-15).

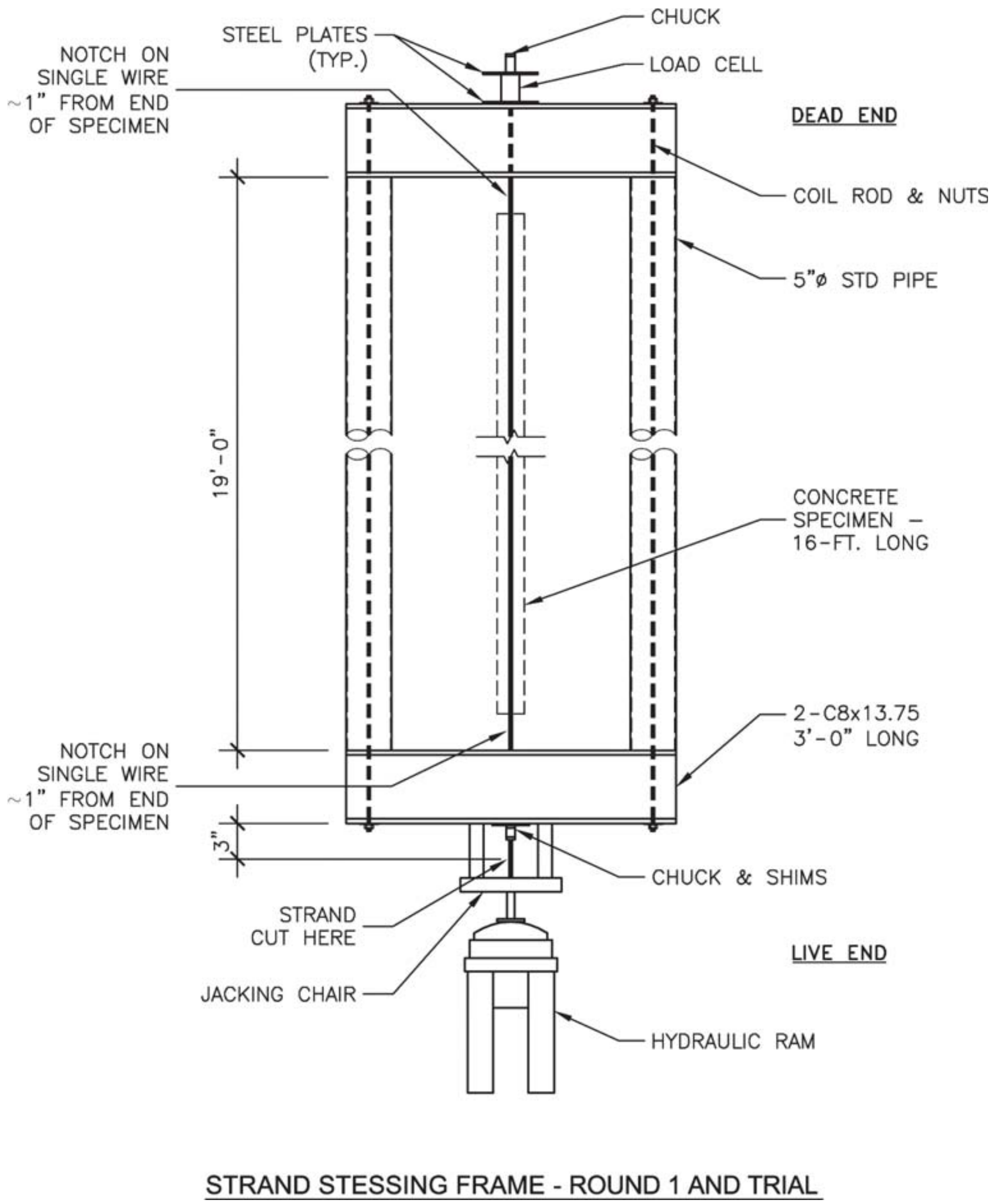


Figure B-4. Transfer length stressing frame and specimen orientation, Trial Test and Round 1 Test.

Two additional issues were experienced in this second round:

1. While making the notch in the wire for the end slip measurement on two of the prisms, the individual wire fractured and some loss of prestress occurred.
2. It was not initially known that one of the strands was 0.52-in. diameter (1/2 in. special) instead of 0.5-in. diameter. Thus, this larger strand had a slightly lower bond stress at transfer.

To account for these issues and enable comparisons of stress transfer behavior between the tested strand sources, the average bond stress over the transfer length was calculated based on the measured transfer length and strand tensile stress. This approach eliminates complications from strands of varying sizes and varying initial stress conditions. The average bond stress, U_t, is calculated as

$$U_t = \frac{f_{se} A_{ps}}{C_p L_t} \qquad \text{(Eq. 1)}$$

where f_{se} is the effective prestress after transfer, A_{ps} is the cross-sectional area of the strand, C_p is the circumferential perimeter of the strand ($4/3\,\pi\, d_b$) and L_t is the transfer length. Average bond stress is thus dependent both on effective prestress as well as transfer length for a given strand geometry. For the specimens from the two rounds of testing, a transfer

Figure B-5. Tensioned strand in form used for casting transfer length specimen.

length of 30 in. translated to an average bond stress of 375 to 425 psi.

The approach used to evaluate the average bond stress is the same as that used to derive the formula for predicting transfer length in ACI 318 Section 12.9, which is

$$L_t = \frac{f_{se} d_b}{3} \qquad \text{(Eq. 2)}$$

Figure B-6. Specimens with Whittemore buttons attached.

Figure B-7. Whittemore gauge readings used to calculate strain.

where L_t = transfer length in inches, f_{se} = the effective stress in the strand after the losses, and d_b = nominal diameter of the strand in inches. The "3" in the denominator is based on an assumed average bond stress of 400 psi and the prevailing strand geometry in the 1950s when the formula was developed (Tabatabai and Dickson 1993). For the purpose of this calculation, f_{se} was taken as the difference between the stresses in the strand before release and the elastic losses only. The elastic loss was determined based on the strain measured immediately after release in the central region of the test prism over which the strain is approximately constant, assuming no relaxation losses in the strand.

Results of Transfer Length Testing

Table B-6 and Table B-7 show the average bond stress over transfer length measured for Rounds 1 and 2, respectively. The combined results of Rounds 1 and 2 are presented in Table B-8 and graphically in Figure B-16. Table B-9 presents coefficients of variation for these results.

The combined Round 1 and 2 results are averages of six transfer length measurements from the two rounds from each source: two from Round 1 and four from Round 2. Other data collected during Round 1 were left out of this inter-round analysis because of the procedural differences between the two rounds relating to prestress release. The data omitted (grayed in Table B-6) represented bond stresses obtained from the end of the prisms at which the strands were released and those from the prisms saw-cut ends.

The end slip was measured by three methods: using a scale, using calipers/depth gauge, and comparing digital photos taken before and after release. Figure B-17 presents an

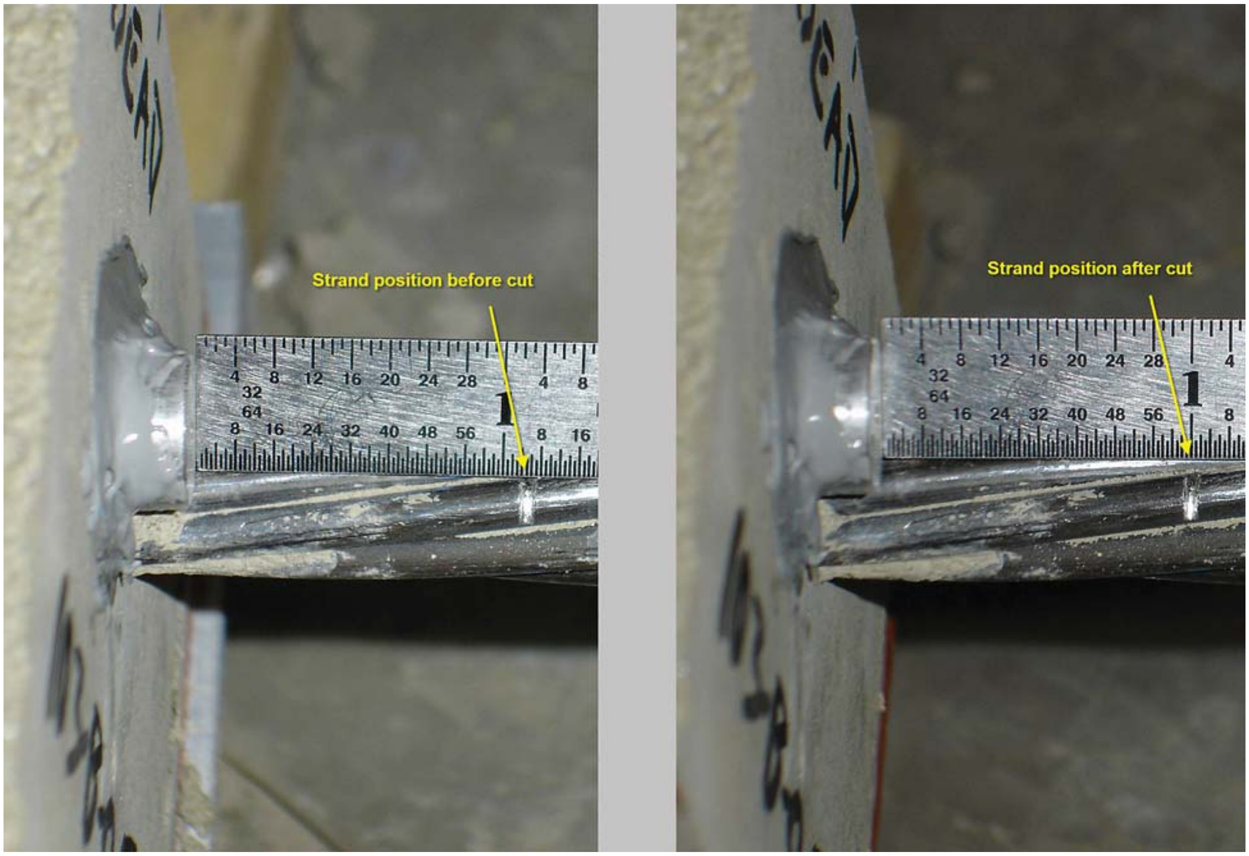


Figure B-8. Measurement of strand slippage from dead end—Strand 103B.

average of the end slips measured with a scale for both rounds of testing excluding data omitted for inter-round bond stress analysis.

Previous researchers (Rose and Russell 1997) demonstrated that transfer length is proportional to end slip. Therefore, since transfer length is in the denominator of the expression given as Eq. 2, it would be expected that average bond stress over transfer length is inversely proportional to end slip. This has been confirmed in this study as shown in Figure B-18, which depicts the correlation between bond stress and the inverse of end slip for measurements made using a scale, photos, and a set of calipers. Each point on this figure represents the average of several measurements made on a single strand source by a given method. The methods of end slip measurement that were judged best

Figure B-9. Fractured end of prism after rapid release of strand.

Figure B-10. Cutting specimen in half.

Figure B-11. Strand appearance after mid-point saw cut of transfer length prism.

were the use of a scale or calipers, because they correlated best with the bond stress calculated from surface strain measurements.

Discussion of Transfer Length Testing

The average bond stress over the transfer length decreased over time. The rate of this decrease was typically largest during the first month after transfer and then was roughly constant during the next two time spans (28 days to 6 months and 6 months to 18 to 22 months). Source 102, initially the poorest bonding strand, displayed less change in bond stress over time.

While the average bond stress decreased, the end slips increased. Strand from the poorest-bonding source (102) had the highest end slips; the best-bonding strand had the lowest end slips. While a correlation exists between end slip and transfer length or bond over transfer length measurements, it is not without some limitations. Examining the end slip measurements for Source 102 over time (Figure B-17) can reveal the limits of the end slip-average bond stress over transfer length correlation. The measured average end slip decreased between 28-day and 6-month measurements; this

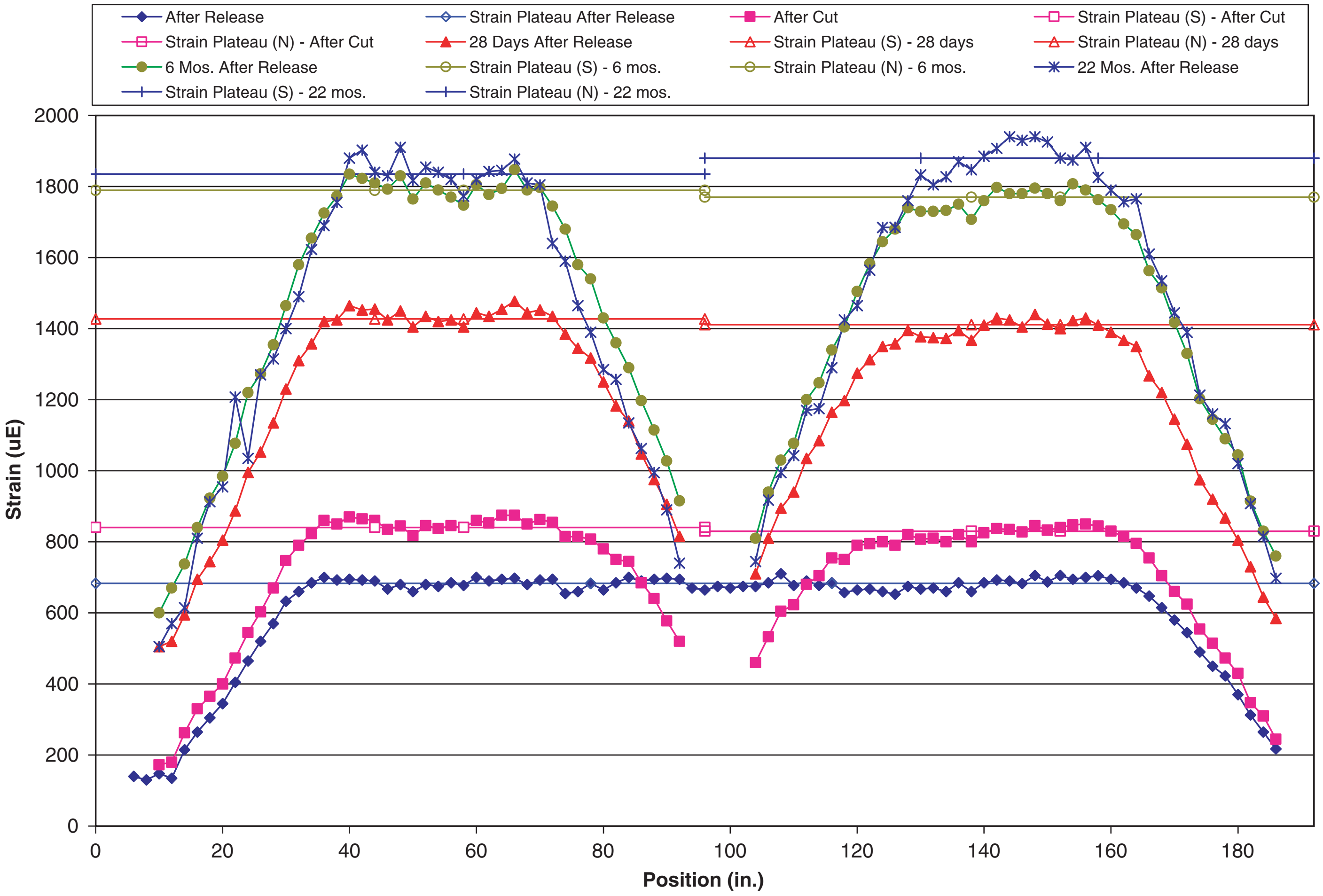


Figure B-12. Strains measured on transfer length beam containing strand from Source 103.

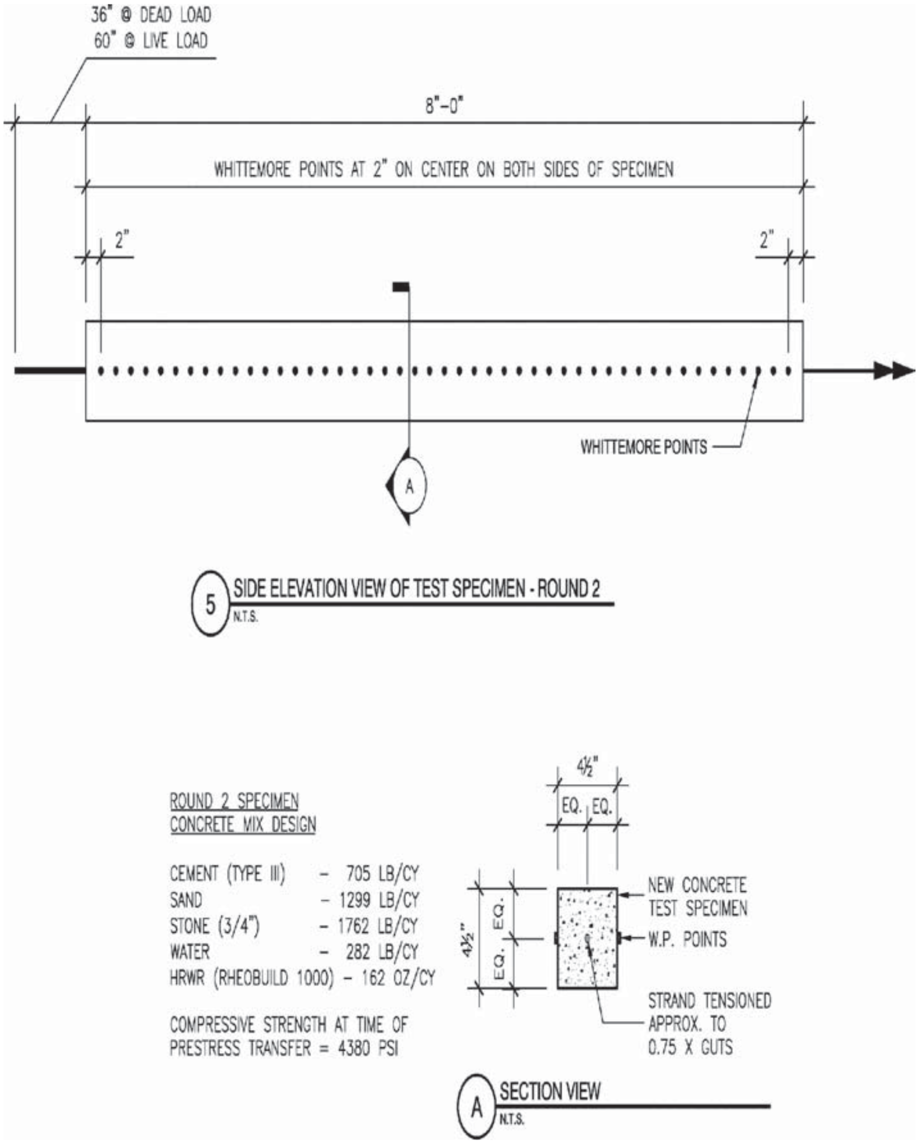


Figure B-13. Transfer length specimen used in the Round 2 Test.

apparent trend is likely related to the measurement technique and no increase was noted in the bond stress for these data points. The largest increase in end slip for all sources occurred between the last two measurements, while the largest decrease in bond stress typically occurred between the first two. Due to the imperfect correlation between end slip and transfer length bond stress, caution should be used when extrapolating transfer length stress or transfer length itself from end slip.

In part, this is because end slip is a difficult property to measure and a certain amount of inaccuracy can be expected in this single measurement. The transfer length and the average bond stress over the transfer length are calculated based on numerous discrete measurements. Multiple methods for measuring end slip were employed in this study; all were difficult to correlate with transfer length bond stress. It was initially thought that using digital photos might decrease error, but instead, this method proved less effective than the alternatives. Due to the large amount of time required to perform the photo analysis and the lack of improvement in the results, it seems that measuring end slip directly, either with a scale or calipers, was a better method. Measurements made using a depth gauge were ineffective, just as were attempted measurements of strand suck-in at cut beam ends for Round 1.

Recent research (Peterman 2007) has indicated that greater and more variable transfer lengths may be experienced by strand located within 8 in. of an as-cast top surface. This may indicate that the specimen size used in this study may have been too shallow. In the transfer length specimens, the strands are about 2 in. below the as-cast top surface and would therefore be expected to suffer from this effect. Furthermore, research has indicated a strong correlation between concrete strength and bond properties (Stocker and

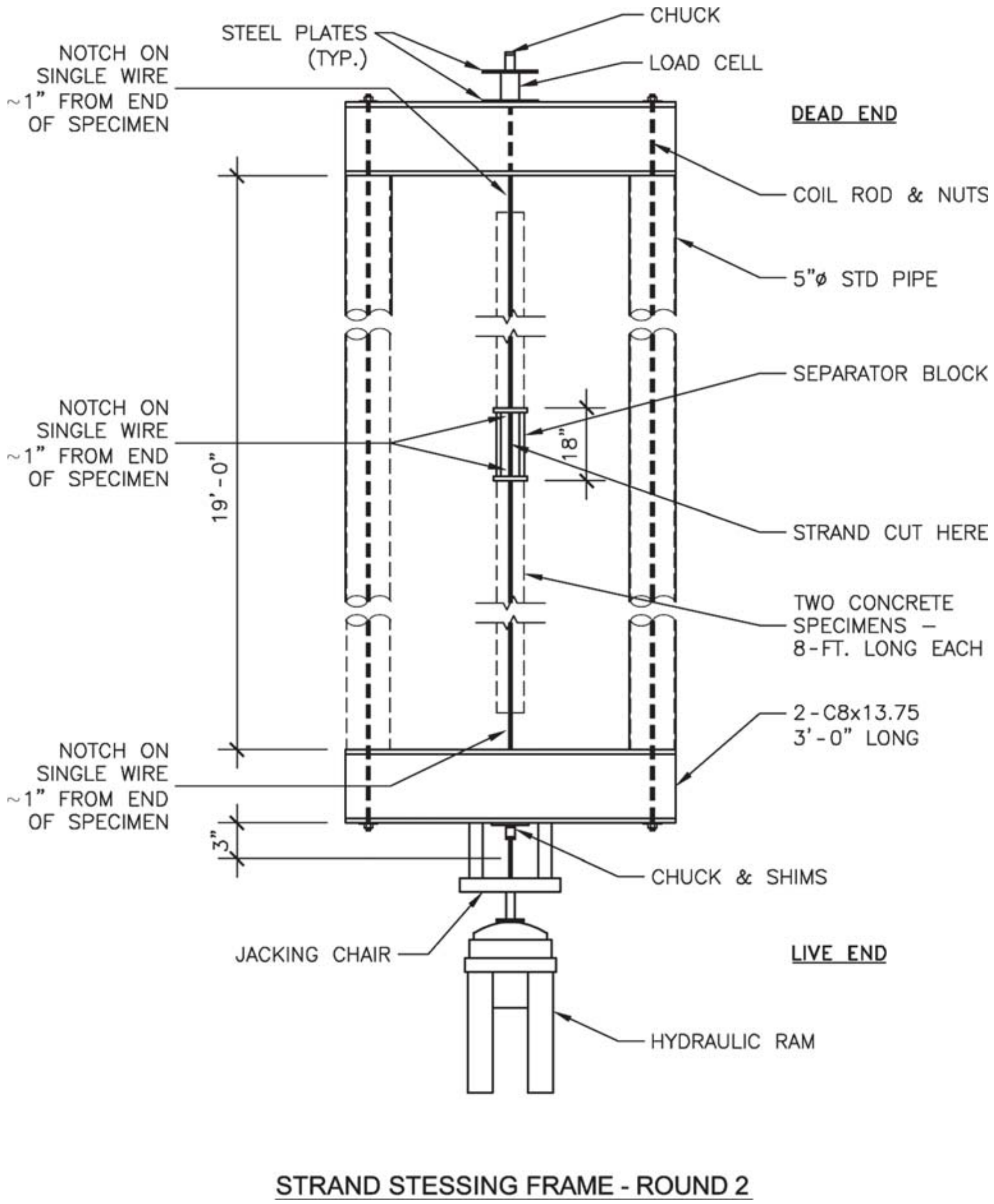


Figure B-14. Transfer length stressing frame and specimen orientation Round 2 Test.

Sozen 1971, Lane 1998, Mitchell et al. 1993). Thus, in an effort to differentiate the innate bond properties of a strand from the results of transfer length tests, a concrete strength correction factor should be employed to interpret transfer length data.

Transfer lengths determined by side strains measured using mechanical strain gages (Whittemore or DEMEC gages) are considered less reliable as the concrete ages. Creep of the concrete results in a non-linear relationship between the strain in the concrete and the strain in the steel. Instead, recent research suggests that the slip of the strand may be a better indicator of transfer length over a long period of time. This measurement has proven to be a better long-term indicator of transfer length and is easier to perform than the mechanical strain measurement.

Transfer Length Testing for Correlation Round of Evaluation

As mentioned, the sampled sources used for the Correlation Round of testing were selected by Bruce Russell of OSU. Transfer length data were provided accompanying two of the sources (see Table B-2 and Table B-3) and it is assumed the testing protocol is as reported by Chandran (2006). Russell reported transfer length data calculated from measured end slips and measured directly from surface strain measurements

Figure B-15. Strands between specimens after torch cut.

(DEMEC). These data were provided for Sources 697 and 717. The transfer lengths reported for Source 717 based on surface strain data were about 25 in. However, based on end slip measurements, the reported transfer lengths for Source 717 were 38 to 75 in. Agreement between the measurement methods was better for Source 697, with surface-strain based transfer lengths ranging from 24 to 27 in. and end slip based transfer lengths ranging from 19 to 24 in. Because of the large unexplained variation between the results of the two transfer length measurement methods, and because there were only two data points available, the transfer length data were not considered in the correlation analysis conducted to evaluate surface and chemical test methods. Lastly, for the same two sources, Russell provided results from flexural beam tests conducted to evaluate development length, which similarly were not analyzed.

Pull-Out Testing

A key component of this project as originally defined was the development of a procedure for qualifying the bond characteristics of prestressing strand using some sort of pull-out test of untensioned strand. Pull-out tests to determine the bond characteristics of steel in concrete have been used for over 100 years. Pull-out tests have been used to judge the bond quality of prestressing strand for almost 50 years. Refinements to historic pull-out test methods continue to develop. These refinements deal not so much with the basic methodology, but rather with the small variations in testing procedures.

At the current time, two types of pull-out tests are most common and appear to be the most viable. The first method involves pulling untensioned strand out of a block of concrete. The second method involves pulling untensioned strand out of a steel cylinder filled with mortar.

Table B-6. Average bond stress over transfer length from Round 1.

Strand Sample ID	Beam End	Average Bond Stress over Transfer Length (psi)				
		Initial	After Cut	28 Days	6 Months	22 Months
102-A-0.5-1A	S-S	437	423	380	364	328
	S-N	--	596	558	477	397
	N-S	--	525	486	437	380
	N-N	375	354	336	345	341
102-A-0.5-2A	S-S	397	436	422	385	364
	S-N	--	569	569	524	468
	N-S	--	513	476	476	416
	N-N	344	364	369	359	331
103-B-0.5-1A	S-S	422	436	429	390	357
	S-N	--	627	547	485	459
	N-S	--	612	485	436	402
	N-N	485	443	436	415	378
103-B-0.5-2A	S-S	514	475	450	428	428
	S-N	--	694	626	571	494
	N-S	--	626	546	514	459
	N-N	597	514	475	467	428
151-Z-0.5-1C	S-S	371	341	319	332	323
	S-N	--	586	413	355	345
	N-S	--	360	341	336	327
	N-N	332	336	319	315	315
151-Z-0.5-2C	S-S	349	359	354	334	309
	S-N	--	399	369	330	302
	N-S	--	434	427	419	359
	N-N	330	309	302	309	258

Bond stress values shaded in gray were measured at central cut or at the end of specimen influenced by release method and were ignored during computation of inter-round averages.

Table B-7. Average bond stress over transfer length from Round 2.

Strand Sample ID	Beam End	Average Bond Stress over Transfer Length (psi)			
		After Release	28 Days	6 Months	18 Months
102-A-0.5-8A	S-S	--	--	--	--
	S-N	--	--	--	--
	N-S	401	322	310	311
	N-N	334	265	250	273
103-B-0.5-5A	S-S	667	442	404	370
	S-N	400	302	278	271
	N-S	538	442	389	333
	N-N	621	466	431	324
151-Z-0.5-5C	S-S	375	318	304	277
	S-N	386	335	312	270
	N-S	430	374	361	291
	N-N	410	358	334	302

In its current form, the concrete pull-out test resembles a method developed by Moustafa (1974). The method was primarily developed to judge the capacity of strand to be used as lifting loops to handle product during shipping and erection. The test developed by Moustafa was modified by Logan (1997) to judge the bond quality of strand in pretensioned applications. Further developments of the method have occurred and are the basis for the testing reported herein.

In its current form, the mortar pull-out test method resembles a method originally developed for the Post Tensioning Institute in 1994 (Hyett et al., 1994, Post-Tensioning Institute 1996). The method was primarily developed to judge the bond quality of prestressing strand used in rock anchors. The method became the basis of ASTM A981-97(2002), *Standard Test Method for Evaluating Bond Strength for 15.2 mm (0.6 in.) Diameter Prestressing Steel Strand, Grade 270, Uncoated, Used in Prestressed Ground Anchors*. Later, this method was modified by Russell and Paulsgrove (1999) for NASPA and became known as the NASPA test. The NASPA test has been modified slightly by this research project to make it less sensitive to the test apparatus.

One of the goals of this project was to try to eliminate variables by using standardized and universally available embedment media referred to in the NCHRP Project 10-62 Request for Proposal as a "surrogate homogeneous material." Accordingly, a third type of pull-out test was attempted: pulling untensioned strand out of a steel pipe filled with a modified gypsum plaster (Hydrocal).

Table B-8. Average bond stress by strand source as a function of time (Rounds 1 and 2).

Strand Source ID	Average Bond Stress over Transfer Length (psi)			
	After Cut or Release	28 Days	6 Months	18-22 Months
102	363	323	316	314
103	531	427	397	351
151	375	334	323	286

Round 1 measurements made at released ends (S-S) or saw-cut ends (S-N or N-S) were ignored in computation of averages.

The three types of pull-out tests were performed on the three sources of strand at KSU in March and May of 2005. In summary, these are: (1) LBPTs, (2) ordinary mortar cylinder pull-out tests, and (3) Hydrocal mortar cylinder pull-out tests. Each of these test procedures and the results are described in detail below.

Large Concrete Block Pull-Out Test

In the LBPT, six prestressing strands are embedded in a 2 ft × 2 ft × 32 in. block of concrete. (Note—the length of the block can vary, depending on the number of strands being tested simultaneously.) The bottom 4 in. and top 2 in. of the strand are debonded, leaving an 18-in. long bond length. The layout of the test specimen is presented in Figure B-19, and the test frame is shown in Figure B-20. The concrete mix and other test parameters are as follows:

Concrete mix (quantities are per cubic yard):

660 lbs Type III cement
1,100 lbs sand
1,900 lbs-3/4 in. top size coarse aggregate with average Mohs hardness > 6.0
26 oz-water reducer
292 lbs water

Required properties:

Required strength at time of test = 3500 to 5900 psi
Required test age = 1 day with heat cure = 2 days with ambient temperature cure

Measured properties in this test series:

Age at test = 2 days
Slump: 1 3/4 in.

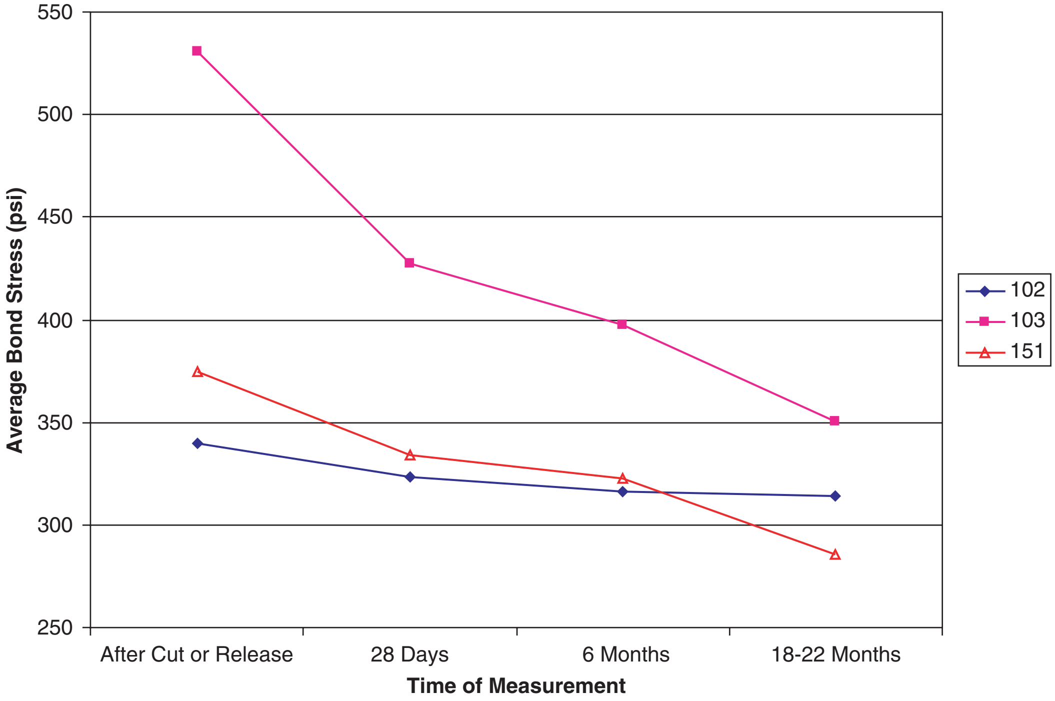


Figure B-16. Average bond stress over transfer length by strand source as a function of time (Rounds 1 and 2).

Compressive strength at 1 day = 5300 psi
Compressive strength at time of testing (2 days): 5300 psi
Modulus of elasticity at 2 days: 3680 ksi (89% of ACI-318 8.5.1)
Splitting tensile strength at 2 days: 487 psi (6.7 × $f_c'^{0.5}$)

Test parameters:
Number of strands per block: 6 from each source
Block dimensions: 2 ft × 2 ft × 32 in.
Strand Embedment: 18 in. (top 2 in. and bottom 4 in. debonded in block, 24 in. − 2 in. − 4 in. = 18 in.)
Load rate: 20 kips/min
Items to record during test: Time, date, load, ram travel, end slip, first movement as observed from live end. Monitor all items continuously during test up to maximum load.

Table B-9. Coefficient of variation for average bond stress by strand source (Rounds 1 and 2).

Strand Source ID	Coefficient of Variation for Average Bond Stresses over Transfer Length (psi)			
	After Cut or Release	28 Days	6 Months	18-22 Months
102	8%	13%	15%	10%
103	19%	15%	16%	15%
151	12%	8%	7%	7%

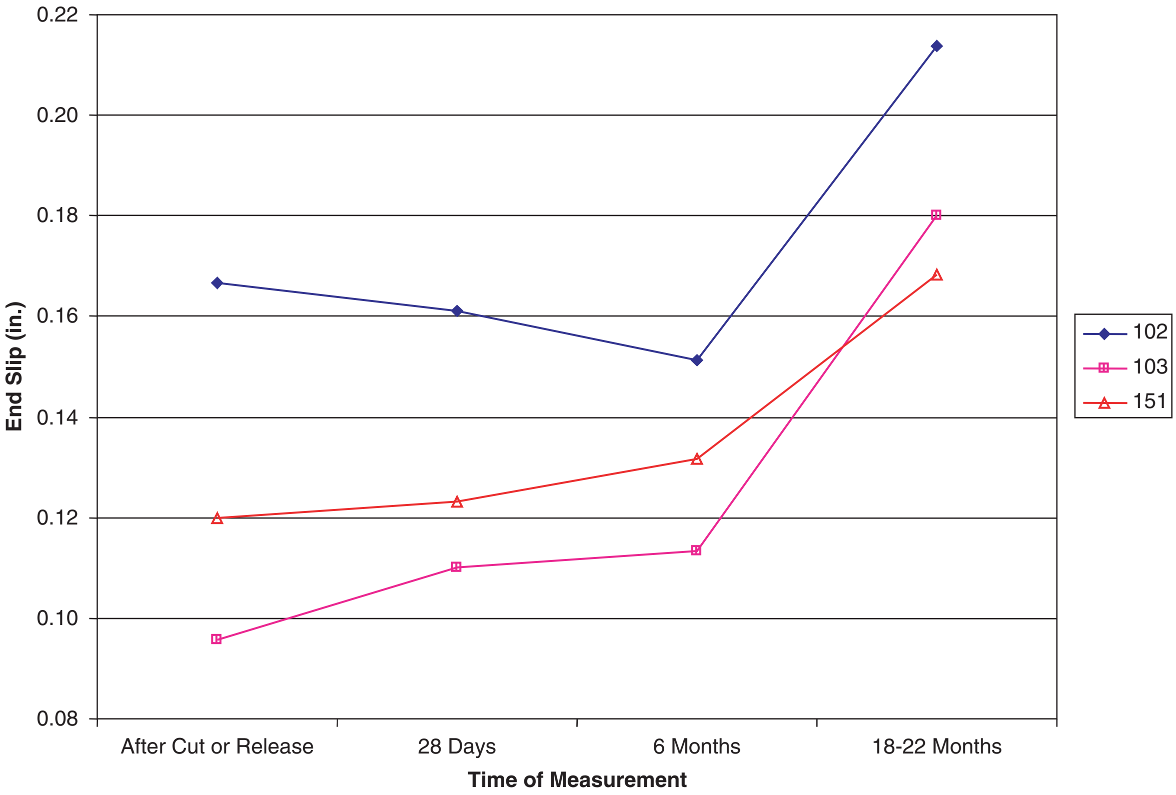
Round 1 measurements made at released ends (S-S) or saw-cut ends (S-N or N-S) were ignored in computation of coefficients of variation.

A photo of the LBPT setup is presented in Figure B-21. The concrete pull-out specimens were cast in the standard (upright) position on blocking to allow the center wires of the strands to pass through the bottom of the form (see Figure B-22 through Figure B-24). The center wire (king wire) of the strand was extended through the bottom of the specimen to be connected to a linear variable displacement transducer (LVDT) so that free-end slip could be measured during testing (see Figure B-25). To test, the block was rotated to the horizontal position, so that the LVDT that measures strand slip could be installed (see Figure B-21). The load apparatus was mounted to the side surface of the block. The particular load frame used at KSU employs a yoke so that the strand is being pulled about 8 in. from the surface of the concrete (see Figure B-26). The strand was loaded at a rate of 20 kips/min. The time, load, ram movement, and strand end slip were all recorded continuously during the test (see Figure B-27). The test was terminated just after the test sample reached the maximum load.

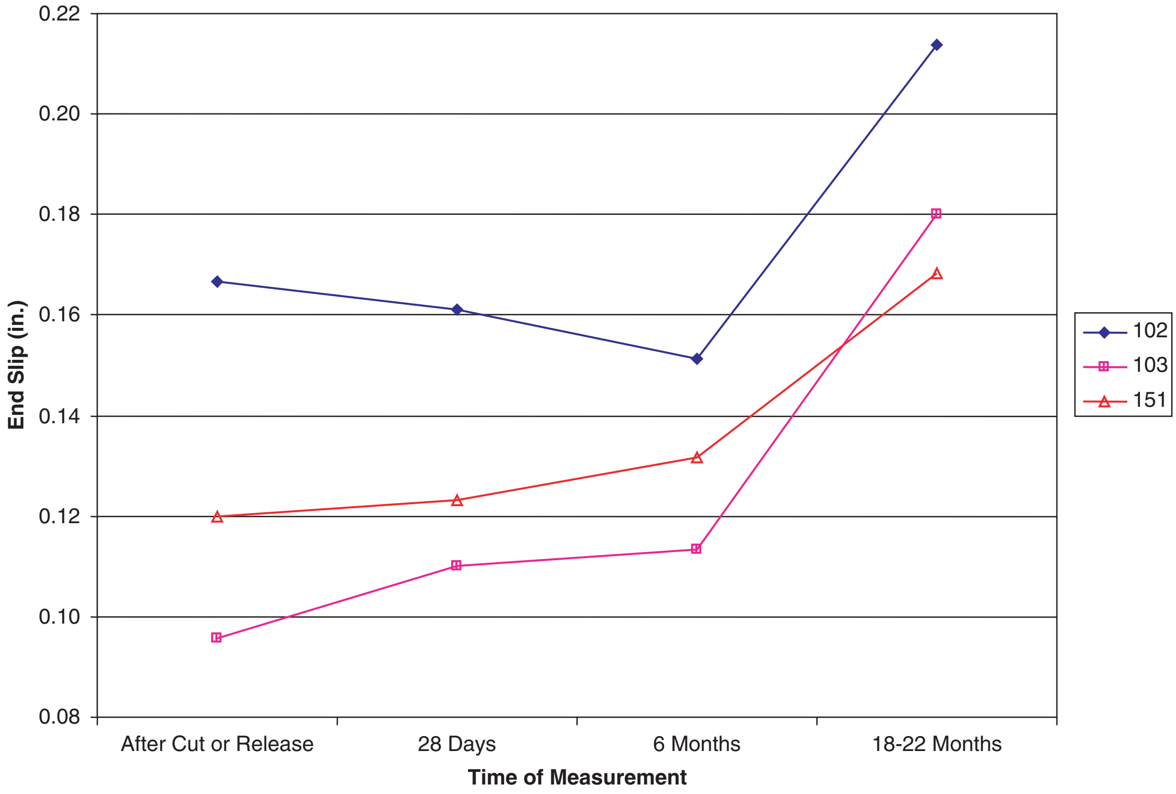
The average bond stress at 0.1-in. slip for each source tested (calculated based on the nominal surface area of the embedded section of the strands) from the concrete pull-out testing is given in Table B-10.

Mortar Pull-Out Test

In the mortar pull-out test, a prestressing strand is embedded in 5-in. diameter by 18-in. long steel cylinders filled with



(a)



(b)

Figure B-17. End slip as a function of time (Rounds 1 and 2) measured with a scale.

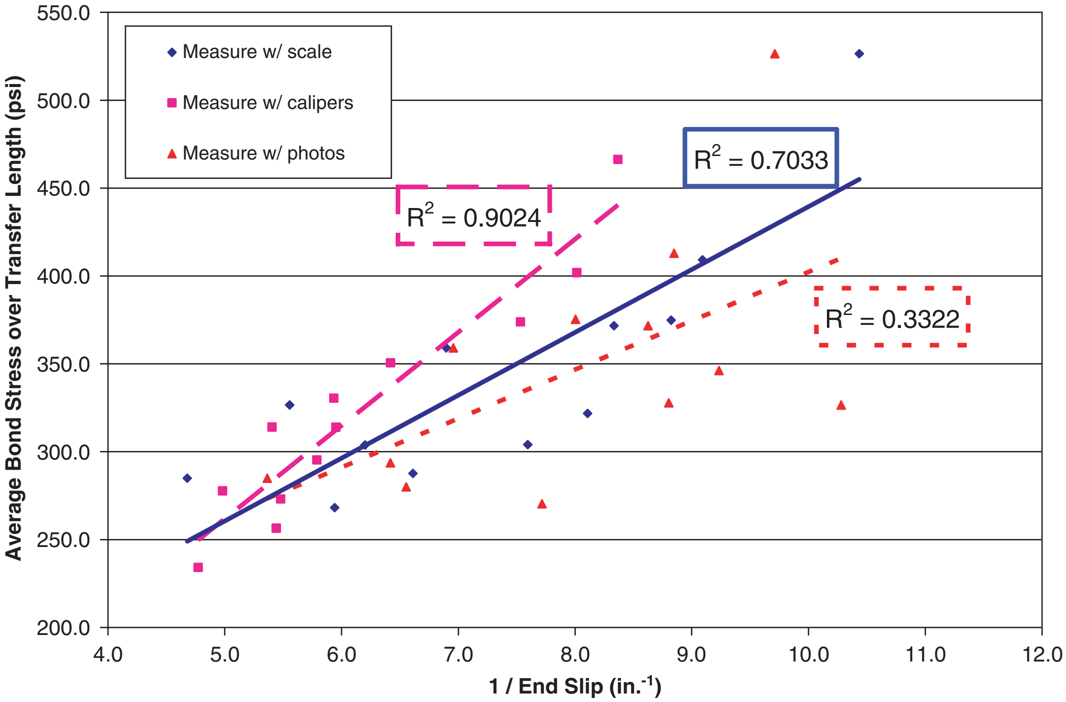
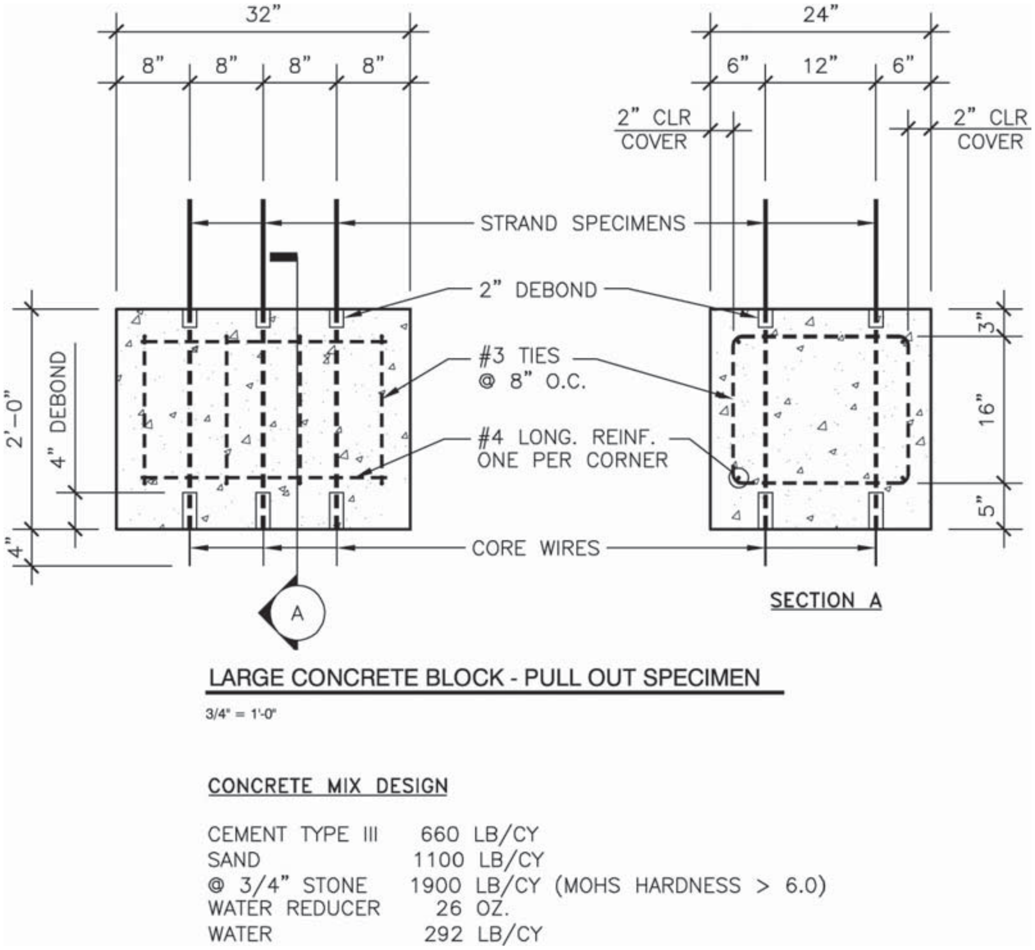


Figure B-18. Correlation between average bond stress over the transfer length and the inverse of end slip as measured with three different methods.



CONCRETE MIX DESIGN

CEMENT TYPE III	660 LB/CY
SAND	1100 LB/CY
@ 3/4" STONE	1900 LB/CY (MOHS HARDNESS > 6.0)
WATER REDUCER	26 OZ.
WATER	292 LB/CY

Figure B-19. Pull-out test in concrete specimen dimensions and details.

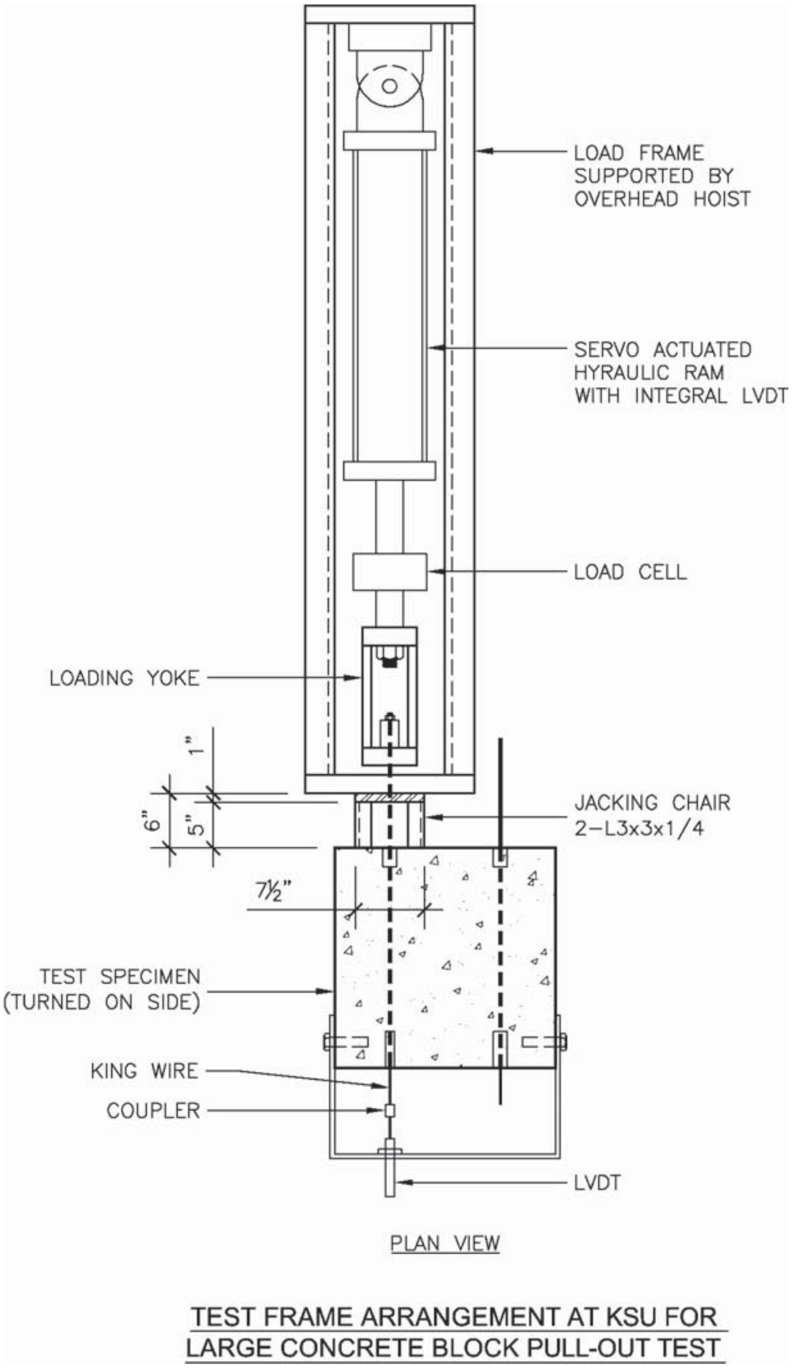


Figure B-20. Test setup for pull-out test in concrete.

mortar (Portland cement, sand, and water). The top 2 in. of the embedded portion of the strand is debonded, leaving a 16-in. bond length. Six cylinders are tested for each source of strand.

This test is a slight modification to the NASPA test reported by Russell and Paulsgrove (1999). The NASPA test uses displacement-rate control (the rate of movement of the hydraulic ram is specified to be 0.1 in./min in the NASPA test) while the test described herein uses load-rate control with a load rate of 5 kips/min, which was found to be similar to the rates used under displacement-rate control (according to a conversation held with Russell). Also, the test described herein continues up to maximum load while the NASPA test stops at an end slip of 0.1 in.

The mortar pull-out test was conducted with load rate control instead of displacement-rate control because it is believed that the load rate is an important test parameter that will influence the test results. For instance, in 2002, tests were

Figure B-21. Test set-up. Concrete block is turned on its side, loading apparatus placed at one side (live end) and measurement of end slip instrumentation mounted at the other side (dead end).

performed at KSU using displacement-rate control conforming exactly to the NASPA procedure at that time. The strands tested were from the same reel as strands tested at the University of Oklahoma (OU). However, the pull-out forces measured at KSU were much higher than measured at OU. Part of the reason for this difference is because OU used a flexible load frame (see Figure B-28), while KSU used a stiff load frame (see Figure B-29). Consequently, while both tests were conducted at similar displacement rates, the rate of load (about 7 to 11 kips/min) at KSU was much higher than at OU (reported to be about 5 kips/min). It is a well known phenomenon that higher load rates can result in higher load values in structural materials testing. For a test to be universally applicable, the test should not be load frame dependant. Accordingly, the test was changed from a displacement-rate controlled test to a load-rate controlled test. In a load-rate controlled test, the design of the test frame is unrelated to the test result. The load rate selected was similar to the rate reportedly achieved at OU, 5 kips/min. (At a presentation to the PCI Prestressing Steel Committee in April 2004, Don Pellow presented some additional information from Russell, indicating that the load rate achieved at OU was actually closer to 7 kips/min.)

The specimen configuration is shown in Figure B-30, and the test frame is shown in Figure B-31.

Figure B-22. View of large block forms during concrete pour. The student is internally vibrating the concrete.

Figure B-23. View of inside of form.

Figure B-24. Large blocks immediately after casting. Concrete is cured at lab room temperature.

Figure B-25. Close-up view of LVDT used to measure end slip. Note single king wire of strand extends through the concrete.

The mortar mix and other parameters used in the test are as follows:

Mortar mix:
1 part Type III cement by weight
2 parts ASTM C33 sand by weight
0.45 parts water by weight

Required properties:
Required strength at time of test = 3500 to 5000 psi
Age at test: 22 to 26 h

Measured properties:
1 day compressive strength: 3700 psi +/– 200 psi

Figure B-26. Loaded end (live end) of strand.

Figure B-27. Data acquisition system.

Test parameters:
Number of strands per cylinder = 1
Number of cylinders per test = 6
Cylinder dimensions: steel tube 5-in. diameter by 1/8-in. wall thickness × 18-in. long with welded steel base plate with center hole to pass strand through
Strand embedment: 16 in. (top 2 in. of specimen unbonded)

Table B-10. Pull-out test results from concrete, Portland cement mortar, and gypsum plaster mortar.

Strand Source ID	Average Pull-Out Stress at 0.1-in. Slip (psi) Concrete	Portland Cement Mortar	Gypsum Plaster Mortar
Historic Strand [A]			
KSU-F	241	--	--
KSU-H	209	--	--
SC-F	223	--	--
SC-H	472	--	--
SC-IS	682	--	--
101	241	--	--
Recently Manufactured Strand			
102	441	315	588
103	944	397	621
151	541	273	619
153	142/406[B]	--	--
OSU Strand			
349	--	156	--
548		623	
697	--	606	--
717	--	206	--
478 *	--	409	--
960 *	--	409	--

* Samples designated 478 and 960 were from same source.

[A] All Historic Strand concrete pull-out values represent average pull-out stress at first slip rather than at 0.1-in. slip.

[B] Sample from India was tested at Stresscon and KSU and found to be highly variable.

Figure B-28. NASPA test specimen at OU. Note flexible frame.

Figure B-29. NASPA test specimen at KSU. Note stiff frame.

Load rate: 5 kip/min (note: the NASPA test uses a live end displacement rate of 0.1 in./min)

Items to record: Load, ram travel, end slip, first visual movement. Monitor continuously during test. Measure mortar cube strength at beginning and end of test period.

Photographs of the mortar pull-out test specimens and testing procedure are given in Figure B-32 through Figure B-38.

The strand displacement rates achieved during the critical portion of the mortar pull-out tests (around the 0.1-in. end slip) are close to 0.1 in./min (ipm), which is the rate specified by the NASPA test, as presented in Figure B-39.

The average bond stresses (calculated based on the nominal surface area of the embedded section of the strands) from the Portland cement mortar pull-out testing are given in Table B-10.

Hydrocal Pull-Out Test

A range of Hydrocal-based mixtures were evaluated as possible surrogate materials for use in a pull-out test.

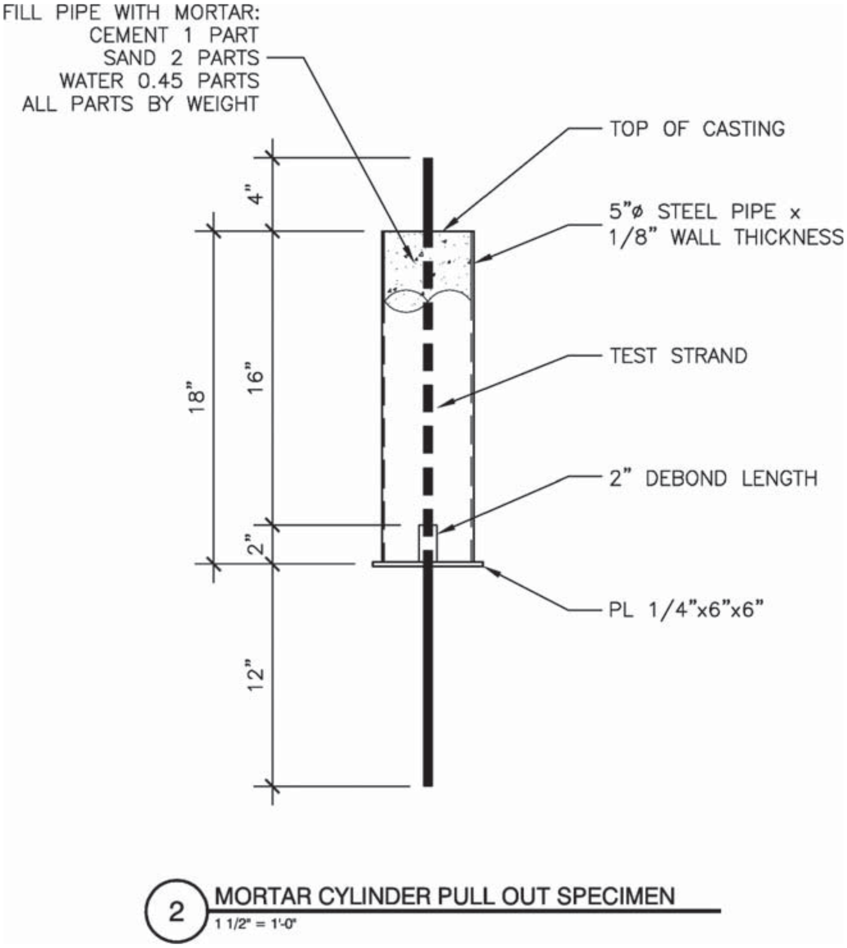


Figure B-30. Pull-out test in mortar specimen.

The final mixture used in this testing contained Hydrocal White (a material similar to plaster of paris made by United States Gypsum), Ottawa graded sand (ASTM C778), calcium hydroxide flakes, USG retarder for lime-based plasters, and water. This formulation of Hydrocal is almost pure plaster of paris and was chosen because it is produced at only one manufacturing facility from consistent raw materials. Also, like cement, it is a calcium compound (plaster of paris is hemihydrated calcium sulfate). Calcium hydroxide flakes were added to simulate the alkalinity of concrete, and the Hydrocal was combined with sand to limit the heat production generated during the rapid plaster hydration.

Aside from water and sand contents, the strength of this Hydrocal mixture is influenced by the mixing procedure (longer, more vigorous mixing results in greater strength) and moisture content after curing (specimens that were dried resulted in higher strengths). Two-inch cubes could be produced that demonstrated compressive strengths of more than 8500 psi at 48 h, if oven-dried for 24 h after demolding. However, that same mixture produced strengths of only 3900 psi if kept moist which is the condition of the Hydrocal mortar within the steel pipe. Because it would take over 7 days to fully dry the plaster, the specimens were tested in a wet condition.

In this test, a prestressing strand is embedded 12 in. in a 3-in. diameter steel cylinder filled with mortar (gypsum/lime plaster, sand, and water). Six cylinders are tested for each source of strand. The test is conducted similarly to the mortar pullout test.

The layout of the test is presented in Figure B-40, and the test frame is shown in Figure B-41. The specimen materials, configuration, and testing procedures are shown in Figure B-42 through Figure B-50.

The Hydrocal mix and other parameters used in the test are as follows:

Hydrocal mortar mix:
1 part Hydrocal White by weight
0.75 parts Ottawa graded sand (ASTM C778) by weight
0.05 parts $Ca(OH)_2$ flakes by weight
0.0002 parts USG retarder for lime-based plasters
0.35 parts water by weight

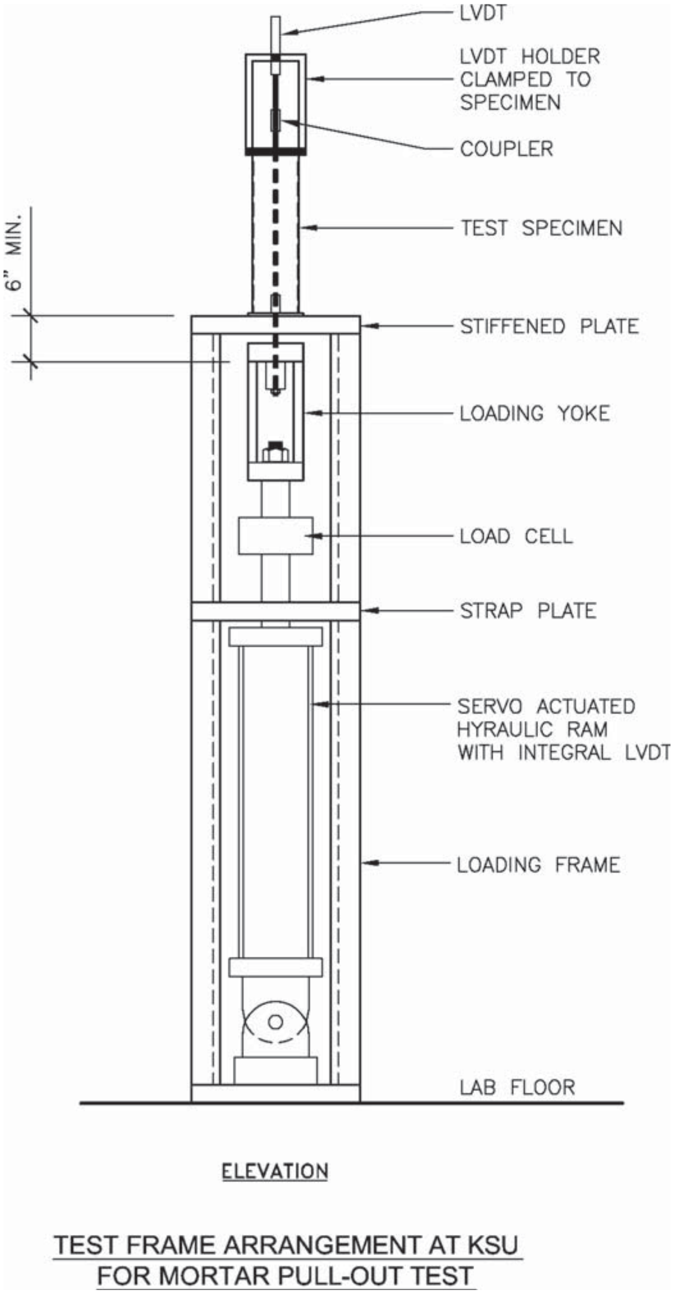


Figure B-31. Test setup for pull-out test in mortar.

Required properties:
Required strength at time of test = 3000 to 4000 psi
Age at test: 22 to 26 h

Measured properties:
1 day compressive strength: 3540 +/– 300 psi

Test parameters:
Number of strands per cylinder = 1
Number of cylinders per test = 6
Cylinder dimensions: standard steel pipe 3-in. diameter by 12-in. long. Welded steel base plate with center hole.

Figure B-32. Mortar cylinder.

Strand embedment: 12 in.
Load rate: 5 kips/min
Items to record: Load, ram travel, end slip. Monitor continuously during test. Measure mortar cube strength at beginning and end of test period.

The average bond stresses (calculated based on the nominal surface areas of the embedded sections of the strands) from the Hydrocal mortar pull-out testing are given in Table B-10.

Pull-Out Test Results

The tests performed in the Screening Round resulted in six data sets for each of the three recently manufactured strand sources and for each of the three pullout test methods for a total of 54 data sets. In each data set is recorded time, load, ram travel, and end slip. For the concrete pullout tests, there is also trigger data corresponding to the point where slip at the loaded end was first noticed. This "observed first slip" is important to maintain because it relates back to historic pullout data recorded by Logan (1997) and others.

To enable comparisons of the shape of the stress-slip curves for each strand source, average curves were calculated using a computer program written in Visual Basic by WJE. The data logging equipment used during testing records data at set

Figure B-33. Top view of empty mortar cylinder.

intervals of time. This results in slightly different load increments for each data set. Accordingly, it is necessary to generate equivalent load increments in each data set so that average slips can be calculated for each increment of load. A load increment of 100 lbs was chosen. The program interpolates the end slips, average, and standard deviation at each increment of load. The average bond stress is plotted against the end slip in the figures shown below with the standard deviation for end slip at that load increment included in a horizontal error bar on each side of each data point.

The averaged bond stress versus slip curves appear in Figure B-51 to Figure B-53 for all three pull-out test methods.

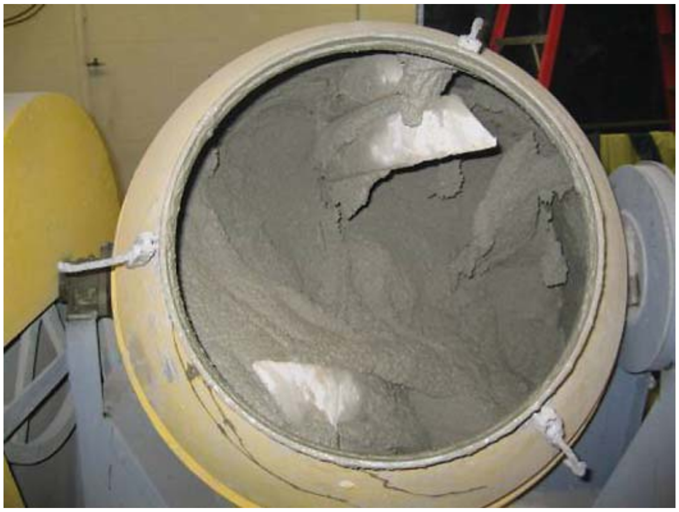


Figure B-34. Mortar in mixer.

Figure B-35. Mortar cubes.

The previously described NASPA test uses a pull-out criteria of 0.1-in. end slip as a way to compare different sources of strand. Based on these test results, this appears to be a good criterion for evaluating the concrete pull-out test as well. This target value replaces the concept of first observable slip in the old Moustafa test because the first observed slip is subject to greater operator error.

The "first observable slip" and "0.1-in. slip" are measured in different ways, yet were found to be close in value to one another. The bond stresses measured at the first observed slip and the 0.1-in. end slip measured in a range of concrete

Figure B-36. Vibrating mortar in specimen.

Figure B-37. Specimen in test frame.

Figure B-38. Specimens ready to test.

pull-out tests are plotted together in Figure B-54. It comes as no surprise that the first observed slip at the live end of the strand occurs at varying values of measured end slip at the dead end of the strand. However, this plot, which also includes a line of equality, shows that these values are generally similar. This is to be expected since the stress at first observed slip and at 0.1-in end slip both occur early in the pull-out test. This similarity is significant because the stress at 0.1-in end slip was not recorded during the historic concrete pull-out tests conducted on the many historic samples that were included in the screening evaluation of the chemical and surface test methods reported in the next section, Surface and Chemical Test Methods and Results. Therefore, in discussing the correlation between bond performance and the chemical test

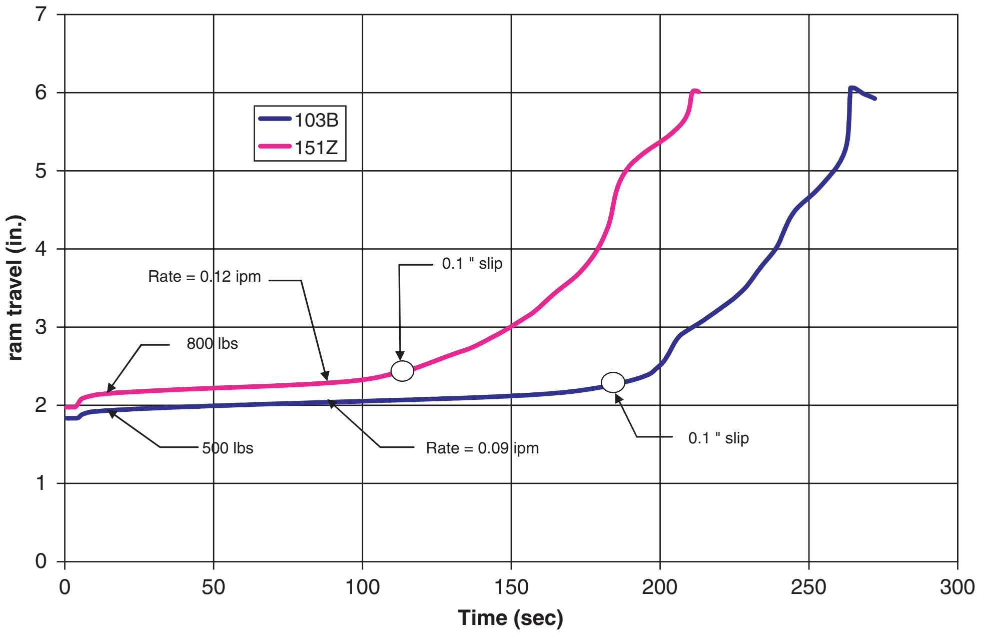


Figure B-39. Ram travel versus time.

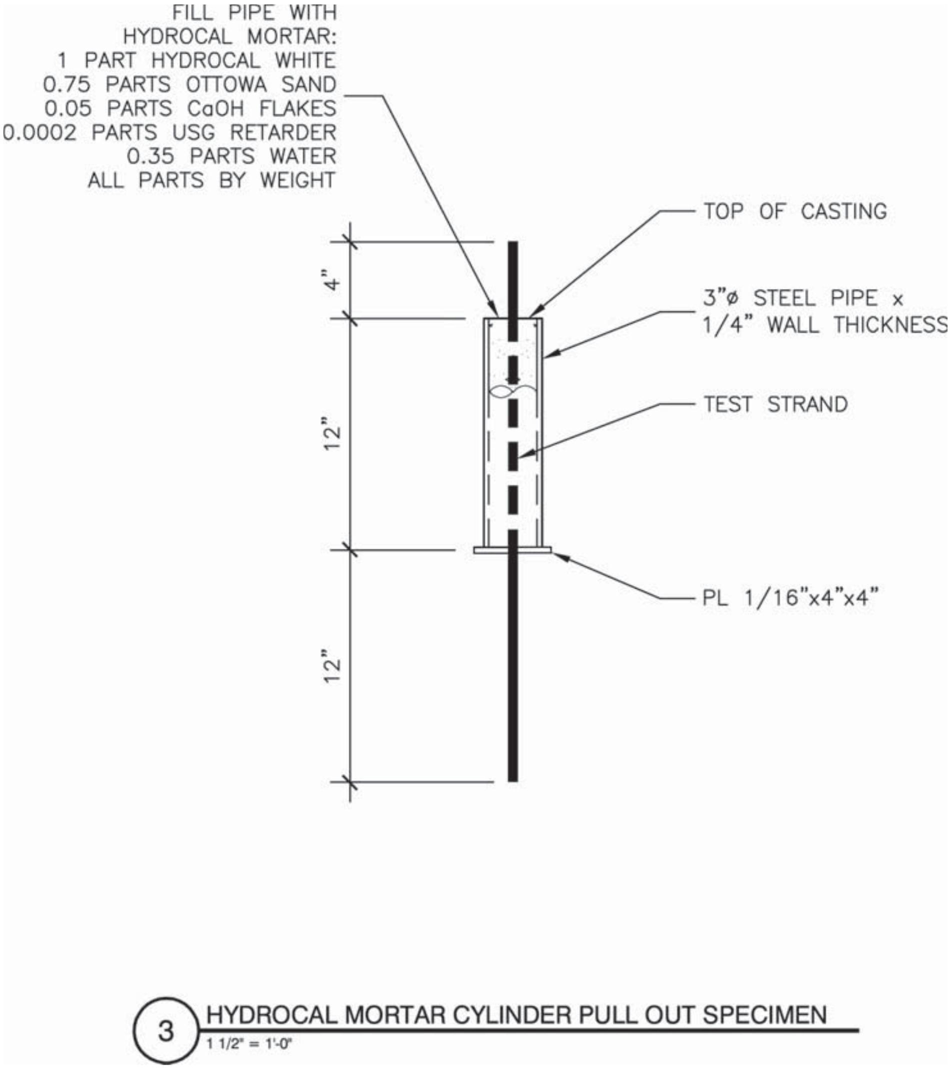


Figure B-40. Pull-out test for the Hydrocal mortar specimen.

results, both types of bond characterization are used together. The average bond stresses, calculated based on the nominal surface area of the embedded section of the strands, for concrete pull-out tests conducted by Logan at Stresscon are given in Table B-10.

Mortar Pull-Out Testing for Correlation Round of Evaluation

As mentioned, the sample sources used for the Correlation Round of testing were selected by Bruce Russell of OSU. Mortar pull-out data were provided for each of the sources (see Table B-2), and the testing protocol is as reported by Chandran (2006). The reported NASPA pull-out forces represent the average load at 0.1-in. slip for multiple (5 to 12) specimens, all tested on the same day with the same batch of mortar. Per the protocol outlined in the thesis, the mortar pull-out force was measured on strands embedded in 5-in. diameter by 18-in. long cylinders (with 16 in. of strand in direct contact with mortar). These mortar pull-out tests were conducted under displacement-rate control, with an additional criterion for load rate. This is in contrast to mortar pull-out tests conducted by this project, which were performed under load-rate control. However, the displacement rate for this project's tests ranged from 0.09 to 0.12 in./min, which is close to the rate of 0.1 in./min specified by the NASPA method.

For comparison with mortar pull-out test results for the Screening Round of testing, the loads at 0.1-in. slip provided from OSU were converted to average bond stresses at 0.1-in. slip. This was done by dividing the given load by the surface area of the strand in contact with mortar (33.51 in.^{2} for 0.5-in. diameter strand). These bond stresses are given in Table B-10. Standard deviations for the OSU bond stresses were not provided along with the data, but were computed based on the full set of data presented in Chandran (2006) for

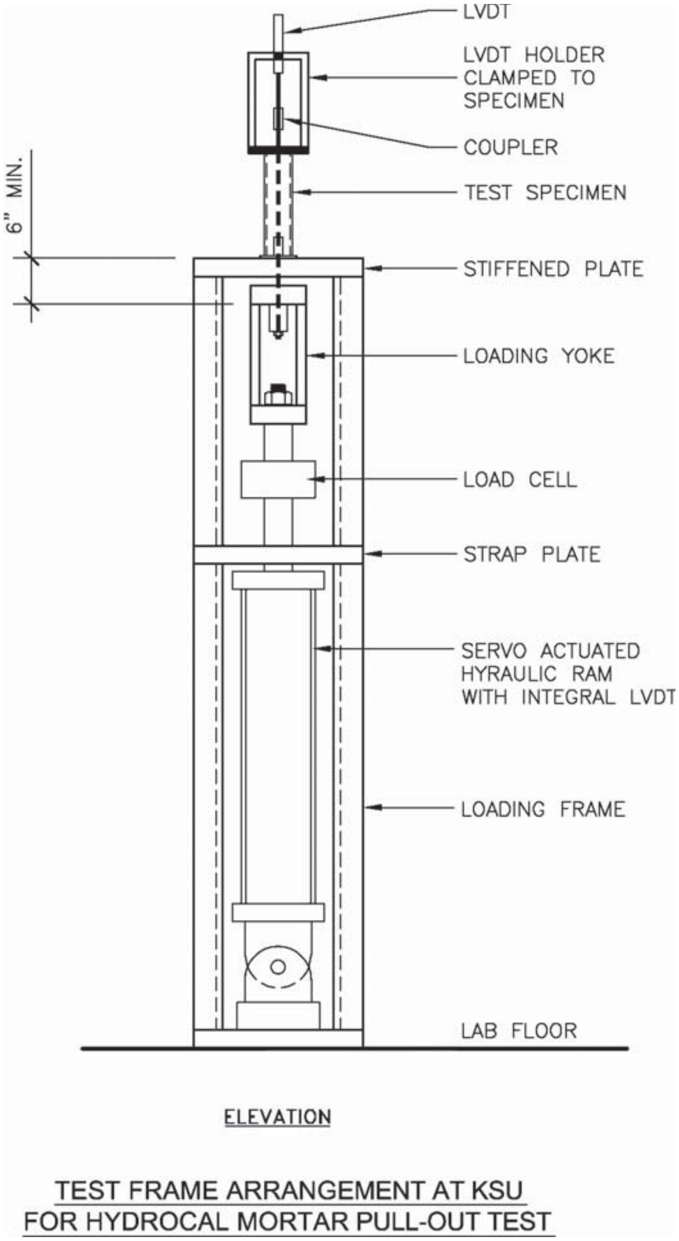


Figure B-41. Test setup for pull-out test in Hydrocal mortar.

all but one source (Source 717). The standard deviation was computed considering all specimens' load data rather than breaking these data into subsets based on batch. Since individual test data were not available for Source 717, the standard deviation plotted with this source was the standard deviation for Batch 8N, which included 12 specimens.

Discussion of Mortar and Large Concrete Block Pull-Out Testing

The relationship between pull-out strength in concrete and pull-out strength in mortar is not well established. However,

Figure B-42. Empty cylinders and mix ingredients.

there is at least one data set for comparison. Pull-out strengths in mortar were measured by Russell in the Round II NASPA tests (Russell and Paulsgrove 1999). These test results were found to correlate quite well with the first observed slip in the large concrete block tests performed on the same strand at Stresscon (unpublished data collected previously by the research team) as shown in Figure B-55. The data suggest that the first observed slip threshold for the large concrete block pull-out test (LBPT) should be 1.5 times that of the NASPA test.

Thus, the NASPA test criteria for 1/2-in. strand of 10,500 lbs would correspond to a first observed slip of 15.8 kips in the LBPT. This value is close to the 16-kip pull-out threshold recommended by Logan.

Surface and Chemical Test Methods and Results

In this section, the experimental study conducted to evaluate the surface and chemical test methods is presented. The test procedure and results are covered separately for each method. The number of samples of each strand source tested by each

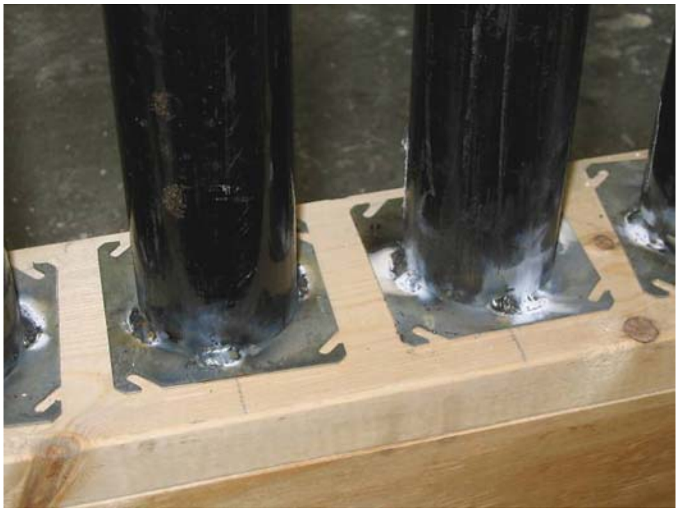


Figure B-43. End plates welded to pipe bases.

Figure B-44. Mixture in bucket and mixing attachment to drill.

surface and chemical test method is shown in Table B-4 and B-5.

Contact Angle Measurement

The contact angle is a measure of surface tension (wettability). It was anticipated that the presence of drawing lubricants would affect this property. Measurements were taken with the strand: (1) in an as-received condition, (2) after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution, and (3) after an ignition process. The calcium hydroxide exposure (also called a lime dip) will convert sodium soaps (e.g., sodium stearates) to insoluble calcium salts. For example, water-soluble sodium stearate (a soap or wetting agent) is converted to a film of insoluble calcium stearate (a wax-like, water-repellent that increases the surface-energy of the strand). This conversion reaction was chosen to simulate the reaction of concrete with surface residues of soaps and is intended to produce a condition where the effect of similar calcium stearate compounds on the contact angle are compared, even if the original residue did not result from a calcium stearate-based lubricant. The ignition process was performed on samples to volatilize organic compounds expected to be present in the drawing lubricants.

Figure B-45. Filling the cylinders.

The contact angle is measured on the projected shadow of a small drop of distilled water that has been applied to the strand surface with a syringe as pictured in Figure B-56.

Figure B-46. Vibrating the Hydrocal mixture.

Figure B-47. Making mortar cubes.

The process of making a measurement with this method is described as follows:

- The strand sample was laid on the stage with the section of wire to be tested facing upward.
- The projection lens was adjusted so that silhouettes of both the wire surface and the syringe needle were in focus.
- The helical wire surface was adjusted to achieve a level silhouette on the projection screen under the syringe needle that was adjusted so that it was directly over the high point of the wire surface.
- A distilled water droplet, with a diameter of seven units on the scale imprinted on the projection screen, was formed using a microdroplet syringe.
- The cylinder stage was raised until the wire surface touched the water droplet and then lowered until the water droplet released from the syringe needle.
- The cylinder stage and projection screen were adjusted to align the left-hand side of water droplet silhouette with the origin of the scale on screen.
- The dial of the protractor component of the instrument was adjusted so that the indicator line intersected the apex of the water droplet silhouette.

Figure B-48. Completed specimens ready for test.

- The contact angle, which is twice the angle measured from the apex of the drop, was read from the protractor scale (the units are already doubled on the projection screen scale shown in Figure B-56) and recorded.

Six individual contact angle readings were taken per strand sample in each of the three conditions that were tested. Readings of a strand sample (one reading per outer wire) were taken in its as-received condition. Readings of a strand sample were also taken after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution to stimulate the environment surrounding the strand when it is in concrete. The immersion time in the calcium hydroxide solution was 10 min and, before testing, the strand was rinsed with water and dried in the following manner: (1) immersion in de-ionized water for 5 min, (2) drying by allowing vertical strand to drip while exposing to a hot air stream from a heat gun, and (3) setting the strand on a clean surface and cooling to room temperature. Finally, readings were taken after an ignition process similar to that discussed for the Weight Loss on Ignition (LOI) test: Pieces of strand approximately 9-in. long were dried for 4 h at 110°C, allowed to cool in a desiccator for at least 12 h, ignited for 30 min at 415°C, allowed to cool in a desiccator for at least 12 h and then tested.

The contact angle values measured are reported in Table B-11. This gives the average contact angle for each source. When possible, contact angle readings were performed on three samples of a strand source.

Contact angle testing after the calcium hydroxide exposure was also included in the Correlation Round. The ignition process to remove organic residue was only used in the

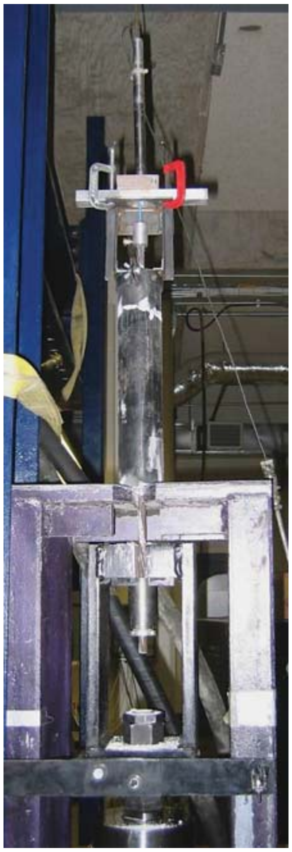


Figure B-49. *Specimen in test frame.*

Figure B-50. Close up of dead end showing LVDT attachment.

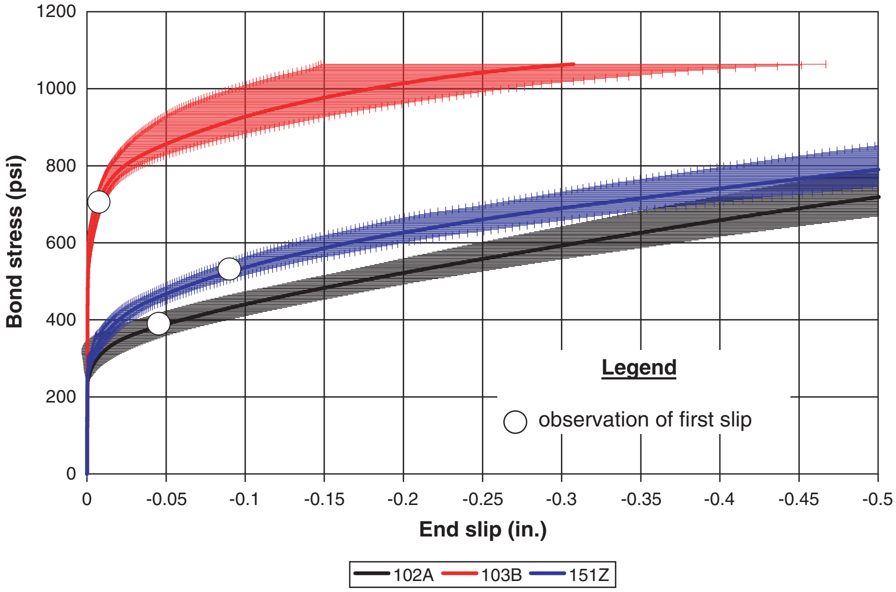


Figure B-51. Bond stress versus end slip from large concrete block pull-out test.

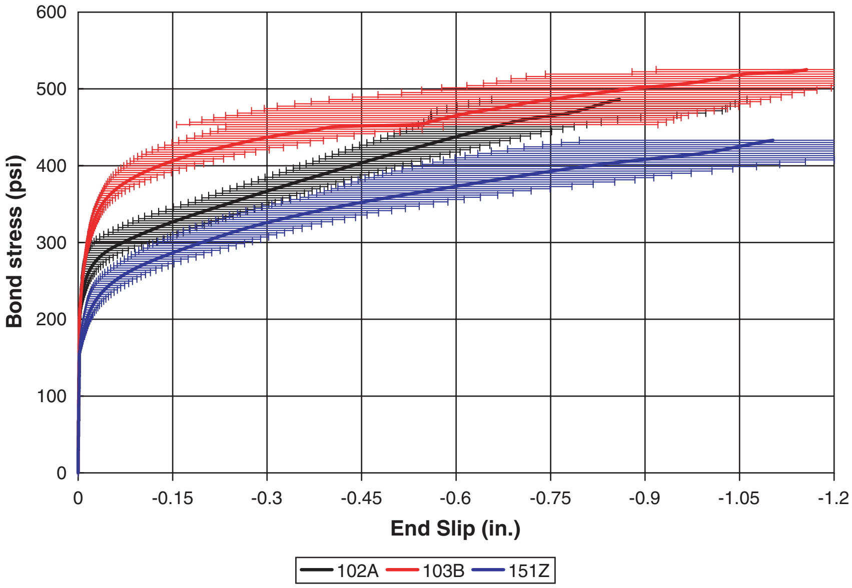


Figure B-52. Bond stress versus end slip from mortar pull-out test.

Correlation Round as a frame of reference representing strand without any residue.

Examination under Ultraviolet Light

The examination under ultraviolet (UV) light was conducted using a range of light sources having different wavelengths. The most promising had a 366-nm wavelength. The examinations revealed detectable fluorescence in three of the strand samples examined. In the first and third, SC-F and 102, the fluorescence was found to be diffuse over the surface of the strand and appeared to coincide with rust product. In the second, SC-IS, the florescence was confined to the interstitial valleys between the wires that make up the strand and appeared as localized speckles (Figure B-57). Observations of the fluorescence are given in Table B-12. This test method was not included in the Correlation Round.

Testing pH

Testing of the pH of the surface was attempted with each of the strand sources to see if measurement of the alkalinity of a solution generated by placing drops of water on the residue could be linked to bond. Testing of the pH of the

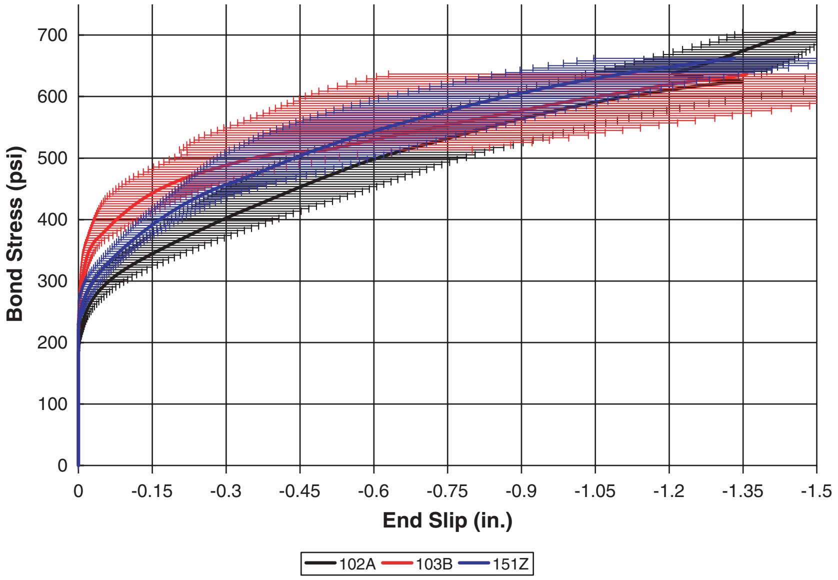


Figure B-53. Bond stress versus end slip from Hydrocal pull-out test.

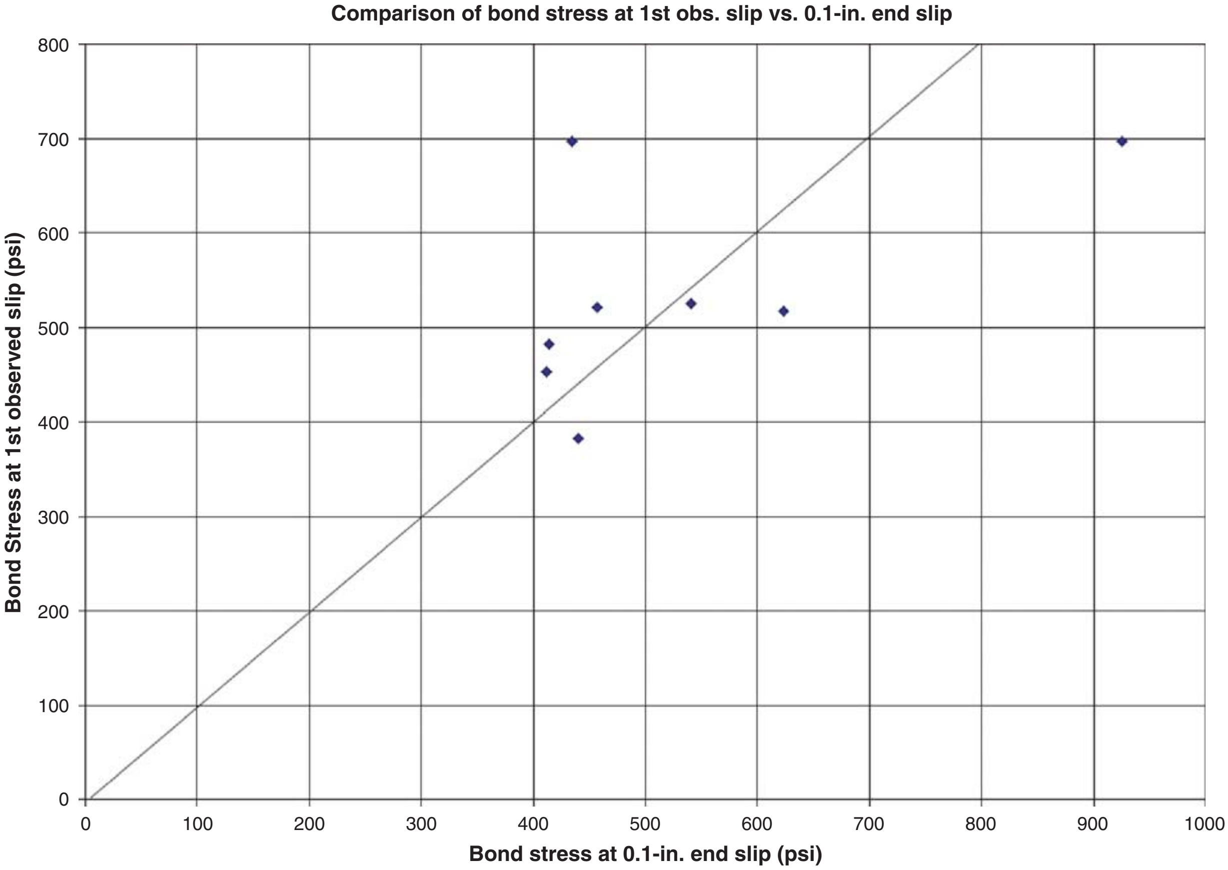


Figure B-54. Correlation between bond stress at first slip and bond stress at 0.1-in. slip.

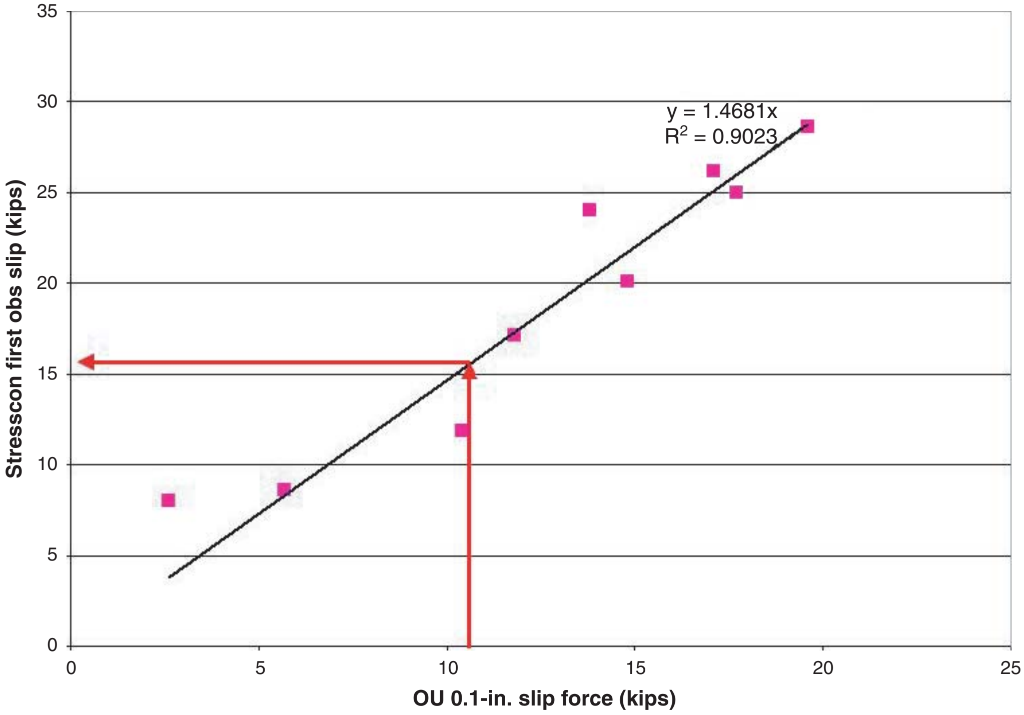
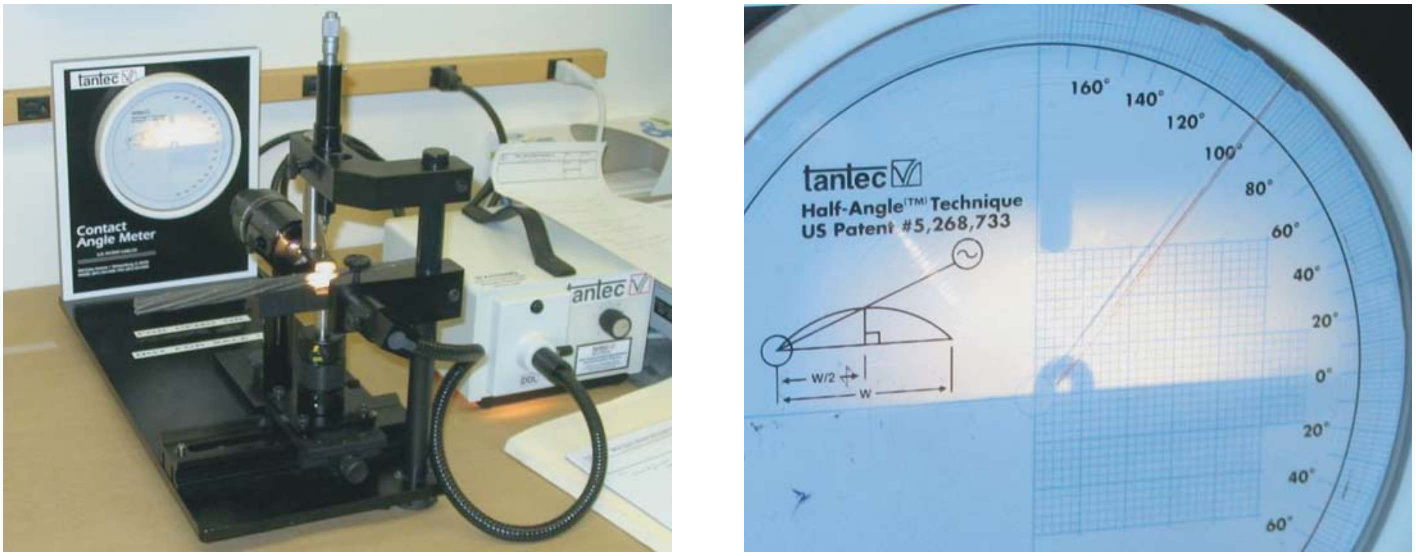


Figure B-55. Comparison of pull-out capacities in mortar versus concrete.



(a)

(b)

Figure B-56. Contact angle meter (a) and close-up of drop projection (b).

surface was conducted using a pH meter, indicator papers, and indicator solutions.

When using universal indicator paper (Tridicator by Filchem, Inc.), droplets of de-ionized water were placed on the strand surface and allowed to dissolve water-soluble surface residue for 30 sec before a piece of indicator paper was touched to the surface. Droplets of water were applied to the outer peak surface of an individual wire in two locations and to the interstitial valley in two locations on each piece of strand tested. A photo of the droplets and the universal indicator paper, after the color change, is given in Figure B-58.

Table B-11. Contact angle.

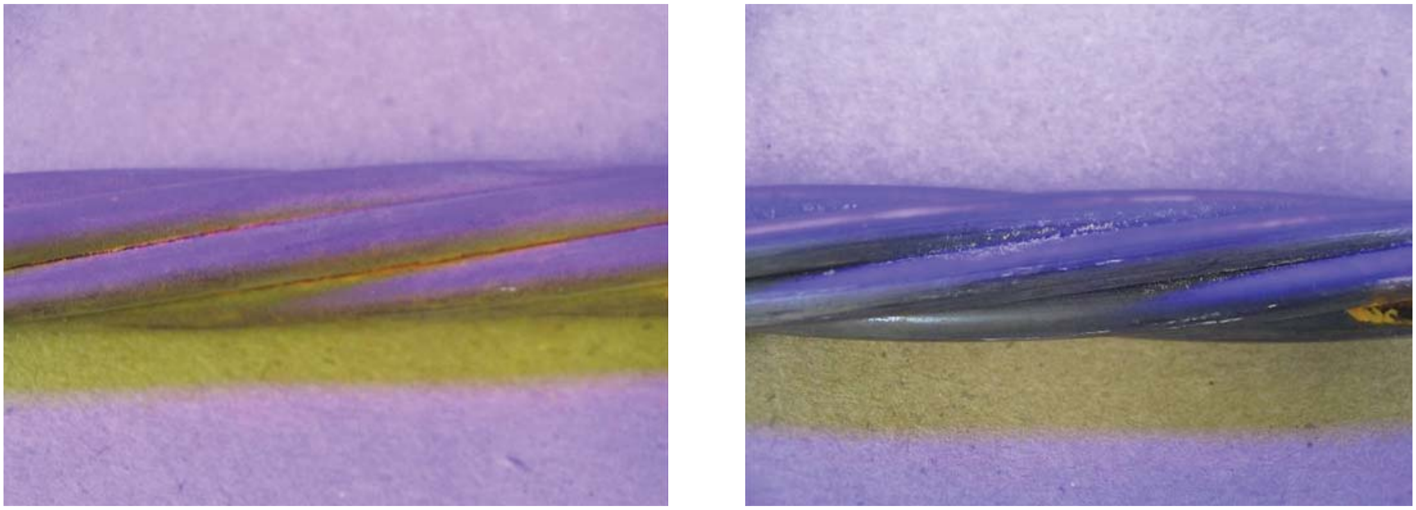
Strand Source ID	Average Contact Angle (°)		
	As-Received	After $Ca(OH)_2$	After Ignition
Historic Strand			
KSU-F	80	106	--
KSU-H	91	103	--
SC-F	--	--	--
SC-H	87	80	--
SC-IS	83	79	--
101	55	94	--
Recently Manufactured Strand			
102	60	87	--
103	71	79	--
151	107	98	--
153	--	75	--
OSU Strand			
349	--	87	11
548	--	79	15
697	--	68	8
717	--	94	12
478 *	--	73	7
960 *	--	76	9

* Samples designated 478 and 960 were actually from same source.

Testing also included experimentation with indicator solutions that change color in a pH range deemed suitable for these tests (7 to 10). These indicators (and the pH range over which they change color) included phenolphthalein (8.2 to 10), curcumin (7.4 to 8.6), thymol blue (8 to 9.6), and cresol red (7 to 8.8). These solutions were found to be inappropriate for testing strand samples since the low surface tension of the solutions caused them to slide over the strand surface. A photo showing the indicator solution colors and the type of color change observed is given in Figure B-59.

Additional pH testing was performed to expand on the resolution of the measurement technique, since this property showed some promise as an identifier of poor bond. This additional testing was done using a hand-held pH meter (Exstik manufactured by Extech) and using specific pH indicator papers (manufactured by Macherey-Nagel), both of which measure pH at a greater resolution than was done with the universal indicator paper. The pH of the aqueous solution produced by applying a de-ionized water droplet to the crevice between two adjacent wires of a strand sample and allowing that droplet to dissolve surface residues for a set period of time was measured.

The Exstik pH meter is pictured in Figure B-60. This instrument was calibrated using pH 7 and pH 10 buffer solutions before each use and reads pH to two decimal places. This meter required that 2 drops of water be placed next to each other on a sample crevice. The reading was taken by placing the meter against the elongated water drop, causing the probe surface to wet sufficiently to wet both ports on the probe tip for each reading. The meter was held against the surface for 10 to 20 sec



(a) (b)

Figure B-57. Surfaces of strand examined under ultraviolet light. Sample SC-F is on the left (a), and sample SC-IS is on the right (b).

to allow the meter to reach an equilibrium reading. Sometimes the meter readings seemed fairly stable, but other readings tended to drift significantly (usually upward) the longer the instrument was held in place.

All four types of high-resolution pH indicator paper from Macherey-Nagel (Part Nos. 90211, 90305, 92160, and 92170) shown in Figure B-61 were tested to measure the pH of the strand surface. The pH indicator papers that appeared most suitable for this application based on the ease of use and the applicable range were the Duotest and pH-Fix 7.5 to 9.5 indicators, and the data presented were collected using these indicators. These read with 0.3 and 0.2 pH unit resolutions, respectively. To measure pH with pH indicator papers, droplets of de-ionized water were placed in crevices and allowed to dwell for 30 sec. The paper edge was then dipped into the water droplet until it made contact with the bottom of the crevice, and it was withdrawn. The Duotest paper was read immediately, but the pH-Fix paper was allowed to stand for 1 min for complete color development before reading.

Table B-12. UV fluorescence.

Strand Source ID	Fluorescence
Historic Strand	
KSU-F	None
KSU-H	None
SC-F	Inconsistent, dull glow, concentrated on one side of strand (rust)
SC-H	None
SC-IS	Speckles in crevices
101	None
Recently Manufactured Strand	
102	Dull glow in crevices (rust)
103	None
151	None

Ten pH measurements were conducted using each of the three high-resolution methods alternately along the length of two strand samples. Each measurement site was separated by approximately 4 in. so the individual test locations for each method were spaced about 1-ft apart. Droplets of water were allowed to stand for 5 min before testing. The averages of the pH readings for each sample are given in Table B-13. The pH of the de-ionized water, according to the pH meter was between 5.5 and 6.2. The average of the pH measured on samples from the same source appears repeatable for Sources 102 and 151, with both sources resulting in a high pH. However, the pH on one of the samples from Source 103 was high (9.02), while the other sample was low (7.62). Nevertheless,

Figure B-58. Surface of strand during pH testing (the bottom of the universal indicator paper, as shown, was touched to the water and has changed color).

Figure B-59. Indicator solutions (from left to right, Thymol Blue, Cresol Red, Curcumin, Phenolphthalein) and strips of indicator-treated paper that had been applied to strand surface.

there is general agreement between the types of measurement, albeit with an apparent offset separating the readings taken with the different methods. Despite the higher resolution of measurement, the pH meter resulted in wider scatter and had a greater standard deviation over the 10 individual readings. The Duotest paper is the easiest to read and resulted in little scatter. The results from each of the three methods are given in Table B-14. The Duotest procedure was included in the Correlation Round.

Figure B-60. Exstik hand-held pH meter.

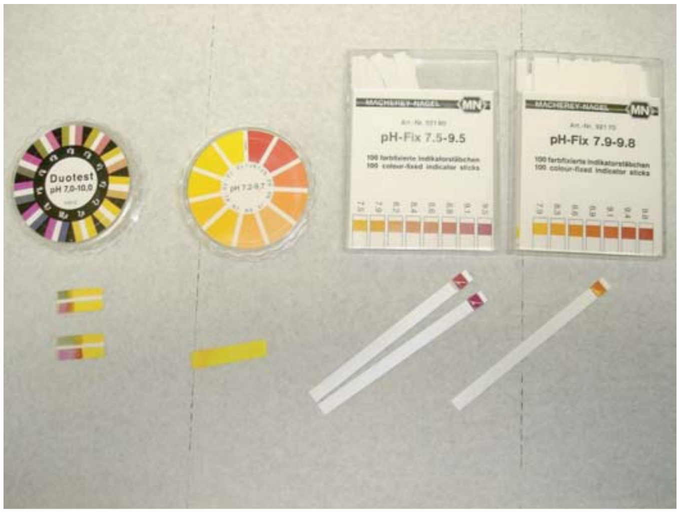


Figure B-61. High-resolution pH indicator papers. The Duotest (left) and pH-Fix 7.5–9.5 (3rd from left) were preferred.

Loss on Ignition

The weight LOI represents the weight of compounds that can be volatilized or burned off the strand surface at high temperature. This property was measured with the expectation that the weight lost may consist mainly of organic residues such as drawing lubricants.

The LOI was measured using the following procedure: Pieces of strand approximately 9-in. long were dried for 4 h at 110°C, allowed to cool in a desiccator for at least 12 h, weighed three times to 0.1-mg precision then ignited for 30 min at 415°C, allowed to cool in a desiccator for at least 12 h, and then weighed again three times on the same balance. The LOI reported is the change in average weight divided by the surface area of the strand. The average LOI for the strands tested are given in Table B-15. This test method was included in the Correlation Round.

Table B-13. pH measurements with universal indicator.

Strand Source ID	Average pH reading Interstitial Valley	Peak
Historic Strand		
KSU-F	9.0	8.0
KSU-H	8.8	7.3
SC-H	7.0	7.0
SC-IS	7.0	7.0
Recently Manufactured Strand		
102	9.0	8.2
103	7.0	7.0
151	7.7	7.0

Table B-14. pH measurements with pH meter and with Duotest and pH-fix indicators.

Strand Source ID	Average pH		
	pH Meter	Duotest	pH-Fix 7.5-9.5
Recently Manufactured Strand			
102	9.36	8.7	9.0
103	8.32	7.4	8.0
151	9.13	8.3	8.4
153	--	7.3	--
OSU Strand			
349	--	7.2	--
548	--	7.1	--
697	--	7.0	--
717	--	7.5	--
478 *	--	7.1	--
960 *	--	7.2	--

* Samples designated 478 and 960 were actually from same source.

Loss in Hot Alkali Bath

The weight loss after hot alkali bath (LAB) represents the weight of compounds that can be washed off the strand surface in a hot sodium hydroxide solution. As with the LOI test, this property was measured with the expectation that the weight lost may consist mainly of drawing lubricants.

The LAB was measured using the following procedures: Pieces of strand approximately 9-in. long were dried for 4 h at 110°C, allowed to cool in a desiccator for at least 12 h, weighed three times to 0.1-mg precision then cleaned according to one of the procedures detailed below, dried in an oven (110°C) for 4 h, allowed to cool in a desiccator for at least 12 h, and then weighed again three times on the same scale. The LAB reported is the change in average weight divided by the surface area of the strand.

The first cleaning procedure (Method 1) used a hot sodium hydroxide solution in an ultrasonic cleaning tank. The sodium hydroxide solution was prepared by slowly adding 500 gm of NaOH pellets to 1,500 mL of de-ionized water (originally about 22°C). (The dissolution of NaOH pellets in water was an exothermic reaction, causing the solution temperature to rise significantly. The temperature of the solution after about 15 min of stirring was 77°C.) The stainless steel tub of the ultrasonic cleaning tank was conditioned by rinsing twice with 80°C water. The hot rinse water was poured out before adding NaOH solution that had been heated to 80°C on a hot plate. The strand samples were disassembled into the individual wires, and the king (center) wire was not included in the testing. Two sets of wires were placed in the tank (each set resting on one half of the tank bottom), and the ultrasonic cleaner was run for 10 min. Each wire was removed individually and thoroughly wiped with a paper towel. After the first wiping procedure, each wire was rinsed with de-ionized water and thoroughly wiped again with a paper towel.

A second round of tests was initiated to see if the original procedure, which removed a large amount of material, was overly aggressive. In the modified tests (Method 2), the strands were tested intact and either the wiping process or the ultrasonic agitation was eliminated. Neither of these modifications appeared to have a significant effect on the results of the test. The average LAB values for the strands tested are given in Table B-16. This test method was not included in the Correlation Round.

Table B-15. Average weight loss on ignition (LOI).

Strand Source ID	Average LOI (mg/cm^2)
Historic Strand	
KSU-F	0.102
KSU-H	0.080
SC-F	--
SC-H	0.036
SC-IS	0.012
101-A-0.5	0.017
Recently Manufactured Strand	
102	0.051
103	-0.021
151	0.002
153	0.375
OSU Strand	
349	0.139
548	0.059
697	0.036
717	0.086
478 *	0.041
960 *	0.045

* Samples designated 478 and 960 were actually from same source.

Table B-16. Average weight loss after alkali bath (LAB).

Strand Source ID	Average LAB - Original Procedure (mg/cm^3)	Average LAB - Modified Procedure (mg/cm^3)
Historic Strand		
KSU-F	0.288	-
KSF-H	0.242	-
SC-H	0.493	-
SC-IS	0.360	-
Recently Manufactured Strand		
101*	0.195	0.197 ^{A}
102*	0.368	0.391 ^{B}
103*	0.416	0.375 ^{B}
151*	0.185	0.167 ^{B}

^{A} - No wipe

^{B} - No ultrasonic agitation

* - Strand tested intact

Change in Corrosion Potential

Past studies of the corrosion resistance of prestressing strand in concrete have suggested that strand with a coating of residue does not corrode as readily as a clean strand. To assess the potential for corrosion, the strand samples were placed in a solution of de-ionized water, and the corrosion potential measured with a reference cell (saturated calomel reference electrode) was monitored versus time. This corrosion potential is determined by the amount of ferrous ions in solution surrounding the sample, and a greater drop in this potential is indicative of a greater tendency to corrode.

Four strand samples were tested at a time using a multiplexer that allowed readings of the potential to be made using a single reference cell. Figure B-62 is a photo of the test setup. While data were collected continuously for 6 h (Figure B-63), an exposure time of 6 h was arbitrarily chosen as measurement cutoff points.

Measurements were taken with the strand in as-received condition, after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution, and after an ignition process. The immersion time in the calcium hydroxide solution was 10 min and, before testing, the strand was rinsed with water and dried in the following manner: (1) immersion in de-ionized water for 5 min, (2) drying by allowing vertical strand to drip while exposing to a hot air stream from a heat gun, and (3) setting the strand on a clean surface and cooling to room temperature. For the ignition samples, readings were taken after an ignition process similar to that discussed for the LOI test: For this process, pieces of strand approximately 9-in. long were dried for 4 h at 110°C, allowed to cool in a desiccator for at least 12 h, ignited for 30 min at 415°C, allowed to cool in a desiccator for at least 12 h, and then tested.

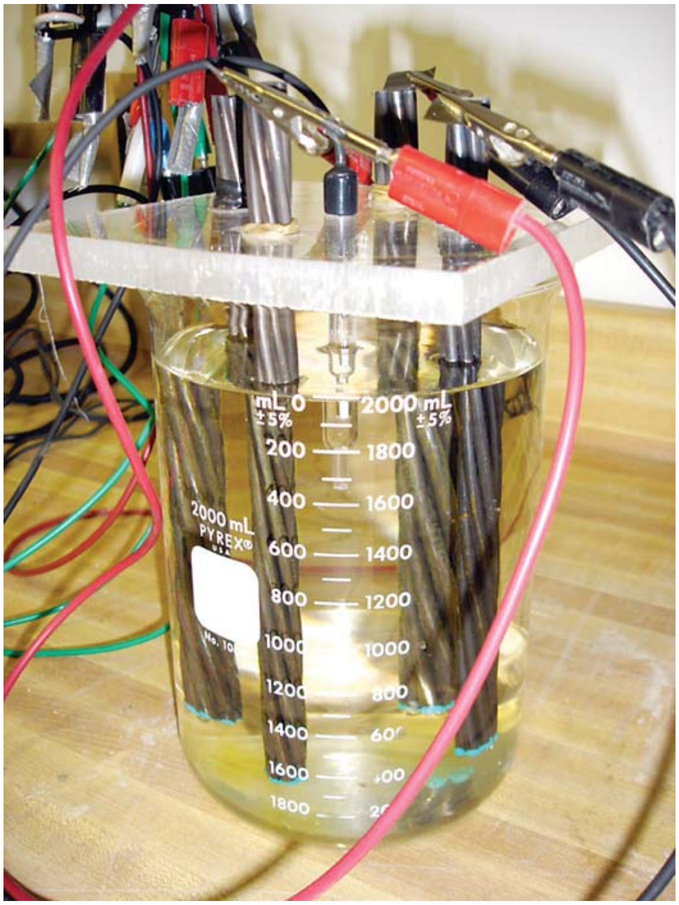


Figure B-62. Setup for monitoring corrosion potential versus time. Saturated calomel reference electrode is in center of figure.

The average changes in corrosion potential from the values initially measured are reported in Table B-17. This test, conducted on samples in as-received condition, was included in the Correlation Round. The ignition cleaning regime to remove organic residues was used only in the Correlation Round as a frame of reference representing strand without any residue.

Surface Roughness

Microscopic examinations of sectioned portions of wire taken from strand have indicated that an observable difference in the surface roughness of the good- and poor-bonding strand sources exists. Based on images captured using a scanning electron microscope, the depth of the roughened surface features is typically 3 μm (0.0001 in.) or less. Trials with a portable profilometer suitable for a QC setting (Figure B-64) were conducted to determine whether these physical measurements could accurately represent the surface roughness and to investigate the correlation with bond performance. This system works by measuring the deflection of a diamond probe, with a tip radius of 2-μm (shown in Figure B-64), as it is dragged 2 mm across the surface of the sample.

The surface profile can be plotted, but it is most commonly interpreted using one of a range of possible parameters calculated based on the profile. The three parameters deemed to be most applicable to this project are R_a, the average roughness; R_z, the average roughness depth; and P_c, the peak count. The definitions of the first two parameters are illustrated in Figure B-65. The peak count is defined by the instrument manufacturer as "the number of profile features whose peaks and following valleys project beyond the upper and lower cut-off thresholds respectively." For our evaluation, the upper and lower thresholds were located a distance 0.13 μm above and below the mean depth of the profile.

The R_a, R_z, and P_c were measured at least twice per wire on each of the six exterior wires on one piece of strand. The average R_a, R_z, and P_c, for each of the tested strand sources is given in Table B-18. This test method was included in the Correlation Round.

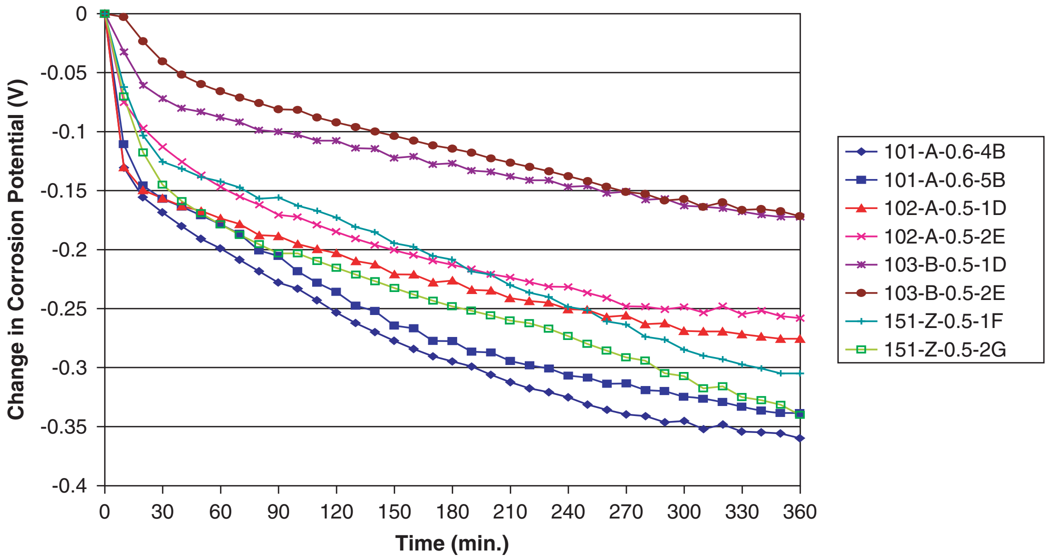


Figure B-63. Change in corrosion potential versus time for recently manufactured strand.

Corrosion Rate

To further explore the interaction between strand bond and corrosion, the instantaneous rate of corrosion of samples of strand in a salt solution was measured with a polarization resistance technique. The polarization resistance technique measures the corrosion current, which quantifies the rate at which the electrochemical corrosion reaction is occurring. This is a much faster test than the test for change in corrosion potential, but requires specialized equipment (a potentiostat).

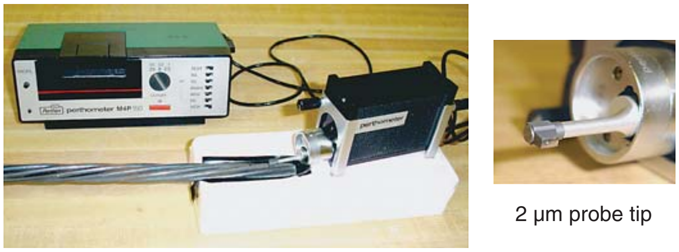
The polarization resistance technique measures the shift in potential of a metal sample from a stable corrosion potential due to an external current. The polarization resistance, R_p, that is, the ratio of the change in potential to the applied current near to the corrosion potential, is inversely proportional to the corrosion current, which quantifies the rate at which the electrochemical corrosion reaction is occurring. This relationship is governed by the Stern-Geary equation: $i_{corr} = B/R_p$, where i_{corr} is the corrosion current density (current per unit area), and B is constant for the metal being testing. The value of B is usually assumed to be 26 mV for steel that is actively corroding, which is the assumption made here.

The potential of the sample is measured relative to a reference electrode, in this case a silver-silver chloride half-cell, which is also submerged in the solution. The solution used in these tests was 0.1% Cl as NaCl by weight. The corrosion rate was measured by varying the corrosion potential ±15 mV from the corrosion potential after the strand sample was

Table B-17. Average change in corrosion potential.

Strand Source ID	Change in Corrosion Potential (V)					
	As-Received		After $Ca(OH)_2$		After Ignition	
	1 h	6 h	1 h	6 h	1 h	6 h
Historic Strand						
101	-0.189	-0.349	--	--	--	--
Recently Manufactured Strand						
102	-0.16	-0.267	-0.188	-0.315	--	--
103	-0.077	-0.172	-0.059	-0.156	--	--
151	-0.16	-0.322	-0.212	-0.377	--	--
153	-0.203	-0.272	--	--	-0.137	-0.268
OSU Strand						
349	-0.228	-0.289	--	--	-0.074	-0.163
548	-0.064	-0.080	--	--	-0.136	-0.222
697	-0.090	-0.154	--	--	-0.142	-0.237
717	-0.170	-0.241	--	--	-0.196	-0.314
478 *	-0.216	-0.272	--	--	-0.080	-0.186
960 *	-0.154	-0.211	--	--	-0.064	-0.140

* Samples designated 478 and 960 were actually from same source.



(a) (b)

Figure B-64. Profilometer (a) and close up of pickup probe (b).

Table B-18. Average roughness parameters.

Strand Source ID	R_a (µm)	R_z (µm)	P_C (µm)
Recently Manufactured Strand			
102	0.66	3.85	183
103	0.40	2.53	210
151	0.54	3.13	199
153	0.41	2.45	244
OSU Strand			
349	0.56	3.33	200
548	0.26	1.65	212
697	0.81	4.72	181
717	1.20	5.83	110
478 *	0.42	2.52	212
960 *	0.50	3.21	203

* Samples designated 478 and 960 were actually from same source.

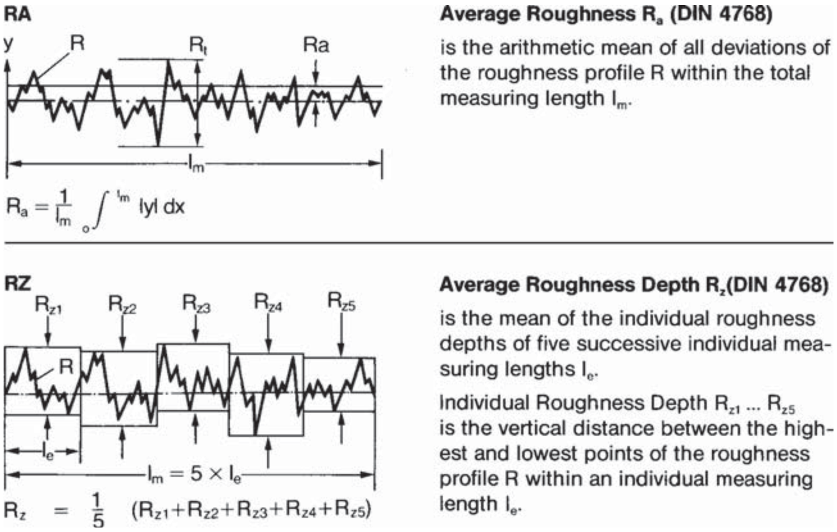
exposed to the chloride solution for 5 min. The exposure length of the samples was 10 cm, and the submerged ends of the samples were coated with epoxy resin so that the cut surface of the sample would not participate in the measurement. Figure B-66 is a photo of the test setup.

Measurements were taken with the strand in an as-received condition, after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution and after an ignition process. The immersion time in the calcium hydroxide solution was 10 min and, before testing, the strand was rinsed with water and dried in the following manner: (1) immersion in de-ionized water for 5 min, (2) drying by allowing vertical strand to drip while exposing to a hot air stream from a heat gun, and (3) setting the strand on a clean surface and cooling to room temperature. For the ignition samples, readings were taken after an ignition process similar to that discussed for the LOI test reported above. For this process, pieces of strand approximately 9-in. long were dried for 4 h at 110°C, allowed to cool in a desiccator for at least 12 h, ignited for 30 min at 415°C, allowed to cool in a desiccator for at least 12 h, and then tested. The ignition cleaning process was used in the Correlation Round as a frame of reference to represent strand without any organic residue.

The average corrosion rates (the corrosion current per unit area) are given in Table B-19. The corrosion rate test was conducted on samples in the as-received condition and after calcium hydroxide exposure in the Correlation Round.

Organic Residue Extraction

The tests for identification and quantification of organic drawing-compound residues were based on solvent extraction procedures, together with gravimetric and FTIR analyses. Essentially, the amount of material extracted from a defined length of strand was determined by weighing the extraction residue on an analytical balance. The material(s) in the



$$R_a = \frac{1}{l_m} \int_0^{l_m} |y|\, dx$$

Average Roughness R_a (DIN 4768)

is the arithmetic mean of all deviations of the roughness profile R within the total measuring length l_m.

$$R_z = \frac{1}{5}(R_{z1}+R_{z2}+R_{z3}+R_{z4}+R_{z5})$$

Average Roughness Depth R_z(DIN 4768)

is the mean of the individual roughness depths of five successive individual measuring lengths l_e.

Individual Roughness Depth R_{z1} ... R_{z5} is the vertical distance between the highest and lowest points of the roughness profile R within an individual measuring length l_e.

Figure B-65. Definition of surface roughness parameters (from Operating Instructions for Perthometer M4P-150).

Figure B-66. Setup for corrosion rate testing.

extraction residue were then identified by FTIR analysis of the residue. The FTIR spectrum obtained is like a fingerprint of the material.

The extraction procedure used is a modification of a procedure found in ASTM C114 for organic materials in cement. Multiple extractions were used to differentiate between various forms of drawing-compound residue. For example, sodium stearate is soluble in warm or hot water, but calcium stearate and stearic acid are insoluble in water. The strand was first washed with warm or hot water to remove water-soluble materials, such as sodium stearate. Then, the strand was exposed to hydrochloric acid and chloroform to extract water-insoluble residues such as calcium stearate and stearic acid.

In the Screening Round of evaluation, the extraction method was done with two successive procedures over a 12-in.-long strand segment: (1) a water-wash procedure, and (2) an acid/solvent-wash procedure. During the water-wash procedure, the strand was washed with either warm (initially 120°F) or hot (initially 212°F) water. The wash solution was acidified, and then the chloroform-soluble organic components were extracted from this acidified solution with chloroform. In the subsequent acid/solvent-wash procedure, the same strand segment was washed with a hydrochloric acid solution (10% HCl), followed by a chloroform-wash. The organic compounds in the aqueous acid wash were then extracted with the chloroform wash solvent. After both of these wash procedures, the chloroform and acidified solutions were separated in a separatory funnel. (The acidified solution was saved for the elemental analysis discussed below.) The chloroform solvent with dissolved organic components was evaporated, and the residue after evaporation was weighed. The weight per unit area was then calculated.

Table B-19. Average corrosion rate.

Strand Source ID	Average I_{corr} ($\mu A/cm^2$)		
	As-Received	After $Ca(OH)_2$	After Ignition
Historic Strand			
KSU-F	32.8	--	--
KSU-H	56.1	--	--
SC-F	--	--	--
SC-H	87	--	--
SC-IS	83	--	--
101	55	--	--
Recently Manufactured Strand			
102	32.7	52.8	--
103	9.4	10.2	--
151	23	44.0	--
153	--	2.9	19.3
OSU Strand			
349	3.1	3.1	13.3
548	3.9	2.0	12.3
697	76.8	46.4	29.4
717	30.5	12.9	14.3
478 *	10.7	10.1	14.8
960 *	10.8	8.7	10.5

* Samples designated 478 and 960 were actually from same source.

The average residue weights generated for three samples from each source by the water-wash procedure and the acid/solvent-wash procedures are given in Table B-20, along with the "total," which is the sum of the two concentrations. The extraction was performed using water at a higher temperature in the second half of the test program since the warm-water wash removed little organic residue on any of the strand. It had been expected that the water wash would differentiate sodium stearate lubricants that are water-soluble from calcium stearate lubricants, which are not. With the exception of Source 101, minimal amounts of water-soluble residue suggest that sodium stearate had not been applied, or had been removed by rain or some other water exposure (washing process), or is less water-soluble than originally thought. The hot water was intended to dissolve more of the sodium stearate, if present.

At the conclusion of the Screening Round, it was observed that the water temperature had little effect in most cases, but that the residue concentrations measured with the warm-water method seemed to generally correlate better with bond tests. Therefore, only a warm-water wash was used in the Correlation

Table B-20. Organic residue extraction concentrations.

Strand Source ID	Average Residue (mg/cm^2) - Warm water			Average Residue (mg/cm^2) - Hot Water			Average Residue (mg/cm^2) - Total [A]
	Warm-Water Wash	Acid/Solvent Wash	Total	Hot-Water Wash	Acid/Solvent Wash	Total	
Historic Strand							
KSU-F	0.006	0.137	0.143	--	--	--	--
KSU-H	0.006	0.112	0.118	--	--	--	--
SC-F	0.005	0.102	0.107	--	--	--	--
SC-H	0.002	0.040	0.042	--	--	--	--
SC-IS	0.001	0.016	0.016	--	--	--	--
101	0.006	0.05	0.055	0.033	0.034	0.067	--
Recently Manufactured Strand							
102	0.005	0.064	0.069	0.005	0.062	0.067	--
103	0.001	0.01	0.012	0.002	0.023	0.025	--
151	0.005	0.034	0.038	0.017	0.024	0.04	--
153	--	--	--	--	--	--	0.186
OSU Strand							
349	--	--	--	--	--	--	0.022
548	--	--	--	--	--	--	0.047
697	--	--	--	--	--	--	0.019
717	--	--	--	--	--	--	0.117
478 *	--	--	--	--	--	--	0.033
960 *	--	--	--	--	--	--	0.035

* Samples designated 478 and 960 were from same source.

[A] All OSU strand tested for total extraction residue with a combined warm-water and acid/solvent wash.

Round. In addition, to minimize the effort spent on performing the time-consuming chloroform organic extraction, the wash solutions from the warm-water and acid/chloroform washes were combined, and a single separation was performed. Therefore, only one FTIR scan and residue weight determination was made per piece of strand. However, this is considered essentially equivalent to the combination of sequential warm-water and acid/chloroform washes performed in the Screening Round. Since quantifying the water-soluble materials was still of interest, because it might provide insight into possible cleaning methods, separate warm-water washes were performed on additional pieces of strand. This wash solution was acidified and saved for elemental analysis. The organic residue concentrations from the single separation of the combined warm-water and acid/chloroform washes for the OSU strand sources is also included in Table B-20.

The FTIR spectra of the residues indicate that a salt of a fatty acid, such as stearic acid, was present in the extractions. A summary of the spectra interpretations, based on multiple spectra for each source, is listed in Table B-21.

Atomic Absorption and Colorimetric Analyses

To identify the chemical composition of residual inorganic components of pretreatment chemicals and drawing compounds, chemical analyses of the acidified water-extract and acid-solvent-extract solutions, which had been separated from the chloroform, were performed. Either zinc phosphate or borax (sodium borate) is often applied to the wire before the drawing process begins to help drawing lubricants stick to the surface of the rod stock. Most common drawing lubricants are expected to include stearate salts, particularly sodium and calcium stearates. The elemental concentrations

Table B-21. Interpretation of FTIR spectra.

Strand Source	FTIR Spectra Interpretation*
Historic Strand	
KSU-F	stearic acid
KSU-H	stearic acid
SC-F	stearic acid
SC-H	stearic acid, acrylic
SC-H	stearic acid
SC-IS	fatty acid, trace styrene
101	stearic acid
Recently Manufactured Strand	
102	stearic acid
103	fatty acid
151	stearic acid
153	fatty acid
OSU Strand	
349	fatty acid, resin
478	stearic acid
548	fatty acid
697	fatty acid, resin
717	stearic acid
960	stearic acid

*Stearate salts are converted to stearic acid by the organic residue extraction process.

of sodium, potassium, calcium, and zinc were determined by atomic absorption spectroscopy. The solutions were also scanned for detectable quantities of aluminum during the Screening Round. Colorimetric analyses of the wash solutions for boron and phosphate (PO_4^{3-} as total phosphate) were performed using visible light spectroscopy.

Atomic Absorption Studies

Dilutions were made with de-ionized water as needed to produce final solutions with concentrations in the linear ranges of the desired elements under study. Lanthanum chloride, 0.1% by volume, was added at the rate of 5 mL/100 mL of solution to prevent ionization of the desired elements. Standard solutions of sodium, potassium, calcium, and zinc were made using purchased NIST traceable 1000 mg/L atomic absorption standards. These standards were diluted to produce linear standards for the concentration ranges desired. The atomic absorption spectrometer was calibrated using the prepared standards. Each diluted solution was run twice with recalibration of the spectrometer for each run to determine the concentration of the selected element. The values were averaged, and the concentration of each element in milligrams per centimeter of strand was calculated. To detect the presence but not exact quantities of aluminum, the diluted wash solutions were checked for the presence of aluminum without instrument calibration.

The average concentrations measured with atomic absorption spectroscopy taken as the average of three samples from each source are given in Table B-22 and Table B-23. The quantity of aluminum was below detectable levels.

Visible Light Spectroscopy

Portions of the filtered wash solutions from the water-wash and acid/chloroform-wash procedures were also analyzed for boron using sulfuric-carminic acid with visible light spectroscopy. An aliquot of the wash solution was mixed with the sulfuric-carminic acid solution and allowed to stand for 25 min. The absorbance of the solution was read using a Hach visible light spectrometer set at 605 nm. Boron standards made from boric acid were used to prepare calibration curves. Examples of the appearance of the solutions are given in Figure B-67. The concentration of boron in milligrams per liter (mg/L) of wash solution was determined from the calibration curves obtained. The average boron concentration of three samples from each source is given in Table B-22 and Table B-23.

During the Correlation Round, portions of the filtered acid extraction solutions were analyzed for phosphate using molybdic acid reaction and visible light spectroscopy. The orthophosphate in the solution reacts with the molybdate in an acidic medium, producing a phosphomolybdate complex that is blue in color. The color of the solution is proportional to the concentration of phosphate. An aliquot of the extraction solution was mixed with the molybdic acid solution and allowed to stand for 2 min. The absorbance of the solution was read using a Hach visible light spectrometer set at 890 nm. Based on this value, the concentration of phosphate in the extraction solution was determined and this was converted to a strand surface concentration. The results of phosphate analyses can be found in Table B-23.

Evaluation of Test Methods

This evaluation was conducted in three evaluation rounds: screening, correlation, and precision. The evaluation of the Screening and Correlation Rounds are presented together, even though correlation testing was not conducted on all the test methods attempted. The precision testing is discussed separately.

Variability in Test Methods

The goal of this project was the identification of test methods that could accurately measure or predict the bond quality of prestressing strand in concrete. To determine whether a proposed method under consideration was able to achieve this goal, the results obtained with that test method were compared with other test results deemed to be a reliable measure of bond quality. It should be noted that all test methods have a certain level of uncertainty and that poor correlation will result if there is variability or inaccuracy in either the method being evaluated or the test result against which it is compared.

Each data point on the plots presented in this section is the average of a series of measurements using each technique. Horizontal and vertical error bars on the plots display one standard deviation in each direction from the data point. Assuming a normal distribution, the range covered by these bars includes 68% of the measured data.

Evaluation of Mechanical Test Methods

Before assessing the correlation of the surface and chemical test methods to bond performance, the correlation between the mechanical test methods was evaluated. This was possible only for the recently manufactured strand, since they were the only available sources that could be obtained in sufficient length to support transfer length testing. The transfer length test, measured based on strain profiles, is considered the most accurate measure of bond performance. This is because this method most closely simulates the in-service conditions in which prestressing strand is used, evaluating the bond performance of strand in concrete in a stressed condition. To allow for comparisons to be made across samples with different

Table B-22. Elemental analysis—average for each source—results from Screening Round testing.

Strand Source ID	Sodium (mg/cm^2)			Potassium (mg/cm^2)			Calcium (mg/cm^2)			Zinc (mg/cm^2)			Aluminum (mg/cm^2)			Boron (mg/cm^2)		
	Water	**Acid**	**Total**	**Water**	**Acid**	**Total**	**Water**	**Acid**	**Total**	**Water**	**Acid**	**Total**	**Water**	**Acid**	**Total**	**Water**	**Acid**	**Total**
Historic Strand																		
KSU-F	0.227	0.033	0.260	0.061	0.014	0.075	0.005	0.073	0.078	0.002	0.004	0.006	N.D.	N.D.	N.D.	0.075	0.024	0.099
KSU-H	0.174	0.031	0.205	0.049	0.012	0.061	0.007	0.065	0.072	0.002	0.004	0.006	N.D.	N.D.	N.D.	0.061	0.024	0.085
SC-F	0.013	0.080	0.093	0.004	0.008	0.012	0.076	0.705	0.781	0.002	0.876	0.878	N.D.	N.D.	N.D.	0.002	0.008	0.010
SC-H	0.018	0.130	0.148	0.005	0.023	0.028	0.004	0.072	0.076	0.002	0.874	0.876	N.D.	N.D.	N.D.	0.004	0.013	0.017
SC-IS	0.009	0.188	0.197	0.002	0.025	0.027	0.005	0.023	0.028	0.002	1.407	1.409	N.D.	N.D.	N.D.	0.002	0.009	0.011
101 (warm)	0.140	0.030	0.170	0.034	0.008	0.042	0.002	0.014	0.016	0.002	0.002	0.004	N.D.	N.D.	N.D.	0.090	0.009	0.099
101 (hot)	0.154	0.026	0.180	0.035	0.008	0.043	0.002	0.013	0.015	0.002	0.002	0.004	N.D.	N.D.	N.D.	0.096	0.007	0.103
Recently Manufactured Strand																		
102 (warm)	0.150	0.037	0.187	0.019	0.008	0.027	0.013	0.269	0.282	0.002	0.002	0.004	N.D.	N.D.	N.D.	0.033	0.018	0.051
102 (hot)	0.174	0.018	0.192	0.029	0.004	0.033	0.019	0.272	0.291	0.002	0.002	0.004	N.D.	N.D.	N.D.	0.040	0.008	0.048
103 (warm)	0.014	0.098	0.112	0.002	0.013	0.015	0.005	0.019	0.024	0.002	0.894	0.896	N.D.	N.D.	N.D.	0.006	0.013	0.019
103 (hot)	0.015	0.101	0.116	0.002	0.014	0.016	0.006	0.017	0.023	0.003	0.937	0.940	N.D.	N.D.	N.D.	0.006	0.013	0.019
151 (warm)	0.063	0.020	0.083	0.005	0.002	0.007	0.002	0.027	0.029	0.002	0.002	0.004	N.D.	N.D.	N.D.	0.042	0.021	0.063
151 (hot)	0.072	0.019	0.091	0.004	0.003	0.007	0.005	0.024	0.029	0.003	0.002	0.005	N.D.	N.D.	N.D.	0.053	0.021	0.074

N.D. = Not detected

Table B-23. Elemental analysis—average for each source—results from Correlation Round testing.

Strand Source ID	Sodium (mg/cm^2)			Potassium (mg/cm^2)			Calcium (mg/cm^2)			Zinc (mg/cm^2)			Boron (mg/cm^2)			Phosphate (mg/cm^2)		
	Warm Water	Acid	Total	Warm Water	Acid	Total	Warm Water	Acid	Total	Warm Water	Acid	Total	Warm Water	Acid	Total	Warm Water	Acid	Total
Recently Manufactured Strand																		
153	0.030	0.086	0.115	0.009	0.024	0.033	0.016	0.579	0.595	0.003	1.209	1.212	0.002	0.009	0.011	--	--	1.467
OSU Strand																		
349	0.133	0.048	0.181	0.003	0.004	0.006	0.030	0.436	0.466	0.004	0.960	0.964	0.002	0.006	0.008	--	--	1.229
548	0.016	0.148	0.164	0.004	0.039	0.043	0.018	0.075	0.093	0.004	1.395	1.399	0.002	0.005	0.007	--	--	1.605
697	0.017	0.048	0.065	0.003	0.003	0.005	0.044	0.385	0.429	0.004	0.946	0.950	0.002	0.007	0.009	--	--	1.206
717	0.019	0.111	0.130	0.002	0.002	0.004	0.016	0.294	0.310	0.004	0.890	0.894	0.003	0.011	0.014	--	--	1.080
478 *	0.029	0.135	0.165	0.009	0.056	0.065	0.004	0.052	0.056	0.004	0.960	0.964	0.002	0.009	0.011	--	--	0.902
960 *	0.026	0.130	0.156	0.009	0.053	0.061	0.003	0.048	0.051	0.004	0.968	0.972	0.003	0.011	0.014	--	--	0.933

* Samples designated 478 and 960 were actually from same source.

strand diameters and to directly account for the stress transferred to the concrete, the average bond stress over the transfer length was calculated in the manner discussed in the previous section on Mechanical Test Methods and Results.

As reported, the bond stresses of the recently manufactured strands were measured at 0.1-in. slip during pull-out testing in concrete, mortar, and Hydrocal. This concrete bond stress is compared to the average bond stress over the transfer length for each strand source in Figure B-68. A nearly linear relationship exists between the concrete bond stress and the bond stress over transfer length and the ranking of the three sources in terms of bond quality is consistent. In Figure B-69, the mortar bond stress has a fair correlation with the mortar bond stress at 0.1-in. slip. Figure B-70 compares the Hydrocal bond stress to the bond stress over the transfer length. A weak relationship between Hydrocal pull-out stress and average bond stress over transfer length is visible, but the correlation is less clear than with the concrete or the mortar, because of the large variability in the pull-out performance.

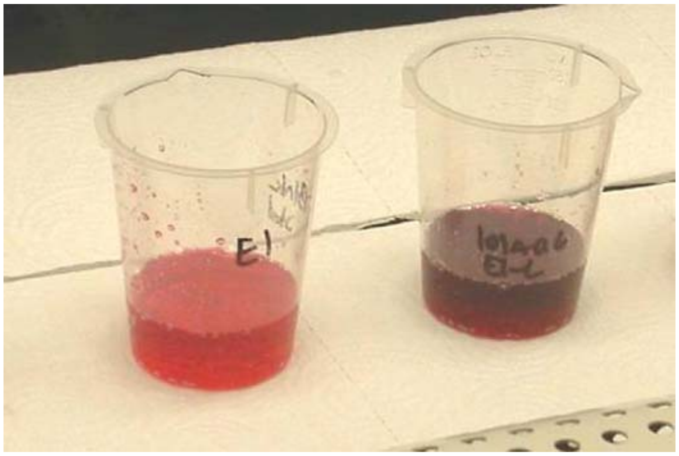


Figure B-67. Boron analyses: lighter sample is a blank while the darker sample on the right is a sample taken from strand and contains boron.

All three methods differentiate between the higher bond control strand (Source 103) and the marginal bond strands (Sources 102 and 151). The concrete pull-out test is more discriminating than the other two test methods since the high bond strand had a pull-out bond stress almost twice the value of the marginal bond strands. In the mortar cylinder test, the high bond strand only had 1.3 times the pull-out value of the marginal bond strand. The Hydrocal mortar pull-out test is the least discriminating, with the high bond strand bond stress of only 1.2 times the marginal bond strand's at 0.1-in. slip. As can be seen in Figure B-53, at 0.5-in. slip, one of the marginal bond strand sources carried an even higher stress in the Hydrocal than the high bond strand, rendering this test method unworthy of further consideration.

The comparatively smaller range in bond stresses measured using the mortar pull-out test was expected, based on the literature. However, more discrimination was expected than was recorded in this test series. During the NASPA tests performed at KSU in 2002, the tested strands had bond stresses at 0.1-in. end slip ranging from 167 to 715 psi. The range in the most recent tests reported herein ranged from 273 to 397 psi, admittedly on a different series of strands. One primary difference between the two test programs is the mortar strength. In the 2002 tests, the mortar strengths were about 5200 psi. In the current series of tests, the mortar strength was about 3700 psi. Another difference is the use of load control versus displacement-rate control. In the tests run in 2002, load rates as high as 11 kips/min were achieved for the high bond strand compared to the 5 kips/min used in the current series.

No additional mechanical testing was conducted in the Correlation Round of the evaluation.

Evaluation of Surface and Chemical Test Methods

To compare the surface and chemical testing to bond behavior, the results of these tests were plotted against pull-out

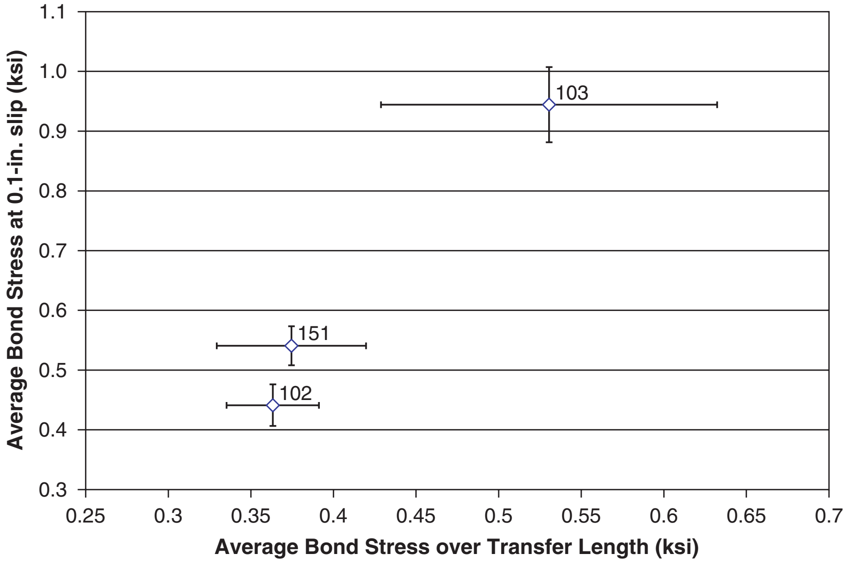


Figure B-68. Concrete pull-out bond stress versus bond stress over transfer length (recently manufactured strand).

test behavior measured in the LBPTs in the Screening Round of our evaluations and in the mortar pull-out tests in the Correlation Round of our evaluations. Additional comparisons were made between the results of the surface and chemical test methods and the transfer length test results for the recently manufactured strand. Since the transfer length was not measured on the historic strand and the available transfer length results for the samples provided by OSU were deemed unreliable, the plots presented here show only correlation with pull-out behavior.

As discussed above, in all of the historic strand sources, first slip measurements were recorded by visual inspection. For recently manufactured strand (strands from Sources 102, 103, and 151), the stress used for plotting is taken as the stress when end slip was 0.1 in.

Contact Angle

The contact angle measured on the as-received strand did not correlate well with average bond stress over the transfer length or the bond stress at first or 0.1-in. slip during concrete pull-out testing. This was probably because the lubricant contained mixed forms of stearate. The contact angle on strand after ignition process also did not correlate well with the bond

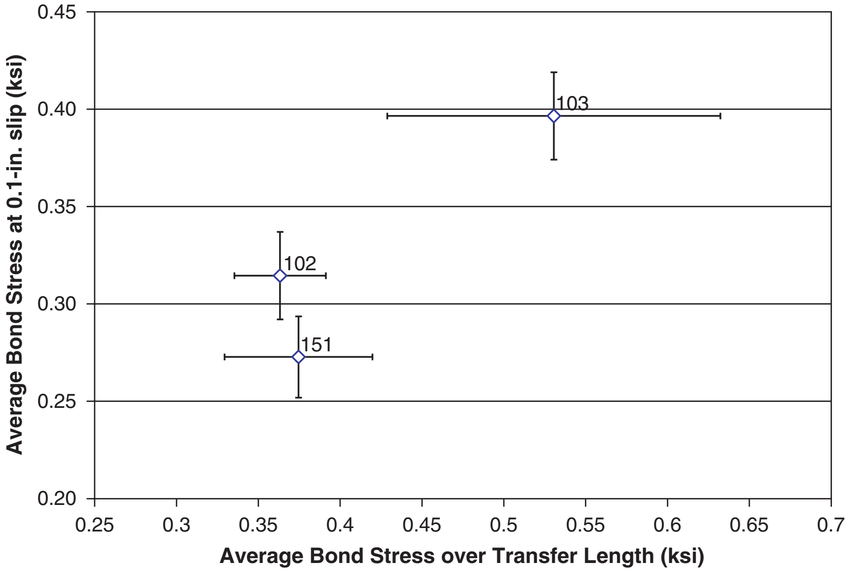


Figure B-69. Mortar pull-out bond stress versus bond stress over transfer length (recently manufactured strand).

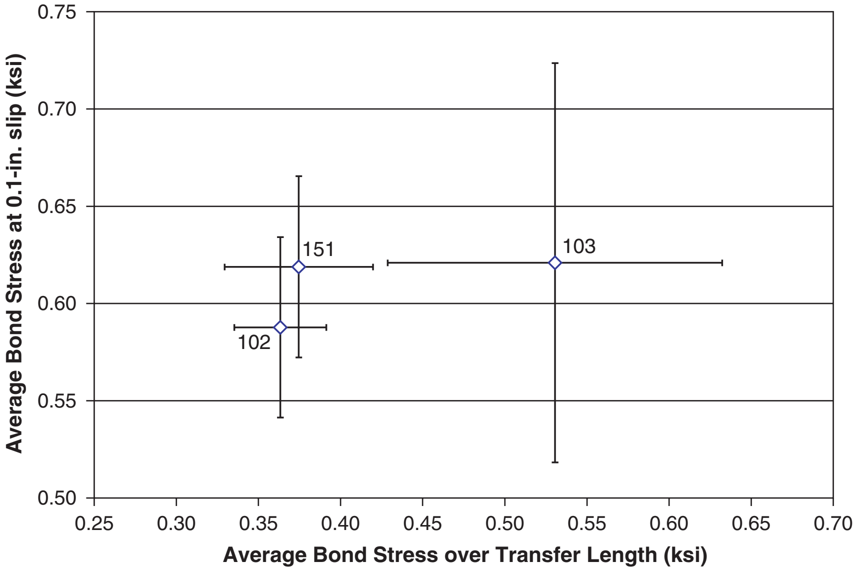


Figure B-70. Hydrocal pull-out bond stress versus bond stress over transfer length (recently manufactured strand).

stress at 0.1-in. slip during mortar pull-out testing. In fact, nearly all ignited sources tested gave similar results.

Figure B-71 is a plot of the contact angle after the strand was dipped in a saturated calcium hydroxide solution versus stress at 0.1-in. slip in concrete. The contact angle after exposure to calcium hydroxide correlated well. The contact angle was lower with greater bond stress. This makes conceptual sense since strand that is more water-repellant will produce a higher contact angle and apparently is less likely to bond well. Measured contact angles below approximately 85° after the calcium hydroxide exposure were indicative of good bond. Because of this good correlation, this test was included in the correlation testing. A comparison of contact angle after exposure to calcium hydroxide and 0.1-in. slip stress in mortar is given in Figure B-72. These results from the Correlation Round confirmed the relationship between and contact angle after calcium hydroxide exposure and bond.

Examination under Ultraviolet Light

Table B-24 shows the bond stress at first or 0.1-in. slip during concrete pull-out testing and the bond stress over the

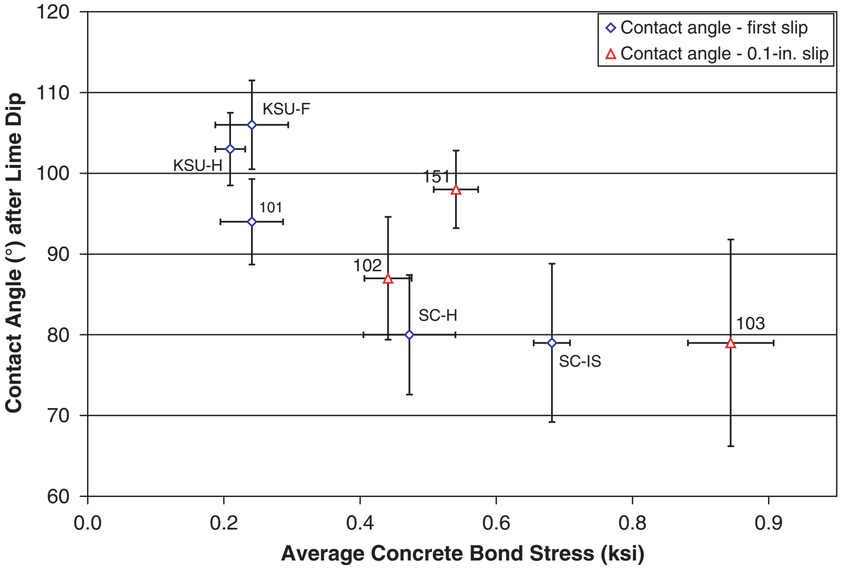


Figure B-71. Correlation between contact angle on strand after a dip in calcium hydroxide solution and bond stress in concrete at first or 0.1-in. slip (historic and recently manufactured strand).

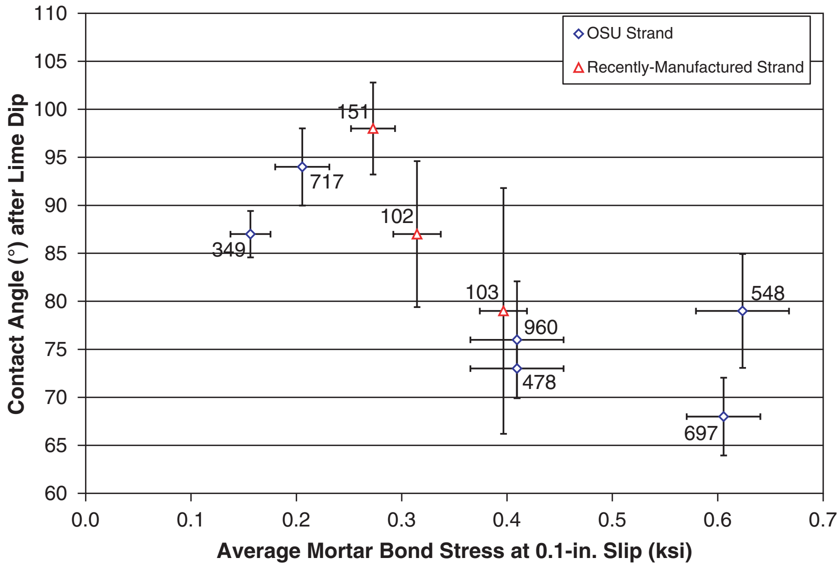


Figure B-72. Correlation between contact angle on strand after a dip in calcium hydroxide solution and bond stress in mortar at first or 0.1-in. slip (OSU strand).

transfer length measured for each of the recently manufactured strand sources and the observed fluorescence under UV light. No correlation was found for either property. It is possible that this approach could be useful if inert, fluorescing tracer compounds were intentionally added to the drawing lubricant materials during their manufacture. However, since the commonly available drawing materials do not contain such materials, this method proved ineffective and was not included in the Correlation Round of evaluation.

Testing pH

Initial testing with the lower resolution, universal Tridicator paper on both the historic and recently manufactured strand indicated a limited correlation with bond. This correlation was strongest when the droplets were applied to the crevice between wires on the strand surface. Therefore, additional testing of the crevice area was conducted on recently manufactured strand (no additional historic strand was available) with three different high-resolution measurement techniques: pH meter, Duotest paper, and pH-Fix paper. A higher pH corresponded to a lower average bond stress for all methods. The quality of the correlations differed depending on the measurement method, with the Duotest paper showing the best correlation. The relationship between pH measured with this method and the 0.1-in. slip stress during concrete pull-out testing is plotted in Figure B-73. As bond stress increased, the pH measured with each of these

Table B-24. UV fluorescence and mechanical properties.

Strand Source ID	Fluorescence	Bond Stress at First Slip or 0.1-in. Slip (psi)	Average Bond Stress over Transfer Length (psi)
Historic Strand			
KSU-F	None	241	-
KSU-H	None	209	-
SC-F	Inconsistent dull glow, concentrated on one side of strand (rust)	223	-
SC-H	None	472	-
SC-IS	Speckles in crevices	682	-
101	None	241	-
Recently Manufactured Strand			
102	Dull glow in crevices (rust)	441	363
103	None	944	531
151	None	541	375

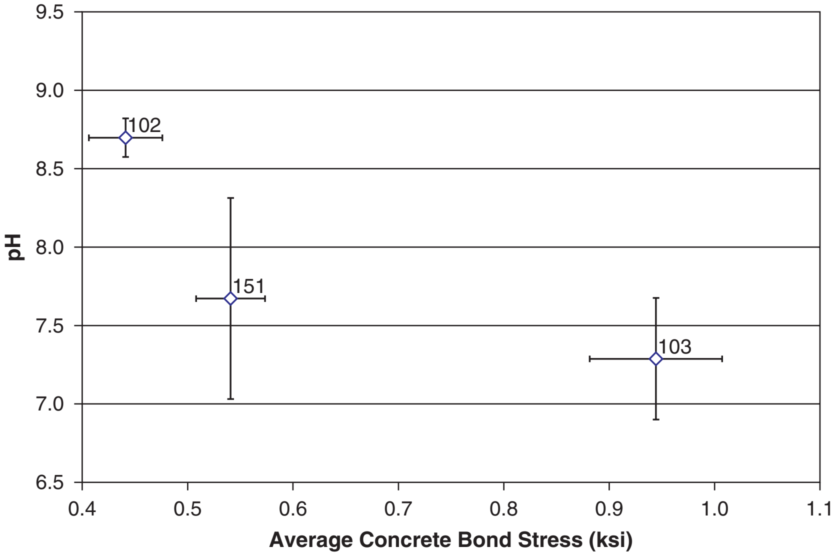


Figure B-73. Correlation between the Duotest paper pH reading and the bond stress in concrete at 0.1-in. slip (recently manufactured strand).

methods decreased for all three sources of recently manufactured strand.

The pH testing conducted in the Screening Round was deemed initially successful at finding a correlation with bond. Of the methods used to measure pH, the Duotest paper was most effective, since it was easiest to use and produced the least scatter. Therefore, correlation testing using the pH test was performed with the Duotest indicator paper. Comparison of pH and 0.1-in. slip stress in mortar for the recently manufactured and OSU strands is given in Figure B-74.

When all the measurements are considered, the relationship between poor bonding performance and pH is unclear. A lower pH (<8) does not guarantee satisfactory bonding performance. However, the pH test results appear to be influenced by the presence of borax pretreatments, which were apparently used on a number of the historic and recently manufactured strands, based on the elemental analysis of the residue extracts. Analysis of the samples from OSU indicated that none of these strands were pretreated with borax. Therefore, distinguishing between these

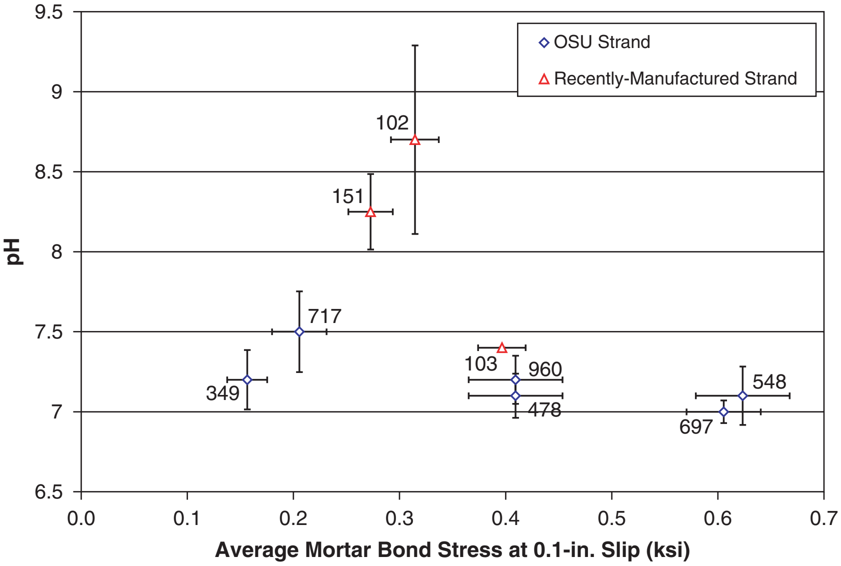


Figure B-74. Correlation between Duotest Paper pH reading and bond stress in mortar at 0.1-in. slip (recently manufactured and OSU strand).

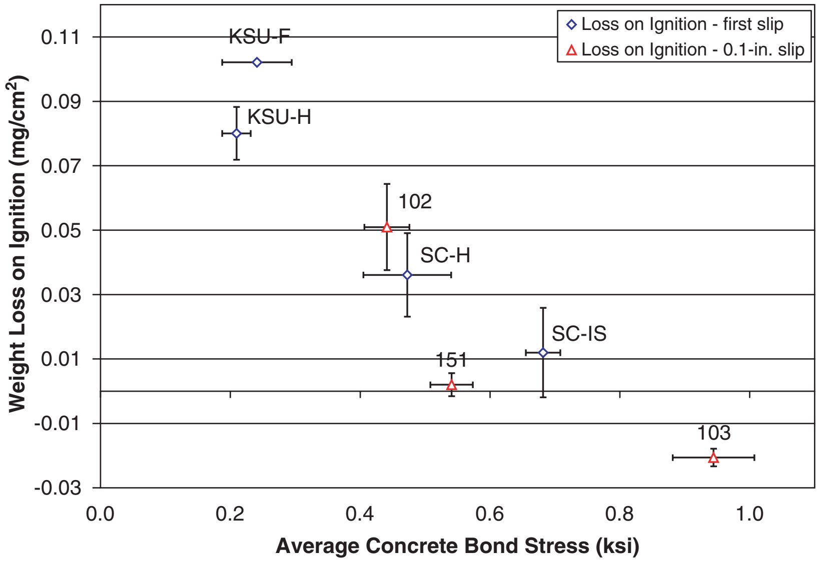


Figure B-75. Correlation between weight loss on ignition and bond stress in concrete at first or 0.1-in. slip (historic and recently manufactured strands).

latter sources using the pH test method is apparently not as useful.

Loss on Ignition

The weight LOI versus concrete pull-out bond stress at first or 0.1-in. slip is plotted in Figure B-75. This shows that, as the average bond stress increased, the weight loss on ignition decreased. The samples of the strand from Source 103 did not lose weight, but in fact gained weight due to oxidation of the steel.

Since good correlation was observed, this test was included in the Correlation Round of testing. The results of this testing are compared to the mortar pull-out bond stress at 0.1-in. slip in Figure B-76. The correlation here is less clear, even among the recently manufactured strand, which showed better correlation when compared to concrete pull-out results. However, high weight losses above 0.7 mg/cm^2 did appear to be consistent with low bond. While this test appears to have moderate effectiveness at predicting mortar pull-out stresses, it is easy to conduct and is therefore recommended as part of a larger

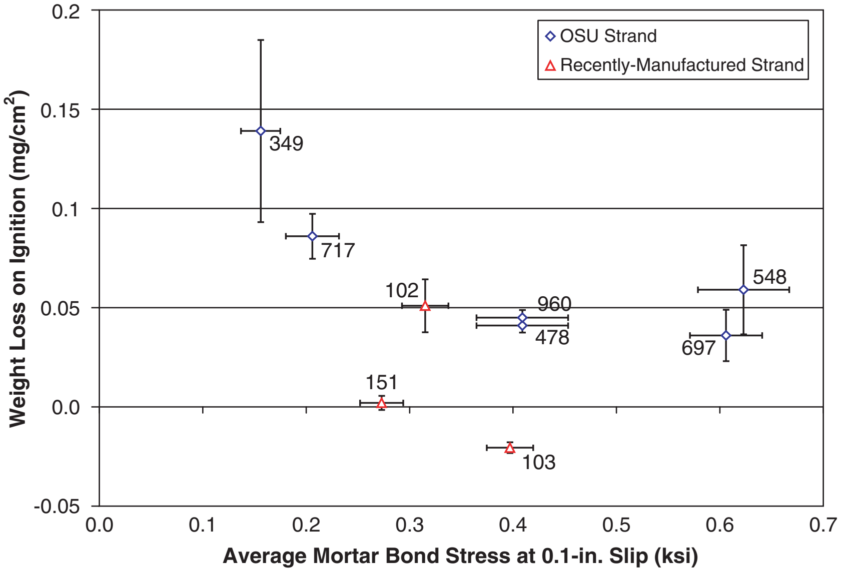


Figure B-76. Correlation between weight loss on ignition and bond stress in mortar at 0.1-in. slip (recently manufactured and OSU strands).

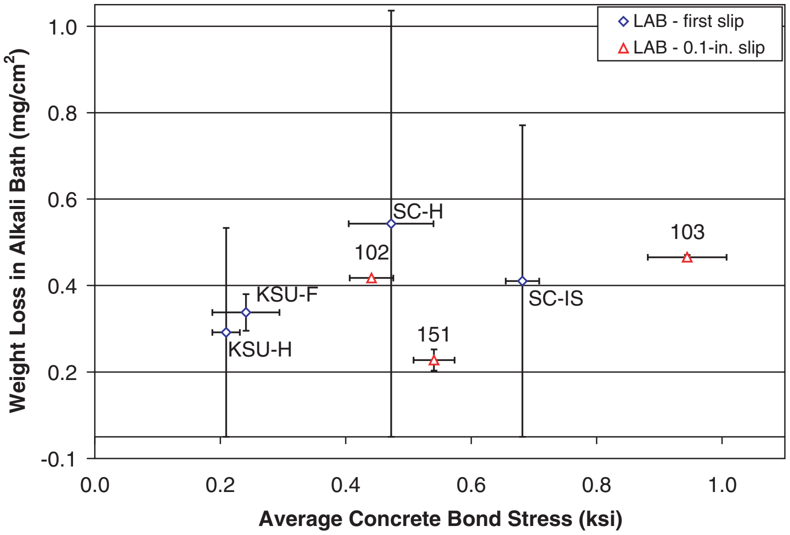


Figure B-77. Correlation between weight loss in alkali bath and bond stressing concrete at first or 0.1-in. slip using Method 1 (historic and recently manufactured strands).

QC program. Since many materials that are not related to bond may be ignited during this test, the use of this method without other companion testing is not recommended.

Loss in Hot Alkali Bath

No trends were apparent between the weight loss after soaking the strand in a hot alkali (sodium hydroxide solution) bath and average bond stress over transfer length or pull-out bond stress at first or 0.1-in. slip using either of the attempted methods. The weight loss in alkali bath and bond stress in concrete at first or 0.1-in. slip using Method 1 (the more aggressive cleaning procedure) is shown in Figure B-77. Based on this lack of correlation, this test method was not included in the Correlation Round of evaluation.

Change in Corrosion Potential

When compared to concrete pull-out bond stresses at first or 0.1-in. slip, a greater drop in corrosion potential correlated with lower bond stress in the strands as-received (Figure B-78). This correlation was also noted after exposure to calcium

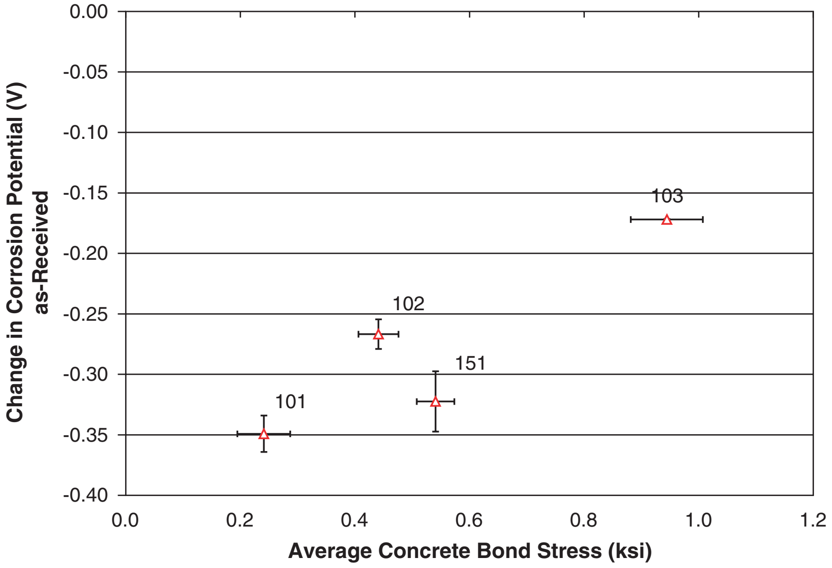


Figure B-78. Correlation between corrosion potential change and bond stress in concrete at 0.1-in. slip (recently manufactured strand in as-received condition).

hydroxide solution, but less pronounced. Similar trends were observed based on the transfer length data. Note that a greater corrosion potential drop implies a greater readiness to corrode.

Based on this apparent correlation, a 6-h test to measure the change in corrosion potential of strand in the as-received condition was included in the Correlation Round. The results of that testing are shown in Figure B-79. Testing after ignition was not helpful for evaluating bond performance.

Since a greater drop in corrosion potential means that the strand corroded more easily, this was contrary to what was expected. Instead, it had been anticipated that a strand with some surface residue would be more likely to resist corrosion. This expectation was based on field observations of flash rusting on clean steel and based on work by Perenchio et al. (1989). The metallurgical examination of the strand surface may explain why the corrosion performance is contrary to expectations. The half-cell tests provide indications of active corrosion, but not where the corrosion is occurring. It is possible that the relatively rough surfaces found in low bond strand provide small corrosion potential sites at the "peaks" of the surface profile, compared to the "valleys," where most of the lubricant presumably resides. Thus, the corrosion activity measured may be occurring in small localized areas. The greater surface area per unit length of the rougher strand when considered on a microscopic scale may also support more corrosion.

Surface Roughness

Increased surface roughness as measured using the parameter R_a corresponded to a lower bond stress in concrete at 0.1-in. slip, as shown in Figure B-80. This was consistent with the visual interpretation of SEM micrographs discussed in Appendix D. Similar trends were observed based on the transfer length data. As a result, this test was included in the Correlation Round of evaluation. The results of that testing are shown in Figure B-81. These data show a slight correlation but, as can be seen from the standard deviations in these figures, there is significant variation in the individual readings. The measurements are relatively quick and easy to take, but this test method does not appear to be effective enough to differentiate the mortar pull-out bond quality of the strand sources.

Corrosion Rate

As seen in Figure B-82, corrosion rate correlated well with the concrete bond stress at first or 0.1-in. slip. Note that a higher corrosion rate implies a greater readiness to corrode. For each test of as-received strand and strand after exposure to calcium hydroxide solution, higher bond stress corresponded to lower corrosion rates. Similar trends were observed based on the transfer length data. Because of this good correlation, this test was included in the Correlation Round of evaluation. In that round, the corrosion rate did not correlate well in any of the three conditions (as-received, after calcium hydroxide solution exposure, and after ignition) tested. The results of the as-received tests are plotted versus mortar pull-out test results in Figure B-83.

The trend that corrosion occurs more readily on strands with poorer bond properties, at least when concrete pull-out testing is considered, is similar to that observed during the

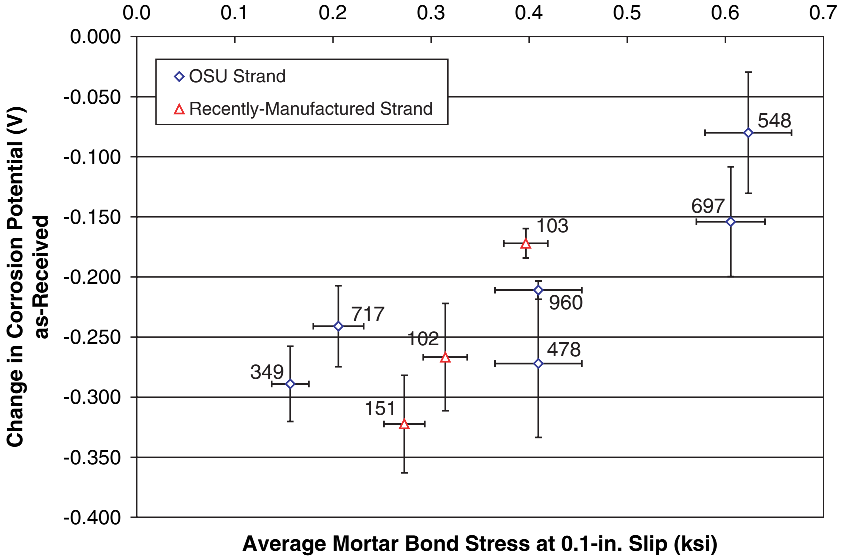


Figure B-79. Correlation between corrosion potential change and bond stress in mortar at 0.1-in. slip (recently manufactured and OSU strand in as-received condition).

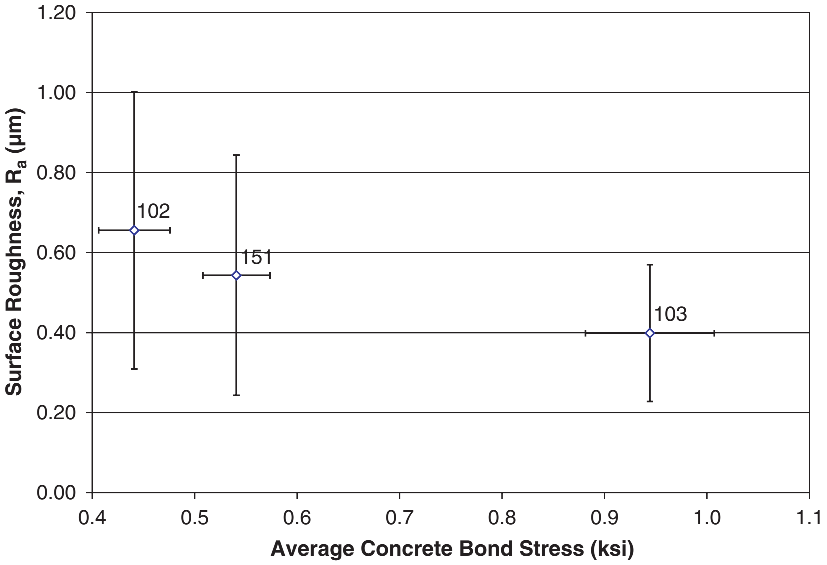


Figure B-80. Correlation between surface roughness parameter R_a and bond stress in concrete at 0.1-in. slip (recently manufactured strand).

change in corrosion potential measurements, and is also contrary to prior expectations. Possible explanation for the unexpected result is described in the section entitled Change in Corrosion Potential.

Organic Residue Extraction

For the sources tested as part of this study, as extraction residue decreased, bond stress increased. The relationship is evident when the "total" extraction residue, that is the combined residue concentration from both the water and acid/chloroform wash solutions, is considered. As shown in Figure B-84, the "total" extraction residue correlates well with the concrete pull-out bond stress at first or 0.1-in. slip for both the hot- and warm-water wash procedures. A similar strong relationship exists between the extracted organic residue and the average bond stress over the transfer length.

Because of this good correlation, this test was included in the Correlation Round of evaluation. In that round the mortar pull-out bond stress at 0.1-in. slip correlated less well with

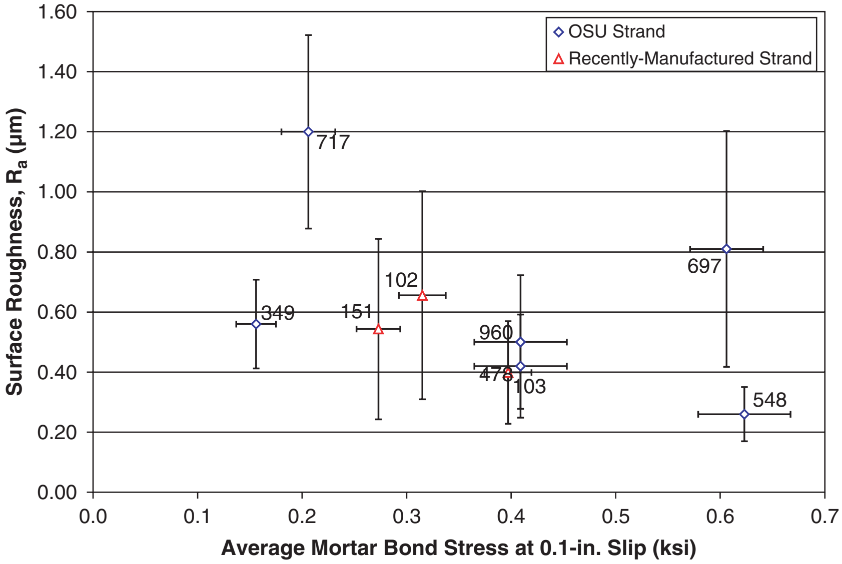


Figure B-81. Correlation between surface roughness parameter R_a and bond stress in mortar at 0.1-in. slip (recently manufactured and OSU strand in as-received condition).

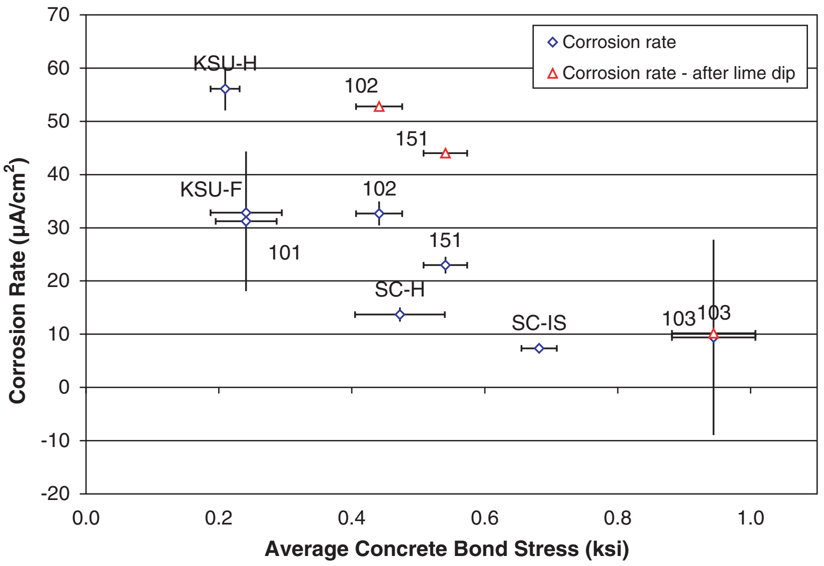


Figure B-82. Correlation between corrosion rate as-received and after calcium hydroxide (lime) solution exposure and concrete bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

total residue (Figure B-85). However, samples with a high concentration of organic residue (greater than 0.05 mg/cm^2) had a bond stress at 0.1-in. slip in mortar lower than 0.4 ksi.

Elemental Analyses

The elemental analysis was conducted on the extraction solutions in both the screening and correlation testing. This testing was performed to provide information about components in pretreatments and lubricants used to manufacture strand and the effect these components may have on bond performance.

Sodium—The surface concentration of sodium in the water wash per unit area of strand versus the concrete pull-out bond stress at first or 0.1-in. slip is illustrated in Figure B-86. The sodium concentration from the water-wash solutions and the combined water and acid/solvent wash concentrations showed a similar relationship to bond stresses: a high concentration of sodium correlates to a low concrete

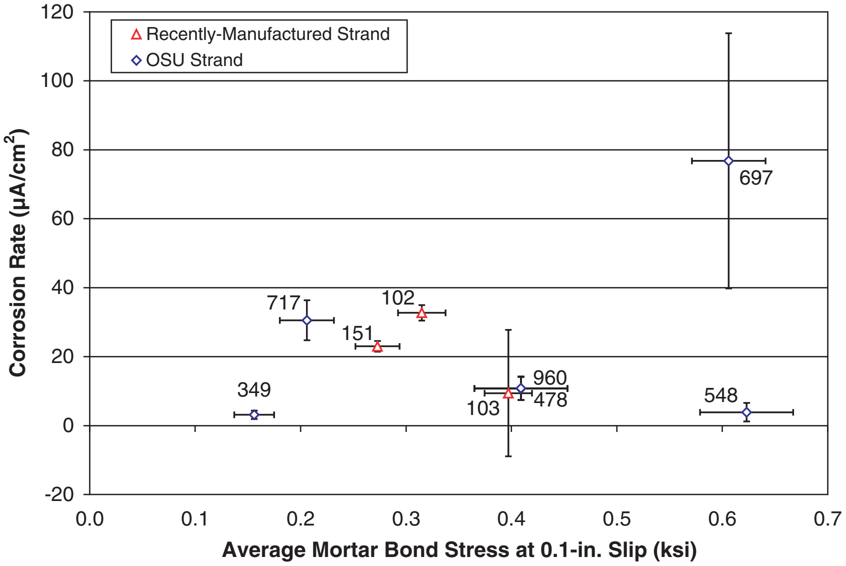


Figure B-83. Correlation between corrosion rate as-received and bond stress in mortar at 0.1-in. slip (recently manufactured and OSU strand).

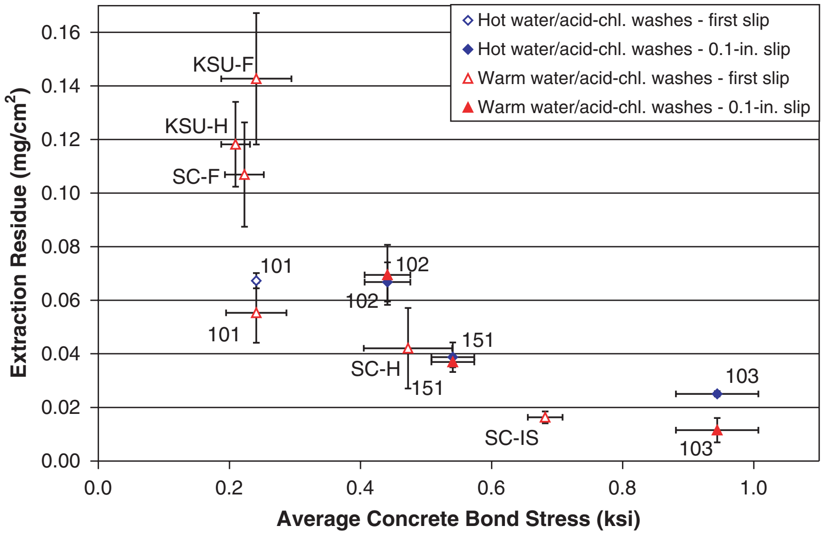


Figure B-84. Correlation between the "total" combined residues (water plus acid/chloroform washes) and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

bond stress, but that a low concentration of sodium does not guarantee a high concrete bond stress. The surface concentration of sodium from the acid/chloroform wash following the warm-water wash did not correlate well with bond. The results from a comparison of water-soluble sodium with mortar pull-out test results in the Correlation Round are similar to that compared to concrete pull out, as shown in Figure B-87.

Calcium—Figure B-88 shows the calcium surface concentration per unit area of strand from the acid/chloroform wash. Nearly all calcium removed from these strands was removed by the acid/chloroform wash. The initial water washes were ineffective at removing compounds containing this element, and the plot of the total calcium concentration mimics that of the acid/chloroform wash. In the Correlation Round, the total calcium surface concentration showed little correlation to bond in mortar (Figure B-89).

Potassium—Figure B-90 shows the relationship between the potassium surface concentration from the water wash and concrete pull-out bond stress, and Figure B-91 illustrates the

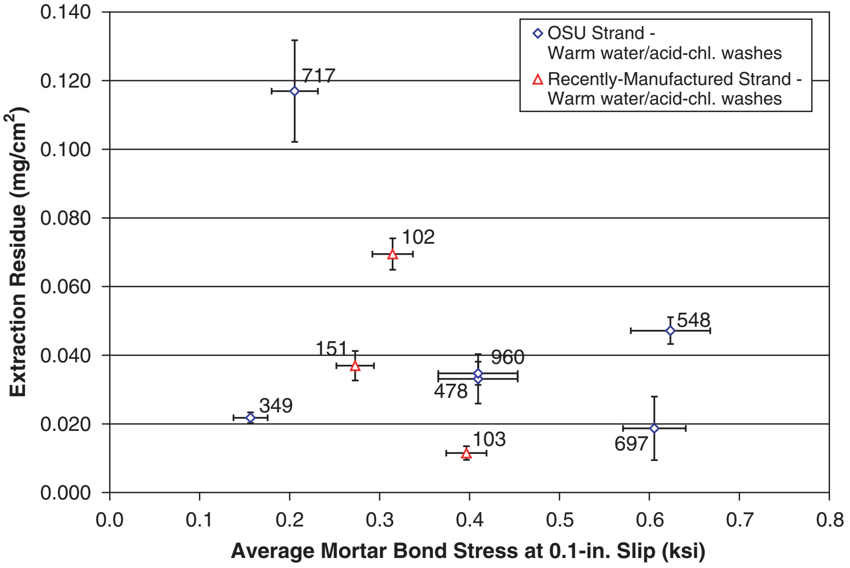


Figure B-85. Correlation between the "total" combined residues (water plus acid/chloroform washes) and bond stress at first or 0.1-in. slip in mortar (recently manufactured and OSU strand).

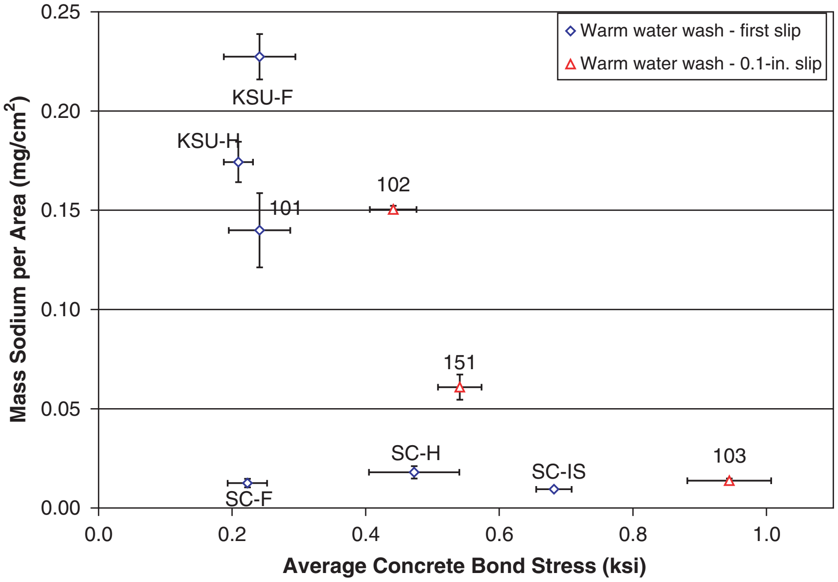


Figure B-86. Correlation between sodium concentration from warm-water wash and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

relationship between the potassium surface concentration from the acid/chloroform wash and the concrete bond stress. As can be seen, for most sources, the majority of the potassium was removed by the water wash procedure. However, for a number of sources, particularly SC-H and SC-IS, potassium compounds of apparently low water-solubility were present, and these compounds required the acid/chloroform wash procedure to remove them. Since the amount of potassium removed by the water wash was generally greater than by the acid/chloroform wash in the Screening Round, it appeared that a high level of water-soluble potassium corresponds to a low concrete bond stress, but at a low level of potassium, the bond stress may be high or low. This relationship was less clear in the Correlation Round, as shown in Figure B-92, which shows the potassium surface concentration from the water wash and mortar pull-out bond stress.

Zinc—Zinc was found in non-negligible concentrations only in the acid/chloroform wash. Figure B-93 shows the total zinc concentration versus the concrete bond stress at first

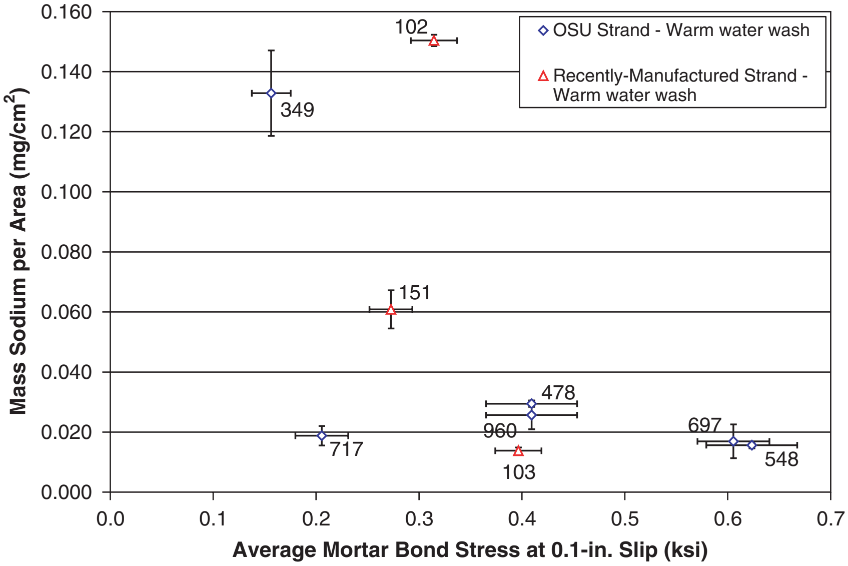


Figure B-87. Correlation between sodium concentration from warm-water wash and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

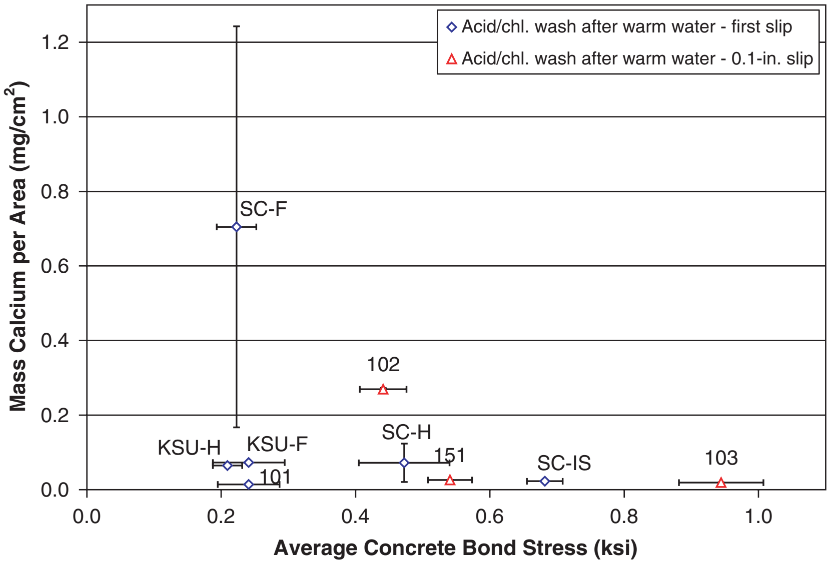


Figure B-88. Correlation between calcium concentration from acid/chloroform wash after warm-water wash and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

or 0.1-in. slip. A high concentration of extracted zinc was associated with both high and low bond stresses and did not correlate well with bond quality. This was confirmed in the Correlation Round (Figure B-94).

Boron—Unlike the zinc compounds, the boron compounds in the strand surface residue were largely water-soluble. Figure B-95 shows the total concentration of boron compared to the concrete pull-out bond stress. Low boron contents appear to correlate with higher average bond stress over the transfer length. Following the trend observed in much of the elemental analysis, a high boron level signifies a low bond stress, but a low boron level does not necessarily guarantee a high bond stress. This was confirmed in the Correlation Round (Figure B-96).

Phosphate—Not surprisingly, like the concentration of zinc with which it probably was applied to the strand in a pretreatment process, the phosphate concentration did not correlate well with bond (Figure B-97). High concentrations of

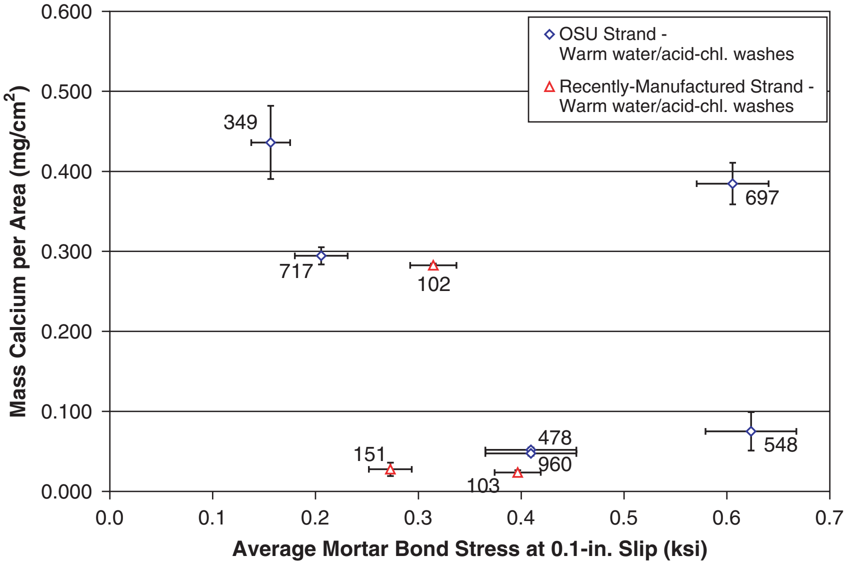


Figure B-89. Correlation between total calcium concentration from warm-water and acid/chloroform washes and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

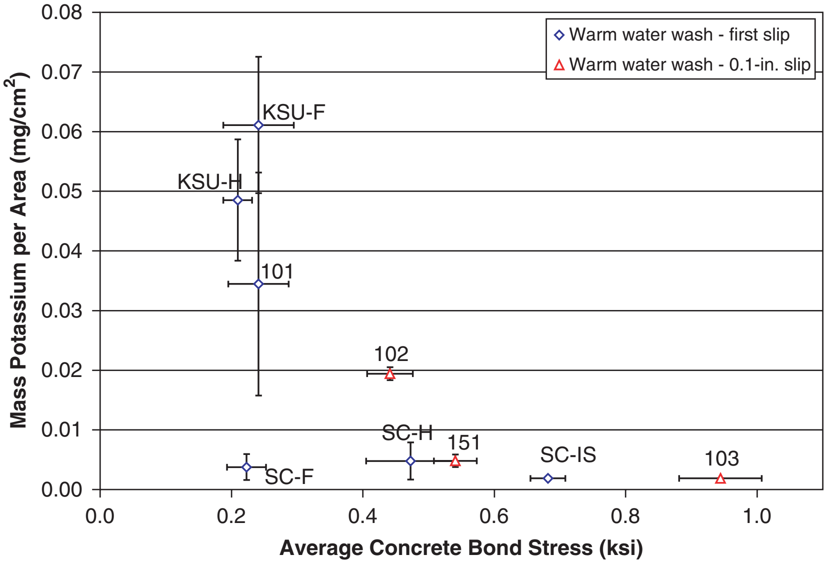


Figure B-90. Correlation between potassium concentration from the warm-water wash and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

extracted phosphate were associated with both high and low bond stresses.

Interpretation of Elemental Analyses Relative to Manufacturing Processes

The atomic absorption and colorimetric analyses done on wash solutions from strands from various sources measured varied levels of sodium, calcium, potassium, zinc, boron, and phosphate. The presence and concentration of these elements is largely governed by the specific pretreatment process and wire-drawing lubricants used in the manufacturing of each specific strand source, though other sources of these elements may be possible. The pretreatments commonly used in strand production are zinc phosphate and sodium borate, also known as borax. Lubricants typically consist primarily of either sodium stearate, calcium stearate, or some other fatty acid. Table B-25 and Table B-26 show each strand source ranked in

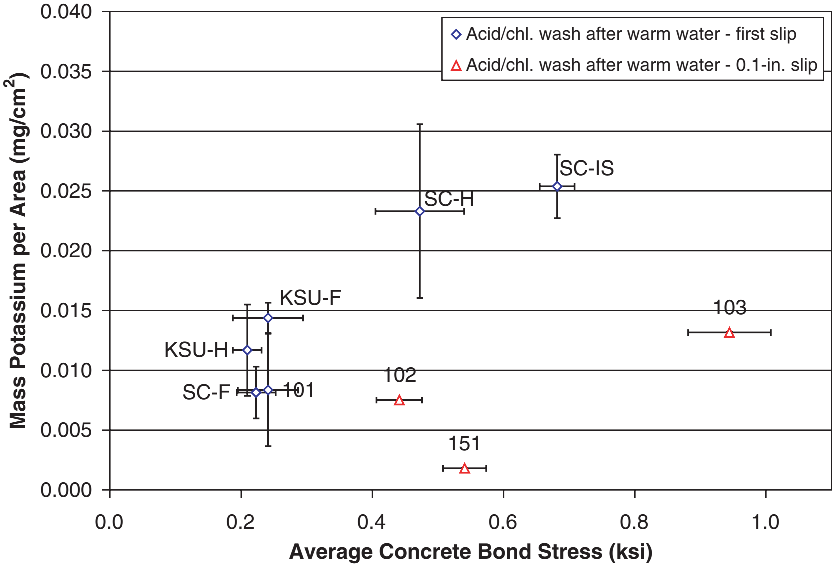


Figure B-91. Correlation between potassium concentration from acid/chloroform wash after warm-water wash and bond stress in concrete at first or 0.1-in. slip (historic and recently manufactured strand).

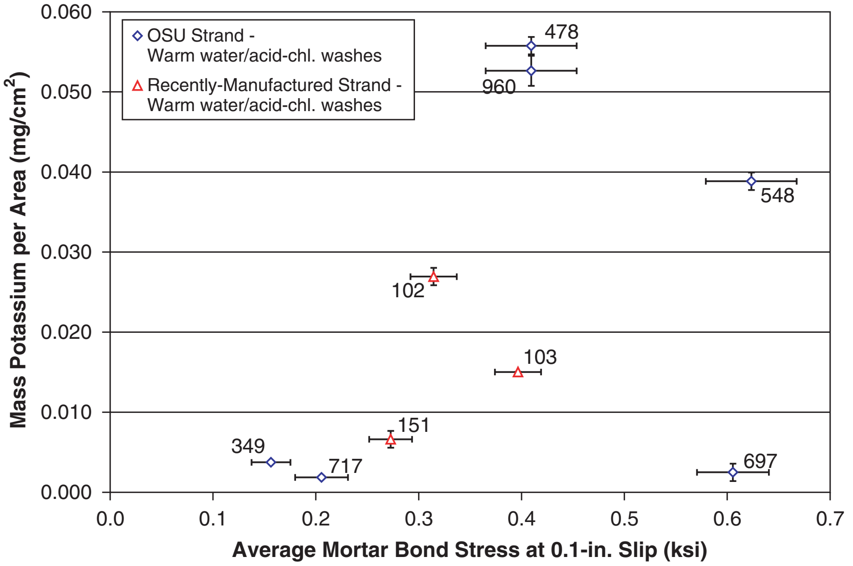


Figure B-92. Correlation between total potassium concentration from warm-water wash and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

ascending order of concrete or mortar pull-out bond performance together with the prominent elements removed during the extraction process, and the presumed pretreatment and lubricants used based on the prominent elements. Knowledge about the types of pretreatments and drawing lubricants can be an important part of the interpretation of the QC test results.

These results of the elemental analyses suggest that the wires in the sampled strands were pretreated using one of two methods during the manufacturing process. Those strands that carried high amounts of boron typically carried low amounts of zinc and phosphate. As expected, the lubricants appeared to be either calcium or sodium/potassium stearates but a number of strands showed evidence of both. This may result from different types of lubricants being used in separate dies in the drawing process.

There is a greater amount of sodium, potassium, and calcium than would be expected from the stearate compounds alone (based on the concentration of the organic residues extracted). Therefore, other sources of these elements are contributing to the values measured here. Possible sources include chemicals

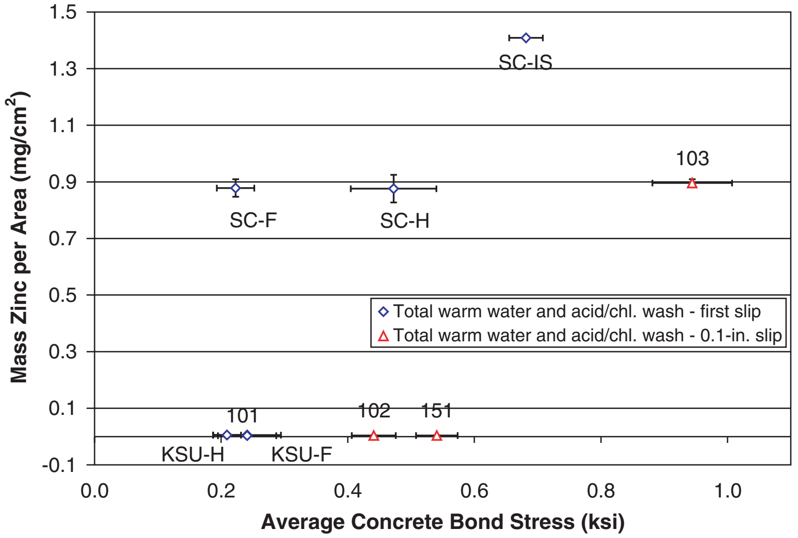


Figure B-93. Correlation between zinc concentration from combined warm-water and acid/chloroform washes and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

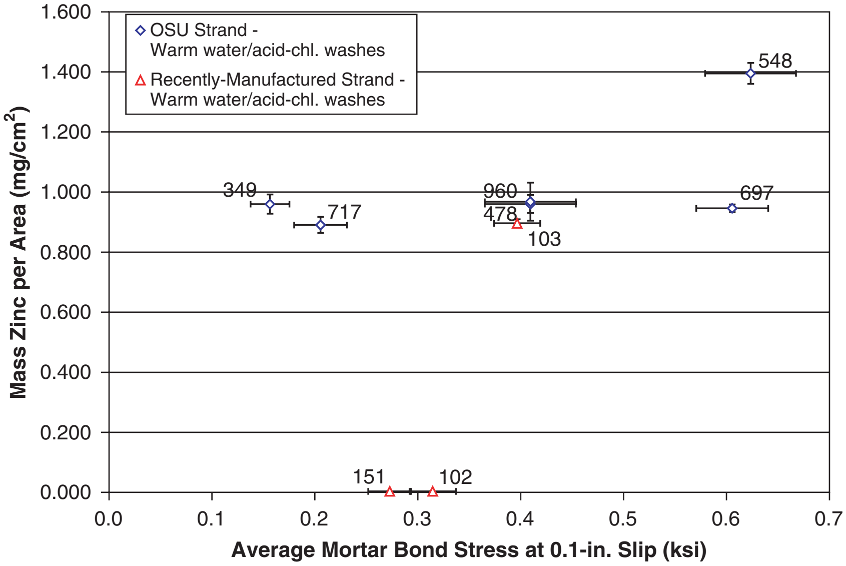


Figure B-94. Correlation between zinc concentration from combined warm-water and acid/chloroform washes and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

in the pretreatment processes described above; detergents used for cleaning; or fillers, such as lime, used in some drawing lubricants.

Combined Elemental Analysis

For any single element or organic residue quantified (as discussed above), good bond performance was observed only with strand sources on which low concentrations were found. However, poor bond performance was found with strands with both high and low concentrations of these single entities. This may be explained by suggesting that high concentrations of any of these element or organic residues has the potential to produce poor bond, and that while a poor bonding strand may have a low concentration of one single element, it is likely to be high in at least one other. To investigate this idea, analysis was done to compare bond performance associated with various combinations of these elements. The objective

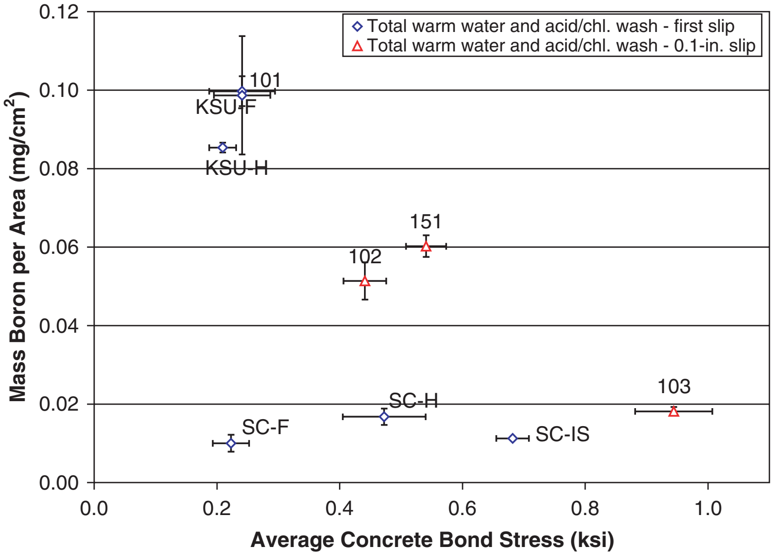


Figure B-95. Correlation between concentration from boron in combined warm-water and acid/chloroform washes and bond stress at first or 0.1-in. slip in concrete (historic and recently manufactured strand).

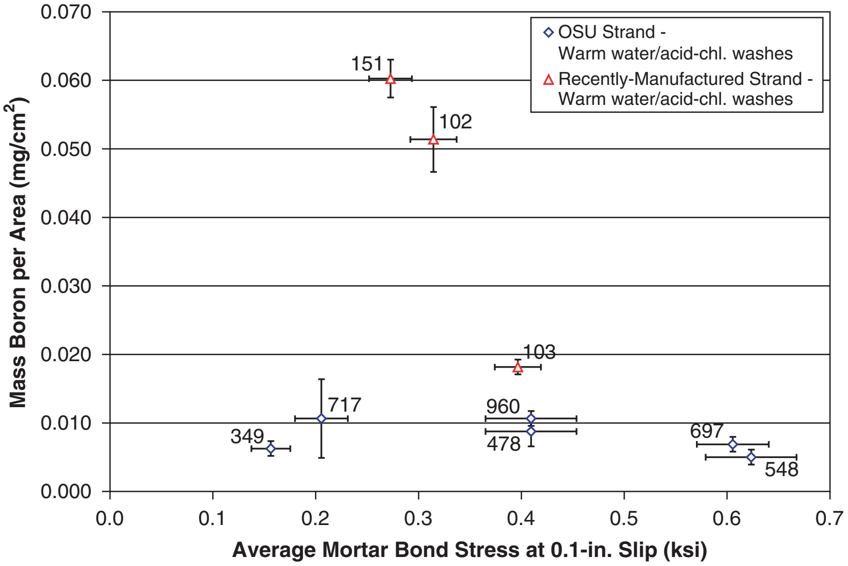


Figure B-96. Correlation between boron concentration from combined warm-water and acid/chloroform washes and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

of this combination approach was to determine which elements had the most significant effect on bond and to identify an indicator or score that might be used to predict poor bond. This score, which has been called the combined index, was defined so that it would increase if any of the included elements were found on the strand.

This combined index was defined as the average of the scaled residue concentrations for certain combinations of elements after the concentrations were scaled from 0 to 1, to account for differences in the magnitude of the measured concentrations. This was done for the total concentrations, that is, the sum of the materials removed using water and acid/chloroform washes in the Screening Round or the combined wash solutions in the Correlation Round, measured with warm water as the first step in the wash procedure. The scaling was performed by dividing the concentrations measured for each source by

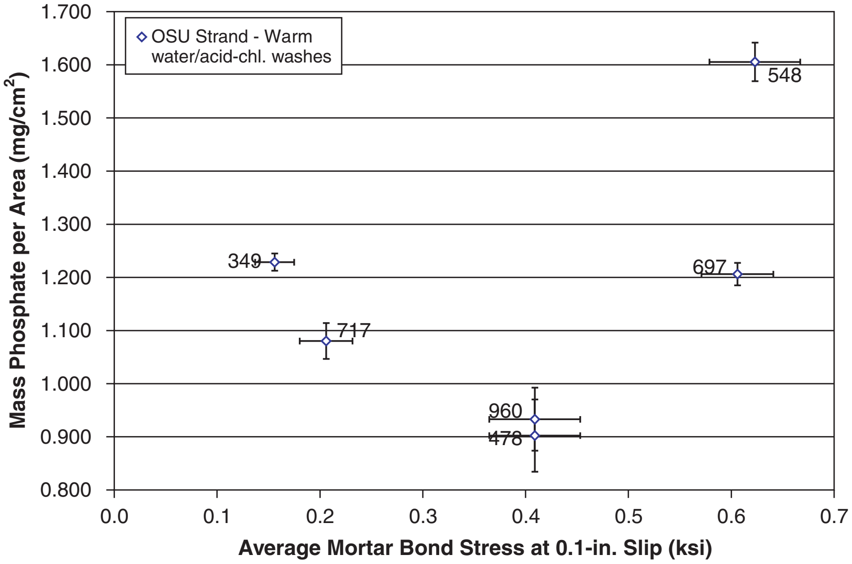


Figure B-97. Correlation between phosphate concentration from combined warm water and acid/chloroform washes and bond stress at 0.1-in. slip in mortar (recently manufactured and OSU strand).

Table B-25. Compounds likely used in manufacture of each source—historic and recently manufactured strands.

Strand	Prominent Elements	Presumed Pretreatment	Presumed Lubricants	Concrete Pull-Out Bond Stress (ksi)
153	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid	-
KSU-H	Na, K, B	borax	Na/K stearate	0.209
SC-F	Ca, Zn	zinc phosphate	calcium stearate	0.223
101	Na, K, B	borax	Na/K stearate	0.241
KSU-F	Na, K, B	borax	Na/K stearate	0.241
102	Na, Ca, B	borax	Na/Ca stearate	0.441
SC-H	Na, K, Zn	zinc phosphate	sodium stearate	0.472
151	Na, B	borax	calcium stearate	0.541
SC-IS	Na, Zn	zinc phosphate	sodium salt of fatty acid	0.682
103	Na, Zn	zinc phosphate	sodium salt of fatty acid	0.944

the maximum measured for that compound in each round. In this way, the maximum concentration was assigned a value of 1, and lower values were distributed between 0 and 1. The idea behind the averaging was that if any of the concentrations were high, it would be reflected in the index for that source.

Table B-27 shows the scaled concentrations for sodium, potassium, calcium, zinc, boron, and the organic residue for those sources for which concrete pull-out testing results were available. The relationship between bond and combined indices based on many element and organic residue combinations were explored, from individual pairs of elements to combinations of all the scaled concentrations. The combined index based on boron, calcium, and extracted organic residue was found to have the highest R^2 value ($R^2 = 0.78$) when plotted against concrete pull-out bond stress, as seen in Figure B-98. This figure illustrates a good correlation between poor bond stress and these three normalized values present together. The final column in Table B-27 is the combined index of boron, calcium, and organic residue used to generate this plot.

A similar analysis was performed for those samples for which mortar testing was available and Table B-28 show the scaled concentrations for sodium, potassium, calcium, zinc, boron, and the organic residue. Once again, the combined index based on boron, calcium, and organic residue was found to have the highest R^2 value (as shown in Figure B-99), but this time the correlation was much less ($R^2 = 0.28$).

Summary of Test Method Correlation

A large experimental program has been conducted to support the evaluation of various proposed test methods for strand bond that were intended for use as part of a QC program. These were classified as performance-based (i.e., mechanical) tests and surface and chemical tests. The correlation between bond and the methods that fall under each of these classifications were evaluated differently based on the strand sources that were collected for testing and the data quantifying bond performance that were available. The evaluation of correlations between the various pull-out testing methods and the bond were based on bond performance as measured with transfer length tests. The correlations between the surface and chemical test methods were evaluated based on the results of

Table B-26. Compounds likely used in manufacture of each source—OSU strands.

Strand	Prominent Elements	Presumed Pretreatment	Presumed Lubricants	Mortar Pull-Out Bond Stress (ksi)
349	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid and resin	0.156
717	Na, Ca, Zn, P	zinc phosphate	Na/Ca stearate	0.206
478	Na, K, Zn, P	zinc phosphate	Na/K stearate	0.409
960	Na, K, Zn, P	zinc phosphate	Na/K stearate	0.409
697	Ca, Zn, P	zinc phosphate	calcium salt of fatty acid and resin	0.606
548	Na, K, Zn, P	zinc phosphate	Na/K salt of fatty acid	0.623

Table B-27. Scaled concentrations and combined index for comparison with concrete pull out.

Source	Zinc	Potassium	Sodium	Calcium	Boron	Organic Residue	Combined Index for B, Ca, and Organic Residue
Historic Strand							
KSU-F	0.004	1.000	1.000	0.100	1.000	1.000	0.700
KSU-H	0.004	0.798	0.790	0.092	0.856	0.829	0.592
SC-F	0.623	0.158	0.356	1.000	0.101	0.749	0.617
SC-H	0.620	0.372	0.568	0.097	0.168	0.295	0.187
SC-IS	1.000	0.361	0.757	0.035	0.113	0.114	0.087
101	0.002	0.567	0.651	0.020	0.989	0.388	0.466
Recently Manufactured Strand							
102	0.003	0.357	0.738	0.362	0.515	0.487	0.455
103	0.636	0.199	0.430	0.030	0.182	0.081	0.098
151	0.003	0.088	0.308	0.035	0.604	0.259	0.300

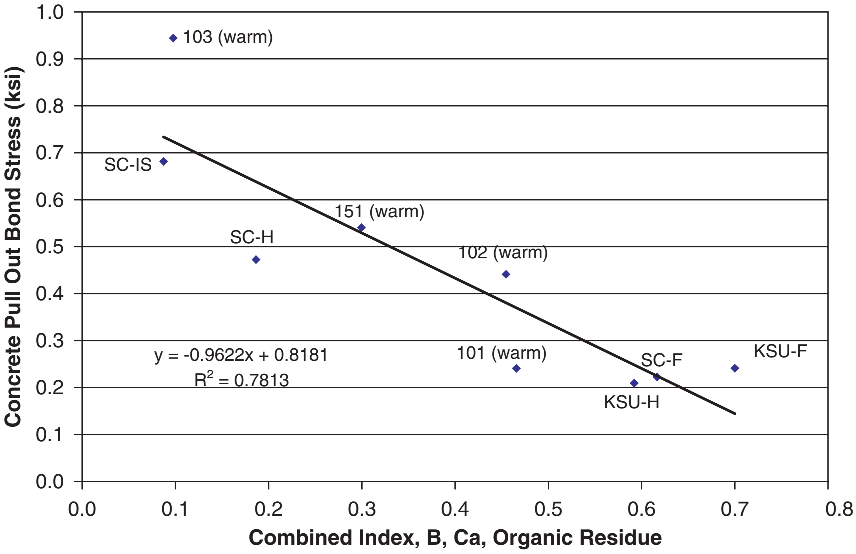


Figure B-98. Comparison between concrete pull-out bond stress at first or 0.1-in. slip and the combined index of boron, calcium, and organic residue.

Table B-28. Scaled concentrations and combined index for comparison with mortar pull out.

Source	Zinc	Potassium	Sodium	Calcium	Boron	Organic Residue	Combined Index for B, Ca, and Organic Residue
Recently Manufactured Strand							
102	0.003	0.483	1.000	0.648	0.853	0.594	0.698
103	0.642	0.270	0.583	0.055	0.302	0.098	0.152
151	0.003	0.119	0.417	0.064	1.000	0.316	0.460
OSU Strand							
349	0.688	0.067	0.251	1.000	0.104	0.186	0.430
548	1.000	0.697	0.769	0.172	0.083	0.403	0.220
697	0.678	0.045	0.248	0.882	0.114	0.160	0.386
717	0.638	0.034	0.577	0.675	0.177	1.000	0.617
478	0.688	1.000	0.704	0.119	0.146	0.283	0.183
960	0.694	0.944	0.678	0.109	0.177	0.297	0.194

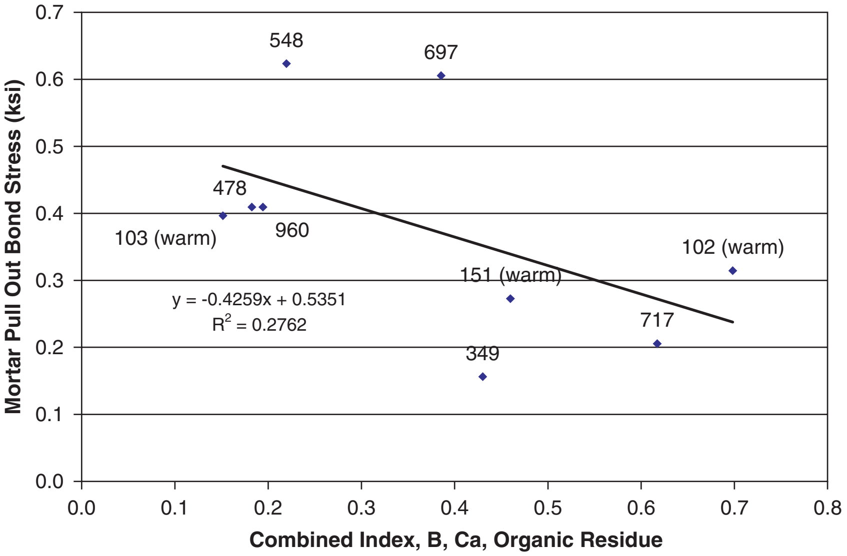


Figure B-99. Comparison between mortar pull-out bond stress at 0.1-in. slip and the combined index of boron, calcium, and organic residue.

pull-out tests from concrete in the Screening Round and based on pull-out tests from mortar in the Correlation Round. The correlations with bond are discussed below for each classification.

To provide a quantitative measure of the goodness-of-fit to aid in the evaluation of these methods, a linear regression has been performed, and the coefficient of determination (R^2) determined for the relationship between each proposed test method and the bond quality measure. No physical basis for a linear relationship between these measures of bond is known; however, the linear relationship was assumed as the simplest model relating the parameters. The coefficient of determination (R^2) is a measure of the adequacy of a regression model; that is, it describes the amount of variability in the data explained by the regression model. The closer the R^2 is to 1.0, the more completely the model describes the relationship between the test method results and the basis for evaluation. These coefficients of determination are presented in Table B-29 and Table B-30.

To further evaluate the validity of these methods, the significance of the linear models developed based on these data was evaluated by the calculation of P-values for the coefficients (slope) from the linear models. The coefficient from the linear model is judged to be significant when there is a sufficiently high confidence that it is not equal to zero. If this is true, the relationship represented by the model is statistically significant and the results of the surface tests are meaningful in the prediction of the pull-out test. A 95% confidence level is commonly used to evaluate significance. The level of confidence of significance on the coefficient is given by $(1 - \text{P-value}) \times 100\%$, so a $\text{P-value} < 0.05$ implies that the confidence interval does not include zero with higher than 95% confidence. The P-value was determined for the relationship between selected test methods and the bond quality measures and these are presented in Table B-31. This concept of P-value is revisited in Regression and Prediction Intervals in the next section, Development of Thresholds.

Table B-29. Coefficient of determination (R^2) from linear regression with average bond stress over transfer length.

Test Method	QC Level	Coefficient of Determination (R^2) from Regression with Average Bond Stress over Transfer Length
Concrete Pull Out	II	0.98
Mortar Pull Out	II	0.85
Hydrocal Mortar Pull Out	II	0.36

Correlation with Bond—Mechanical Test Methods

The concrete pull-out test results correlated better with bond quality than the other pull-out test methods that were evaluated, based on comparisons with transfer length tests conducted on three strand sources. However, pull-out testing from mortar also showed promise as a means to evaluate bond, and the existing correlation is deemed sufficient to justify further study. This limited program cannot be considered a definitive evaluation of these methods. Nevertheless, this conclusion is contrary to that of other studies of pull-out test methods, including that sponsored by NASPA which have concluded that the mortar pull-out test is superior at assessing bond performance (Russell and Paulsgrove 1999, Russell 2001, Russell

Table B-30. Coefficient of determination (R^2) from linear regression with concrete and mortar pull out at 0.1-in. and first slip.

Test Method		QC Level	Coefficient of Determination (R^2) from Regression with Mechanical Test	
			Concrete Pull Out (0.1-in. and First Slip)	**Mortar Pull Out (0.1-in. Slip)**
Contact Angle (°)	As-Received	I	0.04	0.35
	After $Ca(OH)_2$ Dip	I	**0.61**	**0.57**
	After $Ca(OH)_2$ Dip—Stearate Only†	I	**0.44**	**0.84**
	After Ignition	I	N.A.	0.00
pH		I	0.97	0.18
Loss on Ignition		I	**0.86**	**0.16**
Loss on Alkali Bath		I	0.17	0.76*
Change in Corrosion Potential after 6 h	As-Received	I	**0.72**	**0.68**
	After $Ca(OH)_2$ Dip	I	0.80	1.00*
	After Ignition	I	N.A.	0.00
Surface Roughness, R_a		I	0.93	0.16
Corrosion Rate	As-Received	II	0.67	0.09
	After $Ca(OH)_2$ Dip	II	1.00	0.00
	After Ignition	II	N.A.	0.18
Organic Residue Extraction	Total	II	**0.81**	**0.12**
	Total—Stearate only†	II	**0.88**	**0.63**
Sodium	Warm Water	II	0.34	0.31
	Total	II	0.12	0.02
Potassium	Warm Water	II	0.39	0.00
	Total	II	0.28	0.14
Calcium	Warm Water	II	0.17	0.06
	Total	II	0.22	0.07
Zinc	Warm Water	II	0.21	0.05
	Total	II	0.25	0.21
Boron	Warm Water	II	0.30	0.10
	Total	II	0.28	0.11
Phosphate	Total	II	N.A.	0.17
Combined Index for B, Ca, & Org. Res.	Scaled for combination	II	0.78	0.28

R^2 values presented in bold are for those methods recommended for use in a QC program.
* Test method not included in Correlation Round—regression based on three sources.
† Only those sources identified as containing primarily stearate-based compounds by FTIR analysis are considered.

Table B-31. P-value from linear regression with concrete and mortar pull out at 0.1-in. and first slip.

Test Method		QC Level	P-value from Regression with Mechanical Test	
			Concrete Pull Out (0.1-in. and First Slip)	**Mortar Pull Out (0.1-in. Slip)**
Contact Angle (°)	After $Ca(OH)_2$ Dip	I	**0.039**	**0.019**
	After $Ca(OH)_2$ Dip - Stearate Only†	I	**0.262**	**0.029**
Loss on Ignition		I	**0.003**	**0.285**
Change in Corrosion Potential after 6 h	As-Received	I	**0.356**	**0.006**
Organic Residue Extraction	Total	II	**0.002**	**0.353**
	Total—Stearate only†	II	**0.006**	**0.110**

† Only those sources identified as containing primarily stearate-based compounds by FTIR analysis are considered.

2006). This correlation was not explored further in the Correlation Round of testing.

Correlation with Bond—Surface and Chemical Test Methods

At the initiation of this study, the surface and chemical methods were divided into Level I and II QC tests, based on the required effort and complexity of each test. These correlations are discussed separately here, since the level of correlation required to justify the use of each test method is different for each QC level. As can be seen in Table B-30, a number of the surface and chemical test methods that showed good correlation with concrete pull-out test results did not correlate as well with the mortar pull-out test results. This may be indicative of the inadequacy of the surface and chemical methods, but may also be related to inaccuracies in the pull-out test methods.

For the contact angle and organic residue extraction measurement methods, coefficients of determination have been calculated using only data from sources identified in the FTIR analyses as carrying only stearate-based lubricants. This was done to eliminate the potentially confounding influences of non-stearate-based lubricants. Analyzing these data in this manner has a practical motivation, since such models could be useful in a production setting where the lubricant in use is known to be only stearate-based.

Finally, the significance of the relationships between the pull-out test results and the surface and chemical method results given in terms of the P-values for the coefficient are considered for those methods that showed promise based on the coefficients of determination or other factors.

Level I QC tests—The objective of the Level I QC test methods is to quickly and easily determine if strand properties that have been correlated with questionable bond are present. The minimum correlation required for these tests to be useful is somewhat lower than for the Level II QC tests.

- Contact Angle—Contact angle correlated with bond only after the strand sample was subjected to exposure to a saturated calcium hydroxide solution. This correlation is higher for those sources judged to carry only stearate-based lubricants, when performance based on mortar pull-out testing is considered. Both P-values calculated when comparing this test against mortar and concrete pull-out testing are less than 0.05, suggesting that there is greater than 95% confidence that the relationships between the pull-out tests and this test method are significant. It is recommended that this method be included as part of a future QC program.
- Examination under UV light—A limited quantity of fluorescing material was observed, and no correlation to bond was found. This method should be abandoned.
- Testing pH—The pH test was successful in finding a correlation with bond as measured by concrete-based pull-out testing on a limited data set, but it was unsuccessful at finding a similar correlation based on mortar pull-out test results. It also appeared that this test was only effective to differentiate strands produced with a borax pretreatment. Therefore, this method may be applicable only for strand produced with borax pretreatments. More study is needed before a recommendation regarding the adaptation of this method can be made.
- Loss on Ignition—A good correlation was found between the ignition loss and bond performance measured in concrete pull-out tests. The P-value calculated when comparing this test against concrete pull-out testing is less than 0.01, suggesting that there is greater than 99% confidence that this test method is significant at predicting concrete pull out. This correlation or significance was not found based on mortar pull-out test results. This is one of the easiest tests to perform and is recommended for future testing, though not as a sole measure of bond performance.
- Loss in Alkali Bath—Multiple cleaning procedures using sodium hydroxide solutions were attempted, but no correlation was observed between the weight loss and bond in concrete. While a higher correlation was found with the mortar test, this higher correlation is based only on three sources. Interestingly, this is the only method suggested by the Wire Association International manual for measuring surface residues on wire. It is recommended that this test be abandoned.
- Change in Corrosion Potential—Although the mechanism behind the observed trend is somewhat uncertain, the drop in corrosion potential showed a good correlation with bond in both the Screening and Correlation Rounds of evaluation. The P-value calculated when comparing this test against mortar pull-out testing is less than 0.01. When compared to concrete, the coefficient of determination is similarly high, but the P-value is also high (0.356), suggesting that this relationship is not significant based on a 95% confidence threshold. This high P-value is likely influenced by the fact that this analysis is based on only three samples. A nearly perfect correlation ($R^2 = 1.00$) with mortar pull out was measured in the Screening Round for this test when performed on samples after exposure to a saturated calcium hydroxide solution. However, this higher correlation is only based on three sources, and it was judged that this additional effort is not worthwhile. Therefore, it is recommended that this method, conducted on strands in as-received condition, be included as part of a future QC program.

- Surface Roughness—The surface roughness parameter, R_a, correlated well with bond in concrete based on only three sources, but not with bond in mortar. Since an increased roughness was associated with poor bond, it appears that correlation to bond is not a direct effect, but is related to the tendency of the wire surfaces to retain residue. The profilometer used to measure this property is convenient for use in a QC setting, but does not appear to be sensitive enough to measure the roughness at the scale needed, nor does it test a sufficiently large surface of the strand for the test result to be representative of a property that can be tied to bond performance. Therefore, it is recommended this method be abandoned in its current form. However, we believe that this test method could be viable if performed using a different methodology. Unfortunately, project funds did not permit further development of this test method.

Level II QC tests—The objective of Level II QC testing is to provide a more conclusive prediction of bond performance than possible with the Level I QC tests. These tests require either more advanced methods or more complicated equipment. The minimum correlation required for these tests is higher than for the Level I QC tests.

- Corrosion Rate—A strong correlation was measured between corrosion rate and pull-out bond in concrete. However, the correlation between corrosion rate and pull-out bond in mortar was relatively weak. Given this lack of consistent correlation, the uncertainty about the mechanisms involved in establishing the initial good correlation, and the complexity and equipment-dependent nature of this test, it is not recommended for inclusion in a future QC program.
- Organic Residue Extraction—The concentration of the organic residue correlated well with the bond performance in concrete, but only moderately with bond in mortar. This test is time-consuming to perform, but gives the best direct measure of the type and quantity of drawing lubricants left on the strand surface during the manufacturing process. Of all the methods proposed, this method evaluates the property of the strand tied most directly to bond quality. Therefore, it is recommended that this method be included as part of a future QC program. FTIR spectroscopy should continue to be performed on the organic residues to ensure that residues being evaluated are consistent. This is necessary because the effect of residues with different chemistries is unlikely to be proportionally similar (e.g., a stearate-based lubricant residue will likely effect bond differently than a non-stearate-based lubricant residue). FTIR analyses will also identify contamination of the samples from other organic materials, such as oils, greases, or form release agents. The correlation between mortar pull-out stress and residue concentration was much higher when those sources carrying only stearate-based lubricants were included in the correlation analysis. Nevertheless, the P-values calculated when comparing this test against mortar pull-out testing considering all samples was less than 0.01, providing further support to the recommendation to use this test method.
- Elemental Analysis—Atomic absorption and visual light spectroscopy were used to determine the surface concentration of various elements. The concentrations of sodium and boron showed signs of a correlation with the mechanical properties measured in pull-out tests in both concrete and mortar. The concentrations of zinc, however, did not. The elemental analysis gives some insight into the type of pretreatment and lubricant in use and was useful for the purposes of this study. The combined index based on boron, calcium, and organic residue, combines the elemental analyses with the organic residue extraction procedures, and showed good correlation to pull-out bond in concrete. This correlation was not found with mortar pull-out results. Given the cost and equipment-dependent nature of the atomic absorption testing, it is not recommended for inclusion in a future QC program.

In summary, the methods that are recommended for inclusion in future QC programs are as follows:

- Weight Loss on Ignition (LOI) of Steel Strand,
- Determination of the Surface Tension of Steel Strand by Contact Angle Measurement,
- Change in Corrosion Potential of Steel Strand, and
- Organic Residue Extraction with FTIR Analysis.

Precision Testing

The recommended QC methods have been written in AASHTO/ASTM standard method format in Appendix C, where they are titled:

1. Test Method for the Determination of the Surface Tension of Steel Strand by Contact Angle Measurement,
2. Test Method for Weight Loss on Ignition (LOI) of Steel Strand,
3. Test Method for Change in Corrosion Potential of Steel Strand, and
4. Test Method for Identification and Quantification of Residue on Steel Strand by Extraction, Gravimetric, and Spectroscopical Analyses.

Testing was conducted to provide the basis for a precision statement accompanying the proposed test methods developed for identifying strand bond performance.

Table B-32. Strand source for precision testing.

Test Method	Strand Source
Organic Residue Extraction	102
Contact Angle	102
Change in Corrosion Potential	103
Weight Loss on Ignition	717

To determine the precision (i.e., the repeatability) of the methods, the selected tests were repeated up to six times on samples of strand obtained from the same source. This testing was conducted on a single source identified as a middle range performer in that particular test during the correlation testing. The results of this testing are presented below according to ASTM practice. A determination of bias in the testing methods is not possible at this time since a known reference sample cannot be selected in a universally acceptable manner.

The sources of strand that were used for this testing are given in Table B-32. These sources were chosen because they produced middle-of-the-range results obtained during the original testing programs. The results for the four test methods are given in Table B-33 through Table B-36. The precision and bias statements to be added to the standard test methods are in the following discussion.

Test Method for Weight Loss on Ignition (LOI) of Strand

Single-operator precision—The single-operator standard deviation was found to be 0.014 mg/cm^2*. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.041 mg/cm^2*. (Numbers followed by an asterisk in this and the following sections represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670, Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials* [ASTM 2003].)

Table B-33. Precision test results for organic residue extraction.

Sample Set (three pcs. of strand)	Average Concentration (mg/cm^2)
1	0.057
2	0.061
3	0.072
4	0.055
5	0.088
6	0.078
Average for 6 repeats	**0.069**
Standard Deviation for 6 repeats	**0.013**

Table B-34. Precision test results for weight loss on ignition.

Sample Set (three pcs. of strand)	Average Weight LOI (mg/cm^2)
1	0.086
2	0.079
3	0.102
4	0.086
5	0.079
6	0.115
Average for 6 repeats	**0.091**
Standard Deviation for 6 Repeats	**0.014**

Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Test Method for Contact Angle Measurement of a Water Droplet on a Strand Surface

Single-operator precision—The single-operator standard deviation was found to be 4°F*. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 10°F*.

Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Test Method for Change in Corrosion Potential of Strand

Single-operator precision—The single-operator standard deviation was found to be 0.047 V*. Therefore, results of two

Table B-35. Precision test results for contact angle.

Sample Set (three pcs. of strand)	Average Contact Angle after Lime Dip (°)
1	68
2	75
3	71
4	71
5	77
6	73
Average for 6 repeats	**73**
Standard Deviation for 6 Repeats	**4**

Table B-36. Precision test results for change in corrosion potential.

Sample Set (three pcs. of strand)	Change in Corrosion Potential (V) After 6 h
1	-0.402
2	-0.358
3	-0.317
4	-0.280
5	-0.313
Average for 5 repeats	**-0.334**
Standard Deviation for 5 Repeats	**0.047**

properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.133 V*.

Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Test Method for Identification and Quantification of Strand Surface Residue by Extraction, Gravimetric, and Spectroscopical Analyses

Single-operator precision—The single-operator standard deviation was found to be 0.013 mg/cm^2*. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.037 mg/cm^2*.

Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

In Table B-37, the results of the precision testing program are compared to those obtained during the screening and correlation testing program. Two tests, namely the *Test Method for Change in Corrosion Potential of Strand* and the *Test Method for Contact Angle Measurement of a Water Droplet on a Strand Surface*, were conducted on the same sources of strand that were tested as part of the Screening Round of evaluation in 2004. The results of the Precision testing program obtained in 2007 gave average results that differed from the earlier result by an amount greater than the acceptable single-operator ranges listed in the precision statements above. This is explained by the fact that these methods are heavily dependent on the physical condition of the strand surface, and the surface of these strand samples had undergone some corrosion. The results of the other two test methods are less sensitive to such corrosion and gave results that were more consistent with previous testing efforts.

Development of Thresholds

For the recommended surface and chemical test methods to be useful in a QC setting, thresholds for acceptable bond behavior are needed. The usefulness of acceptance/rejection thresholds for the surface and chemical test results is dependent on the precise correlation of these results with minimum acceptable bond strengths established by physical test methods. The validity of these thresholds is also dependent on the validity of the physical test methods (such as pull-out tests) used to measure bond performance. At the direction of the NCHRP supervisory panel, the transfer length testing originally planned for this test program as a basis for developing thresholds for the surface and chemical test results was not conducted. Instead, the basis available for developing thresholds for the chemical and surface test methods are the acceptance limits proposed by Russell and adopted by NASPA.

The bond strength thresholds proposed by Russell are stated in terms of the force at 0.1-in. slip measured by the NASPA mortar pull-out test procedure. They are based on a set of

Table B-37. Results of precision testing compared to correlation testing.

Test Method	Strand Source	Screening or Correlation Testing Result	Precision Testing Result*	Reported Single Operator Precision (d2s)
Organic Residue Extraction (mg/cm^2)	102	0.069	0.069	0.037
Contact Angle (°)	102	87	73	10
Change in Corrosion Potential (V)	103	-0.167	-0.334	0.133
Weight Loss on Ignition (mg/cm^2)	717	0.086	0.091	0.014

*Average of all Precision testing results.

development length tests conducted in parallel with the development of the NASPA Strand Bond Test (Russell 2001, Russell 2006). The thresholds were derived using development length tests on four strand sources (referred to as the NASPA Round III study [Russell 2001]), and they are defined in terms of acceptance criteria for the average force at 0.1-in. slip from six pull outs with a lower criterion for any single measurement of the six pull outs. The Round III report proposed thresholds of 7300 and 5500 lbs, for the minimum permissible average and single test result, respectively for 1/2-in. diameter strand (Russell 2001). These minimum thresholds have since been increased to 10500 and 9000 lbs, but without additional testing (Russell 2006). For 0.6-in. diameter strand, the suggested thresholds are 12600 and 10800 lbs for the minimum permissible average and single test result, respectively (Russell 2006). No threshold has been suggested for other sizes of strand.

Despite the somewhat limited scope of the development process used to establish these NASPA test thresholds, the threshold determination effort for the surface and chemical testing conducted in this study was performed assuming that these thresholds were well-defined lower bounds. As has been done throughout this study, the thresholds were converted to bond stresses calculated as the force divided by the nominal surface area (the nominal perimeter of the strand multiplied by the embedment length) to support comparisons among all of the tested strands. When converted to a bond stress, the minimum threshold on the average of six tests of 10,500 lbs is equal to 0.313 ksi. This value was used as the basis for the threshold analysis.

Note that since rigorous mechanical testing was not performed on many of the strand sources used to develop the correlations between performance and these chemical and surface tests, the scientific basis for such thresholds is less than would be desirable. As a result, conservative thresholds will be proposed.

Regression and Prediction Intervals

While some analysis has already been made using linear regression methods, a background on regression techniques is presented here as a basis for the discussion of prediction intervals, which are less commonly used, but which are needed for the development of thresholds. In this analysis, the pull-out performance is treated as the dependant variable and is plotted on the y-axis, while the variables (i.e., test method results) on which the predictions are based are plotted on the x-axis.

Regression with a Single Predictor

Regression is a standard statistical technique for modeling the relationship between two or more variables. In the case of the mortar pull-out stress of the strand, the goal is to predict the mortar pull-out stress as a function of the proposed QC tests or chemical analyses. Since these tests can be done more easily than the actual pull-out test, it would be helpful to predict the pull-out stress by running a few simple tests and then using a prediction function to estimate the pull-out stress for that strand.

The model for the response (the pull-out stress in this case) consists of two major parts: the prediction formula and the error that captures the variability in the response (the pull-out stress in this case). The response is usually denoted by y and the predictor variable is usually denoted by x. The most common prediction formula is a simple linear model with an unknown slope, β_1, and intercept, β_0. The model for the response is then:

$$y = \beta_0 + \beta_1 x + \varepsilon \qquad \text{(Eq. 3)}$$

where ε is the error term, which is typically assumed to be a normal distributed random variable with mean zero and an unknown variance, σ^2. The idea is that the average response (e.g., pull-out stress) is linearly related to the value of the predictor variable (e.g., the change in corrosion potential as-received); however, due to random variation (for example, variation in the testing and measurement system), the actual pull-out stress for any given strand sample may be somewhat higher or lower than the predicted average pull-out stress based on that predictor (e.g., corrosion potential).

Figure B-100 shows the example of mortar pull-out stress versus the change in corrosion potential as-received. In the plot, the linear trend is apparent, but individual strand samples often deviate substantially from the line due to random error. The standard deviation of the observations from the line is estimated by the value S shown in the box in the upper right-hand corner of the plot. S^2 is the estimator of the variance of ε, the normally distributed random variable used to model the random error. Although the true values of β_0 and β_1 are unknown, the regression provides the estimates that produce the smallest possible value of S, in this case these estimates are $\hat{\beta}_0 = 0.7659$ and $\hat{\beta}_1 = 1.743$, where the "hat" notation indicates that these are estimators of β_0 and β_1. The other important measure provided in the plot, R^2, is a measure of how much of the variability of the data is explained by the linear model. This value ranges between 0 and 1, where zero means that the slope is flat and thus no linear relationship exists between x and y. An R^2 of 100% indicates that the data all fall exactly on the single line generated by the regression. In this case, the R^2 value is 68.2%, which indicates that there is clearly some relationship between slip stress and corrosion; however, there is also a substantial amount of error.

Figure B-101 shows the standard regression output from Excel's Data Analysis Tool Pack. The Regression Statistics

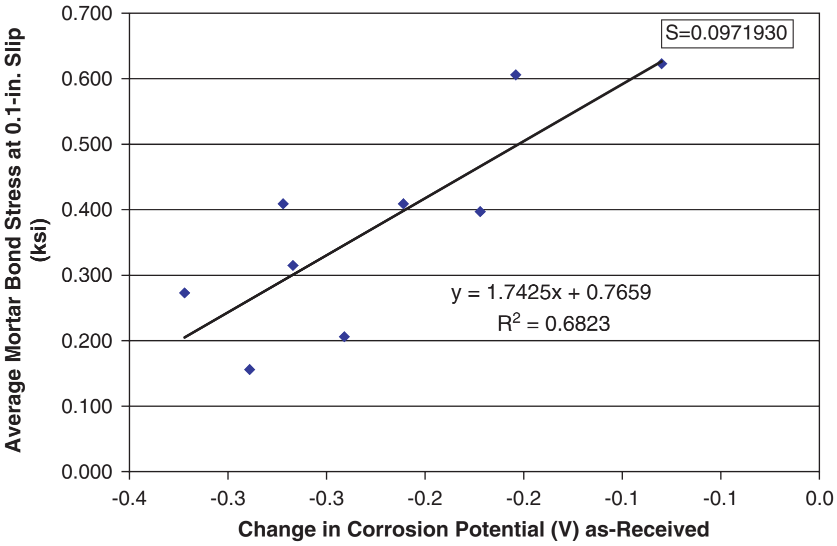


Figure B-100. Fitted line plot for mortar pull-out stress versus the change in corrosion potential as-received.

Table provides Standard Error (S), R^2, and R^2 *adjusted*. The multiple R is just the square root of R^2 and is sometimes called the correlation.

The ANOVA (Analysis of Variance) analysis performed by Excel includes a statistical test to determine if the amount of variability explained by the fitted model is significantly more than would be expected from a model fitted to random data from a normal distribution. All the entries in the ANOVA table are intermediate values for calculating the "Significance F" value. The "Significance F" value shows how likely it would be that random, normally distributed data would fit a linear model as well as this model fits these data. Here, the probability is very low (0.0061) indicating that it is very unlikely that these data are just random data and much more likely that the data are actually following the linear model.

Finally, the bottom table provides the estimates for the coefficients ($\hat{\beta}_0 = 0.7659$ and $\hat{\beta}_1 = 1.7432$). The P-values in the bottom table are the result of statistical tests that test to see if the true intercept, β_0, and the true slope, β_1, are equal to zero. The low P-values indicate that neither the slope nor the intercept is likely to be zero. For the intercept, this test is not very interesting, but if the slope is zero, that would indicate no linear relationship between the pull-out stress and the change in corrosion potential as-received. Notice that the P-value for the slope (in the row labeled "Change in Corr. Pot. As Received") is exactly the same as the "Significance F" value above. For the case of a single predictor variable, these two tests are exactly equivalent. In the next section where multiple predictors are discussed, these tests will no longer be equivalent.

SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.826022976
R Square	0.682313957
Adjusted R Square	0.636930237
Standard Error	0.097193005
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	1	0.142022	0.142022	15.03433	0.00607324
Residual	7	0.066125	0.009446		
Total	8	0.208147			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	0.765888672	0.105371	7.268469	0.000167	0.516724919	1.015052
Change in Corr. Pot. As Received	1.74252891	0.449405	3.877413	0.006073	0.679854919	2.805203

Figure B-101. Excel output for regression with single predictor.

The last two entries in each row of the bottom table are 95% confidence intervals for the intercept and slope. These are ranges that 95% of the time will cover the true values β_0 and β_1. The most useful piece of information here is similar to the conclusions made earlier and that is that with 95% confidence β_1, the slope is greater than zero—again indicating a linear relationship between pull-out stress and change in corrosion potential as-received.

The main reason for developing this regression is to allow for prediction of the pull-out stress by measuring the change in corrosion potential. However, entering the measured change in corrosion potential into the prediction formula $\hat{y}=\hat{\beta}_0+\hat{\beta}_1 x$ gives the average estimated pull-out stress and does not account for variation that is bound to occur in the test results or uncertainty in the regression model. This variation is evidenced by the fact that all the points used to develop the regression did not fall on the line, that is the R^2 value was not 100%. Instead, what is needed to interpret and practically apply a change in corrosion potential test result is the computation of a lower bound on the interval that, with 90% confidence, includes the pull-out stress for a strand sample with that change in potential test result. This type of interval is known as a one-sided prediction interval and is a standard part of regression theory and practice. A two-sided prediction interval is used when both an upper and a lower bound are required. The one-sided prediction interval will be focused on here.

The prediction interval concept is a necessary part of the development of acceptance/rejection thresholds for the recommended QC tests, since, to conservatively verify that a specified pull-out bond stress is likely to be achieved, the threshold on the QC test must be chosen as the value where the prediction interval lower bound is equal to the pull-out stress threshold. The model $\hat{y}=\hat{\beta}_0+\hat{\beta}_1 x$ gives an estimate of the average pull-out stress if the pull-out test was actually conducted repeatedly on the same source of strand. For a given measurement of the predictor, half of the actual pull-out test results would be expected to fall above this average and half would fall below. The distribution of individual pull-out observations about that average pull-out stress is the basis for the prediction interval, which is calculated based on the variability in the data used for the regression.

This concept is demonstrated graphically in Figure B-102, which shows the prediction interval lower bound plotted along with the regression line, and data for the mortar pull out plotted versus the change in corrosion potential. If a specified threshold on mortar pull out is defined as 0.313 ksi, the threshold on the corrosion potential is the value where the pull-out threshold and the curve delineating the lower bound of the prediction interval intersect, shown by the red lines in the plot. In this case, the threshold would be approximately −0.175 V.

Unfortunately, Excel does not provide prediction intervals as a part of its standard output. However, a formula is provided in (Eq. 4) that allows for the calculation of a 90% prediction interval lower bound for pull-out stress of a new strand for the predictor. In the example, this is a prediction interval for pull-out stress with a measured value of change in corrosion potential as received. Of course, this prediction interval assumes that the model in (Eq. 3) is the correct model.

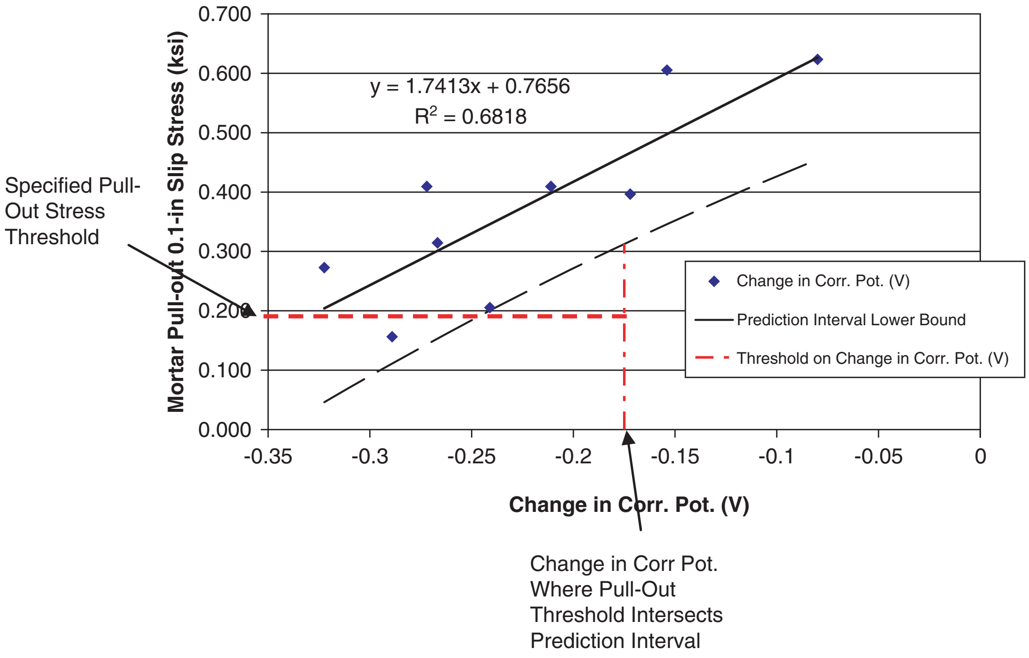


Figure B-102. Threshold determination using the prediction interval.

Prediction Interval (x_0)

$$= \hat{\beta}_0 + \hat{\beta}_1 x_0 - t_{.90,n-p} S \sqrt{1 + \frac{1}{n} + \frac{(x_0 - \bar{x})^2}{S_{xx}}} \qquad \text{(Eq. 4)}$$

Each of the variables in (Eq. 4) is explained in the following table. To help with the calculations below, the vector contains all the values of the predictor variable that were used in the regression (the prime indicates the transpose of the vector, so $\mathbf{x}_D$ is defined as a column vector),

$$\mathbf{x}_D = (-0.289, -0.080, -0.154, -0.241, -0.272, -0.211, -0.267 \\ -0.172, -0.322)'.$$

Explanation of Variables in Eq. 4

Variable	Description	Numerical Value in This Example
$\hat{\beta}_0$	The estimate of the intercept.	0.7658
$\hat{\beta}_1$	The estimate of the slope.	1.7425
S	The standard deviation of the observations from the fitted line.	0.097193
x_0	The value of x for which y is to be predicted.	Any value of corrosion potential for which a prediction is needed. For example, $x_0 = -0.1$.
$\bar{x}$	The average of all the x values used in the regression.	$\bar{x} = -0.228$
S_{xx}	This is a measure of the spread in the x values in the regression. It is defined as $S_{xx} = \mathbf{x}'_D \mathbf{x}_D - n\bar{x}^2$	0.04683
n	The total number of observations in the regression.	9
p	The total number of β's in the model (β_0, β_1)	2
$t_{.90,n-p}$	The 90th percentile of the *t*-distribution with *n-p* degrees of freedom. See the table below for approximate values. *t*-distribution for other confidence levels are available in most standard statistics textbooks and using Excel's TINV() function.	1.42 for *n-p* = 7

Approximate Values of the 90th Percentile of the *t*-Distribution

n-p	$t_{.90,n-p}$
1	3.08
2	1.89
3	1.64
4	1.53
5	1.48
6–7	1.42
8–9	1.39
10–13	1.36
14–27	1.33
>27	1.30

Following (Eq. 4), the 90% prediction interval for a new strand sample that has a change in corrosion potential as-received of $x_0 = -0.1$ V, would be

$$
\begin{aligned}
&= 0.7658 + 1.7425(-0.1) \\
&\quad - 1.42(0.097193)\sqrt{1 + \frac{1}{9} + \frac{(-0.1-(-0.228))^2}{0.04683}} \\
&= 0.425
\end{aligned}
$$

This means that for a measured change in corrosion potential as-received of −0.100 V, the mortar pull-out stress at 0.1-in. slip would be expected, with 90% confidence, to be greater than 0.425 ksi.

For the purpose of determining thresholds on other surface and chemical tests, the prediction interval over the range of the measured responses is needed. This is calculated by varying x_0 in Eq. 4.

Regression with Multiple Predictors

Often the response of interest depends on more than one predictor variable. In that case, additional terms are added to the regression model as shown in Eq. 5 for k predictors.

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_k x_k + \varepsilon \qquad \text{(Eq. 5)}$$

When regression is performed with multiple predictors, the regression output is very similar to the regression output for a single predictor. Again, the R^2 and R^2 *adjusted* can be calculated and should be interpreted as measures of how well the model fits the data. The difference between the two statistics is that, since adding predictors makes the model more flexible and thus better able to fit the data, the R^2 *adjusted* measure includes a penalty for additional predictors in the model. Thus, the goal of multiple regression is to find a model that has a relatively high R^2 value with as few predictors as possible, and maximizing R^2 *adjusted* accomplishes this goal. However, if too many predictors are put into the model, there will be very few degrees of freedom (n-p) for estimating the error variance (S). Again the goal is to have low values of the F-significance and low P-values for each of the slope estimates (i.e., β coefficients). An Excel regression output for a multiple regression is shown in Figure B-103, and it can be seen that this regression is a better model for the data than the single predictor regression shown above, as measured by R^2 and R^2 *adjusted*. However, two of the slope estimates (Weight LOI and Contact Angle After Dip) have P-values that are relatively large. Notice that the 95% confidence intervals of the slopes for these two factors contain zero; this indicates that the data are not conclusively supporting that these slopes are different than zero. Thus, the relationship between these measures and pull-out stress is not conclusive at the chosen confidence level.

If this multiple regression model were to be used for prediction of the pull-out stress, a prediction interval is still needed to determine thresholds. Determining the prediction interval for models based on multiple predictors is possible; however, it is somewhat more complicated and involves the matrix manipulations demonstrated by the following example.

For this example, the data used to develop the model by regression are as follows:

Weight Loss on Ignition x_1	Contact Angle after Lime Dip x_2	Change in Corrosion Potential As-Received x_3	Mortar Pull-Out Stress at 0.1-in. Slip (ksi) y
0.139	87	−0.289	0.156
0.059	79	−0.08	0.623
0.036	68	−0.154	0.606
0.086	94	−0.241	0.206
0.041	73	−0.272	0.409
0.045	76	−0.211	0.409
0.051	87	−0.267	0.315
−0.021	79	−0.172	0.397
0.002	98	−0.322	0.273

Let the matrix **X** be defined as a column of ones (representing the coefficient to be multiplied by the intercept, β_0) and then a column of levels for each of the other predictors in each observation.

$$
\mathbf{X} = \begin{bmatrix}
1 & 0.139 & 87 & -0.289 \\
1 & 0.059 & 79 & -0.080 \\
1 & 0.036 & 68 & -0.154 \\
1 & 0.086 & 94 & -0.241 \\
1 & 0.041 & 73 & -0.272 \\
1 & 0.045 & 76 & -0.211 \\
1 & 0.051 & 87 & -0.267 \\
1 & -0.021 & 79 & -0.172 \\
1 & 0.002 & 98 & -0.322
\end{bmatrix}
$$

The 90% prediction interval is then given by:

$$
\begin{aligned}
&\text{Prediction Interval } (x_1, x_2, \ldots, x_k) \\
&\quad = \hat{\beta}_0 + \hat{\beta}_1 x_1 + \hat{\beta}_2 x_2 \cdots \hat{\beta}_k x_k - t_{.90,n-p} S \sqrt{\mathbf{x}'(\mathbf{X}'\mathbf{X})^{-1}\mathbf{x}}, \qquad \text{(Eq. 6)}
\end{aligned}
$$

where each of the variables in (Eq. 6) is explained in the following table.

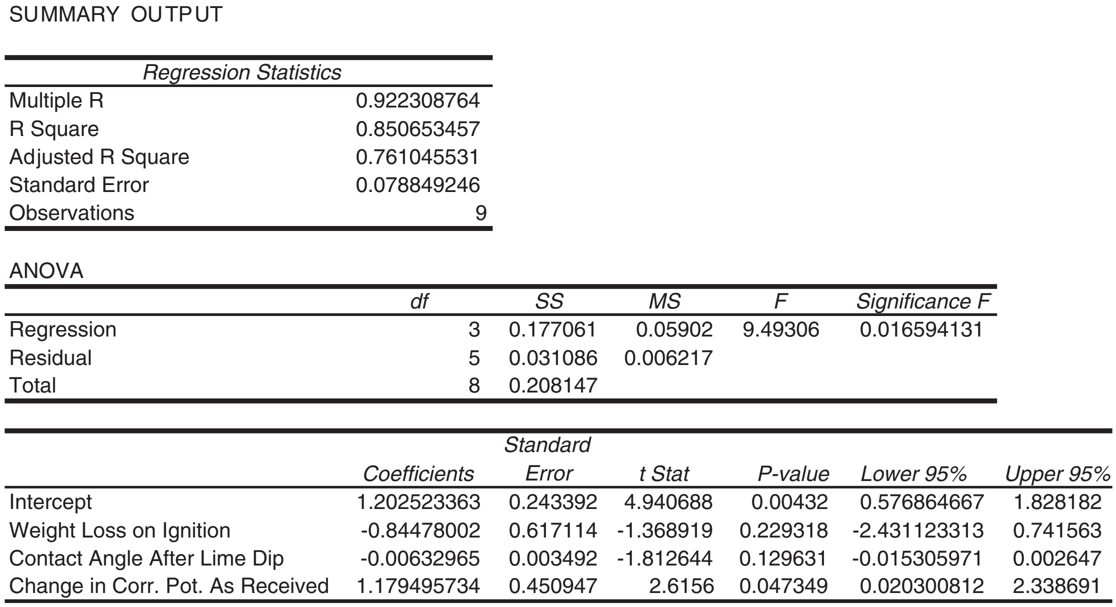
Explanation of Variables in Eq. 6

Variable	Description	Numerical Value in This Example
$\hat{\beta}_0$	The estimate of the intercept.	0.7658
$\hat{\beta}_i$	The estimate of the slope for predictor variable *i*.	1.7425
x	A vector of levels for each predictor at which *y* is to be predicted (including a 1 for the intercept).	For example, $\mathbf{x}_0 = \begin{pmatrix} 1 \\ 0.04 \\ 78 \\ -0.250 \end{pmatrix}$
n	The total number of observations in the regression.	9
k	The number of predictors in the model.	3
$p = k + 1$	The total number of β's in the model.	4
$t_{.90,n-p}$	The 90th percentile of the *t*-distribution with *n-p* degrees of freedom. See the table above for approximate values. *t*-distribution for other confidence levels are available in most standard statistics textbooks and using Excel's TINV() function.	1.48 for *n-p* = 5

The 90% prediction interval lower bound for $\mathbf{x}_0 = \begin{pmatrix} 1 \\ 0.04 \\ 78 \\ -0.250 \end{pmatrix}$ is 0.252 ksi.

This means that for a strand source with measured LOI of 0.04 mg/cm^2, a contact angle after lime dip of 78°, and a change in corrosion potential as-received of –0.250 V, the mortar pull-out stress at 0.1-in. slip would be expected, with 90% confidence, to be greater than 0.252 ksi.

Unfortunately, the prediction intervals based on regression with multiple predictors cannot be plotted in two dimensions as was done in Figure B-103, for the single predictor example. The predicted pull-out stress (y in Eq. 5) is not uniquely determined by a single combination of predictors ($x_1, x_2, \ldots$), but can be found based on numerous combinations. However, the prediction interval for the pull-out stress will be different depending on the specific combination of predictors that is used. That means that when multiple regression is used to improve the predictive ability of the model, a single



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.922308764
R Square	0.850653457
Adjusted R Square	0.761045531
Standard Error	0.078849246
Observations	9

ANOVA

	df	*SS*	*MS*	*F*	*Significance F*
Regression	3	0.177061	0.05902	9.49306	0.016594131
Residual	5	0.031086	0.006217		
Total	8	0.208147			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*
Intercept	1.202523363	0.243392	4.940688	0.00432	0.576864667	1.828182
Weight Loss on Ignition	-0.84478002	0.617114	-1.368919	0.229318	-2.431123313	0.741563
Contact Angle After Lime Dip	-0.00632965	0.003492	-1.812644	0.129631	-0.015305971	0.002647
Change in Corr. Pot. As Received	1.179495734	0.450947	2.6156	0.047349	0.020300812	2.338691

Figure B-103. Excel output for regression with multiple predictors.

Table B-38. Regression coefficients for single-predictor models.

Predictor	Constant (x_0)	Coefficient (β)	Coefficient of Determination (R^2)
Weight Loss on Ignition (mg/cm^2)	0.445	-1.403	0.16
Contact Angle after Lime Dip (°)	1.393	-0.012	0.57
Change in Corrosion Potential After 6 h(V)—As-Received	0.766	1.741	0.68
Extracted Organic Residue (mg/cm^2)	0.453	-1.752	0.12
Extracted Organic Residue (mg/cm^2)—Stearate only	0.436	-1.943	0.63

threshold cannot be defined. Instead, for a specific set of predictors, a new prediction interval must be calculated based on the set of data used to develop a regression model. The lower bound of the newly calculated prediction interval must then be compared with the specified pull-out threshold. For the example calculation performed above, the lower bound is 0.252 ksi. This is lower than the specified pull-out threshold of 0.313 ksi, so it cannot be predicted that this source of strand will exceed that pull-out stress 90% of the time (the defined confidence level). The framework for completing this calculation and comparison is given in the Microsoft Excel spreadsheet developed in this study.

Selection of Confidence Level

For the threshold determinations performed based on the data collected in this study, the confidence level was taken as 90%. This means that for a given surface and chemical test result, 10% of the pull-out results would be expected to fall below that prediction interval. This confidence level is lower than the 95% confidence interval that is most commonly used as the basis for probabilistic design in structural engineering analysis. However, using a confidence level as high as 95% makes determination of the thresholds for the surface and chemical tests very conservative.

Thresholds Based on Regression with Single Predictor

The test methods that were recommended for inclusion are:

- Weight LOI,
- Contact Angle Measurement after Lime Dip,
- Change in Corrosion Potential,
- Organic Residue Extraction.

The efforts made to define thresholds for each of these methods were based on single variable linear regressions and are described individually below. The results are summarized in Table B-38.

Weight Loss on Ignition (LOI)—The prediction interval for LOI with a one-sided confidence level of 90% is shown in Figure B-104. As can be seen in this figure, the prediction interval does not exceed 0.313 ksi anywhere over the range of test results observed in this study. For that reason, no threshold can be determined.

Contact Angle Measurement after Lime Dip—The prediction interval for Contact Angle with a one-sided confidence level of 90% is shown in Figure B-105. As can be seen in this figure, this prediction interval exceeds 0.313 ksi when the

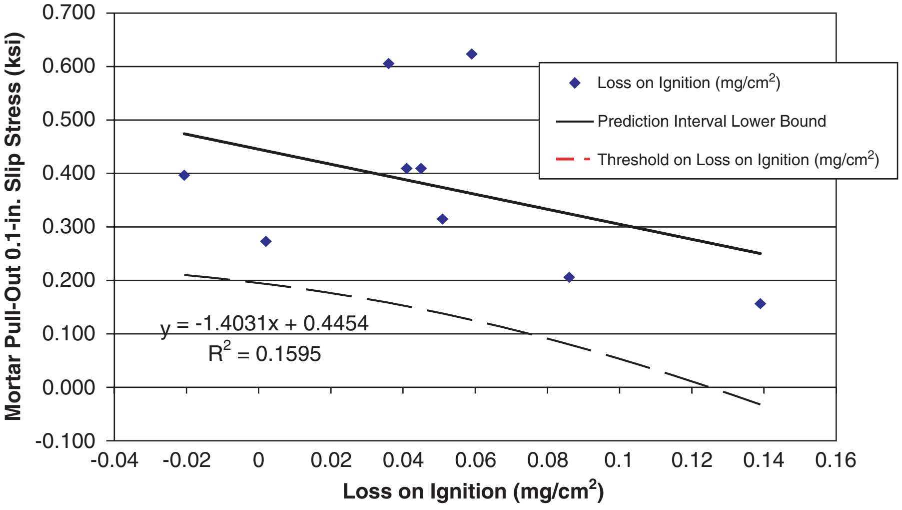


Figure B-104. Prediction interval (confidence level = 90%) for Weight Loss on Ignition. Threshold not possible.

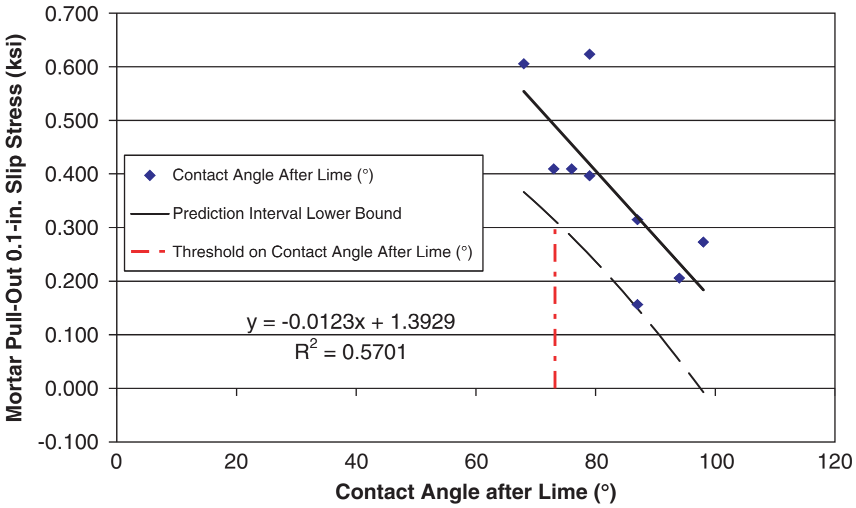


Figure B-105. Prediction interval (confidence level = 90%) for Contact Angle after Lime Dip.

contact angle is less than 73°. Therefore, based on these data and the NASPA defined threshold on mortar pull-out stress at 0.1-in. slip, a Contact Measurement after Lime Dip of 73° or lower is recommended to give a good (90%) confidence of adequate bond. This test must be run on recently manufactured strand with no surface weathering or rust (i.e., bright strand).

Change in Corrosion Potential—The prediction interval for Change in Corrosion Potential with a one-sided confidence level of 90% is shown in Figure B-106. As can be seen in this figure, this prediction interval exceeds 0.313 ksi when the change in the corrosion potential is less negative than –0.175 V. Therefore, based on these data and the NASPA defined threshold on mortar pull out 0.1-in. slip stress, a Change in Corrosion Potential of –0.175 V or more (less negative) is recommended to give a good confidence of adequate bond.

Organic Residue Extraction—The prediction interval for organic residue extraction with a one-sided confidence level of 90% is shown in Figure B-107. As can be seen in this figure, the prediction interval does not exceed 0.313 ksi anywhere over the range of test results observed in this study. For that reason, no threshold can be determined. A similar analysis was attempted considering only those sources with organic residue that the FTIR analyses indicated was primarily stearate. This was done to eliminate potentially confounding influences of non-stearate-based lubricants. The prediction interval for this stearate residue

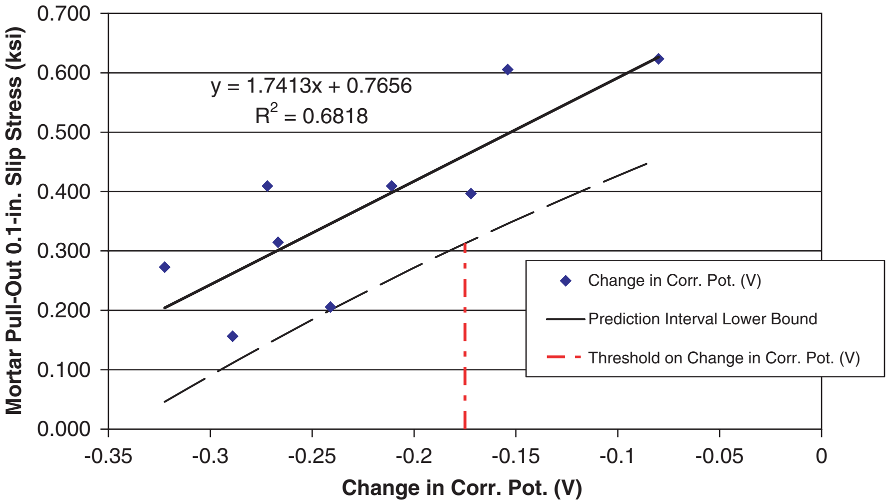


Figure B-106. Prediction interval (confidence level = 90%) for Change in Corrosion Potential.

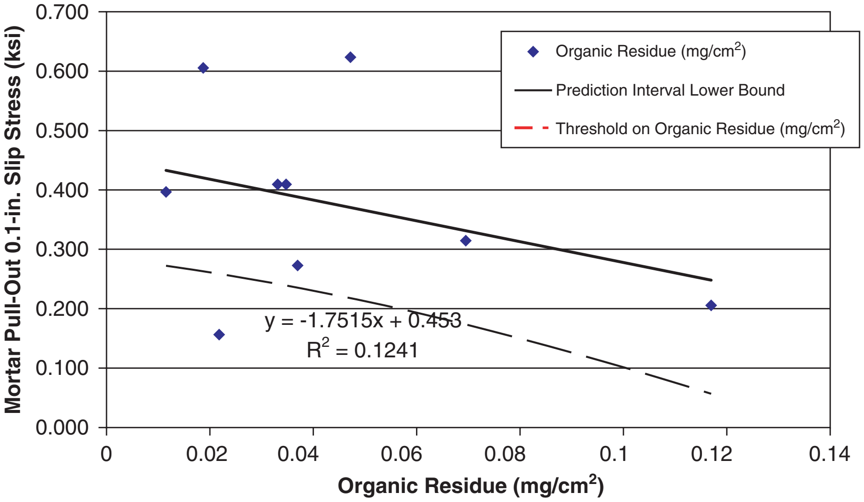


Figure B-107. Prediction interval (confidence level = 90%) for Organic Residue. Threshold not possible.

with a one-sided confidence level of 90% is shown in Figure B-108. As can be seen in this figure, the Coefficient of Determination is higher, but the prediction interval still does not exceed 0.313 ksi anywhere over the range of test results observed in this study, and no threshold can be determined.

Thresholds Based on Regression with Multiple Predictors

An attempt was also made to look for linear combinations of multiple results obtained from all the evaluated methods that would correlate with bond performance. While numerous combinations were examined, the three combinations that showed the best correlation, based on the adjusted coefficient of determination (R^2 *adj.*), were:

- Contact Angle Measurement after Lime Dip & Change in Corrosion Potential,
- Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only),
- Weight Loss on Ignition (LOI) & Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Note that for multiple-predictor regression, a larger number of variables will increase the R^2. Therefore, the adjusted R^2 statistic, which accounts for the number of degrees of freedom in the data set, was calculated as a means to compensate for

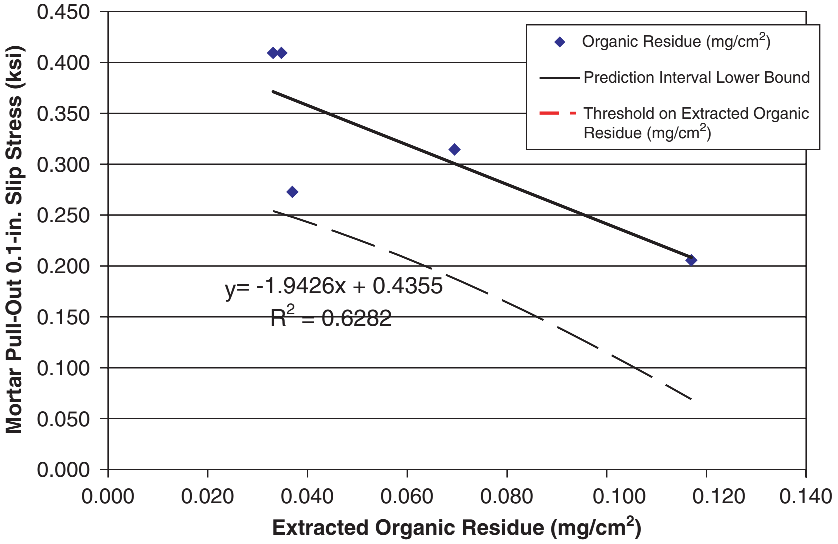


Figure B-108. Prediction interval (confidence level = 90%) for Organic Residue when FTIR analysis indicates organic residue is primarily stearate. Threshold not possible.

Table B-39. Regression coefficients for model based on Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Predictor	Coefficient
Constant	1.209
Contact Angle after Lime Dip (°)	-0.007
Change in Corrosion Potential After 6 h (V)—As-Received	1.233
Adjusted Coefficient of Determination (R^2 *adj.*)	0.727

Table B-40. Regression coefficients for model based on Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only).

Predictor	Coefficient
Constant	0.864
Contact Angle after Lime Dip (°)	-0.006
Extracted Organic Residue (mg/cm^2)	-1.093
Adjusted Coefficient of Determination (R^2 *adj.*)	0.976

Table B-41. Regression coefficients for model based on Weight Loss on Ignition (LOI), Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Predictor	Coefficient
Constant	1.203
Weight Loss on Ignition (mg/cm^2)	-0.846
Contact Angle after Lime Dip (°)	-0.006
Change in Corrosion Potential After 6 h (V)—As-Received	1.178
Adjusted Coefficient of Determination (R^2 *adj.*)	0.761

this potentially misleading effect. The regression coefficients for these three models and the R^2 *adj.* are given in Table B-39 to Table B-41. The R^2 *adj.* values for these combinations were high and equal to 0.73, 0.98 and 0.76, respectively. The R^2 *adj.* for each of these combinations was higher than the R^2 for the single-predictor regression models.

The regression that indicated that the last combination of predictors listed above (Contact Angle Measurement after Lime Dip & Organic Residue Extraction) was a good predictor of bond was performed based only on those strand sources that the FTIR analysis of the organic residue identified as being stearate only. This limited the number of data points used to develop the regression model to five, but was done as a means of eliminating potentially confounding influences of non-stearate-based lubricants on the results obtained by the contact angle and organic residue extraction measurement methods. Given the high level of correlation with the multiple regression approach, this model may be particularly useful in a production setting where the lubricant in use is known.

The prediction interval cannot be shown in a two-dimensional plot as was done with the single variable models. This is because there are multiple combinations of variables that can combine to give the same output. For this reason, a separate prediction interval must be calculated for each combination of variables. A Microsoft Excel-based spreadsheet has been developed for this purpose. To give a sense of how these multiple regression models might be used, tables have been prepared showing the predicted pull out, the lower bound on the prediction interval and the result of a comparison with the specified mortar pull-out threshold of 0.313 ksi, for each of the three multiple regression models. These are shown in Table B-42 to Table B-44.

For example, Table B-42 was developed for the model based on Contact Angle Measurement after Lime Dip & Change in Corrosion Potential. The first row of this table shows the results of these two individual tests obtained for Source 349. Based on the regression model, the predicted mortar pull-out stress at 0.1-in. slip is 0.264 ksi for these two results. The lower bound on the prediction interval for that combination of the two test results must be calculated

Table B-42. Evaluation of prediction interval for model based on Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Strand Source ID	Contact Angle after Lime Dip (°)	Change in Corrosion Potential (V)	Mortar Pull Out 0.1-in Slip Stress (ksi)			Pass/Fail* Based on Prediction Interval from QC Tests	Pass/Fail* Based on Pull-Out Test Result
			Experimentally Determined in Pull-Out Test	Value Predicted by Regression for QC Results	Lower Bound of Prediction Interval		
349	87	-0.289	0.156	0.264	0.131	Fails	Fails
548	79	-0.080	0.623	0.576	0.420	Passes	Passes
697	68	-0.154	0.606	0.559	0.417	Passes	Passes
717	94	-0.241	0.206	0.276	0.136	Fails	Fails
478	73	-0.272	0.409	0.38	0.232	Fails	Passes
960	76	-0.211	0.409	0.435	0.303	Fails	Passes
102	87	-0.266	0.315	0.291	0.161	Fails	Passes
103	79	-0.172	0.397	0.463	0.331	Passes	Passes
151	98	-0.322	0.273	0.149	0.003	Fails	Fails

* Threshold for passing is 0.313 ksi.

Table B-43. Evaluation of prediction interval for model based on Contact Angle Measurement after Lime Dip & Organic Residue Extraction (100% stearate only).

Strand Source ID	Contact Angle after Lime Dip (°)	Extracted Organic Residue (mg/cm^2)	Mortar Pull Out 0.1-in Slip Stress (ksi)			Pass/Fail* Based on Prediction Interval from QC Tests	Pass/Fail* Based on Pull Out Test Result
			Experimentally Determined in Pull-Out Test	Value Predicted by Regression for QC Results	Lower Bound of Prediction Interval		
717	94	0.117	0.206	0.211	0.176	Fails	Fails
478	73	0.033	0.409	0.42	0.388	Passes	Passes
960	76	0.035	0.409	0.401	0.371	Passes	Passes
102	87	0.069	0.315	0.303	0.274	Fails	Passes
151	98	0.037	0.273	0.276	0.24	Fails	Fails

* Threshold for passing is 0.313 ksi.

Table B-44. Evaluation of prediction interval for model based on weight loss on ignition, Contact Angle Measurement after Lime Dip & Change in Corrosion Potential.

Strand Source ID	Weight Loss on Ignition (mg/cm^2)	Contact Angle after Lime Dip (°)	Change in Corrosion Potential (V)	Mortar Pull Out 0.1-in Slip Stress (ksi)			Pass/Fail* Based on Prediction Interval from QC Tests	Pass/Fail* Based on Pull Out Test Result
				Experimentally Determined in Pull-Out Test	Value Predicted by Regression for QC Results	Lower Bound of Prediction Interval		
349	0.139	87	-0.289	0.156	0.193	0.044	Fails	Fails
548	0.059	79	-0.08	0.623	0.558	0.407	Passes	Passes
697	0.036	68	-0.154	0.606	0.56	0.424	Passes	Passes
717	0.086	94	-0.241	0.206	0.25	0.114	Fails	Fails
478	0.041	73	-0.272	0.409	0.385	0.243	Fails	Passes
960	0.045	76	-0.211	0.409	0.435	0.309	Fails	Passes
102	0.051	87	-0.267	0.315	0.294	0.169	Fails	Passes
103	-0.021	79	-0.172	0.397	0.517	0.378	Passes	Passes
151	0.002	98	-0.322	0.273	0.2	0.049	Fails	Fails

* Threshold for passing is 0.313 ksi.

specifically using those values and is 0.131 ksi. Since 0.131 ksi is less than the mortar pull-out threshold of 0.313 ksi, this source fails to meet the minimum required combined Contact Angle Measurement after Lime Dip & Change in Corrosion Potential performance. For Source 548, the prediction interval calculated for the specific combination of test results measured for that source is 0.420 ksi, and this source "passes" since this value exceeds the threshold.

Summary

An experimental program was conducted to evaluate a number of test methods proposed for use as part of a QC program to evaluate bond of strand. This included limited mechanical testing (pull-out testing from concrete, Portland cement mortar, and gypsum plaster-based mortar) and extensive surface and chemical testing (Contact Angle, Examination Under UV light, pH, LOI, Loss in Alkali Bath, Change in Corrosion Potential, Corrosion Rate, Surface Roughness, Organic Residue Extraction/FTIR Analysis, and Elemental Analysis). These tests, as well as transfer length tests, have been conducted on a range of strand sources to help establish correlations between the proposed QC tests methods and bond quality.

To evaluate these test methods, several rounds of evaluation were conducted: screening, correlation, and precision testing. The objective for the Screening Round was to eliminate those tests that do not show promise for predicting bond performance. The Correlation Round included those methods that showed promise in the screening experiments and was conducted to confirm that the selected QC tests correlated with bond performance over a larger sample set and were able to accurately identify good and bad strand. The Precision Testing was intended to form the base for precision statements to be included in the published test methods.

The Screening Round was conducted using strand collected by the project team over the 5 years before this project began. Obtaining additional strand with a range of bonding qualities for the purposes of the Correlation Round directly from strand suppliers proved difficult. Sections of strand were provided by Bruce Russell of OSU, who had previously conducted mortar pull-out tests on these sources of strand in work performed for the NCHRP Project No. 12-60 *Transfer, Development,* *and Splice Length for Strand/Reinforcement in High-Strength Concrete*; the Oklahoma Department of Transportation; and NASPA, also known as the Committee of the American Wire Products Association [AWPA]).

Although pull-out testing from concrete appears to correlate best with transfer length, the most reliable and realistic measure of bond performance, the Correlation Round of this test program had to be based on available mortar pull-out results provided by Russell from the NCHRP 12-60 program.

The four test methods that showed the best correlation with bond and that are recommended for inclusion in future QC programs are:

1. Weight Loss on Ignition (LOI) of Strand (QC-I),
2. Contact Angle Measurement of a Water Droplet on a Strand Surface (QC-I),
3. Change in Corrosion Potential of Strand (QC-I), and
4. Organic Residue Extraction with FTIR Analysis (QC-II).

The QC tests have been divided into two categories, depending on the complexity and time required to conduct the tests: Level I (QC-I) and Level II (QC-II) tests. The QC level is shown in the list above.

Thresholds for these QC tests have been developed where possible based on prediction intervals for the regression calculated from the available data and a minimum criterion on the mortar pull-out stress adopted by NASPA.

Regression with multiple predictors has also been performed to see if results of these methods can be combined to better predict bond. The three combinations that showed the best correlation, based on the adjusted coefficient of determination (R^2 *adj.*), were:

1. Weight Loss on Ignition (LOI) & Contact Angle Measurement after Lime & Change in Corrosion Potential,
2. Contact Angle Measurement after Lime & Change in Corrosion Potential, and
3. Contact Angle Measurement After Lime & Organic Residue Extraction (100% stearate only).

The adjusted coefficients of determination for each of these combinations were higher than the coefficients of determination for the single-predictor regression models. Thresholds for multiple-predictor regressions cannot be determined using the same procedure used for single-predictor regressions. Instead, the lower bound on the prediction interval must be calculated for each combination of test results. A computational tool in the form of a Microsoft Excel spreadsheet has been developed for this purpose.

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APPENDIX C

Specifications for Standard Surface Test Methods

Introduction

Based on the experimental results conducted during this study, four surface and chemical test methods have been recommended for inclusion in a QC program to assess strand bond. The recommended QC methods have been written in AASHTO/ASTM standard method format and presented in this appendix.

They are titled:

1. Test Method for the Determination of the Surface Tension of Steel Strand by Contact Angle Measurement,
2. Test Method for Weight Loss on Ignition (LOI) of Steel Strand,
3. Test Method for Change in Corrosion Potential of Steel Strand, and
4. Test Method for Identification and Quantification of Residue on Steel Strand by Extraction, Gravimetric, and Spectroscopical Analyses.

Proposed Standard Test Method for Determination of the Surface Tension of Steel Strand by Contact Angle Measurement

Scope

1.1 The contact angle of a water droplet on a strand surface is a measure of surface tension (wet-ability). The presence of drawing lubricants will affect this property. A relatively high amount of water-insoluble drawing lubricants will cause a relatively high contact angle value. The contact angle is measured on the projected shadow of a droplet of distilled water applied to the strand surface using a specialized instrument designed for this purpose.

1.2 The contact angle is measured after immersing the strand sample in a saturated calcium hydroxide [$Ca(OH)_2$] solution to simulate the environment surrounding the strand when it is in concrete. Due to the chemistry of Portland cement, concrete mix-water quickly becomes saturated with calcium hydroxide. This calcium hydroxide solution reacts with drawing-compound residues on the strand. This is the form of the residue on the strand when it is in concrete. Thus, this is the form of the residue that affects the bond, and this is the form to be assessed with this test.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Apparatus

2.1 *Contact angle meter*—The contact angle of a water droplet on a strand sample surface is measured using a contact angle meter.[1] The apparatus has a built-in syringe to dispense distilled water droplets of a small, precise size. Also, it has a fiber optic light source and a focusing lens to project the water droplet image onto a graduated screen. A photograph of the apparatus is shown in Figure C-1.

Specimens

3.1 The strand segments shall be cut to a length of 12 in., a length suitable to fit on the V-shaped sample pedestal of the apparatus.

Test Procedures

4.1 The procedure for preparation of each strand segment is described as follows:

4.1.1 Immerse the entire strand segment, or the portion to be tested, in saturated calcium hydroxide solution for 10 min.

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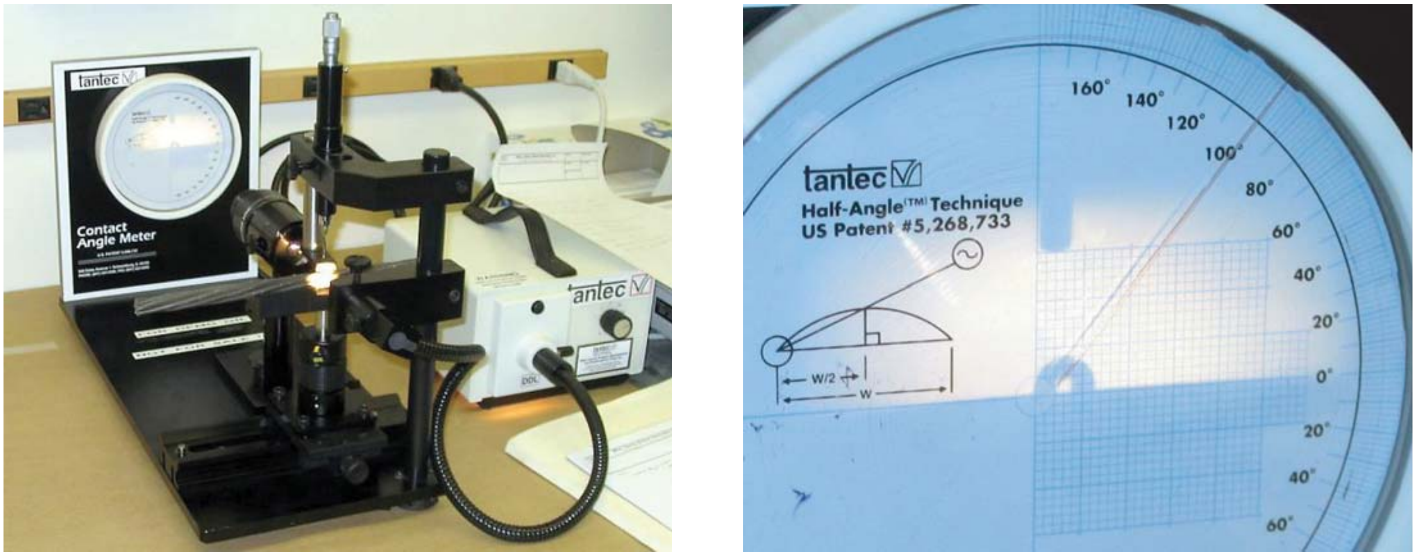
[1] This device is commercially available as Cam Plus Micro contact angle meter manufactured by ChemInstruments, Inc., Fairfield, OH.



(a) (b)

Figure C-1. Contact angle meter (a) and close-up of drop projection (b).

4.1.2 Immerse in de-ionized water for 5 min.

4.1.3 Dry by allowing vertical strand to drip while exposing to a warm air stream from a minimum 100 W heat gun held at a distance of 12 in. for a period of 2 min.

4.1.4 Set strand on a clean surface and cool to room temperature.

4.2 The procedure for contact angle measurement with the aforesaid apparatus is as follows:

4.2.1 Set the strand sample horizontally on the pedestal of the test apparatus with the surface to be tested facing upward.

4.2.2 Adjust the projection lens so that silhouettes of both the wire surface and the syringe needle are in focus.

4.2.3 Adjust the position of the helical wire surface to achieve a level silhouette on the projection screen under the syringe needle.

4.2.4 Adjust the syringe needle so that it is directly over the high point of the helical wire surface.

4.2.5 Form a distilled water droplet at the tip of the syringe. Rotate the syringe barrel to form a droplet with a diameter of 7 units on the scale imprinted on the projection screen

4.2.6 Raise the pedestal holding the strand sample until the wire surface touches the water droplet, and then lower the pedestal until the water droplet is released from the syringe needle.

4.2.7 Adjust the sample pedestal and the projection screen to align the left-hand side of water droplet silhouette with the y-axis and origin of the scale on the screen.

4.2.8 Adjust the dial of the protractor on the projection screen so that the indicator line intersects the apex of the water droplet silhouette.

4.2.9 Measure the angle from the apex of the drop using the protractor scale. Please note that the contact angle, which is twice the angle measured from the apex of the drop, is already doubled on the projection screen scale. Record this contact angle.

Calculation

5.1 Measure and record six individual contact angle readings for each sample. The individual readings are used to calculate a mean average contact angle.

Report

6.1 The mean average contact angle and the source of the strand are reported.

Interpretation

7.1 A lower contact angle corresponds to better bond performance. In our past studies, measured contact angles below 85° after the calcium hydroxide exposure were indicative of, but did not guarantee, good bond potential. As was observed when comparisons were made with transfer length, the contact angle measured on the strand after exposure to calcium hydroxide correlated well with concrete pull-out bond stresses (that is, bond stress at first or 0.1-in. slip during concrete pull-out testing is inversely proportional to contact angle). The contact angle was lower with greater bond stress.

Precision and Bias

8.1 *Single-operator precision*—The single-operator standard deviation was found to be 4°F. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 10°F. (These numbers represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670* [ASTM 2003].)

8.2 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Keywords

9.1 strand; contact angle.

Proposed Standard Test Method for Weight Loss on Ignition of Steel Strand

Scope

1.1 The weight loss on ignition (LOI) represents the weight of compounds that can be volatilized or burned off the strand surface at high temperature. This property is measured with the expectation that the weight loss represents the quantity of volatile residues from organic components of drawing lubricants and organic contaminants such as lubricating oils.

1.2 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Apparatus

2.1 *Balance*—An analytical-grade weighing instrument shall have a maximum capacity of at least 300 g and a precision of 0.1 mg. The sample compartment shall be at least 24.5-cm high to accommodate a vertically set sample.

2.2 *Graduated cylinder*—A 25-mL glass or plastic graduated cylinder is needed to hold the strand sample in a vertical position on the balance pan while weighing. The tare weight of the cylinder is subtracted from the weight reading to obtain the net weight of the sample.

2.3 *Oven and furnace*—The drying oven and ignition furnace instruments shall both have sufficient chamber length and volume to accommodate at least three strand segments. The drying oven shall be capable of heating the samples to 110°C ± 5°C, and the ignition furnace shall be capable of heating the specimens to 415°C ± 5°C.

2.4 *Desiccator*—A glass desiccating chamber shall use granular calcium chloride desiccant and a ceramic disk insert on which to rest strand segments and to separate samples from desiccant. The chamber shall have sufficient volume to accommodate at least three strand segments.

Specimens

3.1 Three segments per strand source shall be cut and loosely wrapped in uncoated aluminum foil for transport to minimize contamination or abrasion of the strand surfaces. Each strand segment shall be cut to a length of 23.0 cm ± 0.5 cm. The operator shall handle the strand segments with powder-free latex gloves.

Test Procedure

4.1 The LOI of each strand segment is measured using the following procedure:

4.1.1 Measure the length of the strand segment to the nearest 0.1 cm.

4.1.2 Determine the nominal diameter of the strand.

4.1.3 Dry the strand segment sample for 4 h at 110°C.

4.1.4 Cool sample in a desiccator for at least 12 h.

4.1.5 Weigh the sample three times to 0.1 mg precision. Record these initial weights and calculate the average weight.

4.1.6 Ignite samples for 30 min at 415°C.

4.1.7 Cool samples to room temperature in a desiccator for at least 12 h.

4.1.8 Weigh each sample again three times on the same analytical balance. Record these final weights and calculate the average final weight.

Calculation

5.1 The LOI reported for the strand source is the change in average weight divided by the surface area of the strand. The surface area can be calculated by $4\pi \times$ nominal diameter $\times$ tested length.

Report

6.1 Report the loss on ignition in milligrams per square centimeter (mg/cm^2) of strand and the source of the strand.

Interpretation

7.1 In past studies, the mass LOI decreased as the pull-out bond stress increased.

Precision and Bias

8.1 *Single-operator precision*—The single-operator standard deviation was found to be 0.014 mg/cm^2. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.041 mg/cm^2. (These numbers represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670* [ASTM 2003].)

8.2 *Bias*—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Keywords

9.1 strand; ignition; mass loss.

Proposed Standard Test Method for Change in Corrosion Potential of Steel Strand

Scope

1.1. This test method covers the estimation of change in the electrochemical corrosion potential of prestressed strands in de-ionized water, for the purpose of indirectly estimating the bond performance of prestressing strand in concrete.

1.2. *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 *ASTM Standards:*

- G3 Practice for Conventions Applicable to Electrochemical Measurements in Corrosion Testing
- G15 Standard Terminology Relating to Corrosion and Corrosion Testing

Summary of Test Method

3.1 Specimens of strands are immersed in de-ionized water at room temperature. Corrosion resistance of strands is characterized by measuring corrosion potential change from initial immersion after 6 h of immersion. Continuous potential monitoring with a potentiostat is optional. At least triplicate specimens shall be used and specimens shall be tested either as received or after ignition.

Significance and Use

4.1. This test method is intended for use as a rapid quality control method to indirectly assess the bonding characteristics of prestressing strand. Laboratory testing has demonstrated that strands with greater changes in potential have reduced bond strength.

4.2. Specimens are tested in as-received condition. It is also possible to test specimens after ignition.

4.3 Contamination of testing solution shall be avoided.

4.4 It is recommended that the test is to be performed in a temperature-controlled environment (e.g., an air-conditioned room), so that variation of solution temperature throughout the test is less than 3°C.

Interferences

5.1. Extended exposure of reference electrodes with ceramic tips of inferior quality may introduce aggressive ions such as chloride into the testing solution, and such contamination might alter testing results. Immersion time of reference electrodes shall be minimized. Copper/copper sulfate reference electrodes with porous wood plugs shall not be used.

Apparatus

6.1 *Reference electrode*—either a standard calomel electrode or Ag/AgCl electrode with controlled rate of leakage (about 3 μL/h) can be used. Such electrodes are durable, reliable, and commercially available.

6.2 *Voltmeter*—The division on the scale shall be such that a potential difference of 0.02 V or less can be read without interpolation.

Test Solution

7.1 De-ionized water shall be used.

Test Specimens

8.1 Strand specimens shall be 9-in. long.

8.2 The cut-end to be immersed in solution shall be sealed with a two-component epoxy. No more than 1/2 in. of the side of the strand shall be coated.

8.3 Specimens shall be tested in as-received condition, and surface contamination shall be avoided.

Procedure

9.1 Place the strand specimen(s) in a 2-L beaker and support firmly. A plexiglass plate with drilled holes shall be used to hold the specimens in place. When multiple specimens are used, the specimens shall be widely separated to avoid any direct contact with each other.

9.2 Introduce de-ionized water to the 2-L mark. Insert a reference electrode into the solution without disturbing any strand.

9.3 Measure the potential of each strand specimen at the start of testing and then after 1 h and after 6 h of the immersion.

9.4 If the potential of strand is being monitored with a potentiostat, the reference electrode shall be introduced before monitoring starts.

Report

10.1 Record the test procedure used, specimen size, temperature, and potentials.

10.2 Calculate changes in potential from the initial measurement to the potential measurement after 1 h and 6 h of exposure.

10.3 Report following information:

10.3.1 Strand identification,

10.3.2 Type of reference electrode, and

10.3.3 Change in potential at 1 h and at 6 h.

10.3.4 When a potentiostat is used to monitor potential change, a plot of potential change versus time shall be reported.

Precision and Bias

11.1 *Single-operator precision*—The single-operator standard deviation was found to be 0.047 V. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.133 V. (These numbers represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670* [ASTM 2003].)

11.2 Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

Keywords

12.1 Corrosion potential, strand, reference electrode.

Proposed Standard Test Method for Identification and Quantification of Residue on Steel Strand by Extraction, Gravimetric, and Spectroscopical Analyses

Scope

1.1 The test methods for identification and quantification of organic drawing-compound residues on strand surfaces are based on solvent extraction procedures, together with gravimetric and Fourier transform infrared spectroscopy (FTIR) analyses. Essentially, the amount of material extracted from a defined length of strand is determined by weighing the extraction residue on an analytical balance. The material(s) in the extraction residue are then identified by FTIR spectroscopical analysis.

1.2. *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Apparatus

2.1 *Balance*—An analytical-grade weighing instrument shall have a maximum capacity of at least 300 g and a precision of 0.1 mg. The weighing instrument shall have a maximum capacity of at least 50 g and a precision of 0.1 mg.

2.2 *Glassware*—The test procedure requires the use of the following glassware: a glass cylinder (Pyrex No. 2962), a 1000-mL glass separatory funnel (Pyrex No. 6402), a 400-mL glass beaker, and a 50-mL glass beaker.

2.3 *FTIR*—Use an FTIR spectroscope capable of analyzing the chemical make-up of the organic residue. Spectroscopic grade potassium bromide (KBr) plates will be required for FTIR analysis.

2.4 *Reagents*—Reagent grade hydrochloric acid (HCl), deionized water, and HPLC grade chloroform are required for this procedure.

Specimens

3.1 Each strand sample shall be cut to a minimum length of 35.5 cm (14 in.). A preferable strand segment length is in the range of 40.6 cm (16 in.) to 45.7 cm (18 in.) long. The upper boundary of the test portion area shall be marked by cutting a small notch at a length of 30.5 cm (12 in.) from the bottom of the strand segment.

Test Procedure

4.1 The organic drawing-compound residues on the strand segment sample are extracted, quantified, and identified using the following procedure:

4.1.1 Perform all steps involving hydrochloric acid and chloroform under a fume hood. Place a straight strand segment with a minimum length of 35.5 cm (14 in.) in a clean Pyrex No. 2962 glass cylinder, which is about 31.8 cm (12.5 in.) deep by 3.6 cm (1.4 in.) inside diameter. While wearing powder-free latex gloves, handle the strand segment only above the test portion length of 30.5 cm (12 in.).

4.1.2 Fill glass cylinder containing strand sample to a 12-in.-deep mark with 10% hydrochloric acid solution (about 280 mL) pre-heated to 120°F. Two-liter quantities of acid solution can be prepared in advance by adding 472 mL of ACS-grade concentrated hydrochloric acid (37.2% hydrochloric acid assay, 1.19 specific gravity) to 1528 mL of de-ionized water to achieve a 10% hydrochloric acid solution.

4.1.3 Allow 12-in.test portion of strand segment to set in 10% hydrochloric acid solution for 10 min, with frequent agitation of solution by stirring with strand segment. Carefully transfer acidic solution to a clean 1000-mL glass separatory funnel (Pyrex No. 6402). Rinse strand and cylinder with 20 mL of 10% hydrochloric acid solution, and transfer acidic solution to separatory funnel.

4.1.4 Fill glass cylinder containing strand sample to a 12-in.-deep mark with HPLC-grade chloroform (approximately 280 mL). Allow 12-in. test portion of strand segment to set in chloroform for 10 min, with frequent agitation of solvent by stirring with strand segment.

4.1.5 Wearing powder-free latex gloves and handling the strand segment only above 12-in. test portion, very slowly lift the strand sample from the solvent while scrubbing the strand at solvent surface using a rubber policeman (a spatula-shaped, latex-rubber tip on the end of a glass rod). The elapsed time for the scrubbing process of the entire 12-in. test portion should be about 3 min. Briefly immerse the 12-in. test portion into solvent and agitate to remove as much solvent-soluble material as possible. Remove strand sample from solvent.

4.1.6 Carefully transfer solvent to the glass separatory funnel that contains the acidic solution. Rinse strand sample over glass cylinder with about 20 mL of chloroform, swirl solvent in glass cylinder, and pour rinse solvent into same separatory funnel. Stopper the separatory funnel, and shake it for 5 min. Vent funnel occasionally to release vapor pressure. After shaking, allow separatory funnel with liquid contents to rest in an upright position so that chloroform settles to bottom of funnel. The acidified aqueous solution becomes the top liquid layer.

4.1.7 Place the beaker below the separatory funnel and open stopcock of separatory funnel slowly and carefully to drain chloroform only into the beaker. Place beaker on a hot plate set at less than 70°C to evaporate chloroform in a controlled manner. The evaporation process can be aided by directing the air flow of a fan over the top of the beaker.

4.1.8 The 400-mL beaker with dried residue is rinsed with about 10 mL of chloroform, and the chloroform with solvent-soluble residue is transferred to a clean, pre-weighed 50-mL beaker. The chloroform rinse procedure is repeated twice (a triple rinse procedure). The chloroform with solvent-soluble residue in the 50-mL beaker is evaporated at less than 70°C. The beaker is reweighed to determine the net weight gain due to solvent-soluble, dried residue.

4.1.9 The dried residue, if any, is transferred with chloroform to a spectroscopical-grade potassium bromide salt plate for analysis by FTIR spectroscopy. The FTIR spectrum is interpreted to identify the residue.

Calculation

5.1 The extraction residue weight shall be determined by weighing on an analytical balance once to the nearest 0.l mg. This weight shall be divided by the surface area of the strand sample's test portion, in square centimeters (cm^2). The surface area of the strand shall be calculated in cm^2 as follows: $30.5 \times {}^{4}/_{3}\, \pi \times D$ where D = nominal diameter of the strand. After weighing, the residue is identified by FTIR spectroscopical analysis, and the FTIR spectrum interpretation is reported.

Report

6.1 The weight of the residue shall be reported in units of mg/cm^2 of strand surface. Our past studies have found that a relationship exists between the extracted organic residue and the average bond stress over the transfer length. As bond stress increased, extraction residue decreased. The relationship is especially evident in the total extraction residue (combined from water and acid/chloroform wash solutions) plotted for both of these procedures.

The report shall also include the infrared spectrum of the residue, and the interpretation of the spectrum by the spectroscopist.

Precision and Bias

7.1 *Single-operator precision*—The single-operator standard deviation was found to be 0.013 mg/cm^2. Therefore, results of two properly conducted tests by the same operator on the same source are not expected to differ from each other by more than 0.037 mg/cm^2. (These numbers represent, respectively, the (1s) and (d2s) limits as described in *ASTM C670* [ASTM 2003].)

Bias—Since there is no accepted reference material suitable for determining the bias in this test method, no statement on bias is made.

APPENDIX D

Supplemental Investigations of Strand Bond

Appendix D to the contractor's final report for NCHRP Project 10-62 is not published herein. For information, contact NCHRP.

APPENDIX E

Bibliography of Strand Bond

Appendix E to the contractor's final report for NCHRP Project 10-62 is not published herein. For information, contact NCHRP.

Abbreviations and acronyms used without definitions in TRB publications:

AAAE	American Association of Airport Executives
AASHO	American Association of State Highway Officials
AASHTO	American Association of State Highway and Transportation Officials
ACI–NA	Airports Council International–North America
ACRP	Airport Cooperative Research Program
ADA	Americans with Disabilities Act
APTA	American Public Transportation Association
ASCE	American Society of Civil Engineers
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATA	Air Transport Association
ATA	American Trucking Associations
CTAA	Community Transportation Association of America
CTBSSP	Commercial Truck and Bus Safety Synthesis Program
DHS	Department of Homeland Security
DOE	Department of Energy
EPA	Environmental Protection Agency
FAA	Federal Aviation Administration
FHWA	Federal Highway Administration
FMCSA	Federal Motor Carrier Safety Administration
FRA	Federal Railroad Administration
FTA	Federal Transit Administration
IEEE	Institute of Electrical and Electronics Engineers
ISTEA	Intermodal Surface Transportation Efficiency Act of 1991
ITE	Institute of Transportation Engineers
NASA	National Aeronautics and Space Administration
NASAO	National Association of State Aviation Officials
NCFRP	National Cooperative Freight Research Program
NCHRP	National Cooperative Highway Research Program
NHTSA	National Highway Traffic Safety Administration
NTSB	National Transportation Safety Board
SAE	Society of Automotive Engineers
SAFETEA-LU	Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (2005)
TCRP	Transit Cooperative Research Program
TEA-21	Transportation Equity Act for the 21st Century (1998)
TRB	Transportation Research Board
TSA	Transportation Security Administration
U.S.DOT	United States Department of Transportation