

TESTING FACTORS THAT AFFECT THE BOND CHARACTERISTICS OF PRESTRESSING STRAND

John S. Lawler, PhD, PE, Wiss, Janney, Elstner Associates Inc., Northbrook, IL, USA
Andrew E.N. Osborn, PE, Wiss, Janney, Elstner Associates Inc., New York, NY, USA
James D. Connolly, Wiss, Janney, Elstner Associates Inc., Northbrook, IL, USA

ABSTRACT

A four-year study, funded through the NCHRP, had been conducted of US-produced prestressing strand aimed at determining the cause of inconsistent strand bond over the past 20 years and at developing quality control (QC) testing procedures that may be used to identify strand with deficient bond properties. Films of lubricant and other contaminants remaining on the surface of prestressing strand after manufacture can undermine bond performance in concrete. Only certain manufacturers were found to be making strand with deficient bond as a result of excess residual wire drawing lubricants and other factors. Four test procedures (Weight Loss on Ignition (LOI), Contact Angle Measurement After Lime Dip, Change in Corrosion Potential, and Organic Residue Extraction with Fourier Transform Infrared spectroscopic analysis) have been developed for use as part of a routine quality control (QC) program to allow rapid assessment of potential bond quality issues. These tests were compared to transfer length tests and pullout tests of untensioned strand embedded in concrete and mortar. These methods and three linear combinations of their results demonstrated ability to predict bond, as measured with pull out tests. Acceptance thresholds for two of the individual QC tests and all of the combinations were then developed, based on prediction interval data and a predefined minimum criterion for the mortar pull out stress. The developed tests are proposed for use in strand production facilities to qualify the bond properties of strand before it is shipped to customers.

Keywords: Strand, Quality control, Drawing lubricant, Bond

INTRODUCTION

The transfer of prestressing force from prestressed concrete 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 prestressed concrete 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. A set of testing procedures to be used as part of routine quality control (QC) program is needed to assess factors that are known to affect bond properties.

To meet this need, the National Co-operative Highway Research Program (NCHRP) funded Project 10-62 to specifically study the surface characteristics of prestressing strands, determine how those characteristics influence bond, and develop test methods to qualify the strand for bond.

BACKGROUND TO STRAND BOND UNCERTAINTY

The bond properties of strand used in the USA were studied in the 1950's and 60's. Those studies resulted in the current equations for bond transfer and development lengths. Transfer length is the distance from the end of the member to the point on the strand where the strand pretension is developed. The development length is the distance from the end of the member to the point on the strand where the ultimate strength of the strand is developed. Currently, the American Concrete Institute (ACI) and the American Association of State Highway and Transportation Officials (AASHTO) specify a transfer length of about 50 to 60 strand diameters and a development length about three times the transfer length. The transfer length can be estimated as about 300 times the amount of end slip that occurs after release of prestress. Therefore, for 1/2-in. (12.7-mm) strand, for example, we would calculate a transfer length of about 27-1/2 in. (700 mm) and an end slip of about 0.1 in. (2.3 mm). End slips greater than about 2.5 mm are a cause for concern because they could indicate a strand with poor bond properties, i.e. require a greater distance to transfer than expected based on the ACI and AASHTO prediction equations.

In the mid-1980's, a major test program was under-taken at the North Carolina State University to compare the bond properties of epoxy coated strand to that of uncoated strand¹. The researchers found to their surprise that uncoated strand had sometimes twice the expected transfer length. As a precautionary measure, the Federal Highway Administration instituted a 1.6 multiplier on the calculated transfer length until the problem could be better understood and resolved². Numerous experimental programs took place in the 1990's to study the bond characteristics of prestressing strand in concrete, but none of them studied the potentially harmful effects of wire drawing lubricants. In 1992, a strand lifting loop pulled out of a member causing the member to be dropped. The ensuing investigation determined that the strand bond properties were less than they should be, based on a simple test where an untensioned strand is pulled out of a concrete block. Seven manufacturer's products were tested and four of them were found to have deficient bond properties.

In spite of efforts made by the strand producers in North America to study the problem and recommend solutions to date, there are still occasional incidents where strand bond problems occur. From the late 1980's to the present, the authors have investigated or are 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.

MANUFACTURE AND SURFACE CONDITION OF PRESTRESSING STRAND

Seven wire prestressing strand is typically 0.5, 0.52, or 0.6 in. (12.7, 13.2, or 15.2 mm) in diameter, consisting of 6 wires twisted around a straight central wire called the king wire. The individual wires are drawn from 1/2-in. (12.7-mm) rod stock, which has been cleaned and pretreated to facilitate the adherence of wire drawing lubricants. The rod stock is drawn through a series of about eight dies to achieve the final wire diameter. Integral with each die is a box containing wiredrawing lubricant that the wire passes through before entering the die. This allows for different lubricants to be used with different dies. The dies and the capstans, which pull the wire through the die, are typically water cooled since the performance of the lubricant and properties of the wire are sensitive to temperature. The wires are spooled, then loaded into a skip strander. The skip strander wraps the six outer wires around the central wire to make the 7-wire strand. The strand then passes through an oven, under tension (to achieve its low-relaxation properties), followed by a water bath, drying, and spooling. Some of the wire drawing lubricant is still adhered to the surface of the wire after drawing. If the residual wire drawing lubricants are excessive, they can interfere with the bond between the strand and the surrounding concrete.

Pre-treatment, lubrication and residual film

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 post-drawing processes. The purpose of the pretreatment, which is typically 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.

For strand production, the most common material used during the pretreatment process is zinc phosphate, which serves as a carrier for the lubricants applied during the wire drawing process. Borax and lime may also be used for pretreatment, either alone or in combination with zinc phosphate.

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 U.S. 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).

Residual films are always present after wire drawing³. 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 stress-relieving operations⁴. However, as noted in a 1982 article⁵, the replacement of open-flame furnaces with more efficient induction furnaces had the effect that residues were no longer burned off during stress-relieving operations.

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"⁵. The link between lower amounts of lubricant and increased bond strength has also been demonstrated more recently by others⁶.

RESEARCH OBJECTIVES AND APPROACH

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 advisory panel, this third objective was modified during the execution of this project to include the use of a pre-existing performance-based test procedure.

The work plan developed to achieve these objectives was divided into three phases:

The initial phase involved gathering information about strand manufacturing and potential test methods from the prestressed concrete, strand manufacturing and wire drawing lubricant industries and 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, were conducted to learn more about the relationship between bond and residual lubricants.

In the third and final phase of work, 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 advisory panel, the bond performance was quantified by another researcher using a 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.

The complete details of the research effort are to be presented elsewhere⁷. This paper summarizes the evaluation of the quality control test methods.

QUALITY CONTROL PROGRAM DEVELOPMENT

The purpose of a quality control 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 quality control 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 quality control test methods for detecting and measuring the level of deleterious residues on strand that could be performed frequently. 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.

The individual tests that were proposed required a varied range of time, expertise and equipment. Therefore, these tests were assigned to be either to Level I or Level 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 more 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.

Several rounds of experimentation were conducted in this research program was to determine if the proposed tests would be applicable for use in a quality control (QC) program.

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. For each source of strand, bond performance was measured in terms of pull out stresses, transfer lengths or both. 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.

The second round of experiments was called “Correlation” testing and was performed for confirmation and calibration purposes using those methods that showed promise in the Screening experiments. These selected tests were conducted on five new strand sources. 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.

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 proposed test methods and is reported elsewhere⁷.

TRANSFER LENGTH AND PULL OUT TESTS

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

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 mortar, concrete or a “surrogate homogeneous material” (a modified gypsum plaster mortar).

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. 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 slip” or the bond stress at 0.1-in. slip, measured at the non-loaded end of the 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, the concrete pull out test showed the best correlation with bond quality. The mortar pull out test was not as discriminating between the samples of strand as the concrete pull out test. As a result, 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 Principle Investigator from that project supplied the strand samples for this portion of the study. No pull out testing was conducted by the authors 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 not possible.

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.

The historic strand were samples of strand from prior tests conducted at Kansas State University (KSU) by Dr. Bob Peterman and at StressCon Corporation, Inc. by Mr. Don Logan that cover a wide range of pull out behavior. These were manufactured between 1997 and 2004.

Recently-manufactured samples were obtained in large quantities for the purpose of this research and were used in the Screening experiments. 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 samples used for the Correlation Round of testing were selected and supplied by Dr. Bruce Russell of Oklahoma State University (OSU). These sources of strand had been tested for the NCHRP Project 12-60 Transfer, Development, and Splice Length for Strand/Reinforcement in High-Strength Concrete, the Oklahoma Department of Transportation, and for the NASPA (also known as the Committee of the American Wire Products Association [AWPA]). Complete mortar pull out and some transfer length test results were provided in tabular form by Dr. Russell after the chemical and surface testing had been completed. 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 Ref. 8, 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.

PROPOSED QUALITY CONTROL TEST METHODS

The test methods that were proposed and that were conducted as part of the Screening and Correlation testing are summarized in Table 2. These tables also list the QC levels for these tests, if applicable. The surface and chemical tests methods that were attempted included:

- Contact Angle Measurement
- Examination under UV light
- pH testing
- Loss on Ignition
- Loss in Hot Alkali Bath
- Change in Corrosion Potential
- Surface Roughness
- Corrosion Rate
- Organic Residue Extraction
- Elemental Analysis

As is discussed in the following section of this paper, four of these methods showed promise. Each of these promising methods is described briefly below. The details of the other methods can be found in Ref. 7.

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.

Contact Angle Measurement - The contact angle is a measure of surface tension (wet-ability). 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. While measurements were taken with the strand as-received and after an ignition process, the strongest correlation with pull out performance was found after immersing the strand sample in a saturated calcium hydroxide $[\text{Ca}(\text{OH})_2]$ solution. The calcium hydroxide exposure (also called a lime dip) will convert sodium soaps (e.g., sodium stearates) to insoluble calcium salts, which are typically water-repellent. 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.

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 tendency for corrosion to develop, 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. 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 calcium hydroxide $[\text{Ca}(\text{OH})_2]$ solution, and after an ignition process. Testing in the as-received condition was judged to be the most appropriate.

Organic Residue Extraction - The test 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. The extraction procedure used is a modification of a procedure found in ASTM C114 for organic materials in cement. During evaluation of the method, multiple extractions were used to differentiate between water-soluble materials, such as sodium stearate, and water-insoluble residues such as calcium stearate and stearic acid. However, the final recommended method involves washing the strand with hydrochloric acid and chloroform. The chloroform-soluble organic components in this wash solution are then extracted with chloroform. The chloroform solution is then evaporated leaving the organic residue.

The amount of material extracted from a defined length of strand is determined by weighing the extraction residue on an analytical balance. The material in the extraction residue is then identified by FTIR analysis of the residue. The FTIR spectrum obtained is like a fingerprint of the material.

Statistical Evaluation of Results

Statistical analyses of the collected data 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 on both of these analysis methods is given elsewhere⁷.

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.

To further evaluate the validity of these methods, the significance of the linear models developed based on this 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 that 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 $(1 - P\text{-value}) \times 100\%$, so a 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. While non-stearate-based lubricants may also impact bond, the responses of sources with non-stearate-based lubricants were excluded for this calculation since the response of such lubricants may not be similarly proportional to bond performance as the response of strand with only stearate-based lubricants. Analyzing this 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. Other models specific to the lubricant type could also be developed where non-stearate-based lubricants are consistently used.

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, which is the most appropriate measure of goodness-of-fit for multiple-predictor regression, was calculated and used to interpret how well the model fits the data.

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.

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 prediction interval is calculated based on the variability in the data used for the regression.

This concept is demonstrated graphically in Figure 1, 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 cannot be shown graphically. When multiple regression is used, a single 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 the regression model. The lower bound of the newly calculated prediction interval must then be compared with the specified pull out threshold.

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.

FINDINGS OF TEST METHOD EVALUATION

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 the recommended QC methods are given in Table 3. The P-values for these methods are given in Table 4.

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. Some 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 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 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.

Loss on ignition - A good correlation was found between the weight loss on ignition (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, though not alone. Some other measure of bond performance should be included along with LOI in a QC program.

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. 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.

Level II QC Test

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. This test requires either more advanced methods or more complicated equipment. The minimum correlation required for these tests is higher than for the Level I QC tests.

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 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.

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 Dr. Russell and adopted by NASPA.

The bond strength thresholds proposed by Dr. 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 Test⁸. The thresholds were derived using development length tests on four strand sources, (in what is referred to as the NASPA Round III study⁹), 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 strand⁹. These minimum thresholds have since been increased to 10,500 and 9,000 lbs., but without additional testing. The justification for these new criteria can be found in Ref. 8.

Despite the somewhat limited scope of the development process used to establish these 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. The threshold was converted to a bond stress (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.

Thresholds Based on Regression with Single Predictor

The efforts made to define thresholds for each of these recommended QC methods based on single predictor linear regressions are described individually below. The results are summarized in Table 5.

Weight Loss on Ignition (LOI) - The prediction interval for LOI with a one-sided confidence level of 90% is shown in Figure 2. 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 3. 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 this 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 1. 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 this 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 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. 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 and other surface contaminants. The prediction interval for this stearate residue with a one-sided confidence level of 90% is shown in Figure 5. 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 was also 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. While numerous linear 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

The R^2 adj. values for these combinations were high and equal to 0.73, 0.98 and 0.76, respectively.

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 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 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, a table has been prepared showing the predicted pull out, the 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 one of the three multiple regression models. This is shown as Table 6, 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 6 as the example, the first row shows the results of these two individual QC tests obtained for Source 717. The lower bound on the prediction interval for mortar pull out stress at 0.1-in. slip for that combination of the two test results must be calculated specifically using those values and is 0.176 ksi. Since 0.176 ksi is less than the mortar pull out threshold of 0.313 ksi, this source fails to meet the minimum threshold for the combined performance Contact Angle Measurement After Lime Dip & Organic Residue Extraction for those residues determined to be stearate only. For Source 478, the lower bound on the prediction interval calculated for the specific combination of test results measured for that source is 0.388 ksi, and this source “passes” since this value exceeds the 0.313 ksi threshold. The last column shows whether or not that strand would be expected to pass based on the actual pull out test result. Sources 478 and 960 are judged to produce acceptable performance based on both the prediction interval and the actual pull out test results. However, both evaluations are not always in agreement; Source 102 is judged to fail based on the prediction an interval, even though it barely passed in the actual pull out test. 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. Source 102, incidentally, was from a separate reel of strand that exhibited excessive strand slip in hollowcore planks.

DISCUSSION

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. 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. That program only generated two transfer length tests of the five sources of strand submitted to our study. Hence the data from which to make comparisons remain scarce.

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, while requiring some training and the acquisition of some specialized equipment, could be conducted by the 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. While, 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 of ½-in. strand on a quarterly basis. This currently represents only 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 quality control program requirements for at least one other reinforcing steel product - during production of epoxy coated reinforcing steel at many manufacturing facilities, a number of quality control 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 wire drawing process is ongoing. While the development of such a test program was beyond the scope of this research, greater quality control as part of the lubricant acquisition process also would add to the confidence in bond quality.

Thresholds for two of these QC tests (Contact Angle Measurement After Lime Dip and Change in Corrosion Potential recommended) have been developed. The available data was 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 same source) included in the program, only two and three samples would be judged to be acceptable based on these test methods, respectively. While 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 samples 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 has also been performed to determine if results of selected QC methods could be combined to better predict bond. One of the models (for the combination of Contact Angle Measurement After Lime Dip & Organic Residue Extraction) for stearate based residues predicted two of five samples would be judged to be acceptable in the pull out testing, while three actually met the minimum threshold. The two models that included all nine of the strand samples predicted that three of nine samples would pass based on the pull out test, in contrast to the six of nine that were found acceptable by the pull out test. While 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.

Based on the research that has been conducted, there are a number of possible reasons that the prediction intervals are not smaller:

- The QC test methods themselves are inadequate.
- The QC test methods or the mortar pull out test method were susceptible to large scatter.
- The sampled sources were too closely grouped in terms of bond performance.
- A limited number of data points were available for the regression analysis.

At this point, while recognizing the possibility of the first reason, there appears to be sufficient promise in the recommended test methods to support additional effort aimed at eliminating the potential contributions of the remaining three reasons. The most straightforward method to address this need is to obtain additional data points over as wide a range of bond performance as possible. This would increase the confidence in the regression model estimates' ability to accurately predict performance.

While 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 NASP, 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 data set 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.

The threshold on the mortar pull out test result adopted by NASPA is based on transfer (based on end slip) and development length (based on beam tests) 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, while 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.

CONCLUSIONS AND RECOMMENDATIONS

A main objective of NCHRP Project 10-62 was to develop a set of QC procedures for use by strand manufactures or their customers as part of a routine quality control 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. These 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 appeared to correlate best with transfer length, the most reliable and realistic measure of bond performance, this test program was based on available mortar pull out results obtained by others.

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:

- Weight Loss on Ignition (LOI)
- Contact Angle Measurement After Lime Dip
- Change in Corrosion Potential
- Organic Residue Extraction with FTIR analysis

The first three are easily performed by strand production QC staff. The fourth test is somewhat more difficult and would be better performed by an experienced chemical testing laboratory.

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 were:

- 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
- Contact Angle Measurement After Lime Dip & Organic Residue Extraction (when organic residue is primarily stearate)

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 adopted by NASP. Thresholds for multiple-predictor regressions must be calculated based the lower bound on the prediction interval for each combination of test results.

It is recommended that the three Level I QC tests be adopted as part of a routine quality control 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 was 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.

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.

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TABLES

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.										

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	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)
	After Ca(OH) ₂ dip			
	After Ignition			
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	I	pH of surface	Detect presence of pretreatment lubricant residues containing alkaline salts or alkalis
	Indicator solutions			
	pH meter			
	High-res. indicator			
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	I	Weight washed off strand	Determine amount of material that can be washed off the strand surface after soak in a NaOH solution
	Method 2			
Change in Corrosion Potential	As received	I	Average change of potential	Assess the potential for corrosion by comparing the corrosion potential to a reference cell monitored versus time
	After Ca(OH) ₂ dip			
	After Ignition			
Surface Roughness	-	I	Roughness parameters R _a , R _z , P _c	Quantify surface profile

Table 2 (cont.)

Test Method	Condition/ Type of test	QC Level	Property Measured	Objective
Corrosion Rate	As received	II	Corrosion current	Determine the shift in potential of a metal sample from a stable corrosion potential due to an external current
	After Ca(OH) ₂ dip			
	After Ignition			
Organic Residue Extraction	Warm water/acid-chloroform wash	II	Weight of extracted organic residue	Determine amount of individual components of stand manufacturing lubricants from a warm/hot water wash procedure then an acid/solvent-wash procedure
	Hot water/acid-chloroform wash			
Atomic Absorption (AA) Spectroscopy	Sodium	II	Concentrations of inorganic components of extraction residue	Quantify inorganic elements (sodium, calcium, potassium, zinc, and boron) in residue
	Calcium			
	Potassium			
	Boron			
	Zinc			
	Phosphate			
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 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 ($^{\circ}$)	After Ca(OH) ₂ Dip	I	0.61	0.57
	After Ca(OH) ₂ Dip - Stearate Only [†]	I	0.44	0.84
Loss on Ignition		I	0.86	0.16
Change in Corrosion Potential after 6 hrs.	As Received	I	0.72	0.68
Organic Residue Extraction	Total	II	0.81	0.12
	Total - Stearate only [†]	II	0.88	0.63

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

Table 4. 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 ($^{\circ}$)	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 hrs.	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.

Table 5. Regression and Threshold for Single-Predictor Models

Predictor	Coefficient of Determination (R^2)	Threshold Corresponding to Mortar Pull out stress of 0.313 ksi
Weight Loss on Ignition (mg/cm ²)	0.16	Not Possible
Contact Angle After Lime Dip ($^{\circ}$)	0.57	73
Change in Corrosion Potential After 6 hrs. (V) - As Received	0.68	-0.175
Extracted Organic Residue (mg/cm ²)	0.12	Not Possible
Extracted Organic Residue (mg/cm ²) - Stearate only	0.63	Not Possible

Table 6. 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

FIGURES

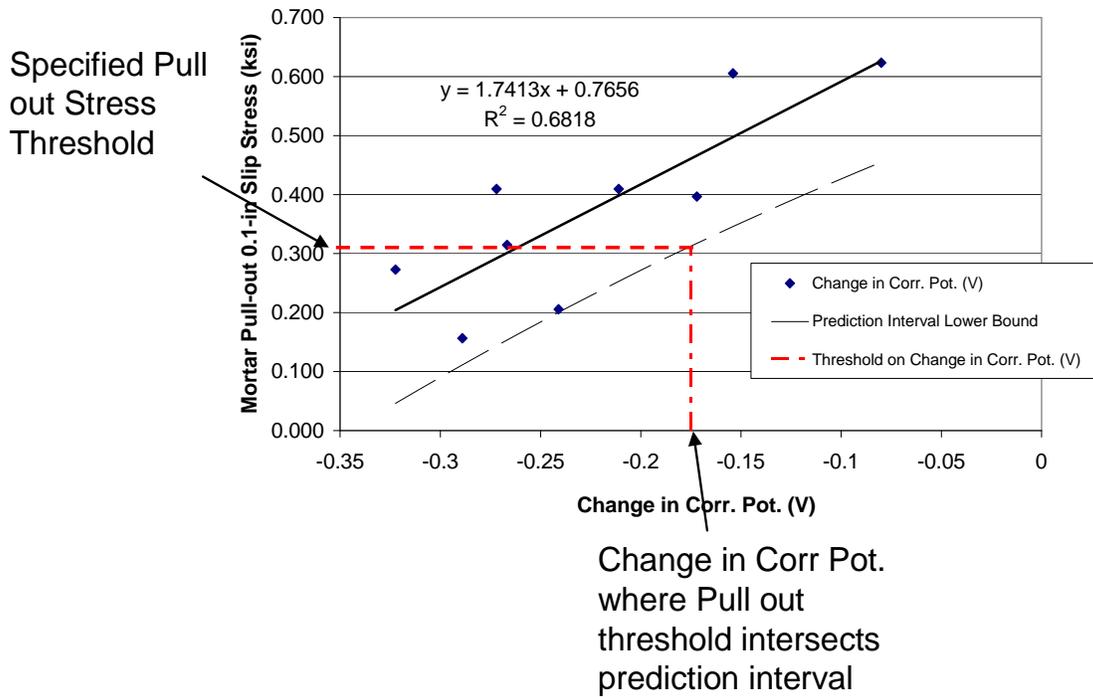


Figure 1 - Threshold determination using the prediction interval for Change in Corrosion Potential (Confidence Level = 90%).

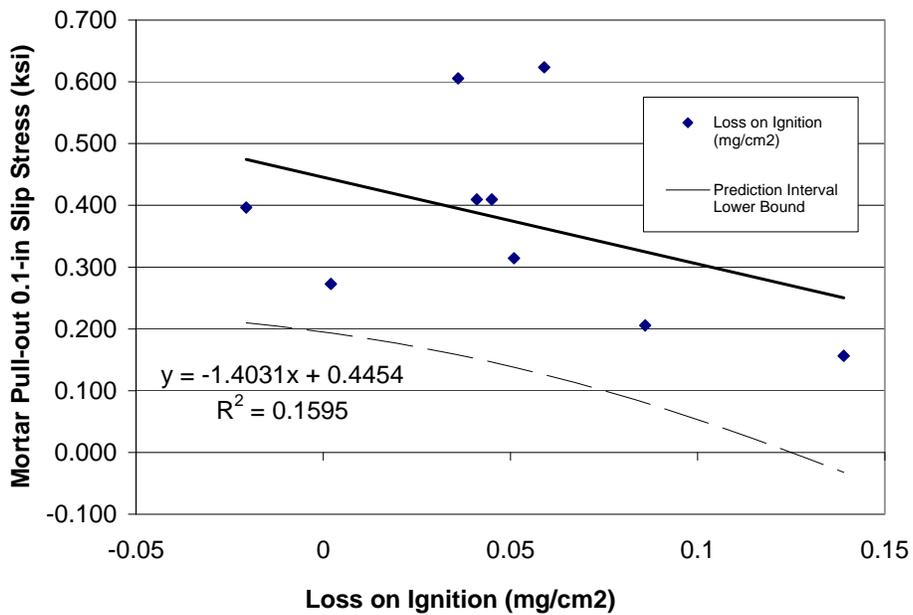


Figure 2 - Prediction Interval for Loss on Ignition (Confidence Level = 90%). Threshold not possible.

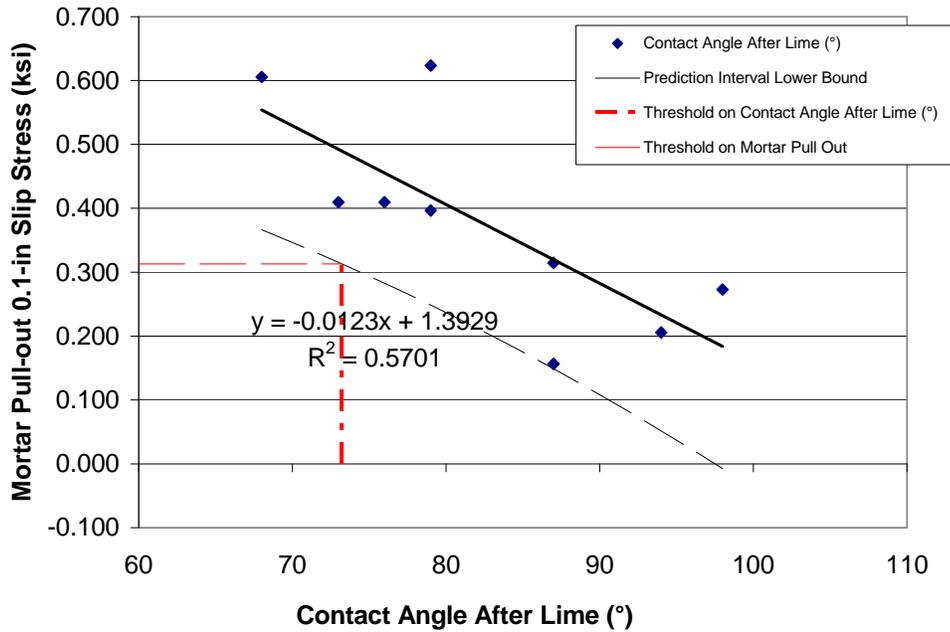


Figure 3 - Prediction Interval for Contact Angle After Lime Dip (Confidence Level = 90%).

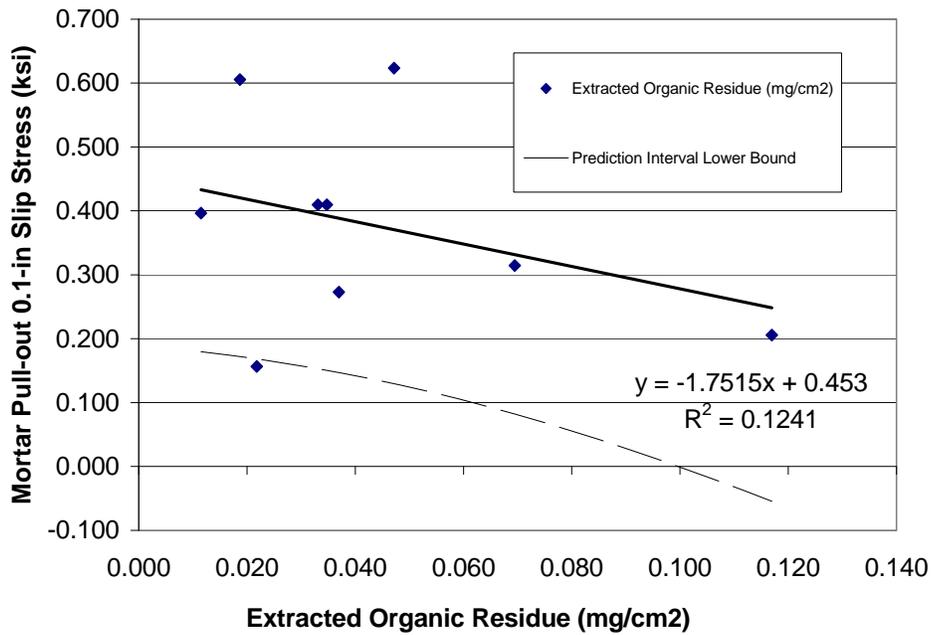


Figure 4 - Prediction Interval for Organic Residue (Confidence Level = 90%). Threshold not possible.

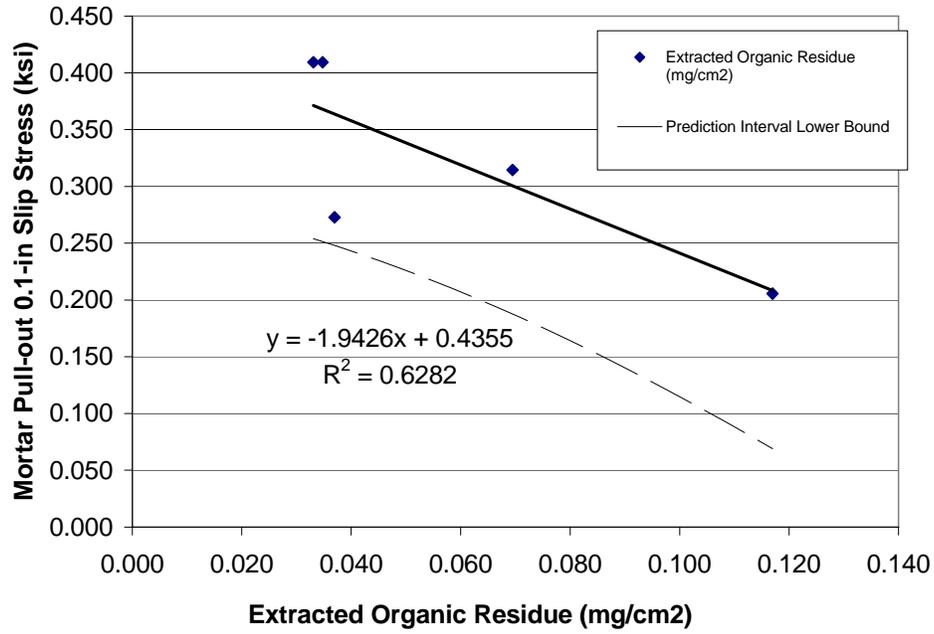


Figure 5 - Prediction Interval for Organic Residue when FTIR analysis indicates organic residue is primarily stearate (Confidence Level = 90%). Threshold not possible.