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Tensile Test to Measure Adhesion Between Old and New Cement Paste

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Progress Report

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U.S. DEPARTMENT OF COMMERCE, C. William Verity, *Secretary*

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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ABSTRACT

A tensile test has been developed to measure the adhesion of repair material to hardened concrete using specimens of repair material cast between two cylinders of hardened substrate material. Cement paste was used as the model material for both the repair material and the hardened substrate. Aspects of the test procedure that appeared to affect the strength data included the jig used to cut the hardened substrate surface in preparing the composite specimen, the grips used during the tensile test, the crosshead speed, exposure condition during loading, and possibly the age of the repair material. The adhesion of cement paste to a hardened cement paste substrate was approximately 2.7 MPa, with a coefficient of variation of 15 percent. The test provides a method for measuring tensile strength of the bond between repair material and concrete substrate under controlled, laboratory conditions, for use in evaluating materials, surface preparation methods, and application procedures.

Key words: adhesion, bond, cement, concrete, tensile strength, test method.

INTRODUCTION

Repair and rehabilitation of concrete are becoming increasingly important as the nation seeks to extend the life of existing concrete structures. Therefore, increased attention should be given to the technology and practice of concrete repair. The goal of this project is to contribute to understanding the factors controlling the performance of cementitious materials for concrete repair, and the formation of a technical basis for developing guidelines for selection of repair materials.

Strong adhesion of repair material to the underlying concrete is considered essential to the repair of concrete structures. Achieving strong adhesion generally involves both effective preparation of the old concrete surface, and proper selection and application of the repair material. Typical cementitious repair materials are concrete or mortar containing some organic polymer. Measuring strength of adhesion between the repair material and its concrete substrate can be used to evaluate both effectiveness of the surface preparation and performance of the repair material.

Test methods for adhesion strength, reviewed by Sasse and Fiebrich [1], and more recently by Knab [2], include measurements of shear strength [3-5] and tensile strength [5-7]. The shear methods are variants of the slant-shear test, in which the concrete is bonded on a plane oblique to the loading axis and loaded in compression to attain an indirect shear loading. The slant-shear test has been adopted by ASTM for measuring strength of the bond between epoxy-resin materials and concrete [3]. However, no similar standard test exists based on tensile strength. It is considered that an available method for measuring tensile test strength would contribute to understanding adhesion and to assessing material performance.

The objective of the present investigation is development of a method for measuring the uniaxial tensile strength of the adhesion of repair material to a concrete substrate, and to carry out preliminary studies to assess the method. There appears to be little information concerning expected levels of strength and variance for the test being developed. Mindess and Struble [8] measured the strength between cement paste and aggregate using a tensile test, and reported strength values of 2 to 4 MPa at 7 days, a slightly higher value at 28 days, and a coefficient of variation (1σ/mean) of ~30 percent. This coefficient of variation is probably excessive if the test is to allow assessing the performances of materials. The strength of concrete in a direct tensile test has a coefficient of variation of approximately 7 percent [9], probably a reasonable target level for a bond tensile test.

EXPERIMENTAL APPROACH

The experimental approach consisted of developing a general test procedure, then varying certain test parameters to evaluate their effect on the strength level and on the precision of strength data. Finally, preliminary tests were carried out using a commercially available repair material.

The loading configuration, shown schematically in Fig. 1, involved composite specimens with repair material sandwiched between two cylinders of substrate, loaded in uniaxial tension. In these preliminary studies, cement paste was used as a model material for both cementitious repair material and concrete substrate. In addition to the cement paste, one commercially available repair material, a cementitious grout[1,2], was tested to determine whether the procedure may be suitable for repair materials. All cement pastes were prepared from the same Type I[3] portland cement with a water-to-cement ratio of 0.35 (by weight). The grout was mixed with water using a water-to-solid ratio of 0.20, following the manufacturer's instructions. The pastes and grout were mixed according to a standard procedure [10].

Substrate paste was cast in cylindrical molds of waxed cardboard, 38 mm (1-1/2 in) diameter and 76 mm (3 in) height. Paste was placed in the mold and consolidated using a vibrating table until no further air bubbles were observed. Specimens were cured for 24 hours in a moist cabinet, at 27°C and 92 percent relative humidity. They were then demolded and cured at room temperature (~23°C) until the desired age, usually 7 to 14 days. Samples were immersed in an aqueous solution saturated with $Ca(OH)_2$ so solid $Ca(OH)_2$ from the hydrated cement would not dissolve during curing.

One face of each hardened cement-paste substrate cylinder was prepared for casting fresh cement paste. This face was cut to form a smooth, planar surface normal to the loading axis, using a low-speed, diamond saw[4] lubricated with water during

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[1]Certain trade names and company products are identified to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the products are necessarily the best available for the purpose.

[2]Masterflow 713, obtained from Master Builders, Inc., Cleveland, OH.

[3]As defined by ASTM C 150-85, Standard Specification for Portland Cement.

[4]Buehler Isomet with low-density blade.

cutting. One of two jigs was used to hold the cylinder during cutting: Jig No. 1, supplied with the saw, and Jig No. 2 (Fig. 2), designed in the course of these studies (See results).

The composite specimens were prepared as a sandwich of cement paste or other repair material between two cylinders of hardened cement paste. A mold was utilized such that fresh paste or other repair material of desired thickness could be cast between the two cylinders. Cement paste was prepared as described previously, and inserted into the mold. The composite specimens were vibrated to consolidate, as described previously, with the axis of the cylinder horizontal to avoid weakening the bond by entrapping air bubbles at the interface. Specimens were allowed to harden in the mold for 24 hours in the moist cabinet, then demolded and immersed in a solution saturated with $Ca(OH)_2$ for the desired curing time, usually 7 to 30 days.

An objective of this investigation was to identify grips for the tensile test that would provide uniaxial tension with minimum shear or torque, would hold specimens tightly enough to prevent slippage, and would not cause stress concentrations that affect the strength measurement. We initially considered using a cable puller, a wire mesh tube designed to grip cable in order to pull it through conduit, which had been used in an earlier study of the bond between cement paste and aggregate [8]. This grip appeared to provide adequate gripping but only moderate precision of tensile strength data (as discussed previously, the typical coefficient of variation was ~30 percent). Two grips were used in the present study. Grip No. 1 (Figs. 3 and 4) was based on a grip designed in our laboratory for calibrating a pneumatic testing device [11]. Key features of the design included clamps to prevent slippage and universal joints to maintain a loading axis free of shear and torque. This design was modified in Grip No. 2 (Fig. 3) to allow more convenient mounting of the specimen, retaining the univeral joints but replacing the clamps with rigid brackets. This grip, however, showed evidence of slipping (See results).

Composite specimens were loaded using a universal testing machine[5] equipped with an electronic load cell and monitored using an XY recorder. The crosshead speed (loading rate) was varied between 0.1 and 100 mm/min. For most tests, specimens were immersed in a solution saturated with $Ca(OH)_2$ during the tensile loading, using a tank designed for this purpose (Fig. 5); for comparison, a few specimens were tested in laboratory air.

With only one exception, all specimens failed at the interface between the inner material and one of the cylinders of

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[5]Instron 1125.

hardened paste. Little inner material was observed adhering to the substrate material after specimens were tested.

To calculate tensile strength, the load at failure is divided by the cross-sectional area of the specimen. Measurements of the diameters of 10 specimens provided a cross-sectional area of 895 mm^2 with coefficient of variation of only 0.3 percent. Based on this low variance, the cross-sectional area was assumed to be the same for all specimens.

RESULTS

The experimental test design consisted of seven variables with fourteen different data sets (Table 1). The tensile strength data are presented in Table 2 and Fig. 6. Both the strength level and the standard deviation varied among the treatments. Strength values ranged from 0.83 MPa to 3.31 MPa, and standard deviation values ranged from 0.21 to 0.78 MPa, with coefficient of variation between 10 percent and 68 percent, depending on the specific treatment.

Assessments of certain test procedures were made during the course of the study. Procedures appeared satisfactory except for problems with one jig used to hold the substrate material while cutting the face against which the inner material was subsequently cast, and one type of grip used during tensile loading. The surface of the substrate materials using Jig No. 1 was not always normal to the cylindrical axis. A new jig, designated Jig No. 2, was designed and constructed to hold this cylinder such that the surface was more nearly normal. Slipping of the grips, indicated by abrupt and transient decreases in load, was often observed when using Grip No. 2, while Grip No. 1 showed no evidence of slipping.

DISCUSSION

Procedures to Analyze Test Data

Test data were analyzed using statistical methods to determine which treatments appeared to influence either the level or the precision of the tensile strength. In order to determine whether the strength data of any treatment were significantly different from the overall strength data, an analysis of variance was performed on all data. The F-ratio of 2.04 calculated in this analysis is significant at the 0.05 level[6], indicating that there are significant differences in strength data for one or more treatment. Another statistical test, Bartlett's test, was performed to determine whether the standard deviation of any one treatment is significantly different from the standard deviation of all data. The Bartlett parameter, which is tested for significan using a Chi-squared distribution, is 22.17, significant at the 0.05 level, indicating that one or more procedures have standard deviation levels that are significantly different from the levels of the overall strength data.

Neither the analysis of variance nor the Bartlett's test indicate which specific procedures affect the strength level or standard deviation. In order to determine which procedures affect the test results, pairs of treatments were compared using statistical methods. The pairs of procedures for these analyses were selected so as to vary only one parameter within each pair. To pair the treatments for this comparison, each test parameter was assigned a binary value (Table 3) for convenience in selecting treatment pairs (Table 4). It should be noted that two parameters, specimen age and crosshead speed, were tested at more than two levels, so analysis of test data using this binary approach provides only a preliminary indication of their effect.

Each pair of treatments was tested for equality of variance using an F-test and for equality of mean using a pooled or unpooled variance t-test[7]. The pooled variance t-test assumes equality of variance, so those pairs of treatments that were found to be unequal in variance were tested for equality of mean using an unpooled variance t-test. Since both tests assume a normal distribution, data for each treatment were first tested for normality using the Wilk-Shapiro test. This test indicated that the distribution of data for each treatment was normal.

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[6]That is, there is a 95% probability that the null hypothesis of no significant difference may be rejected.

[7]The statistical analyses were carried out using RS/1, a data analysis system for personal computers from BBN Software Products Corp.

Results of Analysis of Test Data

Results of the statistical analysis are listed in Table 5. Many of the parameters were varied in more than one pair of tests, sometimes with conflicting results in terms of equality of variance or mean. As summarized in Table 5, a few treatment parameters appeared to affect the variance of the test results, and a number of treatment parameters appeared to affect the mean strength.

One treatment parameter that clearly influenced the mean strength was the cutting jig. In nearly every case, Jig No. 2 produced a lower mean strength than Jig No. 1. There was no apparent effect of the jig on variance. As discussed previously, it was observed that Jig No. 2 provided a bonding surface more nearly and consistently normal to the central axis of the specimen. Thus the jig appears to be an important aspect of the tensile test procedure.

The exposure condition, whether or not the specimen is immersed in $Ca(OH)_2$ solution during loading, affected the mean and possibly the variance. It was considered from previous studies that the specimens should not be allowed to dry. Drying is known to cause microcracks; and if the two materials dry at different rates, the cracks may occur in paste near the interface and reduce the measured bond strength. Thus loading in air was expected to decrease strength level and increase variance. However, the results were not entirely consistent with this expectation. The tests with the specimen loaded in air produced a higher variance, but also a higher mean strength, compared to two similar treatments with the specimen immersed, though the difference in variance was significant only for one of the immersed treatments. So exposure condition appears to be an important parameter.

The crosshead speed did not affect variance, but affected the mean strength in some treatment pairs. Most tests were run using a crosshead speed of 1 mm/min. When crosshead speed was increased to 10 or 20 mm/min or decreased to 0.1 mm/min, there was no effect on mean strength. However, increasing crosshead speed to 100 mm/min did appear to increase significantly the strength level.

The two grips did not affect either the variance or the mean strength. Since Grip No. 2 tended to slip, Grip No. 1 was preferred.

It is not clear from the statistical results whether the age of the sample affected the bond strength. Ages of both the inner cement paste, used to model a repair material, and the outer paste, used to model the concrete substrate, were not varied appreciably or systematically in these tests. The variation of age of the inner paste did not affect either the mean or variance of bond strength. There was an indication that variation of age of the outer paste affected the mean strength in one pair of tests. Age of the inner material is expected to have a significant effect on bond strength if varied more widely, e.g. between a few days and several months. However, such a wide variation in age was not included in the present study, and additional tests are required to confirm any age effect.

In addition to the tests utilizing cement paste as the inner material, one test utilized a cementitious grout. The grout did not affect the variance, but may have affected the mean. The mean strength using the grout was 3.14 MPa, significantly higher than the mean strength using cement paste for one comparable treatment, but not significantly different than the mean strength for the other comparable treatment. The test using grout provided a preliminary indication that the test can be used to assess performance of cementitious repair materials.

Recommended Testing Procedures

Based on these statistical analyses, procedures for measuring tensile strength of the bond between old and new concrete may be described. Test parameters that appear to be important, based on qualitative assessments and on the statistical analysis, and the recommended procedures (Table 6) are as follows: cutting the surface of the hardened substrate material using Jig No. 2, loading the composite specimen using Grip No. 1, keeping the specimens immersed in saturated $Ca(OH)_2$ solution while loading, and loading at a constant crosshead speed, e.g. 1.0 mm/min.

Four treatments (Numbers 4, 6, 11, and 13) utilized these parameters. Data in these four treatments had a total sample size of 24, an overall mean of 2.39 MPa, and a standard deviation of 0.51 MPa, for a coefficient of variation of 26 percent. This variance is still considered excessively high. An analysis of variance produced an F-ratio of 7.51, which is significant at 0.05, indicating that there are still unexplained differences in strength data between one or more individual treatment.

The difference may be due to variation in specimen age. Strength data plotted against age of the inner material (Fig. 7) indicate that results at the two intermediate ages, 15 and 21 days, are higher than the results at the lowest age, 7 days, and the highest age, 28 days. As discussed previously, an effect of age was expected, but the binary analysis showed no age effect, perhaps as a result of that particular statistical approach. If there is an effect of age, either of the substrate material or the inner material, then the bond test should be carried out at a single specimen age. In that case, the test results may better be estimated from data of each individual treatment: the mean strength levels of these four treatments ranged from 1.9 to 2.7

MPa, the standard deviations ranged from 0.2 to 0.5 MPa, and the coefficient of variation levels from 11 to 17 percent. This range of variance is lower than the 26 percent obtained with treatments at various ages, and closer to the 7 percent reported by Neville [9] for the tensile strength of concrete. The effect of specimen age on strength level and variance needs additional study.

Applications of the Tensile Test

The test developed in the present study allows direct determination of tensile strength of the bond between new concrete or repair material and old concrete. This bond is more typically evaluated using a shear test. The present work provides a method for measuring tensile strength in addition to shear strength of this bond. Although additional studies are needed, it appears that under controlled, laboratory conditions, the test can be used to evaluate materials, surface preparation methods, or application procedures. In particular, a combination of a tensile test, such as described in this study, and a shear test, such as ASTM C 882, would allow selection of repair material based on strength of adhesion to the concrete substrate. If the substrate material is found to affect adhesion of a particular repair material, then the test can be used with a specific concrete substrate to select a repair material.

The tensile adhesion test was developed for cementitious repair materials. In the preliminary studyes, the test appeared to provide a satisfactory method to measure adhesion of cement paste or grout to hardened cement paste. The test has not been applied to repair materials that are based on materials other than cement. It may not be suitable for materials that exhibit substantial elastic or plastic deformation.

CONCLUSIONS

A tensile test has been developed to measure adhesion strength of cementitious repair material to concrete substrate. A preliminary study of the tensile test indicates that certain test parameters affect strength results: the jig used to cut the substrate surface, the grips, crosshead speed, exposure condition, and possibly age of the inner material. Recommended procedures are summarized in Table 6. From those tests in which these parameters were not varied, the adhesion of cement paste to a hardened cement paste substrate was 2.7 MPa, with a coefficient of variation of 15 percent. Although the strength data were scattered, the test appears to provide an acceptably low variance. It offers a potential method for assessing the adhesion of repair materials to the concrete substrate. Additional studies are needed to better define test parameters that affect precision and accuracy.

ACKNOWLEDGEMENTS

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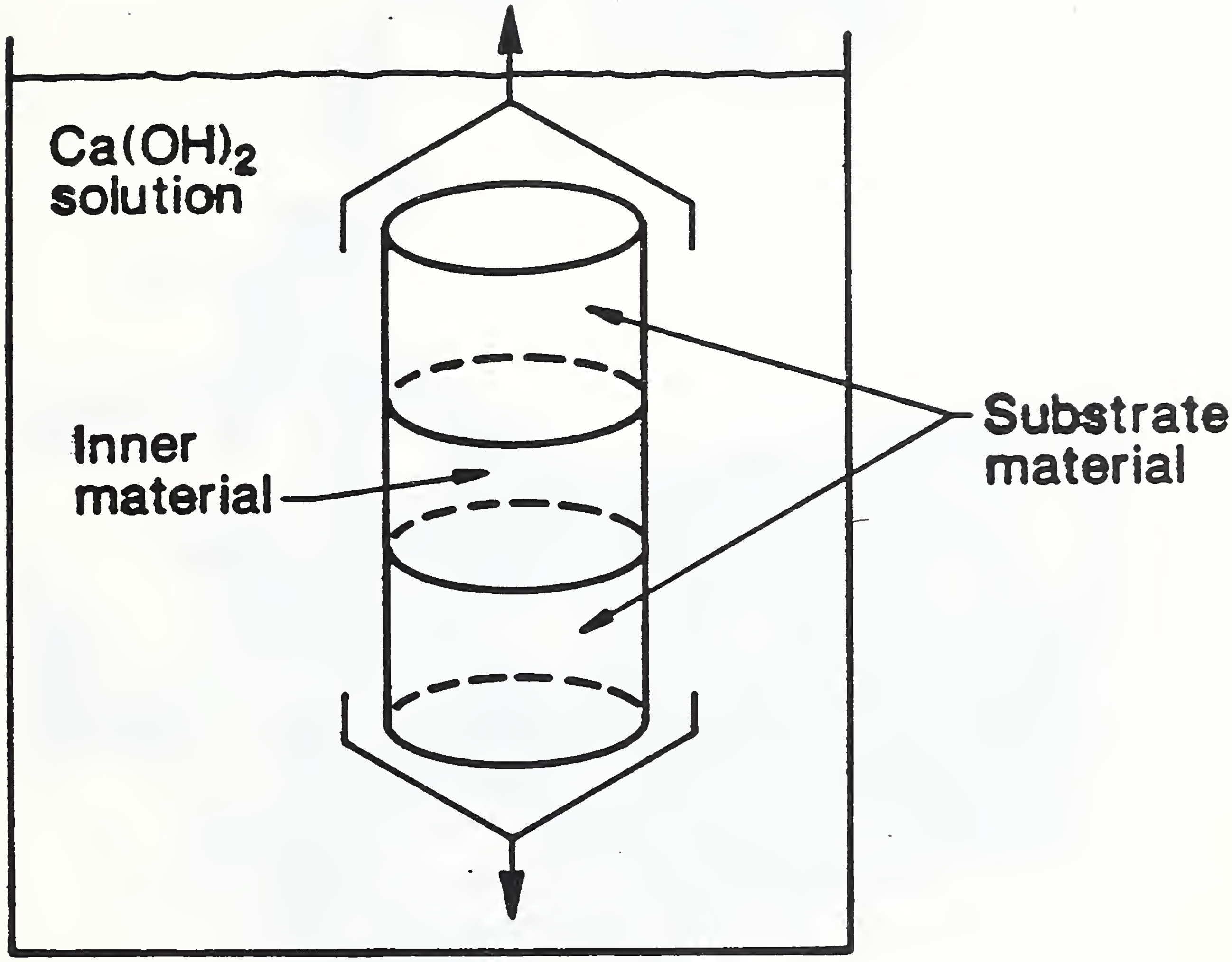


Figure 1. Schematic diagram of loading configuration, showing inner material (cement paste used as a model for cementitious repair material) cast between two cylinders of substrate material (cement paste used as a model for hardened concrete).

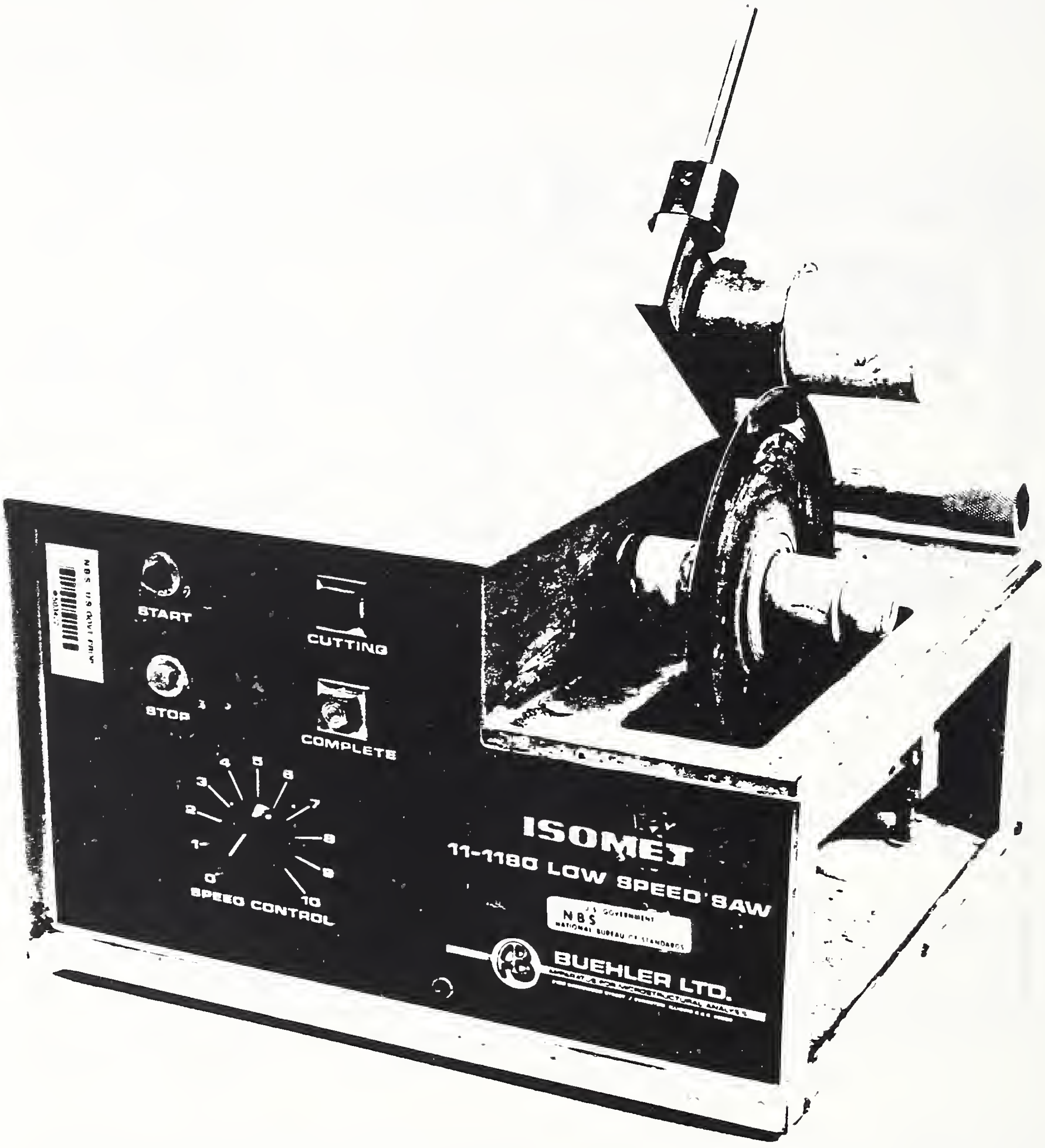
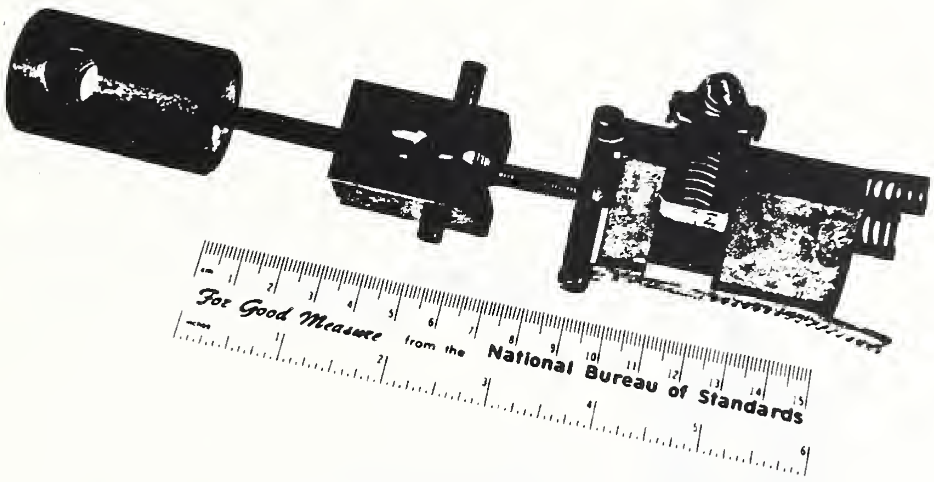
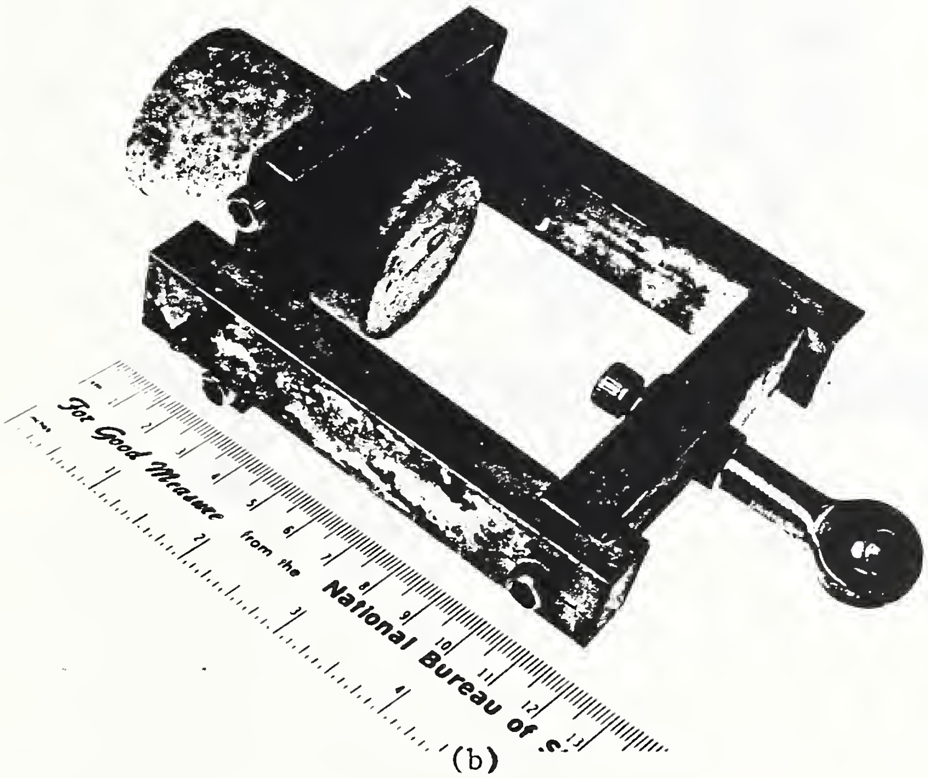


Figure 2. Saw and jig for preparing surface of substrate material.



(a)



(b)

Figure 3. Grips; (a) Grip No. 1, and (b) Grip No. 2.

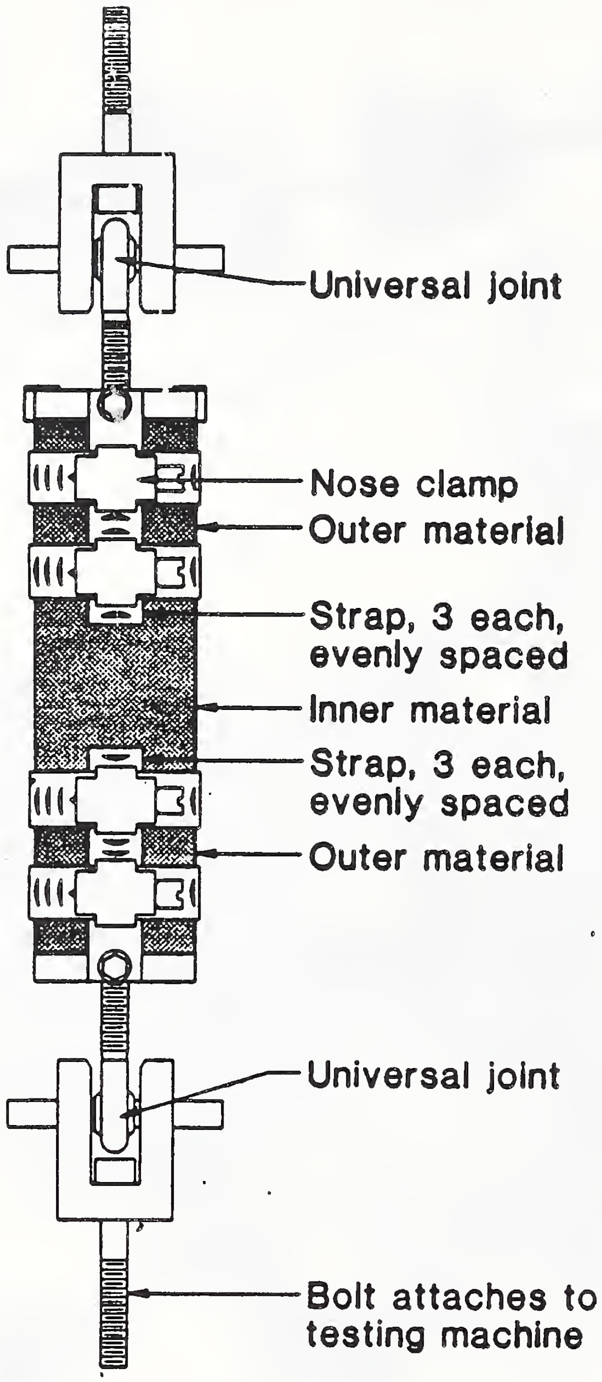


Figure 4. Schematic diagram showing Grip No. 1.

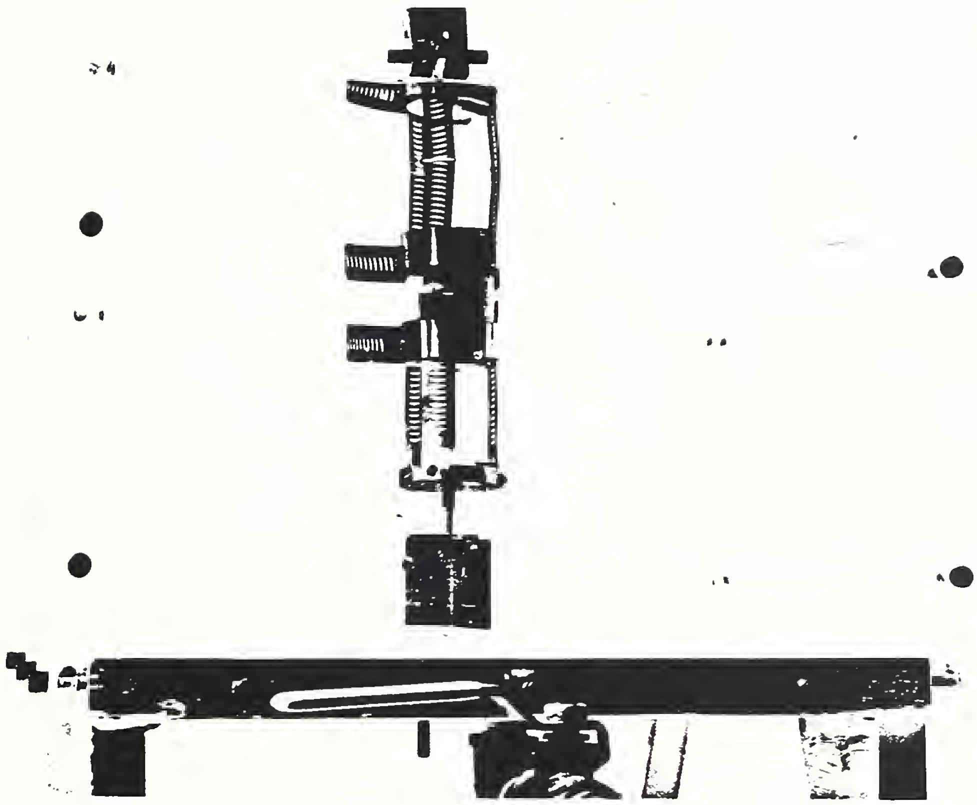


Figure 5. Tank for immersing specimens during tensile loading.

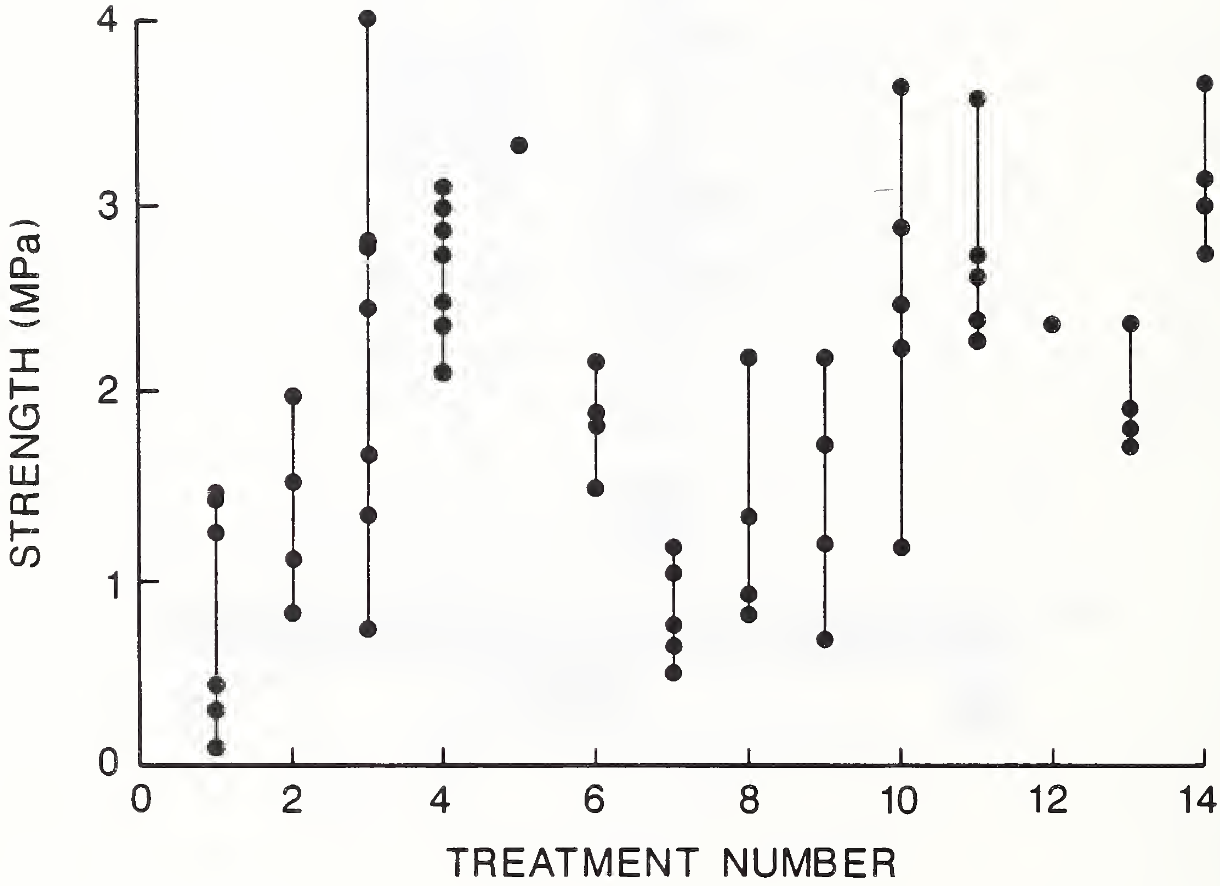


Figure 6. Tensile strength data for each of the 14 treatments.

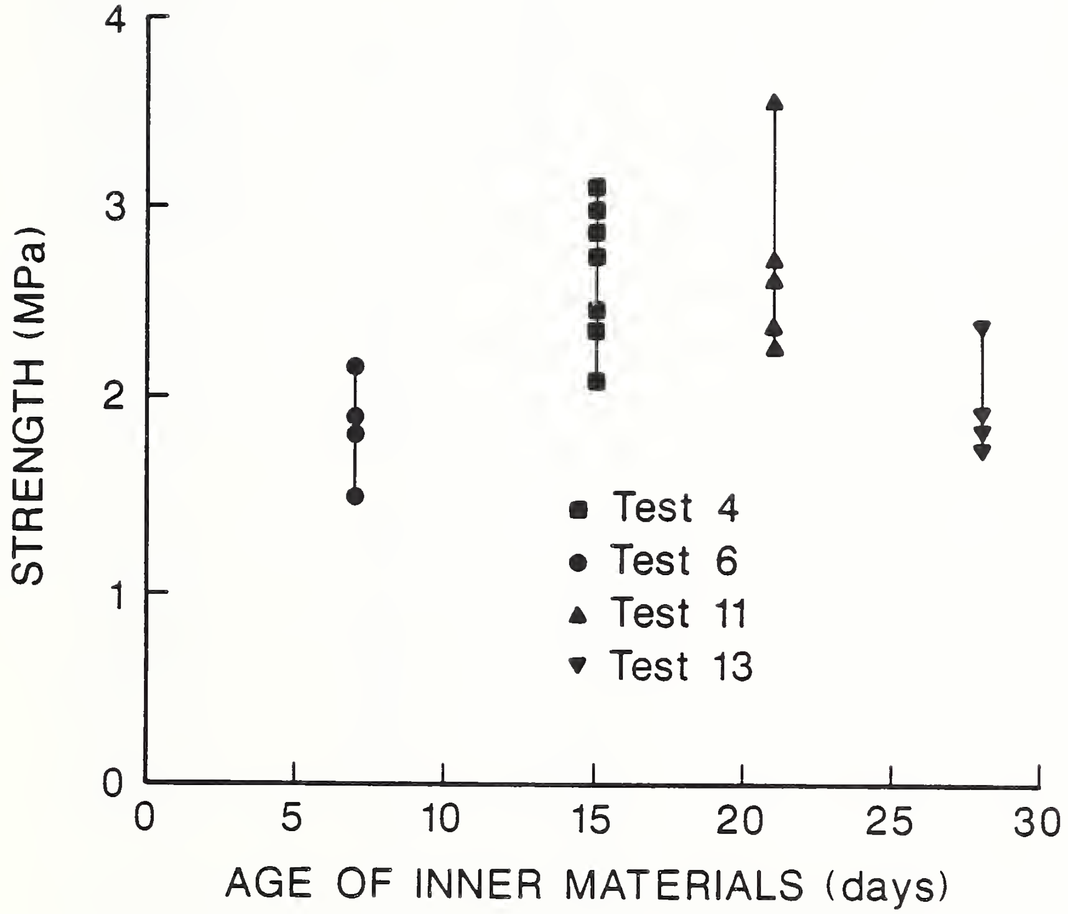


Figure 7. Tensile strength versus age of inner material for the 4 treatments determined to have no affect on strength level and variance.

Table 1. Summary of treatments.

Number	Sample (Inner)	Cutting Jig	Age (Days) Inner	Age (Days) Outer	Grip	Exposure Condition	Crosshead Speed (mm/min)
1	PASTE	1	30	42	2	IMMERSED	1
2	PASTE	1	31	43	1	IMMERSED	1
3	PASTE	1	14	28	1	IMMERSED	1
4	PASTE	2	15	22	1	IMMERSED	1
5	PASTE	2	15	22	1	IMMERSED	100
6	PASTE	2	7	14	1	IMMERSED	1
7	PASTE	1	7	14	1	IMMERSED	0.1
8	PASTE	1	15	22	1	IMMERSED	1
9	PASTE	1	15	22	1	IMMERSED	10
10	PASTE	1	14	21	1	IN AIR	1
11	PASTE	2	21	28	1	IMMERSED	1
12	PASTE	2	28	35	1	IMMERSED	20
13	PASTE	2	28	35	1	IMMERSED	1
14	GROUT	2	14	24	1	IMMERSED	1

Table 2. Tensile strength data.

Procedure Number	Specimen Number	Load (kg)	Strength (MPa)	Mean Strength (MPa)	Standard Deviation (MPa)	Coefficient of Variation (%)
1	S27-29	168	1.44	0.83	0.57	68
	S16-19	12	0.10			
	S35-37	171	1.47			
	S17-21	52	0.45			
	S31-39	145	1.25			
	S18-32	34	0.29			
2	S28-22	129	1.11	1.31	0.40	31
	S26-33	131	1.13			
	S30-40	95	0.82			
	S23-38	177	1.52			
	S34	230	1.98			
3	S42-54	325	2.79	1.97	0.78	40
	S43-52	285	2.45			
	S48-49	155	1.33			
	S47-56	330	2.84			
	S44-45	195	1.68			
	S41-53	85	0.73			
	S46-55	465	4.00			
4	S74-76	320	2.75	2.71	0.31	11
	S57-72	345	2.97			
	S62-75	350	3.01			
	S61-67	360	3.09			
	S59-66	320	2.75			
	S58-71	290	2.49			
	S63-64	275	2.36			
	S60-68	245	2.11			
	S69-73	335	2.88			
5	S65-70	385	3.31	3.31	--	--
6	S118-128	250	2.15	1.86	0.21	11
	S136-153	175	1.50			
	S130-137	215	1.85			
	S146-151	220	1.89			
	S142-154	220	1.89			
7	S78-85	60	0.52	0.83	0.24	29
	S91-103	120	1.03			
	S89-95	135	1.16			
	S84-92	75	0.64			
	S81-88	90	0.77			

(Table 2, cont.)

8	S96-101	105	0.90	1.32	0.49	37
	S77-110	95	0.82			
	S87-100	155	1.33			
	S102-108	155	1.33			
	S88-93	255	2.19			
9	S82-97	200	1.72	1.74	0.76	44
	S94-105	140	1.20			
	S80-90	80	0.69			
	S99-109	255	2.19			
10	S116-120	335	2.88	2.19	0.64	29
	S133-138	260	2.23			
	S134-144	135	1.16			
	S124-131	290	2.49			
	S117-119	425	3.65			
11	S129-143	415	3.57	2.72	0.45	17
	S123-126	280	2.41			
	S148-152	320	2.75			
	S127-141	305	2.62			
	S115-147	265	2.28			
12	S121-145	275	2.36	2.36	--	--
13	S122-125	200	1.72	1.94	0.25	13
	S139-140	210	1.81			
	S132-149	275	2.36			
	S135-150	220	1.89			
14	S190-203	350	3.01	3.14	0.33	10
	S176-197	425	3.65			
	S193-201	365	3.14			
	S198-202	320	2.75			
	S175-200	DID NOT BREAK AT INTERFACE				

Table 3. Values assigned to parameters for pairing test treatments.

Parameter	Value = 1	Value = 2
Sample (inner material)	paste	grout
Cutting jig	No. 1	No. 2
Grips	No. 1	No. 2
Age of inner specimen	<=18 days	>18 days
Age of outer specimen	<=26 days	>26 days
Crosshead speed	<=1 mm/min	>1 mm/min
Exposure condition	immersed	in air

Table 4. Treatments paired for statistical analysis.

Varied parameter	Numbers of Paired Treatments Test 1	Test 2	Parameter Values Test 1	Test 2
Age inner	2	3	31	14
Age outer	3	8	28	22
	3	7	28	14
Crosshead speed	8	9	1	10
	12	13	20	1
	7	9	0.1	10
	11	12	1	20
	5	6	100	1
	4	5	1	100
Cutting jig	2	11	1	2
	4	8	2	1
	5	9	2	1
	2	13	1	2
	6	8	2	1
	4	7	2	1
	6	7	2	1
Grips	1	2	2	1
Exposure condition	8	10	immersed	air
	7	10	immersed	air
Sample	4	14	paste	grout
	6	14	paste	grout

Table 5. Results of statistical analyses comparing each pair of treatments for variance (F-test) and mean (t-test).

Varied parameter	Treatment Pair Test 1	Test 2	Variance	Mean
Age inner	2	3	Equal	Equal
Age outer	3	8	Equal	Equal
	3	7	Not equal	Not equal
Crosshead speed	8	9	Equal	Equal
	12	13	---[a]	Equal[b]
	7	9	Equal	Equal
	11	12	---[a]	Equal[b]
	5	6	---[a]	Unequal[b]
	4	5	---[a]	Unequal[b]
Cutting jig	2	11	Equal	Unequal
	4	8	Equal	Unequal
	5	9	---[a]	Unequal[b]
	2	13	Equal	Unequal
	6	8	Equal	Equal
	4	7	Equal	Unequal
	6	7	Equal	Unequal
Grips	1	2	Equal	Equal
Exposure condition	8	10	Equal	Not equal
	7	10	Not equal	Not equal
Sample	4	14	Equal	Equal
	6	14	Equal	Not equal

[a]One treatment had a sample size of one, so this pair was not tested for equal variance.

[b]One treatment had a sample size of one, so its single value was compared to the mean of the other treatment.

Table 6. Summary of test parameters.

Parameter	Recommended procedur
Jig for cutting surface of substrate material	Jig No. 2
Grip	Grip No. 1
Crosshead speed	1.0 mm/min
Exposure Condition	Immersed
Age of inner material	Constant

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

A tensile test has been developed to measure the adhesion of repair material to hardened concrete using specimens of repair material cast between two cylinders of hardened substrate material. Cement paste was used as the model material for both the repair material and the hardened substrate. Aspects of the test procedure that appeared to affect the strength data included the jig used to cut the hardened substrate surface in preparing the composite specimen, the grips used during the tensile test, the crosshead speed, exposure condition during loading, and possibly the age of the repair material. The adhesion of cement paste to a hardened cement paste substrate was approximately 2.7 MPa, with a coefficient of variation of 15 percent. The test provides a method for measuring tensile strength of the bond between repair material and concrete substrate under controlled, laboratory conditions, for use in evaluating materials, surface preparation methods, and application procedures.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

adhesion, bond, cement, concrete, tensile strength, test method

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