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Performance Evaluation of Mechanical Pinning Repair of Sandstone

Marco J. Federico

University of Pennsylvania

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Advisor: Frank G. Matero

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Performance Evaluation of Mechanical Pinning Repair of Sandstone

Marco J. Federico

A THESIS
in
Historic Preservation

Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of the Requirements for the Degree of
MASTER OF SCIENCE IN HISTORIC PRESERVATION
2008

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1.0 Introduction

1.1 Research Objectives

The present of research seeks to analyze basic performance characteristics of pinning as a repair method of sandstone. Pinning, or the insertion of rods or other solid connectors, with or without adhesive, to rejoin or re-establish mechanical continuity of broken or potentially fractured stone is often necessary in the remedial treatment and conservation of historic masonry. On the most basic level, mechanical repairs are used to re-attach fragments and typically consist of an inert dowel or rod and adhesive. The performance characteristics of simple mechanical pinning can be best evaluated by considering the following:

- individual properties of the pin including physico-chemical stability,
- individual properties of the adhesive including rheology curing and set time, re-treatability, and bond strength,
- bond strength of the pin-adhesive system as measured by pull-out strength, and durability as measured by accelerated weathering.

1.2 Mechanical Pinning Of Cemetery Markers

Architectural conservators are concerned with mitigating the deterioration of historic fabric by conserving as much original fabric as feasible while making an intervention as minimally intrusive as possible. Blind, or concealed, pinning accomplishes this through the joining of fractured masonry by inserting one or more internal pins within the fractured adherends.
In the case of Trinity Episcopal Church, Pittsburgh, mechanical pinning was typically used to re-attach marble or sandstone slabs and headstones along the lines of breakage. What makes mechanical pinning so valuable as a conservation technique is the ability of the pins to redistribute loads and stress through an area much larger than the repair which utilizes a grout or adhesive as a means to join multiple fragments. Previous research has identified materials for pins and adhesives which possess optimal tensile and shear strength. The effects of the outdoor environment on such materials or material assemblies have been less studied. What still needs to be studied is the weathering response of these repairs once subjected to the elements and how this can potentially affect the critical and expected performance of these treatments.
1.3 Sandstone

Sandstone or “Connecticut brownstone” was selected as the stone for this study due to its common use for building facades and cemetery markers and its often extreme deterioration to natural weathering. At Trinity Church Burial Ground in Pittsburgh, Pennsylvania, the most problematic grave markers are those of a local argillaceous and ferruginous sandstone, displaying bedding delamination and fracturing from natural joints and extrinsic trauma (e.g. impact damage). These decay phenomena were addressed through both blind and face pinning as the preferred treatment strategy in 2007. As a result, these various methods were selected for subsequent testing to expand current knowledge on their durability over time through accelerated weathering.

1.4 Conservation Literature Review

Attempts to arrest the deterioration of sandstone have been a serious issue for beginning with the painting of the Acquia Creek sandstone in the early nineteenth century. But it has been the brown sandstone from the northeast belt, and especially the “brownstone” from the Portland-Middletown, Connecticut quarries that have become increasingly difficult to repair due to weathering and poor installation. (Matero & Teutonico. 1982) Being a sedimentary stone, sandstone can be characterized as a densely packed composition of sand, clays, and other accessory minerals. Most of the architectural sandstone seen today came from the vast quarries of Connecticut, New York, Pennsylvania, New Jersey and Ohio. It has suffered greatly from weathering as a result of intrinsic and extrinsic factors, the latter associated to its high demand in the nineteenth century. (Winkler 34, 1973)
Sandstone can fall victim to delamination, erosion, spalling, scaling, blistering and flaking. (Snethledge & Wendler, 1974; Grimmer, 1984) It has been carefully studied in terms of its ability to withstand weathering, yet still much more remains to be understood in its relationship to mechanical repairs. (Amoroso & Fassina 1983; Charola 2000; Paradise 2000)

Weathering cycles play a large role in determining the service life of architectural sandstone. Inter-granular bonds are what hold sandstone together. Movement of the grain structure causes damage to these bonds, and is typically a result of moisture intrusion. (Harris, 2001) Moisture can damage a stone in any number of ways ranging from the introduction of salts and their eventual crystallization to decay stemming from freeze-thaw cycling. In the case of many sandstones, and especially those containing a high clay content, water can cause the clays to expand, contract, and sometime rupture at the surface resulting in delamination, incipient spalling and eventual loss. Pinning will most likely be affected by this weathering and may in fact exacerbate such hygric and hydric responses.

Mechanical pinning for masonry conservation has been a subject little published when compared to consolidation and cleaning. It has long been used to re-attach broken sculptural elements and details. (Plenderleith, 1971) It has also been used for repairing architectural elements, as a means to re-attach spalls and loss, as well as for cemetery conservation. (Grimmer. 1984; Veterans Affairs Canada 2008; Cross 2005)

Pinning has been extensively studied as a method for treatment of the delaminations and incipient spalls of Pennsylvania Blue Marble at the Second Bank of the United States, (Glavan, 2004) and at El Morro National Monument. (Kreilick & Matero 1996) Pinning
has also been used in the construction industry as a means to secure a more visually aesthetic veneer stone or cladding to a more function masonry structural wall. Pins can be adhered with grouts or epoxy adhesives depending on the amount of stiffness required and the depth and volume of the void. (Prudon 1979)

While epoxy resins have demonstrated their strength and durability in the conservation of historic masonry, these applications are highly irreversible and if improperly treated or aggressively applied, their removal will almost certainly be more damaging to the historic fabric. Epoxy and polyester resins applied in the 1970s have subsequently yellowed and become brittle or not exhibited proper depth of penetration in the case of the First Bank in Philadelphia after treated by Gauri. (Selwitz 1995)

Both epoxy resins as well as acrylic resins such as Paraloid B-72 have been employed as adhesives, films, and consolidsents. Acrylic polymer resins were used at early as the 1930s in the form of picture varnishes under the proprietary name of Lucite 44 and 45. (Horie, 106, 1987) Later Paraloid B-72 was used in the field of conservation as an adhesive in 18- 20% concentrations relying on solvents such as xylene and toluene. Paraloid B-72 has become somewhat of an industry standard for the conservator for use as a reversible resin. It is stiffer and more durable than rubber but lacks the high stiffness and strength found in many epoxies. (see chart 1.1) Available in pellet form, it is dissolved in a wide range of solvents, yet the solvent selected can affect its performance.(Hansen 1991) Certain solvents such as tricholoethane and xylene are effective in dissolving B-72, but they are extremely toxic. While their use in a controlled laboratory setting can be executed safely, their use the field setting would be more difficult.
Table 1.1 Physical Properties of Polymers. (Horie, 22, 1987)

Other considerations for solvent selection include the solvent’s evaporation rate from the adhesive. For example, acetone evaporates faster than toluene, but does not result in the same bond strength. (Podany et al, 1, 2001) The converse must also be stated when considering at reversibility; that total immersion of an adhesive bond in a certain solvent will result in a more expedient dissolution.

The testing program outlined in this thesis relies on published standards, independent studies, conferences, and graduate theses. Weathering is best analyzed through observation of existing conditions on structures, grave markers, and sculpture or through accelerated weathering in a laboratory environment. When observing mechanical failure in a cemetery, the result is often discoloration, loss of adhesive or bond strength from embrittlement or associated damage to the original stone.
Aggressive repairs in earlier restoration campaigns often damaged monuments through the boring of deeper holes into markers and attempts to reinforce them with corrosive ferrous pins.

The analysis of a treatment’s response to environmental weathering can be a difficult task given the complexity of agents and factors responsible. Variables in the environment and their cycling time favor accelerated testing in a laboratory environment. Since moisture intrusion is one of the most common and damaging agents of stone decay, the American Society of Testing Material (ASTM) as well as The International Union of Laboratories and Experts in Construction Materials, Systems and Structures (RILEM) have published a variety of test standards for performance evaluation related to accelerated moisture exposure.
2.0 Preliminary Testing

2.1 Overview

The development of the testing protocol required two phases. The first or preliminary phase involved the identification of the optimal Paraloid B-72 adhesive formulation. Three series of tests were performed on solutions of B-72 adhesive and sought to find the ideal type and ratio of solvent and filler. The first test, the injection simulation test, sought to evaluate the working time, set time, and injectibility of the adhesive to flow into a hole and around the pin. The second test, the flow test, sought to assess the rheology, or flow of the pin on a smooth vertical surface. The third test, for cure time, sought to assess hardness over time by injecting the adhesive on a coupon of sandstone and leaving it to cure in open air at room temperature. The final outcome of this segment of preliminary testing was the selection of one formulation of Paraloid B-72 for use in fabrication of assemblies later evaluated through accelerated weathering and pull-out testing.

A wide range of solvents have successfully been utilized with Paraloid B-72, yet each type displays different advantages and disadvantages. Different fillers also impart different properties to B-72 solutions. Some fillers aid in achieving ideal thixotropy while others result in an increase in strength. These tests also sought to examine the effect of fillers in different ratios noting how they reacted with the respective solvents once combined with the Paraloid B-72.

The preliminary tests were undertaken in the laboratory with the intention of evaluating the following performance characteristics:
- thixotropy - an adhesive’s ability to be used as a gel or paste, and then upon agitation to become liquid. An ideal injectable adhesive will be thixotropic.

- viscosity - an adhesive’s viscosity is crucial to the mode by which it is applied. An adhesive which is too viscous may prove difficult to inject as would be the case when using it in a pinning application. It must be of a low enough viscosity so that it may flow around the pin.

- set and curing time - the time needed for an adhesive to set and cure can affect the work schedule and sequence of treatments that may follow as well as the viability of that adhesive during certain weather conditions.

- reversability - a treatment which can be retreated or reversed is always ideal in conservation, but may not always be attainable. While the adhesive may be removed through solvents, heat, or other means, a hole drilled into an adherend is permanent.

- low toxicity - VOC laws and EPA regulations have rendered some treatments too hazardous to be implemented. Health risks to the conservator should be avoided or mitigated with proper safety precautions as per OSHA specifications.

- bond strength - the strength of a bond must be great enough to re-establish the continuity of the fragments, but formulated so that its tensile and shear strength is less than that of the cohesive strength of the stone. A break in the bond components (i.e., pin or adhesive) would be preferred to a new break in the stone adherends.
2.2 Methodology for Formulation of Paraloid B-72

Toluene, acetone, and ethanol were all evaluated as potential solvents to be formulated with B-72 for the final assemblies given their reported success in dissolving B-72. Acetone and ethanol evaporate more quickly than toluene and were preferred due to their faster cure time. Fillers, to strengthen and thicken the formulation were also evaluated: fumed silica (Cabosil) and marble dust (Calcium Carbonate)(Kremer).

Two different methods were employed to prepare the B-72. (Koob 1986.) 100g of Paraloid B-72 pellets were suspended in cheesecloth in a sealed jar with 100g of solvent. After the solution appeared to be of uniform consistency, the cheesecloth was strained of the remaining adhesive and removed. This was performed after 48 hours, with the solution being agitated with a tongue depressor after the 24 hour mark was reached. The second method involved doubling the amount of solvent, adding 5% filler by weight, and again, suspending the Paraloid B-72 pellets by cheesecloth and leaving the lid of the jar open until half the solvent evaporated. This allowed the B-72 pellets to be completely submerged in solvent, greater facilitating dissolution while later achieving the desired weight ratio after the excess solvent evaporated. It also allowed the filler to be more evenly dispersed in the solution. Fillers are commonly added in the field after the initial B-72 formula has been created, and often cannot be effectively dispersed into the solution due to the pot life of the adhesives.

2.2.1 Observations

In formulating the B-72 adhesive, there are readily discernible differences in the way that the solvents behave. The toxicity of toluene makes its handling cumbersome.
Using acetone and ethanol as solvents does not remove these safety requirements but the toxicity risks are less.

Upon pouring the acetone and acetone/toluene mixture into the jars with the suspended cheesecloth of B-72 pellets, after 30 seconds, one could see the pellets begin to dissolve quickly. When the toluene alone was added to the jars, the pellets dissolved more slowly. Visually observing the different solutions after the initial 24 hours, the jar containing toluene appeared discernable thicker on the bottom and thinner at the top of the jar, lacking a uniform consistency.

2.3 Injection Simulations

The injection simulation test used test tubes, aluminum injection ports, and fiberglass dowels. The primary objective of this test was to gauge the injectability, working time, and distribution of the adhesive around the pin. The advantage of injecting into a glass test tube is that one has the opportunity to observe the distribution of the adhesive flow around the pin. The presence of air bubbles can also be observed as well as whether there has been settling or segregation of the filler in the adhesive. The most crucial aspect of this test is that it confirms that the pin is fully seated in the adhesive-free of voids and discontinuities, and also that the pin is seated at uniform distance from the edges.
The injection ports were fabricated at the Fabrication Laboratory, School of Design, University of Pennsylvania. They were milled on a metal lathe using carbide-tipped cutters.

![Figure 2.1. Injection Port and Fluorosilicone Washer. Photo by author](image)

The purpose of the injection ports is twofold. First it allows the adhesive to be injected via a syringe through a canula, or needle, while simultaneously allowing the air in a given hole to escape. Since the neck of the canula is square tapered and the aperture of the injection port is rounded, the port allows air from the hole to escape on either side of the canula’s neck. Second, the injection port ensures that the pin or dowel stays
centered within the adhesive-filled hole during set. The edge of the flanged neck is milled so that it fits snugly around the outer edge of the hole (or test tube in this case). Fitted over the flanged neck of the injection port is a solvent resistant fluorosilicone washer which serves to eliminate airflow from the sides surrounding the hole and acts a release gasket should the adhesive flow out.

The injection port is placed into the test tube and a calculated amount of adhesive is injected into the hole. After the adhesive has been injected (4 ml), the syringe is withdrawn. Fiberglass rods are then cut with a hacksaw to the length of 3 inches. The pin is inserted into the hole, kept perpendicular to the surface and parallel to the outer walls of the tube by the injection port.

### 2.3.1 Formulae

<table>
<thead>
<tr>
<th>SAMPLE</th>
<th>PARALOID B-72</th>
<th>TOLUENE</th>
<th>ACETONE</th>
<th>ETHANOL</th>
<th>FUMED SILICA</th>
<th>CALCIUM CARBONATE</th>
</tr>
</thead>
<tbody>
<tr>
<td>AC1</td>
<td>1</td>
<td>--</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>AC2</td>
<td>1</td>
<td>--</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>AC3</td>
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<td>--</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>TN1</td>
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<td>1</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>TN2</td>
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<td>1</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>TN3</td>
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<td>--</td>
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<td>--</td>
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<tr>
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<td>0.4</td>
<td>0.05</td>
<td>--</td>
</tr>
<tr>
<td>AE3</td>
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<td>0.6</td>
<td>0.4</td>
<td>0.05</td>
<td>0.02</td>
</tr>
<tr>
<td>AE4</td>
<td>1</td>
<td>--</td>
<td>0.6</td>
<td>0.4</td>
<td>0.05</td>
<td>0.05</td>
</tr>
</tbody>
</table>

Table 2.1: Injection Simulation Formulae
2.3.2 Observations

*AC series*

Samples AC1, AC2, and AC3 were drawn up into a 30 cc syringe with relative ease. The adhesives were easily dispersed and flowed evenly to the base of the test tube uniformly seating the pins with no gaps or bubbles. After 4 hours all of the test assemblies displayed settling of the calcium carbonate filler out of the adhesive solution; a 1/4” layer of filler was deposited at the base of the test tube. After 6 hours the adhesive had not yet fully hardened but the pins remained at a 90 degree angle without the aid of the injection port. After 12 hours time the pins could still be flexed within the adhesive. After 24 hours the adhesive appeared to have set.

*TN Series*

Samples TN1, TN2, and TN3 were drawn up into the syringe with slight difficulty. After the first injection, the syringe could no be re-used as the toluene in the solution swelled the rubber stopper within the syringe, compromising the airtight seal needed to draw up the adhesive. A new syringe was needed for each injection. Dispersal of the adhesive also proved difficult as the solution appeared to have an uneven consistency, even after 96 hours of the Paraloid B-72 pellets dissolved. It was noted that the toluene B-72 formula, samples TN1, TN2 and TN3, were of a more varied consistency, ranging from thin and runny to viscous and gelatinous. All of the other
admixtures displayed the same consistency. This could be due to the solvent’s rate of evaporation. The adhesive flowed poorly around the pin and cured in such a way that it was up to 2 inches higher on one side of the test tube wall. After 72 hours, it still had not fully cured. After one week’s time, the injection port was able to be removed. These samples also exhibited settling of the filler as with the AC series. A ¼ inch deposit of filler was found at the base of all 3 test assemblies.

**AE Series**

The primary objective of this last set of test assemblies was to determine the appropriate filler or combination of fillers to thicken the unbulked adhesive. AE1 contained fumed silica at 2% by weight in proportion to the solvents and solute. Upon mixing and injection it displayed better dispersion than calcium carbonate but did not have the ideal viscosity. It flowed evenly around the pin leaving no air bubbles or discontinuities. After 8 hours, the adhesive had cured to the point of allowing the injection port to be removed. After 24 hours, the sample had appeared to set. There was no settlement at the bottom of the test tube.

AE2 was bulked with 5% fumed silica. Once again the silica proved to disperse better into the solution than the calcium carbonate. It flowed evenly around the pin but left several air bubbles towards the base of the test tube. After 8 hours, the adhesive had not yet cured to the point of allowing the injection port to be removed. After 12 hours of time had elapsed the injection port was removed without compromising the seating of the pin. After 24 hours, the sample had appeared to set. There was no settlement at the bottom of the test tube. This admixture was more opaque than AE1.
AE3 was bulked with 5% fumed silica and 2% calcium carbonate. It was drawn up easily into the syringe and was also discharged with relative ease. It flowed evenly around the pin leaving no air bubbles or discontinuities. After 8 hours, the adhesive had not yet cured to the point of allowing the injection port to be removed. After 12 hours, the injection port could be removed, but it was not until 24 hours that the adhesive appeared to set. There was no filler segregation at the bottom of the test tube. This admixture exhibited the ideal viscosity.

AE4 was bulked with 5% fumed silica and 5% calcium carbonate. The adhesive was drawn up into the syringe with relative ease. It completely seated the pin and left no apparent air bubbles or discontinuities. After 12 hours, the injection port could be removed, but it was not until 24 hours had elapsed that the adhesive appeared to set. There was no filler segregation at the bottom of the test tube. This admixture exhibited the ideal viscosity.
2.3.3 Conclusions

Base on the above observations, the fumed silica appears to keep the marble dust in suspension. The admixtures which used larger quantities of filler all exhibited a more desirable injectability and viscosity. The long cure times could possibly be attributed to the impermeable qualities of the glass vials, not allowing the solvent to evaporate easily. In a porous stone such as sandstone, the solvents will presumably evaporate more quickly, accelerating cure time in comparison to these preliminary test assemblies.

2.4 Flow Tests

2.4.1 Objectives
The primary objective of the Flow Test was to assess the rheology, or flow, of different adhesive admixtures. (Bass 1998) The tests were performed on a flat, smooth, nonabsorbent surface. The adhesive was discharged in 1.5 milliliter droplets and then the board was turned vertical. These tests permit multiple adhesives to be assessed quantitatively through the measurements marked on either end of the test board. Qualitative assessments can also be made in terms of whether the adhesive flowed straight or whether it bubbled up in certain area and if the drips appear to be of a uniform consistency.

Figure 2.3 Flow tests with board upright. Photo by Author.
### 2.4.2 Formulæ

<table>
<thead>
<tr>
<th>Sample</th>
<th>Paraloid B-72</th>
<th>Toluene</th>
<th>Acetone</th>
<th>Ethanol</th>
<th>Fumed Silica</th>
<th>Calcium Carbonate</th>
</tr>
</thead>
<tbody>
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<td>1</td>
<td>0.5</td>
<td>0.5</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>TA2</td>
<td>1</td>
<td>0.5</td>
<td>0.5</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>T1</td>
<td>1</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>T2</td>
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<td>--</td>
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<td>0.02</td>
</tr>
<tr>
<td>A1</td>
<td>1</td>
<td>--</td>
<td>1</td>
<td>--</td>
<td>--</td>
<td>0.02</td>
</tr>
<tr>
<td>A2</td>
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</table>

Table 2.2. Flow Test Formulæ
2.4.3 Observations

The acetone based adhesives performed the best. Good performance is generally characterized as flowing no more than 5 inches over the span of 45 seconds. The toluene and acetone adhesive formulas performed as well as the acetone and ethanol adhesive formulas. This is noteworthy because the acetone and ethanol formula contained up to 20 times the amount of filler, both used calcium carbonate as a bulking agent. Toluene alone flowed irregularly and took up a larger surface area while running down the length of the test board. Acetone alone had mixed results, A1 flowing all the way down to 6”, A2 stopping at the 5” mark. The acetone ethanol formula which flowed to a length of 4” appeared to perform well in terms of its rheology; however the mixture which used silica as a filler displayed tiny air bubbles.

2.4.4 Conclusions

The adhesives bulked with fumed silica and calcium carbonate, appeared to exhibit a desirable viscosity, however the resulting air bubbles may compromise the strength of the adhesive. It is known that fine calcium carbonate imparts good strength to Paraloid B-72 resin solutions (Glavan, 74, 2004) Toluene based adhesives flow too irregularly and may take considerable time to cure if the solvent is not fully evaporated.

2.5 Hardness and Cure Time

2.5.1 Objectives

This series of tests gives qualitative data which helped to assess hardness of the adhesive over a 3 week cure time. 1.5 ml of adhesive was placed onto a 2”x 2” sandstone...
surface and then given a fixed amount of time to cure, 14 days. The drops of adhesive were sliced open with a razor to judge hardness, relative density and cure time.

2.5.2 Formulae

<table>
<thead>
<tr>
<th>Sample</th>
<th>Paraloid B-72</th>
<th>Toluene</th>
<th>Acetone</th>
<th>Ethanol</th>
<th>Fumed Silica</th>
<th>Calcium Carbonate</th>
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<td>0.6</td>
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<td>EAS2</td>
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<td>0.6</td>
<td>0.4</td>
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<tr>
<td>EAS3</td>
<td>1</td>
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<td>0.6</td>
<td>0.4</td>
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<tr>
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<td>0.6</td>
<td>0.4</td>
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</tr>
<tr>
<td>EAM1</td>
<td>1</td>
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<td>0.6</td>
<td>0.4</td>
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<td>EAM2</td>
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<td>0.4</td>
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Table 2.3 Hardness and Cure Time
### 2.5.3 Observations

#### Hardness and Curing Time: Observations 1

<table>
<thead>
<tr>
<th>Sample</th>
<th>Time Elapsed (Days)</th>
<th>Moh Hardness</th>
<th>Facility in Cutting</th>
<th>Description of Cross-section</th>
<th>General Observations</th>
<th>Fumed Silica</th>
<th>Calcium Carbonate</th>
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<tr>
<td></td>
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<td>Sliced Easily</td>
<td>Very porous, bubbles of</td>
<td>Skin has hardened,</td>
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<td>void in center</td>
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<td>EAS3</td>
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<td>Slight Resistance</td>
<td>Very porous, bubbles of</td>
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<td>surface but still</td>
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<td>Adhered well to</td>
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**Table 2.4.1**
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<th>Moh Hardness</th>
<th>Facility in Cutting</th>
<th>Description of Cross-section</th>
<th>General Observations</th>
<th>Fumed Silica</th>
<th>Calcium Carbonate</th>
</tr>
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<tbody>
<tr>
<td>EAM1</td>
<td>1</td>
<td>0.2</td>
<td>Sliced Easilly</td>
<td>Paste-like</td>
<td>Skin has hardened, interior still paste-like. Large void in center</td>
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<tr>
<td>EAM2</td>
<td>5</td>
<td>0.5</td>
<td>Sliced Easilly</td>
<td>Very porous, bubbles of varying size</td>
<td>Skin has hardened, gum-like. Large void in center</td>
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<tr>
<td>EAM3</td>
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<td>0.8</td>
<td>Slight Resistance</td>
<td>Slightly porous, bubbles of varying size</td>
<td>Adhered well to surface but still gummy</td>
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</tr>
<tr>
<td>EAM4</td>
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<td>Some Resistance</td>
<td>Slightly porous, bubbles of varying size</td>
<td>Adhered well to surface but still gummy</td>
<td>--</td>
<td>0.05</td>
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<td>EAM5</td>
<td>17</td>
<td>1</td>
<td>Some Resistance</td>
<td>Slightly porous, bubbles of varying size</td>
<td>Slightly difficult to remove from substrate. Very flexible</td>
<td>--</td>
<td>0.05</td>
</tr>
<tr>
<td>EAM6</td>
<td>21</td>
<td>1.5</td>
<td>More Resistance</td>
<td>Pores have hardened</td>
<td>Difficult to remove from substrate. Very flexible</td>
<td>--</td>
<td>0.05</td>
</tr>
<tr>
<td>EAM7</td>
<td>7</td>
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<td>Some Resistance</td>
<td>Small and larger pores, slightly tacky</td>
<td>Adhered well to surface but still gummy</td>
<td>--</td>
<td>0.2</td>
</tr>
<tr>
<td>EAM8</td>
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<td>1.8</td>
<td>More Resistance</td>
<td>Small and larger pores, slightly tacky</td>
<td>Adhered well to surface but still gummy</td>
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Table 2.4.2
# Hardness and Curing Time: Observations III

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<th>Time Elapsed (Days)</th>
<th>Moh Hardness</th>
<th>Facility in Cutting</th>
<th>Description of Cross-section</th>
<th>General Observations</th>
<th>Fumed Silica</th>
<th>Calcium Carbonate</th>
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</thead>
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<tr>
<td>EAM9</td>
<td>21</td>
<td>2.5</td>
<td>Difficult</td>
<td>Pores have hardened</td>
<td>Slightly hard around edges. Hardness of rubber</td>
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<tr>
<td>EAM10</td>
<td>7</td>
<td>0.5</td>
<td>Some Resistance</td>
<td>Small and larger pores, slightly tacky</td>
<td>Slightly hard around edges.</td>
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<td>0.4</td>
</tr>
<tr>
<td>EAM11</td>
<td>14</td>
<td>1.8</td>
<td>More Resistance</td>
<td>Pores have hardened</td>
<td>Harder on edges, slightly flexible</td>
<td>--</td>
<td>0.4</td>
</tr>
<tr>
<td>EAM12</td>
<td>21</td>
<td>2.5</td>
<td>Difficult</td>
<td>Some small Pores still visible</td>
<td>Harder on edges, Difficult to pull apart</td>
<td>--</td>
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</table>

**Table 2.4.3**

### 2.5.4 Conclusions.

These tests corroborate the previous preliminary testing that maximum strength, hardness, and optimum cure time is found in the EAM series of adhesives which use the 60/40 mixture of acetone and ethanol, respectively, combined with 0.4 fumed silica and 0.4 parts calcium carbonate. This mixture yields the most desirable adhesive for the purpose of mechanical pinning for conservation standards. It displayed maximum hardness after 21 days, and exhibited good consistency.
3.0 Repair Criteria and Experimental Treatment Design

3.1 Overview

Fragmentation of grave markers in historic cemeteries is a significant problem affecting the material integrity of the stone as well as the historic integrity and character of the cemetery as a whole. Grave markers typically fracture as a result of many causes. Vegetation and falling branches can displace, dislodge and break stones; if a falling branch does not crack the stone, the force of one marker upon another can potentially cause damage. Vandalism, particularly prevalent but not limited to urban cemeteries, can be a major factor, as many cemeteries are not gated or patrolled.

Deformation through poor design can also be problematic, because it is a result of the stone’s inherent qualities or construction. Excessively ornamented or tall slender designs may lack the structural or tensile strength to withstand the effects of weathering. Sandstone is particularly susceptible to weathering and freeze-thaw cycling as it is often times placed with the edges of the bedding directly into the ground. This allows moisture to rise up easily into the stone bringing with it impurities such as salts, which can lead to spalls, fractures, and detachment.
3.2 Use of Mechanical Pinning as a Remedial Conservation Treatment

Fragmentation from delamination most frequently occurs across the bedding planes of a sedimentary stone in a cemetery context. To remedy fragmentation, mechanical pinning is the most effective method in terms of durability, cost, and ease of application. Usage of structural grouts and adhesives can reinforce mechanical pinning treatments. Blind pinning allows the repair to go unnoticed if properly implemented, which should be the standard for all conservation repairs. Typically, a hole is drilled into the broken surface of both adherends and filled with an adhesive, a pin is placed inside of equal length of the combined drilled holes, and the entire assembly is then secured under compression using clamps until the cure time of the adhesive has been exceeded.
Face pinning can be used to re-attach or secure thinner detachments or incipient spalls that are still attached and requires drilling through the face of the spall into the parent adherend. Blind pinning requires drilling into the ends of broken fragments making the repair less noticeable as well as minimizing damage to the fragment. Mechanical pinning is effective because it allows for a connection to be made which is referred to as a load transfer mechanism. (Glavan, 88, 2004) Load transfer lends tensile reinforcement through the transfer of stresses from the broken fragments through the pin and adhesive. Sleeves and dry-fit friction systems such as Heli-fix™ require a pin to be tightened in the hole. This would be impossible in the case of blind pinning as the morphology of the breaks would have to be planar and level and the two substrates would have to be turned into place. This type of break would never occur in two stone
fragments, unless they were intentionally separated using a saw thus providing an even and planar break perfectly perpendicular to the base of the stone.

3.3 Conservation Principles in Mechanical Pinning

Professional conservators in the United States are bound to follow the principles set forth by the American Institute for Conservation’s (AIC) code of ethics. This presents a unique set of criteria that must be met in designing and executing treatments. As the masonry contractor seeks out a repair which ensures strength and durability, the conservator seeks to design solutions that satisfy these criteria while also ensuring compatibility, minimum intervention, and reversibility or retreatability. Repairs made must recognize the significance of the cultural property being treated, and acknowledge that repairs made may need to be removed in the instance that a superior technology or system becomes available. Since the most successful blind pinning repairs are undetectable from the surface, it makes no visual impact and does not detract from the visual character or integrity of the restored marker. Blind pinning, if properly implemented, makes the most minimal intervention while restoring structural integrity to a fragmented marker. Ideally, the least number of required pins are used, as drilling can potentially damage the stone. However; pins must be installed in a manner that distributes the load across the break. Reversibility is perhaps the most discussed principle in conservation, but can also be the most difficult criterion to satisfy. While conservators can use inert pins with appropriate thermal coefficients relative to the stone and acrylic adhesives that can be re-dissolved, it is impossible to un-drill a hole. Material fabric has been lost. Potential micro-cracks that have been formed and the seepage of
adhesive into the stone cannot be easily reversed. Reversibility, re-termed as retreatability, is a more attainable goal. Retreatability implies irreversibility of any repair, and that removability of the repair should be attainable without inordinate damage to the said cultural property.

3.4 Repair Criteria and Material Compatibility

Compatibility is perhaps the most significant requirement for long term durability. One of the most common incompatible repair methods found in cemeteries is with the use of ferrous pins for pinning. Ferrous pins can result in two significant problems: corrosion jacking and cracking from the volumetric expansion of the pin from iron oxide corrosion. Most metallic pins (except for titanium) also have very different thermal coefficients of expansion and contraction when compared to stone which can result in cracking and loss. An ideal pin is chemically inert (i.e. non corrosive) and possesses a similar thermal coefficient as the stone. Pins should have good tensile strength or modulus of elasticity.

Adhesives meet the criteria outlined in Section 2, which can be summarized as exhibiting good thixotropic properties, reasonable cure time, good bond strength, low toxicity and reversibility.
3.5 Material Selection.

3.5.1 Pins

Based on research and independent field work, Conservepoxy’s Fiberglass Rebar 800™ was selected for evaluation for the Phase 2 confirmatory tests. These pins consist of continuous drawn glass roving saturated with vinyl ester resin. A single strand of glass fiber circumscribes the exterior diameter providing a spiral indentation in the pin providing maximum bonding due to increased surface area. The rods have a gritted surface finish, providing additional increased surface area. The larger surface area provides more points of contact for the adhesive to bond with the pin. Fiberglass Rebar 800 is corrosion resistant, non-conductive and lightweight; it is one-fourth the weight of steel rebar. It can be easily cut with a bimetal hacksaw blade and available at prices comparable to stainless steel and nylon, but not as costly as alumina pins.

Rebar 800™ should function well as part of the load transfer mechanism needed for the repair of fragmented masonry when used with an adhesive. In the field, its chemically inert properties and its thermal coefficient suggest it will not corrode, will volumetrically expand less than the stone at the same temperature, and will not cause staining. The thermal coefficient of sandstone is $6.1 \times 10^{-6}$ in/in/ºF. The thermal coefficient of Conservepoxy’s Rebar 800™ is $5.5 \times 10^{-6}$ in/in/ºF. In the event that the repaired stone experiences low level additional loads or stress from external factors, the fiberglass rebar will give allowing the adhesive to break and will return to its previous position prior to loading. Under larger loads, these pins will cause less damage than more rigid, stronger pins such as those of stainless steel or titanium which will cause the stone to spall and shatter.
Table 3.1: Material Characteristics of Pins

<table>
<thead>
<tr>
<th>Pin Material</th>
<th>Tensile Strength Psi.</th>
<th>Coefficient of Thermal Expansion (10^-6) Psi.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stainless steel grade 304</td>
<td>85,000</td>
<td>9.2</td>
</tr>
<tr>
<td>Stainless steel grade 316</td>
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<td>9.2</td>
</tr>
<tr>
<td>Titanium Grade 2</td>
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<tr>
<td>Rebar800 Fiberglass</td>
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<td>5.5</td>
</tr>
<tr>
<td>Nylon 6/6</td>
<td>11,500</td>
<td>80</td>
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</table>

3.5.2 Adhesives

The adhesive is a critical component in pinning repairs, acting as a bridge between the adherend and pin, completing the load transfer mechanism. The two adhesive resins tested in this thesis are Paraloid B-72 and Sika Anchor Fix-4. Both are synthetic organic resins but possess very different properties. Paraloid B 72 is a thermoplastic polymer. Thermoplastics generally do not crosslink, meaning that the long strands of monomer chains attach one monomer to the subsequent monomer in a two dimensional sequence. They are not held together by formal chemical bonds, they flow past each other, thus allowing them to be dissolved by solvents and melt when heated. (Horie, 12, 1987) The Sika Anchor Fix-4 is an epoxide resin and a thermoset which forms three dimensional monomer chains when heated. These bonds are formal
chemical bonds which are not easily broken between the monomers. Epoxy resins cure through an exothermic reaction made when the hardener comes into contact with the resin. They are stiffer and more durable than thermoplastics. These chains cannot be undone when heated or put in contact with solvents.

The glass transition temperature of an adhesive determines its stiffness once it has set and cured. Thermoplastics have a lower glass transition temperature than thermosets, ultimately determining their performance characteristics in terms of modulus of elasticity and tensile strength.

3.5.2.1 Sika AnchorFix-4®

Sika AnchorFix-4® is a two part system which is sold in disposable 22oz. caulk tubes and mixed at the nozzle of the caulk gun from which they are dispensed. Bulking agents have been added to the hardener and resin to give it the desired thixotropy and rheology. Some epoxy resins such as manufactured by West System, allow the user to bulk the final mix to the ideal density. For the purposes of meeting testing parameters, Sika AnchorFix-4® is capable of holding a pin upright suspended in the adhesive. It has a higher glass transition temperature than Paraloid B-72 which explains why it is stiffer at room temperature. Its higher glass transition temperature accounts for its higher modulus of elasticity and tensile strength.

3.5.2.2 Paraloid B-72

Paraloid B-72 is an acrylic-based adhesive manufactured by Rohm and Haas. It is based on a poly methyl methacrylate and a methyl methacrylate copolymer. It is a general
purpose resin with high flexibility. Typically purchased in granular or pellet form, it is also available in pre-made paste form. It has been an effective adhesive with wide variety of applications and used in conservation since the 1930s in its earlier manifestation. Its uses range from commercial varnishes, to a fixative for adhering paper and canvas, to conservation purposes as a consolidant, coating, and structural adhesive. Its most attractive feature is its UV light stability and reversibility. Commonly employing acetone as its solvent, it is also commonly used with toluene, ethanol, xylene, and methanol. As determined in Chapter 2, it was tested at a 1/1 weight/weight ratio with its solvent component. The solvent component was then mixed with the filler and consists of a 60/40/40 mix by weight of acetone, ethanol, and filler; consisting of calcium carbonate and fumed silica.
4.0 Testing Methodology and Assembly Fabrication

4.1 Overview

To evaluate the performance of the two adhesive pinning methods, the following assemblies were prepared for mechanical testing (pull out) and durability by accelerated weathering tests. Holes were drilled into cubes of Portland sandstone cubes and the pins were seated with two distinct adhesives: Paraloid B-72 and Sika AnchorFix-4®. Four additional samples included a thin barrier of Paraloid B 72 1/1 by weight with no filler as an isolating layer between the sandstone substrate and the pin. This was done to assess what, if any, differences could be observed between the bond strength and durability of the repair when reversibility was insured through the use of an acrylic release or isolating layer for the Sika adhesive system. After 21 days of accelerated weathering at the Architectural Conservation Laboratory, the samples were tested for pull-out strength at the Laboratory for Research on the Structure of Matter at UPenn.

4.2 Assembly Fabrication

4.2.1 Stone Preparation

The sandstone arrived at the University of Pennsylvania in two 2” x 12” x 9” slabs from Portland Brownstone Quarries. The slabs were cut down to 2” x 2” x 2” cubes using a Felker Radial Wet Saw in the School of Design’s Fabrication Laboratory. The samples were then drilled to produce ½” diameter holes at a depth of one inch using a Bosch ½” Hammer Drill. The samples were washed in distilled water. They were then dried at 60° until they reached a constant mass, which was achieved over the course of
approximately 3 weeks. After 3 weeks the surfaces and drilled holes were degreased with acetone using cotton swabs and allowed to air dry.

4.2.2 Preparation of Fiberglass Dowels

ConservEpoxy’s Rebar 800™ dowels were washed in distilled water, blotted with a paper towel and then degreased with acetone. The pin was measured at 1.33 inches in length with a digital micrometer and then cut with a bi-metal blade hacksaw.

4.2.3 Injection of B-72

A resin solution of B-72 was prepared in a 1/1 weight to weight ratio. The solvent consisted of 40% ethanol, 60% acetone. The solution was bulked with calcium carbonate from Kremer pigments. The solvent was pre-bulked with the calcium carbonate in order to allow the solvent, solute, and filler to achieve good consistency. A 12 cc Luer Lock syringe was used to draw up 2.3 cc of adhesive. A size 10 canula was used to dispense the adhesive. The drilled hole was fitted with the aluminum injection ports. The flanged neck, which fits into the hole to ensure the pin is seated at a 90° angle, was coated with Vaseline to inhibit a bond from being formed between the adhesive and the port. A fluorosilicone washer was fit over the port’s neck to seal any air gaps. The unit was fit onto the hole and the canula was inserted into the port’s hole. The injection port’s rounded ingress allowed two gaps on either side of the square necked canula for air to escape from the hole upon injection of the adhesive. 2.3 cc’s of adhesive was dispensed and immediately after, the pin was seated erect, secured by the injection ports. The injection ports were removed after one week’s time at which point the B-72 had hardened.
sufficiently to allow the pin to stand independent of the port. This was also done to ensure maximum solvent loss as part of the curing process.

4.3 Embedment Depth

For the purposes of evaluating pull-out strength of load transfer mechanism, the embedment depth is a critical variable in determining the performance of the completed assembly. In a deeper hole, the adhesive has greater surface area to bond. Depth was determined based on ASTM C 1242 which states that dowel embedment in stone should be a minimum of two-thirds of the thickness of the stone. Sources cite that the stresses experienced during pull-out testing put a majority of the load around the perimeter of the joint at the opening of the hole. (Edwards 1991)

The embedment depth should be at least equal to the depth of the pin, and should be no less than four times the dowel diameter, in this case 3/8 inch. While many of the standards cited a minimum depth, this will be dependent on the geometry of the break and the size of the adherends. The greater the depth of the hole, the greater the loss the risk of increased damage.

4.4 Spacing and Edge Distances

Spacing and edge distance are another critical component which can affect the strength of the repair. Drilling too close to the edge can result in spalls, premature stone failure, and a weakening of the testing assembly. In field practice, the minimum edge distance is generally 4” from the edge of the joint or spall. Other research states that a minimum edge distance should be established at 6 anchor diameters. (ACI Vol 318-02,
02) Most of the research for adhesive bonded anchors has been developed for the concrete industry. The American Concrete Institute (ACI) relies on testing on a case by case basis, as the variety of masonry, concrete mixtures, adhesives, anchors, and dowels can be configured in so many ways that a single conclusive standard is difficult to set forth. For pull out testing, the primary concern for edge spacing is based on drilling the initial hole into the test assembly. For evaluating tensile strength, it can be a greater issue.

### 4.5 Diameter of Pins

Adhering to the conservator’s principle of minimum intervention in the laboratory as well as in the field, the diameter of the pins should be kept as small as possible. In field practice, the diameter and number of pins used is gauged by the shear and tensile strength requirements of the fracture. (Glavan, 59, 2004) Number also relates to edge spacing. An optimal treatment would be one that safely utilizes pins larger in diameter with a greater depth but being fewer in number. Overuse of pins results in unnecessary stresses on the substrate. It can cause cracking and weakening of the stone, especially a bedded stone such as sandstone.

Guidelines for the maximum diameter of the pins come from ASTM C 1242 “Standard guide for design, selection and installation of anchor systems.” Rod anchors and dowels are not to exceed ¼ of the stone thickness. The thickness being 2” makes the maximum allowable diameter of the dowel to be ½”. At 3/8” the fiberglass dowels will fit the requirements outlined herein.
5.0 Accelerated Weathering

5.1 Overview

In order to evaluate the performance of the anchor bond in the test samples, the RILEM test V.3 for Frost Resistance was selected to simulate the effects of freeze/thaw damage typically experienced by stone outdoors. Grave markers are commonly partially submerged in the soil, and given the inherent porosity and permeability of sandstone, they can potentially absorb a great deal of moisture.

5.2 Methodology

Accelerated weathering was carried out on 2 sets of 4 sandstone assemblies. Sample set WP, consisting of fiberglass pins seated in filled Paraloid B-72, was allowed to cure for 1 month at room temperature. Sample set WS, consisting of fiberglass pins seated in Sika Anchor Fix-4®, was allowed to cure for 48 hours. Manufacturer’s data specified its initial set time as 4 hours with 24 hours recommended for maximum strength. The samples were dried in an oven at 60°C until they reached a constant mass ± 5°C. The samples were then submerged in deionized water for 6 hours at 20°C. After 6 hours they were placed in a freezer at -15°C for 6 hours. Each cycle lasts 12 hours allowing for 2 cycles in a 24 hour period. The samples underwent 41 cycles. The samples were visually inspected and photographed prior to accelerated weathering and after every 14 cycles.
5.3.1 Results: Sample Set WP (pins embedded in Paraloid B-72)

Figure 5.1: WP Initial

Figure 5.2: After Week 1 WP
Figure 5.3: After Week 2 WP

Figure 5.4: After Week 3 WP
5.3.2 Results: Sample Set WS (pins embedded in Sika AnchorFix-4®)

Figure 5.5: Initial WS (Prior To Accelerated Weathering)

Figure 5.6: After Week 1 WS
Figure 5.7: After Week 2

Figure 5.8: After Week 3
5.4 Results & Discussion

After 41 cycles of accelerated weathering, little if any significant visual change was observed in the assemblies. Samples were examined where the adhesive meets the pin, where the stone meets the adhesive, erosion of the corners of the assemblies, and changes to the pin itself. Two assemblies, WP1 and WP4 were noted as having small losses at the junction of the adhesive and the rim of the bored hole. This was not thought to have affected the strength or integrity of the assembly or the load transfer mechanism.

Figure 5.9 Small Loss at Rim of Hole

While no significant damage was readily discernable, a second set of pull-out tests on the weathered assemblies was performed.
6.0 Mechanical Testing Program

6.1 Objectives

The fabricated assemblies described in Chapter 4.0 were allowed to cure for three weeks. Eight assemblies then were selected for accelerated weathering for another three weeks. At the end of the six week period, the twenty assemblies were taken to the University of Pennsylvania’s Laboratory for Research on the Structure of Matter for an evaluation of bond strength (Pull-out) according to ASTM E 488-96, Standard Test Methods for Strength of Anchors in Concrete and Masonry Elements. The assemblies were tested using an Instron 4206 electro-mechanical testing machine with a load capacity of 5000 lbs. This test measures the tensile strength of the load transfer mechanism between the pin and the sandstone cube. Values are given as load (in pounds of force) required to displace the pin and measure the corresponding displacement (in inches). The acquired data identifies the tensile profile of the assembly for a comparison of different adhesive systems and will serve to make recommendations for the most compatible conservation intervention.

6.2 Methodology

Each assembly was placed on a metal platen and secured to the Instron 4206. A wedge grip was affixed to the one inch fiberglass pin which protrudes from the top of the assembly. The universal joint is a flexible apparatus which allows the machine to correct a test sample which may not sit perpendicular to the platen. Each assembly was then fastened to the platen using triangular clamp knives. The assemblies were then pre-
loaded with a load of no more than 2 pounds, the data logger was initiated, and the machine slowly pulled up on the assembly with the wedge grip.

Figure 6.1 Instron 4206 Electro-mechanical Testing Apparatus
### 6.3 Pull-Out Test Assemblies

<table>
<thead>
<tr>
<th>Assemblies and Listed Components</th>
<th>Paraloid B-72</th>
<th>Sika AnchorFix-4®</th>
<th>Sika + B-72 barrier</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>P2</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>P2</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>P2</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>WP1</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>WP2</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>WP3</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>WP4</td>
<td>Y</td>
<td>N</td>
<td>N</td>
</tr>
<tr>
<td>PS1</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>PS2</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>PS3</td>
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<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>PS4</td>
<td>Y</td>
<td>Y</td>
<td>Y</td>
</tr>
<tr>
<td>S1</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>S2</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>S3</td>
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<td>Y</td>
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</tr>
<tr>
<td>S4</td>
<td>N</td>
<td>Y</td>
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</tr>
<tr>
<td>WS1</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>WS2</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>WS3</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
<tr>
<td>WS4</td>
<td>N</td>
<td>Y</td>
<td>N</td>
</tr>
</tbody>
</table>

| Table 6.1: Assemblies and Components |
## Results of Pull-Out Tests

<table>
<thead>
<tr>
<th>Assembly Series</th>
<th>Mean Maximum Load (lbs.)</th>
<th>Standard Deviation</th>
<th>Standard Error</th>
</tr>
</thead>
<tbody>
<tr>
<td>P</td>
<td>57.37</td>
<td>9.93</td>
<td>2.48</td>
</tr>
<tr>
<td>WP1</td>
<td>44.94</td>
<td>2.67</td>
<td>0.67</td>
</tr>
<tr>
<td>S</td>
<td>677.28</td>
<td>169.96</td>
<td>42.49</td>
</tr>
<tr>
<td>WS1</td>
<td>854.86</td>
<td>84.72</td>
<td>21.18</td>
</tr>
<tr>
<td>PS</td>
<td>621.21</td>
<td>66.73</td>
<td>16.68</td>
</tr>
</tbody>
</table>

Table 6.2: Results of Pull-Out Tests

### 6.4 Reversal of the B-72 Isolating Barrier

As a final test, the PS assembly series was immersed in acetone and ethanol in order to confirm the reversibility of the pin provided by the B-72 barrier coating. Each cohort was placed in an individual 300 ml container, submerged halfway up the length of the pin, and a watch glass was placed on top. After 48 hours, the cubes began to bloom with white residue, presumably from the Paraloid B-72. After 52 hours, a thin layer around the rim of the adhesive was able to be displaced with a scalpel. After 1 week, the edges surrounding the hole began to separate from the sandstone substrate and the pins loosened.
7.0 Conclusions

7.1 Introduction

The primary objective of the above testing program was to compare the performance characteristics of two different adhesives in an adhesive-based pinning system for masonry repair. Pull-out tests were performed to evaluate both strength and failure behavior; the latter to guarantee minimal damage and retreatability or reversibility. Prior to implementation of the testing program, it was assumed that the epoxy assemblies would display a higher bond strength than the filled Acryloid B-72 assemblies. (See Table 1.1) What was unknown was if and how accelerated weathering would affect the bond strength of all the assembly types. The accelerated weathering definitely showed a reduction in strength for both the epoxy and acrylate resin assemblies.

Figure 7.1: Cross Section of two cohorts of the P Series
### 7.2 Categorization of Failure Modes

<table>
<thead>
<tr>
<th>Assemblies</th>
<th>Stone Failure</th>
<th>Pin Failure</th>
<th>Adhesive Failure</th>
<th>Failure at interface of Pin and Adhesive</th>
<th>Failure at Interface of Stone and Adhesive</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
<td>N</td>
</tr>
<tr>
<td>P2</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
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</tr>
<tr>
<td>P3</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
<td>N</td>
</tr>
<tr>
<td>P4</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
<td>N</td>
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<td>WP1</td>
<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
<td>N</td>
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<tr>
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<td>N</td>
<td>N</td>
<td>Y</td>
<td>Partial</td>
<td>N</td>
</tr>
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<td>WP3</td>
<td>N</td>
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<td>Partial</td>
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<td>WP4</td>
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<td>Y</td>
<td>N</td>
<td>N</td>
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</tbody>
</table>

Table 7.1: Assemblies and Failure Modes
7.3 Discussion of Failure Modes

The failure modes of the sandstone assemblies aid in evaluating their mechanical performance as well their reversibility and degree of damage to the stone. Failure at the stone-adhesive interface or the pin-adhesive interface suggests poor surface bonding of the adhesive with the pin or stone adherend. Failure of the stone indicates excessive bond strength of the adhesive and is undesirable. Failure within the adhesive suggests good adhesive bond interface with the adherends and is the most desirable failure response as it safeguards additional damage to the stone. Breakage of the pin, while not causing damage to the stone, however does inhibit full evaluation of the adhesive assembly. The extent of the adhesive within the hole and on the pin’s embedment surface and its uniformity after cross-section (segregation) are also significant factors in determining pull-out strength and load distribution.

7.3.1 Assemblies formulated with Paraloid B-72 (Series P-WP)

7.3.1.1 Un-weathered Assemblies (P)

The un-weathered assemblies consistently failed within the adhesive whereby adhesive residue was found on both the pin and within the hole. This was confirmed by making a singular vertical cut with a masonry saw, which longitudinally bisected the hole’s length. The adhesive appeared evenly distributed between the pin and the hole’s surface also indicating injection methods were successful in distributing the adhesive within the pin holes. Within the adhesive, however, there did appear to be settling or segregation of the chalk filler. No samples revealed any damage to the sandstone adherends and all samples failed with a load in excess of 55 Lbs. (see table 6.3)
Appendix C confirms that this series exhibited elastic behavior as expected for thermoplastic resins.

7.3.1.2 Weathered Assemblies (WP)

The weathered assemblies also consistently failed within the adhesive. This was also confirmed by making a singular vertical cut, longitudinally bisecting the hole’s length. The adhesive appeared evenly distributed between the pin and the hole’s inner surface. Weathered samples failed under less load than the un-weathered assemblies. The loss of strength was nearly 15 Lbs less than the weathered samples or a loss of strength of 21.8 %. Both sets of numbers fall into the range of previous testing with filled B-72 (Glavan.2004, 89) (See Table 6.2) None of the assemblies displayed any damage to the sandstone adherends from the pull-out tests. These also exhibited elastic behavior as shown in Appendix C.

7.3.2 Assemblies formulated with Sika AnchorFix -4® (Series S-WS)

7.3.2.1 Un-weathered Assemblies (S)

The un-weathered assemblies with Sika AnchorFix-4 all failed in the same manner within the pin. Neither the adhesive nor the sandstone failed during the test. When the assemblies were loaded at 800 Lbs., the fiberglass strands within the pin debonded from their adhesive matrix as the wedge grip continued to pull upwards.
7.3.2.2 Weathered Assemblies (WS)

This set of samples was the only one to exhibit failure within the stone, at the adhesive-stone interface, at the interface of the adhesive and the pin, and in the pin. Assemblies WS1 and WS4 both exhibited failure in the pin similar to the S series. Cohort WS2 failed first within the stone breaking ¾” from its base, and then the pin pulled out a section of the stone attached to the pin. Characteristic of cone failure exhibited during pull-out tests for epoxy-stone assemblies. (Glavan, 91, 2004) WS4 exhibited failure within the stone and partially at the stone-adhesive interface. We cannot be sure if the stone gained or lost strength due to pin failure of the un-weathered assemblies. Either these two samples were weaker than the other two, or the freeze/thaw weathering strengthened the pins. The weathered assemblies withstood 177 more pounds of stress but failed at the stone.

7.3.3 Assemblies formulated with Sika AnchorFix -4 with Isolating Barrier of Paraloid B- 72 (Series PS)

In examining the failure modes of this assembly set, all cohorts exhibited failure within the pin similar to Series S-WS. The adhesive exhibited no damage as neither did the sandstone. When the assemblies were loaded with a stress of 800 Lbs., like series S, the fiberglass strands within the pin de-bonded from their adhesive matrix as the wedge grip continued to pull upwards.
7.4 Analysis of Assembly Components Based on Performance Criteria

<table>
<thead>
<tr>
<th>Assembly Series and Type of Anchor Bond</th>
<th>Injectability</th>
<th>Set Time at 60 F</th>
<th>Cure Time</th>
<th>Bond Strength</th>
<th>Failure Mode</th>
<th>Reversibility</th>
<th>Toxicity (LD) in mg (rat)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S (Sika AnchorFix-4)</td>
<td>Excellent</td>
<td>12 (min)</td>
<td>8-24hrs</td>
<td>677.28 lbs</td>
<td>within stone</td>
<td>No</td>
<td>5,000</td>
</tr>
<tr>
<td>P (Paraloid B-72)</td>
<td>Fair</td>
<td>15 (min)</td>
<td>3-7 weeks</td>
<td>57.3 lbs</td>
<td>within adhesive</td>
<td>Yes</td>
<td>5,500</td>
</tr>
<tr>
<td>PS (Barrier coat of B-72 &amp; Sika)</td>
<td>Fair</td>
<td>15 min</td>
<td>30 hrs</td>
<td>621.21 lbs</td>
<td>at pin</td>
<td>Yes</td>
<td>5,500</td>
</tr>
</tbody>
</table>

Table 7.2 Performance Analysis

7.5.1 Discussion and Conclusions

Table 7.1 lists the three distinct assembly systems based on the two adhesives and assesses their performance according to critical properties deemed significant for their overall evaluation. These were identified in Chapter 2 as: injectability, set time, cure time, bond strength, failure mode, reversibility and toxicity. Injectability is defined as how well the adhesive flows, in this case under hand pressure through a syringe fitted with a #10 Gauge canula. Injectability is also affected by thixotropy, a rheological property of the adhesives which affects viscosity and can be useful in controlling the placement of the adhesive in the holes. Set time is defined as the time available for the adhesive to be installed (i.e., injected) prior to permanent changes in rheology. Good set time should allow ample working time for injection and pin insertion but also allow the pin to be supported in the hole without deflection. Thixotropy complements set time.
Cure time is the time required for the adhesive to solidify and reach its maximum strength. Both set and cure times are affected by ambient temperature and humidity for both adhesives.

Bond strength is defined as the ability of the pin-adhesive-stone assembly to withstand loading and stress. It assumes full cure of the adhesive and is measured by ASTM E48. Failure mode is a critical aspect of performance behavior as it predicts how the system will respond to stress in the field and defines the degree of damage to the masonry. In conservation, repairs should never damage the original fabric, in this case, the stone. Reversability or re-treatability, is a related concern and is defined as those actions that will allow a reversal or additional treatment without further damage to the stone. Toxicity here is defined by the Lethal Dose as listed in the material’s safety data sheets (MSDS).

7.5.2 Series S

Series S performed well in most performance categories. All cohorts displayed results that fell within the limits of acceptability. The epoxy displayed excellent controlled injectability and its use was largely aided by the manufacturer’s pre-packaging as a 2-part injectable gel. Sufficient viscosity and fast set provided good support for the pin after installation; however as set time is affected by temperature, this must be determined depending on ambient working conditions. Epoxy’s short cure time is also desirable, especially for complex, large-scale structural repairs where pinning is one phase of a larger program of treatment. Although the bond strength of the epoxy was found to be over 100 times higher than the acrylate adhesives, its strength also surpassed
the cohesive strength of the stone, causing damage to the adherend during pull-out tests. Its irreversibility as a cross-linked polymer also makes its incompatible as a pin adhesive on sandstone. Its toxicity is nearly equal to the Paraloid B-72 resin solutions in acetone/ethanol; however the epoxy has a lower VOC.

7.5.3 Series P

Series P also met most of the criteria above for a conservation grade adhesive and performed to expectations. The issue of filler could be the primary factory in explaining why it does not inject as well as the Sika or the un-bulked B-72 adhesive. While this thesis examined different bulking proportions, more research is needed. While a filler is required to support the pin, the greater the quantity of filler, the more difficult it is to inject. The segregation or settling of the filler observed in the filled holes may be due to insufficient dispersion during preparation and might be improved by the additional of combined fillers such as microballoons and fumed silica. The filled B-72 adhesive displayed the same set time as the Sika epoxy adhesive with the isolation barrier. Its cure time varies, but 3 weeks might be be considered too long in many applications, especially outdoors,. Its bond strength can be rated as adequate, and certainly its overall strength was not compromised more that that of the epoxy after weathering. Its failure within the adhesive layer itself makes it a successful conservation treatment as no damage is done to the stone. Its reversibility is its best characteristic and makes it optimum for conservation. Its toxicity is just below that of Sika.
7.5.4 Series PS

Series PS met the required performance characteristics and would appear to perform very well as a conservation treatment. Injection of B-72 without filler increases its injectability and the Sika Anchorfix-4 uses proprietary ingredients to enhance its injectability. The combination of these two factors greatly increases ease of application. Since only a thin coating of B-72 was used and then allowed to air-dry, it hardened on the outside within 8 hours. This compounded with the short cure time of a thermoplastic resin gave it a very desirable cure time. Failure of the pin at 621 lbs of load does not determine the exact strength of the bond. Instead of failing at the stone adhesive interface, this series failed at the pin. It is favorable that the combination of epoxy, isolating layer, and fiberglass pin caused no damage to the stone in any of the test assemblies. The reversibility of the B-72 which upon dissolution should release the layer of Sika make these adhesive system very attractive alternatives to epoxy alone. It possesses a standard amount of toxicity found in most adhesives and should be handled with care like any chemical. All of these characteristics suggest a very compatible conservation treatment but will it require further testing so that a better assessment of strength can be made.

It was also unknown how the Sika would behave with the Paraloid B-72 isolation layer. As reported in the literature, similar assemblies failed, several failed at the stone-adhesive interface. (Podany. 2001. This could be due to the fact that Podany used a B-72 formula of 22% by weight. These isolation barriers used a B-72 formula which was 1 to 1, solvent to solute by weight. The thicker film of B-72 may have prevented the epoxy from permeating the isolation layer.
7.6 Recommendations for Future Research

There is a very limited amount of published work on Paraloid B-72 as a structural adhesive and there are many different possible methods for its formulation. Fillers and solvents play a significant role in establishing its performance characteristics. More testing could yield very valuable data using different variables. The bubbles that were observed along the shaft of the pin in the injection simulations as well as in the pins that were pulled from the assemblies may have been caused by the solvents evaporating from the adhesive after injection. This could result in variability of bond strength by the voids created from the entrapped bubbles in the adhesive. It is recommended that more work be done in experimenting with the amounts and types of fillers. The limited time for completion of this course of study limited the use of solvents. It may be of value to look at additional solvents with slower evaporation rates. Different proportions of solvents mixed with B-72 could also be considered. While the fiberglass rods met the criterion of being non-corrosive and of sufficient tensile strength, they were an impediment to evaluating the pull-out strengths for the epoxy due to their failure. To better analyze the adhesive, a more uniform and rigid material such as stainless steel or alumina would be very useful. This testing program relied on the same embedment depth, hole diameter and pin size throughout the its tests. This is another variable which could be expanded on for further research.

There are hundreds of other accelerated weathering test programs which could expand the data listed herein. It is recommended that they also be examined, particularly those created by ASTM and RILEM.
8.0 BIBLIOGRAPHY


“Cutting-Edge Masonry Repair.” Architecture 87, no. 4 (1998)


Hansen, Eric F. “The Effects of Solvent Quality on Some Properties of Thermoplastic Amorphous Polymers used in Conservation”. *JAIC 1991, Volume 30, Number 2, Article 8* (pp. 203 to 213).


9.0 APPENDICES

9.1 Appendix A: MANUFACTURERS AND SUPPLIERS OF MATERIALS
9.2 Appendix B: TECHNICAL DATA OF ADHESIVES AND FILLERS
9.3 Appendix C: TESTING DATA
9.1 APPENDIX A: MANUFACTURERS AND SUPPLIERS OF MATERIALS

**Fiberglass pins- Rebar800™**
Mfg. by & Distributed by
ConservEpoxy
PO Box 454
Northford, CT 06472
(203) 484-4123
www.conservepoxy.com

All Laboratory Supplies
Fisher Scientific
26 Liberty Lane
Hampton, NH 03842
800.766.7000
www.fishersci.com

**Sika AnchorFix-4®**
Mfg. by Sika Corporation
Distributed by Kenseal Production Corp.
1540 Delmar Drive
Folcroft, PA, 19032
610-532-5391
www.kenseal.com

Washers, aluminum, drill bits
Distributed by
McMaster-Carr Supply Co.
Dayton, NJ 08810
732-329-3200
www.mcmaster-carr.com

**Sandstone**
Portland Brownstone Quarries
311 Brownstone Avenue
Portland, CT, 06480
860-342-2920
www.portlandbrownstone.com

Paraloid B-72
Mfg. by Rohm and Haas
Distributed by Talas
20 West 20th Street
New York, NY 10011
212-219-0770
www.talas.com
9.2 APPENDIX B: Technical Data of Adhesives and Fillers.

**Sikadur® Injection Gel**

High-modulus, high-strength, structural, non-abrasive, smooth epoxy paste adhesive

**Description**
Sikadur Injection Gel is a 2-component, 100% solids, moisture-tolerant, high-modulus, high-strength, structural epoxy. When mixed it gives a smooth, non-abrasive, paste adhesive. It conforms to the current ASTM C-381 and AASHTO M-209 specifications.

**Where to Use**
- Structural crack repairs not exceeding 1/4 in. (6 mm) width.
- Mechanical grouting... bolts, dowels, pins, machine and robotic baseplates, bearing pads, etc.
- Waterproofing tunnels, cable vaults, tanks, basements, etc.
- Re-anchoring of veneer masonry.
- Wood-timber repairs.
- Preventive maintenance ... new or existing structures to seal off reinforcing steel from the elements of corrosion.
- Anchor grouting... bolts, dowels, pins and special fasteners.
- As a pick-proof sealant around windows, doors, lock-ups, etc. inside correctional facilities.

**Advantages**
- Unique, non-abrasive texture permits application with automated pressure injection equipment.
- Tolerant of moisture before, during, and after cure.
- High-modulus, high-strength, structural paste adhesive.

**Typical Data (Material ensuring conditions @ 73°F (23°C) and 50% R.H.)**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shelf Life</td>
<td>2 years in original, unopened container</td>
</tr>
<tr>
<td><strong>Storage Conditions</strong></td>
<td>Store dry at 40°-60°F (5°-26°C). Condition material to 65°-75°F (18°-24°C) before using.</td>
</tr>
<tr>
<td><strong>Color</strong></td>
<td>Gray</td>
</tr>
<tr>
<td><strong>Mixing Ratio</strong></td>
<td>Compound 'A' : Compound 'B' = 1:1 by volume</td>
</tr>
<tr>
<td><strong>Consistency</strong></td>
<td>Smooth, non-aggregates</td>
</tr>
<tr>
<td><strong>Pot Life</strong></td>
<td>Approximately 30 minutes. (60 gram mass)</td>
</tr>
<tr>
<td><strong>Tensile Properties (ASTM D-638)</strong></td>
<td></td>
</tr>
<tr>
<td>14 day</td>
<td></td>
</tr>
<tr>
<td><strong>Tensile Strength</strong></td>
<td>4,000 psi (28.6 MPa)</td>
</tr>
<tr>
<td><strong>Elongation at Break</strong></td>
<td>1.3%</td>
</tr>
<tr>
<td><strong>Modulus of Elasticity</strong></td>
<td>4.1 x 10⁶ psi (28.6 MPa)</td>
</tr>
<tr>
<td><strong>Flexural Properties (ASTM D-789)</strong></td>
<td></td>
</tr>
<tr>
<td>14 day</td>
<td></td>
</tr>
<tr>
<td><strong>Flexural Strength (Modulus of Rupture)</strong></td>
<td>6,000 psi (42.2 MPa)</td>
</tr>
<tr>
<td><strong>Tangential Modulus of Elasticity in Bending</strong></td>
<td>7.5 x 10⁶ psi (51.7 MPa)</td>
</tr>
<tr>
<td><strong>Shear Strength (ASTM D-712)</strong></td>
<td>14 day</td>
</tr>
<tr>
<td><strong>Shear Strength</strong></td>
<td>3,700 psi (25.5 MPa)</td>
</tr>
<tr>
<td><strong>Bond Strength (ASTM C-662)</strong></td>
<td></td>
</tr>
<tr>
<td>Hardened concrete to hardened concrete</td>
<td></td>
</tr>
<tr>
<td>2 day (dry cure)</td>
<td>Bond Strength 3,000 psi (20.6 MPa)</td>
</tr>
<tr>
<td>2 day (moisture)</td>
<td>Bond Strength 2,500 psi (17.2 MPa)</td>
</tr>
<tr>
<td>14 day (moisture)</td>
<td>Bond Strength 2,000 psi (17.9 MPa)</td>
</tr>
<tr>
<td>Hardened concrete to steel</td>
<td></td>
</tr>
<tr>
<td>2 day (dry cure)</td>
<td>Bond Strength 3,300 psi (22.7 MPa)</td>
</tr>
<tr>
<td>14 day (moisture)</td>
<td>Bond Strength 2,600 psi (17.0 MPa)</td>
</tr>
<tr>
<td><strong>Heat Deflection Temperature (ASTM D-640)</strong></td>
<td>120°F (48°C)</td>
</tr>
<tr>
<td>7 day</td>
<td>(Riser stress loading = 264 psi (1.8 MPa))</td>
</tr>
<tr>
<td><strong>Air Absorption (ASTM D-570)</strong></td>
<td>7 day (24 hr immersion)</td>
</tr>
<tr>
<td><strong>Compressive Properties (ASTM D-459)</strong></td>
<td>0.11%</td>
</tr>
<tr>
<td><strong>Compressive Strength, psi (MPa)</strong></td>
<td></td>
</tr>
<tr>
<td>4 hour</td>
<td>300 (2.1)</td>
</tr>
<tr>
<td>6 hour</td>
<td>300 (2.1)</td>
</tr>
<tr>
<td>16 hour</td>
<td>100 (0.7)</td>
</tr>
<tr>
<td>1 day</td>
<td>1,400 (9.6)</td>
</tr>
<tr>
<td>3 day</td>
<td>7,500 (52.4)</td>
</tr>
<tr>
<td>7 day</td>
<td>9,000 (62.1)</td>
</tr>
<tr>
<td>14 day</td>
<td>16,000 (108.9)</td>
</tr>
<tr>
<td>28 day</td>
<td>16,000 (108.9)</td>
</tr>
<tr>
<td><strong>Compressive Modulus</strong></td>
<td>2.7 x 10⁶ psi (1830 MPa)</td>
</tr>
</tbody>
</table>

*Gaskets and seals at the temperatures indicated.*

Product Data Sheet
Edition 2003
Identification no. 386
Sikadur Injection Gel

[Image of Sikadur Injection Gel]
Excellent adhesion to masonry, concrete, wood, steel and most structural materials.

Paste consistency ideal for vertical and overhead applications.

Fast-setting (FS) version also available.

Sufficient to mask any rate of A:B = 1:1 by volume.

Excellent bond strength for deep penetration.

Coverage: 1 gal. yields 239 cu. in. of epoxy paste adhesive.

Packaging: 4 gal. units.

**How to Use**

**Surface Preparation**

Surface must be clean and sound. It may be dry or damp, but free of standing water. Remove dust, balancers, graffiti, curing compounds, impregnations, waxes and any other contaminants.

**Preparation Work: Concrete**

- Should be cleaned and prepared to achieve a laitance and contaminant free, open textured surface by blastcleaning or equivalent mechanical means.

**Steel**

- Should be cleaned and prepared thoroughly by blast cleaning.

**Mixing**

SilKlad Injection Gel is specially designed and formulated to be mixed and applied with automated pressure-injection equipment. Follow the recommendations and directions supplied by the equipment manufacturer.

Pre-mix each component. Proportion equal parts by volume of Component 'B' and Component 'A' into a clean mix. Mix thoroughly for 3 minutes with a Sika paddle on low-speed (400-600 rpm) drill until uniform in color. Mix only that quantity that can be applied within its pot life.

**Application**

As a structural adhesive - Apply the pre-mixed SilKlad injection Gel to the prepared substrates. Work into the substrate for positive adhesion. Secure the bond completely into place until the adhesive has cured. Gaps should be kept as thin as possible, not to exceed 1/4 in. (6 mm).

To seal injection ports and cracks for injection grouting - Place the pre-mixed SilKlad injection Gel in the prepared substrates. Allow sufficient time to set before pressure-injecting.

To anchor bolts, dowels, pins - Annual space around bolt should not exceed 1/4 in. (6 mm) depth of embedment is typically 10-15 times the bolt diameter. Grout with neat SilKlad Injection Gel.

To grout cracks - Use automated injection equipment or manual method. Set appropriate injection ports based on the system used. Cracks up to 1/4 in. (6 mm) wide may be grouted.

To anchor bolts, dowels, pins in hollow masonry or concrete block - Consult Technical Service.

To seal base plates and bearings pads - Inject in-place baseplate and bearing pads with SilKlad Injection Gel. Apply up to 1/4 in. (6 mm) thick. As a pick-proof sealant - Use abraded or manual method. Apply an appropriate size bead of material around the area being sealed. Seal with neat SilKlad Injection Gel.

**Limitations**

- Minimum substrate and ambient temperature 40°F (4°C).
- Do not add. Additives or solvents will prevent proper cure.
- Material is a vapor barrier after cure.
- Not for sealing cracks under hydrostatic pressure.

**Caution**

Component 'A' - Irritant: Sensitizer - Contains isocyanate resin, which can cause skin sensitization after prolonged or repeated contact. Skin and eyes irritant. Use with adequate ventilation. Use of safety goggles and chemical-resistant gloves is recommended. In case of excessive fumes of PELA, use an appropriate, properly fitted NIOSH approved respirator. Remove contaminated clothing. Consult MSDS for more detailed information.

Component 'B' - Irritant: Sensitizer - Contains amine. Use with adequate ventilation. Use of safety goggles and chemical-resistant gloves is recommended. In case of excessive fumes of PELA, use an appropriate, properly fitted NIOSH approved respirator. Remove contaminated clothing. Consult MSDS for more detailed information.

**First Aid**

Eye: Hold eyes open and flush thoroughly with water for 15 minutes. Skin: Remove contaminated clothing. Wash skin thoroughly for 15 minutes with soap and water. Inhalation: Remove person to fresh air. Ingestion: Do not induce vomiting. In all cases, contact a physician immediately if symptoms persist.

**Clean Up**

Vapour area. Confine spill. Collect with absorbent material. Dispose of in accordance with current, applicable federal, state, and local regulations. Uncured material can be removed with approved solvent. Cured material cannot be removed mechanically.

KEEP CONTAINER TIGHTLY CLOSED

KEEP OUT OF REACH OF CHILDREN

NOT FOR USE IN THERMAL WELDING

KEEP OFF INTERNAL CONSUMPTION

CONSULT MATERIAL SAFETY DATA SHEET FOR MORE INFORMATION

Silka warrants this product for one year from date of manufacture to be free from manufacturing defects and to meet the technical properties on the current technical data sheet if used as directed within shelf life. User determines suitability of product for intended use and assumes all risks. Buyer's sole remedy shall be limited to the purchase price or replacement of product whichever is greater cost or cure of labor.

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Fax: 514-897-2312

Silka Mexico S.A. de C.V.
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| P.O. Box 145
Queretaro, Queretaro
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Queretaro, Queretaro
C.P. 76000

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Paraloid® B-72
Solid Grade Thermoplastic
Acrylic Resin

Paraloid® B-72 general-purpose thermoplastic acrylic resin is similar to Paraloid® B-66 acrylic resin but capable of forming softer films. The approximate hardness (KHN) is 10-11 compared to 12-13 for Paraloid® B-66 resin.

Paraloid® B-72 acrylic resin is unique in possessing a high tolerance for ethanol. The property allows its use in applications not tolerant of strong solvents. The alcohol dispersions may be cloudy or milky. However, they form clear, coherent films.

Paraloid® B-72 has low reactivity with sensitive phosphorescent and luminescent pigments to produce stable, durable, non-yellowing coatings. It is compatible with vinyls, celluloses, chlorinated rubbers, and silicones. It is well suited for white and metallic aerosols, clear coatings for wood, nitrocellulose modified coatings for general product finishing, pigment dispersion (fluorescent), flexographic printing inks, and gravure plastic coatings.

Solubility

Information about the solvent compatibility of Paraloid® B-72 acrylic resin can be found in Rohm and Haas brochure 82A114—Paraloid® Solid Grade Resins, Solvent Selection Chart.

Typical Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Physical Form</td>
<td>Pellets</td>
</tr>
<tr>
<td>Chemical Composition</td>
<td>EMA Cb polymer</td>
</tr>
<tr>
<td>Tg, °C</td>
<td>40</td>
</tr>
<tr>
<td>Bulk Density, 25 °C, lb/gal</td>
<td>9.6</td>
</tr>
<tr>
<td>Solubility Parameter</td>
<td>9.3</td>
</tr>
<tr>
<td>Ultimate Hardness of Clear Films, KHN</td>
<td>10 to 11</td>
</tr>
</tbody>
</table>
Product Specifications

Appearance, as-is visual Free of visible foreign matter.
Appearance of solution visual

Color of solution, APHA
30, maximum

Turbidity, bentonite, scale 3, maximum

Viscosity, corrected, cps 470 – 770

Brookfield LV
spindle #2, 12 rpm, 25°C
corrected to 40% solids

*Solution preparation: weigh into a pint jar 120 g sample and 180 g toluene. Solution is about 40%
solids.

Safe Handling Information

Rohm and Haas Material Safety Data Sheets (MSDS) contain pertinent information that you
may need to protect your employees and customers against any known health or safety
hazards associated with our products. Under the OSHA Hazard Communication Standard,
workers must have access to and understand MSDS on all hazardous substances to which
they are exposed. Thus, it is important that you provide appropriate training and information
to your employees and make sure they have available to them MSDS on any hazardous
products in their workplace. Rohm and Haas Company sends MSDS on non-OSHA-hazardous
as well as OSHA-hazardous products to its customers upon initial shipment (including
samples) of all its products (whether or not they are considered OSHA-hazardous). If you do
not have access to one of these MSDS, please contact your local Rohm and Haas
representative for an additional copy. Updated MSDS are sent upon revision to all customers
of record. MSDS should be obtained from your suppliers of other materials recommended in
this bulletin.

Rohm and Haas Company is a member of the Chemical Manufacturers Association and is
committed to CMA’s Responsible Care® Program.

PHILADELPHIA, PA 19109

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82A123 December 1996 Printed in U.S.A.
PERFORMANCE MINERALS FOR SPECIALTY APPLICATIONS

ULTRA-PFLEX®
precipitated calcium carbonate (pcc)

ULTRA-PFLEX® precipitated calcium carbonate (pcc) is a surface treated, premium quality, high performance, white pcc. In specialty, particularly extrusion, printing, paper and polystyrene systems, ULTRA-PFLEX® pcc is a functional additive, providing significant cost savings of the system. In injection molding and injection molding applications, ULTRA-PFLEX® pcc enhances both notched flexural strength and impact strength of the end product. The multifunctional aspect of ULTRA-PFLEX® pcc makes it the unique tool with which to solve demanding performance problems, while offering significant cost savings potential.

**Typical Properties**

- **Average Particle Size**: 2.97
- **5% Mark Reserve**: 6.1
- **Specific Gravity**: 2.7
- **Dry Densities (1% air):** 55
- **Bulk Density (gallon)**: 12
- **Talc Density (gallon)**: 12
- **Surface Area (m²/g)**: 16

**Chemical Composition**

- **Calcium Carbonate (CaCO3)**: 99%
- **Magnesium Carbonate (MgCO3)**: <0.1%
- **Iron oxide (Fe₂O₃)**: <0.1%
- **Moisture (H₂O)**: <0.2%

---

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9.3 APPENDIX C: PULL OUT TESTING DATA
Appendix C: Pull Out Strength Test Results
Series P: Paraloid B-72 Assemblies

**P1 Pull-Out Test**

Displacement (1v=0.04 inch) vs. Load (1v=100 lbs)

**P2 Pull Out Test**

Displacement (1v=0.04 inch) vs. Load (1v=25 Lbs)
Appendix C: Pull Out Strength Test Results
Series P: Paraloid B-72 Assemblies

P3 Pull Out Test

P4 Pull Out Test
Appendix C: Pull Out Strength Test Results
Series WP: Weathered Paraloid B-72 Assemblies

WP1 Pull Out Test

WP2 Pull Out Test
Appendix C: Pull Out Strength Test Results
Series WP: Weathered Paraloid B-72 Assemblies

WP3 Pull Out Test

WP4 Pull Out Test
Appendix C: Pull Out Strength Test Results
Series S: Sika AnchorFix-4 Assemblies

S1 Pull Out Test

![S1 Load vs Displacement Graph](image)

S2 Pull Out Test

![S2 Load vs Displacement Graph](image)
Appendix C: Pull Out Strength Test Results
Series S: Sika AnchorFix-4 Assemblies

S3 Pull Out Test

S4 Pull Out Test
Appendix C: Pull Out Strength Test Results
Series WS: Weathered Sika AnchorFix-4 Assemblies

**WS1 Pull Out Test**

Displacement (1V = .04 in.)

Load (1V = 100 lbs.)

**WS2 Pull Out Test**

Displacement (1V = .04 in.)

Load (1V = 100 lbs.)
Appendix C: Pull Out Strength Test Results
Series WS: Weathered Sika AnchorFix-4 Assemblies

**WS3 Pull Out Test**

Displacement (1V = .04 In.)

Load (1V = 100 Lbs.)

**WS4 Pull Out Test**

Displacement (1V = .04 In.)

Load (1V = 100 Lbs.)
Appendix C: Pull Out Strength Test Results
Series PS: Sika AnchorFix-4 with B-72 Barrier Coating Assemblies

PS 1 Pull Out Test

PS2 Pull Out Test
Appendix C: Pull Out Strength Test Results
Series PS: Sika AnchorFix-4 with B-72 Barrier Coating Assemblies

PS3 Pull Out Test

Load (1V = 100 Lbs.) vs. Displacement (1V = .04 In.)

PS4 Pull Out Test

Load (1V = 100 Lbs.) vs. Displacement (1V = .04 In.)
10.0 Index

acetone, 6, 10, 11, 13, 19, 20, 21, 24, 33, 35, 47, 55
acrylic resins, 5
adhesive, 1, 2, 6, 8, 9, 10, 11, 12, 13, 14, 15, 16, 18, 20, 24, 26, 27, 28, 30, 31, 32, 34, 35, 36, 37, 43, 44, 47, 48, 50, 51, 52, 53, 54, 55, 56, 57, 58, 60
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