

Penetrating Sealers:

A Comparison of Epoxy, Moisture-Cured Urethane, and Siloxane Technology on Concrete, Rust, and an Inorganic Zinc Coating

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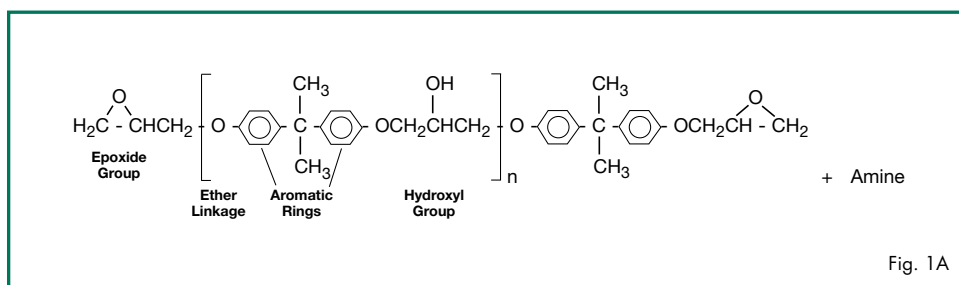


Some things never change—or do they? While great strides have been made during the last two decades with the development of specialty maintenance coatings for rehabilitating both concrete structures and lesser prepared steel surfaces, the war against corrosion is still bedeviled by catastrophic coating failures. And for the coating manufacturer it is almost axiomatic that, when the coating comes off, it's “first off, first to blame.”

Failures. The word conjures up images of expense, wasted effort, misery, lawyers, and worse. So where do we begin to gain greater insight into preventing failures? The answer is usually to examine the primer-substrate interface in the given system. First, let's quickly survey the coating scene. The received wisdom is that successful performance depends on good surface preparation and tenaciously adherent coating systems. Traditionally, the preferred surface preparation method is abrasive blasting of surfaces and ensuring that soluble chlorides and sulfates are kept below threshold levels where they would initiate failure.

This is all well and good, but for steel structures, abrasive blasting is not always feasible given environmental and economic constraints and the desire for quick turnaround maintenance work. Hence, it is imperative to judiciously select any so-called “surface-tolerant” coatings for application after surface preparation with hand tool cleaning, power tool cleaning, or waterjetting. Materials used should be designed to be applied to marginally prepared surfaces. These materials should provide excellent adhesion, barrier, penetrating, and wetting properties to cope with the chemical environment and hygrothermal stress. This is especially true when applying coatings to tight rust, where soluble salt removal is a prerequisite.¹

In some early coating applications, contractive curing stress failures due to poor compatibility have been known to occur from the application of high-build epoxy coatings on marginally prepared steel/old coating systems. For the successful coating of concrete—a substrate often far more difficult to coat—the same multifactorial performance criteria apply. Penetrating and sealer surface porosity is one of



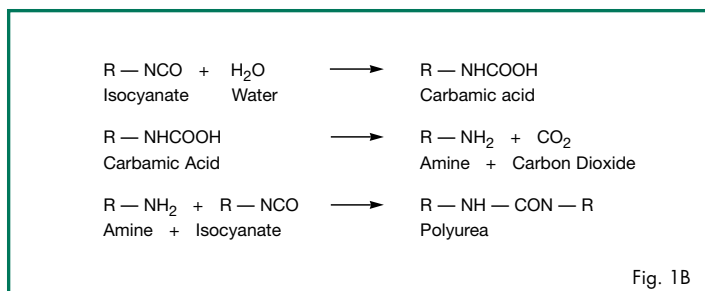
From top to bottom

Fig. 1A - Structure for epoxy resin

Fig. 1B - Urethane linkage from moisture-cured polyurethane

Fig. 1C - Epoxy polysiloxane structure

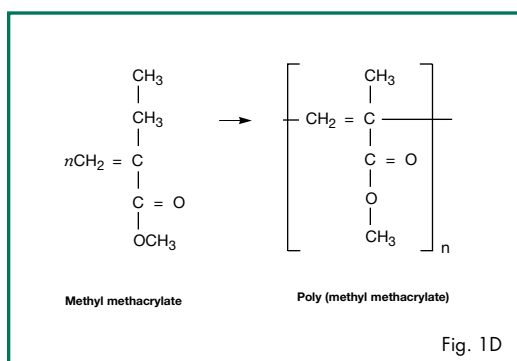
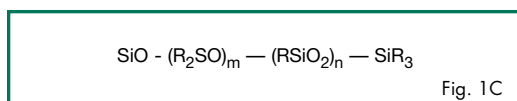
Fig. 1D - Modified methyl methacrylate



Candidate Coatings

Generic Classes Selected

Acknowledging that there is excellent chemistry in several generically different penetrant sealers, 12 proprietary coatings were investigated that have



rapidly gained in popularity in recent years. These are listed in Table 1. Coatings A-H were considered good primers in coating systems and well-suited to protect concrete and rusted steel in harsh environments. Five unpigmented two-component epoxy penetrant sealers were compared and contrasted with three pigmented single-component moisture-cured urethane penetrant sealers. The remaining coatings were three two-component high-build epoxies, a methacrylate, and one polysiloxane coating. Other generic types of penetrant sealers not included in this study are alkyds, acrylics and other latexes, calcium sulfonate wax systems, silicates, silicones, and silanes.

the keys to good adhesion and longevity of the coating system.

This article describes research to determine the penetration, wetting, and adhesion abilities of different coatings when applied to various porous substrates such as cured and green concrete, rust, and an alkyl silicate inorganic zinc coating. Scanning electron microscopy (SEM), optical microscopy, and various physical tests were employed to characterize the action of candidate penetrating sealers.

This study is not intended to be an exhaustive investigation of the behavior of penetrant primers. Rather, the intention was to investigate the behavior of representative primers on one set of typical field construction substrates and is therefore only a snapshot of the behavior of the materials over their range of usefulness.

This investigation was spurred by the authors' professional curiosity regarding the penetrating abilities and behavior of surface-tolerant coatings in fine porosity and in the presence of moisture. Specifically, the range of chemistries and physical properties between the coating products studied indicated that the performance of the coatings on firm rust and concrete in less-than-ideal conditions would be particular to properties of the coatings, and probably not equal.

By examining the microenvironment at the rust-steel and coating-concrete interfacial regions, the authors ranked certain performance aspects of these coatings and correlated these aspects with various physico-chemical attributes of the coatings.



Fig. 2 - Spray application of coatings for testing
Photos and figures courtesy of the authors

Two-Component Epoxies

Figure 1a shows an idealized structure for an epoxy resin and indicates the functional groups responsible for their characteristics. The hydroxyl group contributes to the outstanding adhesion of these resins to most substrates while the aromatic rings are important contributors to their good thermal and corrosion properties. The ether linkage adjoining the aromatic ring is cleaved under strong exposure to ultraviolet light, giving rise to the poor outdoor weathering of epoxy coatings; this is usually unimportant for primer-sealers.

The choice of curing agent for the epoxy resin is of particular importance in determining the final film properties of an effective penetrating sealer for rust, concrete, or old coatings. For enhanced flexibility and lower cross-link density, typical curing agents include low functionality aminoamines, blocked aliphatic amines, and polyamides. This is in marked contrast to the use of multi-functional amines, synonymous with high cross-link density, which give very good chemical resistance.² The curing agent plays a critical role in defining the viscosity, pot life, dry time, and molecular mobility for high penetration/wetting and low stress development. For best results, the formulated coating is unimpent-

ed and carefully designed so that the spacing of active hydrogen atoms produces the desired slower speed of reaction.

Moisture-Cured Urethanes

Single-component moisture-cured urethanes are often used as penetrating sealers on rust, concrete, and old coatings. In this class of coatings, the isocyanate groups react with water vapor in the air to ultimately form polymers with substituted urea linkages (Fig. 1b). Carbon dioxide is released during cross-linking, but this generally is not problematic unless the film is applied too heavily, whereupon significant bubbling can occur. Although some moisture-cured urethane primer sealers are clear, several are pigmented with flat, platy pigments such as micaceous iron oxide or aluminum. Upon application, these pigments are partitioned from the resin/solvent, which then migrates through porous surfaces.

Key advantages of this single-component technology are its inherent abilities to react with water in damp surfaces and afford low temperature cure (20 F, -7 C). Ironically, the fast cure of some of these coatings may prove counterproductive when utilized in the penetrating sealer mode, where slow reaction times are particularly advantageous.

Epoxy Polysiloxane

The novel chemistry of these coatings is based on the hybridization of organic epoxy and inorganic polysiloxane resins. The result is a polymer with an Si-O-Si polysiloxane backbone that provides excellent gloss retention, very good chemical resistance, and apparently good penetration properties for porous surfaces. Figure 1c shows a polysiloxane resin where R is an organic group used for cross-linking. Epoxy polysiloxanes do not cure properly at low humidity, and, under such conditions, their performance is adversely impacted.

**Table 1
List of Candidate Coatings and Properties**

| Coating Code | Generic Type | Volume of Solids | Color | Pot life @ 77 F (hrs) Cure | Lowest Temperature F | (C) |
|--------------|--------------|------------------|-----------|----------------------------|----------------------|-------|
| A | EPS | 100 | Clear | 4 | 50 | (10) |
| B | EPS | 100 | Clear | 1 | 25 | (-4) |
| C | EPS | 100 | Clear | 1 | 32 | (0) |
| D | EPS | 100 | Clear | 6 | 50 | (10) |
| E | EPS | 98 | Green | 0.5 | 20 | (-7) |
| F | MCU-PS | 70 | Off-White | NA | 20 | (-7) |
| G | MCU-PS | 67 | Off-White | NA | 20 | (-7) |
| H | MCU-PS | 61 | Aluminum | NA | 20 | (-7) |
| I | E-Polys | 90 | Blue | 4 | 32 | (0) |
| J1 | Epoxy-HB | 68 | Grey | 4 | 0 | (-18) |
| J2 | Epoxy-HB | 68 | Grey | 4 | 0 | (-18) |
| K1 | Epoxy-HB | 75 | Yellow | 6 | 25 | (-4) |
| K2 | Epoxy-HB | 75 | Yellow | 6 | 25 | (-4) |
| L1 | Epoxy-HB | 80 | Off-White | 3 | 35 | (2) |
| L2 | Epoxy-HB | 81 | Off-White | 3 | 35 | (2) |
| M | Methacrylate | 100 | Clear | 2 | 50 | (10) |

EPS=epoxy penetrant sealer
 MCU-PS=moisture-cured urethane penetrant sealer
 E-Polys=epoxy polysiloxane
 Epoxy HB=epoxy high-build
 Methacrylate= modified methyl methacrylate

Methacrylate

Two-component (solventless) methacrylate penetrating sealers are often used for flooring systems. They are low viscosity materials with the ability to penetrate the surface and fill capillaries of concrete floors. Their usefulness is limited by the fact that they are not compatible with a variety of generically different topcoats (Fig. 1d).

Preparation of Substrates

Surfaces of Concrete

Concrete is highly heterogeneous, particularly at the surface that is usually destined to receive a coating. The surface of concrete has several distinctive characteristics that affect the success of coatings. Due to the wall effect, aggregate sizes are strongly graded against cast surfaces, resulting in a

concentration of fine aggregates and consequently higher mortar fraction at surfaces. As soon as formwork is removed, evaporation from the cast face begins, and the surface of concrete begins to lose water needed for hydration. Hydration is affected when the relative humidity in the concrete falls below about 80%. Therefore, unlike the bulk of concrete, cement in the zone near the surface rarely gets the benefit of extended curing. Surfaces of curing concrete often absorb form oil and receive coatings of film-forming curing agents (intended to reduce evaporation of water from the surface) that affect the ability of the surface to receive coatings.

Effects of Water in Concrete Surfaces

The matrix of cement paste shrinks and swells as changing humidity levels in concrete change the forces of water in fine

capillary spaces. Differential movement between cement paste and aggregates, which are volumetrically stable and cause restraint, results in cracking of the matrix between and around aggregate. Shrinkage and temperature cracking often originate at the surface and are therefore important to coating behavior.

The porosity of concrete is distributed throughout the matrix as a wide variety and size of spaces. Pore sizes available to coatings range from about 100 nm to 5 mm in size. They are typically formed as capillary voids left behind by water and cementitious particles consumed in the hydrating cement paste, by air bubbles entrained or entrapped in the cement paste, by aggregate porosity, and by cracking. Much of the porosity of concrete is not directly accessible to coatings since large internal pore spaces are often occluded by cement paste containing small pores. The large internal pore spaces are not typically linked to the surface by large diameter spaces unless they are intercepted by cracking. Figures 3 and 5 show images of typical cracks, porosity, surface roughness, and blasting damage on cured and green concrete.

Water is mixed into typical plastic structural concrete at about 7% by mass, $1/2$ to $2/3$ of which is eventually consumed in hydration. Water is free to move in the hardened pores of concrete. The unconsumed mixing water and any water absorbed from the outside environment into the system are held at quite high energies because of the surface tension in the menisci, or curved water surface, across small diameter pores. Internal relative humidity in concrete is strongly linked to degree of saturation and the pore size distribution. In typical Portland cement paste at 73 F (23 C), a relative humidity of about 97% is reached when pores smaller than 40 μm are filled with water. The potential energy of water in pores of this size is calculated to be about 400 m (1,300 ft) of water

head in tension. Typical rates of wetting for concrete can be described as sorptivity and range from 0.5 to 2.5 $\text{mm}/\text{s}^{1/2}$. Drying rate for a typical good quality concrete has been measured, and results show that at 8 mm from the surface, it takes about 20 days of continuous drying for the internal humidity to drop to about 85%. (This corresponds to pores greater than about 10 μm being emptied.)³

Effect of Surface Preparation on Concrete

Surface preparation on concrete usually involves abrasive or water blasting to produce a clean, sound surface to receive a coating. Most surface preparation has the deliberate effect of selectively removing softer material, usually cement paste. This effect exposes aggregate surfaces and porosity behind a cast surface. The abraded surface usually has some remnant surface damage or "bruising" that is of a scale consistent with the abrasive effort that went into the surface preparation.

It is axiomatic that the quality, and, hence, longevity of a coating on concrete depend upon the ability of the coating to contend with several factors: contamination; roughness; large and fine open porosity; water and water vapor (sometimes not very deep) in that porosity; and often, unconsolidated material at the surface. The coating with water surface tensions and water vapor must affect its curing, adhesion, and depth of penetration when applied to concrete in the field.



Fig 3 - Coatings on green concrete and paste; aggregate is fractured from blasting



Fig 4 - Penetration of coating A into fine porosity of the surface

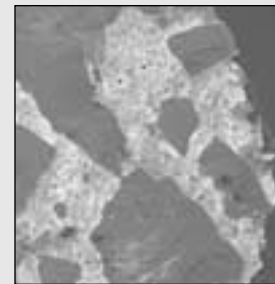


Fig 5 - Cracks, porosity, surface roughness, and blasting damage on cured concrete

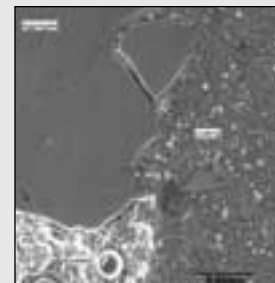


Fig 6 - Coating K2 applied to mature concrete is adhering well but has not fully saturated the crack



Fig 7A - Coating A on rust infiltrates larger porosity and rust-steel interface



Fig 7B - Coating B on rust penetrates rust and interface



Fig 7C - Coating C on rust — no penetration



Fig 7D - Coating D on rust — no penetration

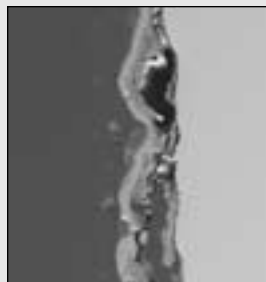


Fig 7E - Coating C on rust — good penetration into large porosity; some rust consolidation

Behavior of Coatings on Rust

Corrosion of steel results in metal loss at the anode where metallic iron goes into solution as ferrous ions accompanied by two electrons. In the immediate vicinity of the anode, these ferrous ions react with hydroxyl ions (produced by oxygen de-polarization at the cathode), and ferrous hydroxide is produced. This area closest to and above the anode is referred to as Zone 1. Ferrous hydroxide then reacts with oxygen to form black Fe_3O_4 , and this area of incompletely oxidized iron is known as Zone 2. Moving further above the anode is Zone 3, where oxygen reacts with black Fe_3O_4 to provide fully oxidized, hydrated rust, $Fe_2O_3 \cdot nH_2O$. This rust is recognized by its brown-red color.⁴

Two approaches have been adopted with respect to coating application over rust, namely, rust conversion and rust stabilization. The former involves applying oxidizing solutions that fully oxidize (convert) the rust into the trivalent state of iron, followed by the application of a coating. The second, and much more successful approach, involves direct application of penetrant sealer primers to the rust with subsequent application of high-performance coatings that act as barriers to the ingress of oxygen, water, and corroding ions.

Rust is heterogeneous and somewhat loose and friable unless removed by mechanical means—say brushed with a firm wire brush. If one applies a penetrating sealer that soaks into the rust, then the sealer acts as a

binder that consolidates it, making it cohesively stronger and potentially assisting in binding it to the steel (substrate). The authors have undertaken pull-off adhesion tests that confirm this phenomenon, and SEMs in Figs. 7 a-k show the rust being partially wetted out and penetrated by 10 tested coatings: A, B, C, D, E, F, H, I, J, and K.

Low molecular weight, 100% solids epoxy, and solvented moisture-cure urethane penetrant sealers are therefore considered by some to be suitable for this task and thus warrant investigation. It has been posited that pigments in thinned down high-build epoxies and solvented moisture-cured urethanes are largely unable to pass through crevices in the rust and thus sit on its surface while the resin partitions and wicks into the rust matrix.⁵

Behavior of Coatings on Inorganic Zinc

A three-day-old inorganic zinc coating was chosen as an experimental substrate because of its very porous matrix and poor cohesion properties. This substrate is not typically coated with penetrating primers in service but was chosen as a test substrate to view the interaction of the coatings with a highly porous substrate 50 to 150 μm (2 to 6 mils) thick. The porosity would therefore show how well the candidate coatings penetrate and fill the interstices in the zinc film. Furthermore, when the porous inorganic zinc is topcoated, air displacement occurs. The air displacement is potentially problematic because it can pinhole and crater coatings during cure.

Properties of an Ideal Penetrant Sealer

Ideally, penetrant sealers for concrete and rust will be molecularly mobile, solventless, and flexible. The cured films should contain low cross-link density so that good penetration volumes are achieved and in-

ternal stress is minimal. Low-build primers result in minimal shrinkage and curing stresses. At the outset of this study, a concern with the application of thick films of high-build epoxies was the predicted lack of penetration into rust and stress attenuation, leading to possible delamination.

The box on p. 39 is a schematic diagram that compares the theoretical mechanical adhesion and capillary action of a pigmented/solvented high-build epoxy or moisture-cured urethane with that of a 100% solids, unpigmented epoxy penetrant sealer. In the case of the pigmented/solvented coatings, the cure process involves shrinkage (hence internal stress development) as well as pigment particles being filtered out on the surface of the substrate. Penetration is depicted as poorer than in the case of the water-thin unpigmented/solventless epoxy which, in contrast, fills voids and crevices.

Desirable properties of a penetrant sealer or primer for use on concrete and rust include the following:

- significant penetration into voids;
- high degree of wetting, adhesion, and capillary action (low viscosity);
- 100% solids (solvent-free);
- unpigmented;
- zero or low shrinkage/internal stress;
- a prolonged period where the penetrant sealer remains wet prior to cure;
- moisture tolerant—displace or react with water;
- carefully balanced rate of cure;
- optimal application (brush, roller, and spray) and flow characteristics;
- minimal stress at the substrate-coating interface;
- capable of consolidating concrete or rust;
- low dry film thickness;
- broad spectrum of compatibility with generically different coatings; and
- high surface tension that provides more driving potential for absorption, low molecular weight, and low cross-link density.

Experimental and Test Procedures, and Results

Concrete Panel Preparation

Two sets of concrete panels were used in the experiments to simulate typical field cure conditions in structural concrete. The concrete mixes used were 30 MPa (4,500 psi) with 5% entrained air, and W/C ratio of about 0.45, maximum 20 mm (0.75 in.) siliceous aggregates, and 15% fly ash replacement of cement. The first set was cast as flat work in the autumn of 1995, cured under damp burlap for 3 days, and then field-cured stacked in the shade outdoors in Vancouver. The second set was cast flat in wooden forms in July 1998, cured for three days under burlap, and then left outdoors until required for painting on the seventh day.

As a result of the sample preparation process, both concretes represented a typical commercially produced CSA A23.1 Class F-1 structural concrete⁶ from the same ready mix supplier, which had similar compositions but with different maturities. The second set was considered to be green concrete: it had higher moisture content than the first set, less paste density, more shrinkage cracking, and more abrasion damage. It had a strength of slightly more than 20 MPa (3,000 psi) at the time of coating. Both concretes had significant moisture content because of their exposure to Vancouver climate without direct insolation. Both concretes were conditioned indoors for almost 48 hours and then abrasive blasted on the bottom (cast) face the day before coating application.

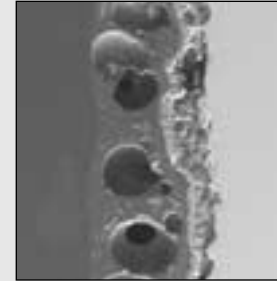


Fig 7F - Coating F on rust — no penetration; voids in coating

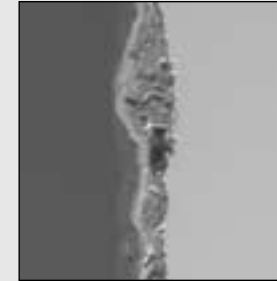


Fig 7H - Coating H on rust — some absorption of binder into small surface porosity of rust



Fig 7I - Coating I on rust

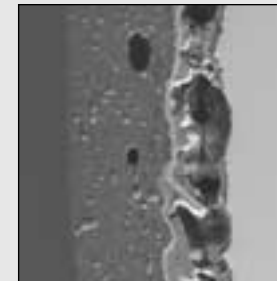


Fig 7J - Coating J on rust — some bubbling

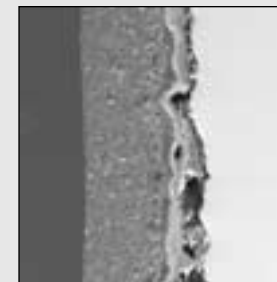


Fig 7K - Coating K on rust — some bubbling above rust; no penetration

Table 2
Application of Coatings to Test Substrates

| Coating Code | Temperature of Steel F (C) | Atmospheric Relative Humidity (%) | Power Mixing Required | Application Tool |
|--------------|-------------------------------|-----------------------------------|-----------------------|------------------|
| A | 21 (-6) | 72 | No | Cup Gun |
| B | 19 (-7) | 78 | No | Cup Gun |
| C | 17 (-8) | 79 | Yes | Pressure Pot |
| D | 17 (-8) | 78 | Yes | Cup Gun |
| E | 19 (-7) | 83 | No | Cup Gun |
| F | 19 (-7) | 82 | Yes | Cup Gun |
| G | 25 (-4) | 68 | Yes | Cup Gun |
| H | 19 (-7) | 90 | Yes | Cup Gun |
| I | 18 (-8) | 78 | Yes | Cup Gun |
| J1 | 20 (-7) | 62 | Yes | Pressure Pot |
| J2 | 20 (-7) | 62 | Yes | Pressure Pot |
| K1 | 19 (-7) | 90 | Yes | Pressure Pot |
| K2 | 19 (-7) | 90 | Yes | Pressure Pot |
| L1 | 19 (-7) | 70 | Yes | Pressure Pot |
| L2 | 19 (-7) | 70 | Yes | Pressure Pot |
| M | 22 (-6) | N/A | No | Squeegee |

Rust and Inorganic Zinc Panel Preparation

Four 3 ft x 3 ft x 0.25 in. (1 m x 1 m x 6 mm) carbon steel plates were abrasive blasted to Near-White Metal (SSPC-SP 10) with a surface profile of 1.5 to 2 mils (38 to 50 µm). Two plates were placed at 68 F (20 C) in an International Electrotechnical Commission weathering chamber, IEC 1109. For 96 hours, a 5- to 10-micrometer (0.2- to 0.4-mil) diameter distilled water fog was passed across the panels at a flow rate of 400 ml/m³/hr. Having uniformly rusted, the plates were taken from the chamber, laser cut into 7 in. x 7 in. (180 mm x 180 mm) panels, and stored in polyethylene bags. The thickness of rust build on the steel ranged from 25 to 170 µm (1 to 7 mils).

The other two abrasive blasted 3 ft x 3 ft x 0.25 in. (1 m x 1 m x 6 mm) carbon steel plates were saw cut into 7 in. x 7 in. (180 mm x 180 mm) panels. One side of each panel was coated with a single coat of an alkyl silicate inorganic zinc at either 2 to 4 mils (50 to 100 micrometers) dry film thickness or 4 to 6 mils (100 to 150 mi-

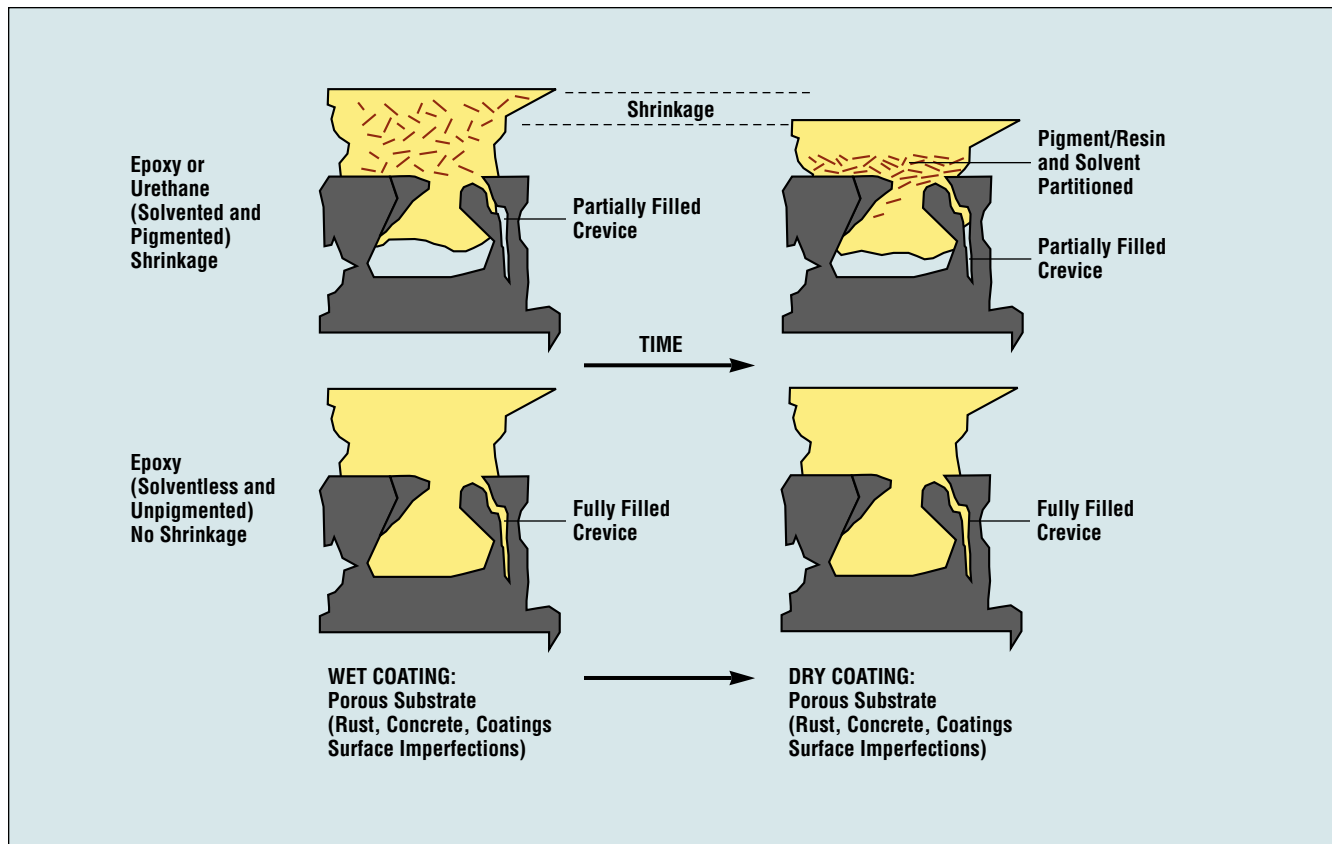
cometers) dry film thickness. The thicker film thickness was specified to accentuate mudcracking and to increase air release. The panels were topcoated with one of the 12 candidate coatings within three days.

Spray Application of Coatings

All coating systems were applied on the same day using a cup gun, pressure pot, or squeegee, depending upon the viscosity of the materials used (Fig. 2). Details are shown in Table 2. After application of each coating system to a set of different substrate/panels in a tray, the tray was placed on a shelf in a custom-made cabinet and the coatings were cured for seven days at approximately 70 F (21 C).⁷

Scanning Electron Microscopy/Optical Microscopy

Samples for the microscopy were prepared by cutting the steel samples with a fine bandsaw, potting in epoxy pigmented with silica fume, and polishing. Concrete samples were cut to thin slices with diamond saws, and polished to 6 µm thin sections



Theoretical Mechanical Adhesion and Capillary Action

on glass. The scanning electron microscopy (SEM) instrument used in the work was a Hitachi S2500. All images were Backscattered Electron Images (BEI) and produced at 25kV accelerating voltage with high resolution digital imaging and editing. Micrographs of the coating to substrate interface were taken to examine the penetration and saturation of the substrate under the coating. Where the SEMs did not clearly present the depth and nature of the coating penetration, optical micrographs were taken.

Behavior of the Coatings under the Microscope

Figures 3 to 7 are representative of the results obtained from the examination of the coating-substrate interfaces. The entire series of nearly 130 optical and SE micrographs could not be published here. Table 6 summarizes measurements and observations taken from the entire series.

Micrographs in Figs. 3 to 6 show representative behavior of the coatings on concrete substrates. In Fig. 3, the green concrete aggregate and paste, and coating

A are shown. Fracturing of the aggregate and paste due to abrasive blasting can be seen at the concrete surface. Penetration of the coating into the cracks and paste up to 0.3 mm (12 mils) from the surface is visible. It can be seen that without wetting and penetrating ability to bind together the damaged surface, the coating would be vulnerable to delamination on this type of surface. Figure 4 shows the interaction of coating A and the surface of the mature concrete. Penetration of the epoxy into the fine porosity at the surface is visible.

Figure 6 shows the surface of the mature concrete with coating K2 applied to it. The paint is well adhered but has not fully saturated the crack in the broken aggregate particle.

The series of micrographs in Figs. 7a to k show the behavior of the coatings on a loose rust substrate. Some penetration of coating into the rust is visible in Figs. 7a, b, e, and h. It must be said that none of the coatings thoroughly saturated the rust or completely consolidated the rust to the steel surface. Under the conditions tried,

coatings A, B, E, and M were the best rust penetrants. Coating H had interesting mobility into rust, given that it is pigmented.

The amount and depth of absorption into the zinc were difficult to quantify, and, therefore, the inorganic zinc substrate did not serve well to differentiate individual penetrating abilities.

Adhesion and Dry Film Thickness Tests

The adhesion of each coating to rust, concrete, steel, or inorganic zinc was measured by adhesion testing to ASTM D 4541, Standard Test Method for Pull Off Strength of Coatings Using Portable Adhesion-Testers. In this test, three aluminum dollies are bonded to the coating film; the force required to remove the dollies is measured; and the area of disbondment is observed and documented.

Coating thicknesses were measured in accordance with SSPC-PA 2, Measurement of Dry Coating Thickness with Magnetic Gages. Averaged results of the coating adhesion and thickness tests are shown in Table 4. A large majority of the fracture surfaces in this work occurred within the zinc primer, the concrete paste, or rust layers. An explanation of this phenomenon is that the internal strength of the coatings was superior to the strength of the substrates they were attempting to penetrate.

Viscosity Measurements

Viscosities of the coatings were measured using a Ford #4 cup, ASTM D 1200 and/or a Krebs Stormer viscometer, ASTM D 562. Table 3 shows viscosity measurements.

Wicking Experiments/Halo Effect

A test described by Rosler and Buesing was used to compare the absorption potential of the coatings.⁸ One- milliliter samples of the products were drawn up into syringes and dropped onto flat samples of each substrate (steel, rusted steel, zinc, mature concrete, green concrete) and observed. A

Table 3
Measured Viscosities of Coatings

| Coating Code | Viscosity Ford #4 Cup (s)* ASTM D1200 | Viscosity Krebs Stormer ASTM D562 |
|--------------|--|--------------------------------------|
| A | 13 | — |
| B | 17 | — |
| C | 334 | 105 |
| D | 54 | 33 |
| E | 49 | — |
| F | 192 | 93 |
| G | 180 | 90 |
| H | 63 | — |
| I | 38 | — |
| J1 | 43 | 61 |
| J2 | 14 | 50 |
| K1 | 16 | 53 |
| K2 | 10 | 49 |
| L1 | 45 | 63 |
| L2 | 17 | 52 |
| M | 8 | — |
| J unthinned | — | 110 |
| K unthinned | 168 | 89 |
| L unthinned | — | 96 |
| Water | 7 | — |

*s = seconds

sixth substrate consisting of sheets of fiberglass cloth lying on glass plate covered with a polyethylene sheet was included in the experiment. The sample products were left to flow or diffuse into the substrates until they hardened. The area of substrate and cloth saturated by the coating and the radius of the “fringes” where they were observed were measured (Table 5 and Fig. 8).

The fringes or halos occur at the wetting front where coating product drawn ahead of the puddle has not saturated the substrate, or where pigmentation or filler particles have been filtered out as the coating's binder wicks through the substrate. Filtering occurs when coatings are moved by absorption into porous spaces smaller than the size of the pigment particles. Figures 9a and 9b show the spreading of 1 ml of coatings I and M on fiberglass mat and the resultant wicking or capillary action.

Table 4
Measured Adhesions of Coatings to Substrates (Averages of Three Tests)

| Coating Code | Film Thickness (mils)** on Rusted Steel | Adhesion on Substrate (psi)* | | | | | | |
|--------------|---|------------------------------|------------------------|---------------------|-------------------|--------------|-----------------|----------------|
| | | Viscosity Ford #4 Cup (s)* | Shot-Blasted Steel**** | Zinc Primer Regular | Zinc Primer Thick | Rusted Steel | Mature Concrete | Green Concrete |
| A | 4 | 13 | 683 | 700 | 1,000 | 708 | 458 | 383 |
| B | 4.4 | 17 | ***** | 817 | 717 | 375 | 425 | 467 |
| C | 9 | 334 | — | 633 | 692 | — | 428 | 342 |
| D | 6.5 | 54 | — | 667 | 750 | 325 | 442 | 542 |
| E | 7.4 | 49 | — | 925 | 408 | 150 | 392 | 383 |
| F | 11 | 192 | — | 542 | — | 125 | 233 | 392 |
| G | 8.9 | 180 | — | 425 | — | 167 | 350 | 442 |
| H | 7.9 | 63 | — | 1000 | — | 483 | 350 | 367 |
| I | 7.6 | 38 | 1,000 | 433 | — | 350 | 475 | 533 |
| J1 | 9.5 | 43 | — | 867 | — | 500 | 408 | 567 |
| J2 | 13.4 | 14 | — | 758 | — | 792 | 442 | 367 |
| K1 | 10.4 | 16 | 1,000 | 700 | — | 600 | 575 | 417 |
| K2 | 13.4 | 10 | — | — | — | 683 | 517 | 392 |
| L1 | 8 | 45 | — | 767 | 1,000 | 317 | 375 | 367 |
| L2 | — | 17 | — | 600 | — | 293 | 700 | 633 |
| M | 1 | 8 | 633 | 542 | — | 750 | 492 | 392 |

* 1 psi x 6.895=kPa

** 1 mil = 25.4 μm

*** s = seconds

**** Although not discussed in the text, shot-blasted steel was included for comparative purposes

***** Data were omitted where failure mode was not representative.

Discussion

The adhesion test results show that the strength of bond to surface-dry but internally damp green and mature concretes is governed by more than penetrating ability. In general, the best adhesion values were produced by the epoxies, with the thinned high-build epoxies performing as well as or better than the penetrating epoxies. The methacrylate and polysiloxane also bonded well to both materials. The adhesion values on both substrates were nearly equal, indicating that maturity of the cement paste was not a strong factor, and possibly meaning that adhesion to exposed aggregate surfaces dominates the adhesion results.

Adhesion of primers to rust and the penetration results correlate strongly to the viscosity of the materials (Tables 4, 6, and 3), suggesting that the abilities to penetrate

loose rust and consolidate the rust to steel interface are functions of viscosity.

In this work, the spreading area of the various coatings on fiberglass mat and rusted surfaces correlates strongly with the observations of penetration in the SEM photographs as shown in Table 7, but not viscosity. This indicates that a spreading or wicking test could be used as an indicator of penetrating ability and could be used as a simple means to review the suitability of coatings as penetrating primers.

As shown in Table 6, the epoxy primers, the methacrylate, and the polysiloxane materials (A, B, C, D, E, M, and I) penetrated the concrete cracks and pores. The binders from pigmented urethane coatings F, G, and H also achieved some penetration into the concrete porosity and wetted the concrete surface even though adhesion values for these materials

Table 5
Measured Spreading and Wicking of Coatings on Substrates

| Coating Code | Wetted Area on Substrate (mm ²) | | | | | Average Radius of Halo Effect on Fiberglass (mm) | | |
|--------------|---|-----------------------|--------------|-----------------|------------|--|----------|--------|
| | Fiberglass | Inorganic Zinc Primer | Rusted Steel | Mature Concrete | Shot Steel | Fringe | Absorbed | Puddle |
| A | 10,608 | 12,272 | 12,272 | 14,849 | 20,869 | 0 | 50 | 0 |
| B | 11,124 | 5,674 | 9,590 | 10,843 | 7,620 | 0 | 50 | 0 |
| C | 6,648 | 4,418 | 6,720 | 8,252 | 6,013 | 4 | 11 | 31 |
| E | 7,088 | 2,507 | 3,318 | 4,717 | 3,848 | 4 | 7 | 38 |
| F | 1,400 | 661 | 755 | 573 | 683 | | | |
| G | 1,452 | 1,018 | 755 | 531 | 908 | 2 | 3 | 17 |
| I | 1,963 | 804 | 661 | 1,104 | 1,018 | 2 | 6 | 17 |
| M | 11,322 | 12,568 | 14,000 | 21,000 | 3,5074 | 0 | 50 | 0 |

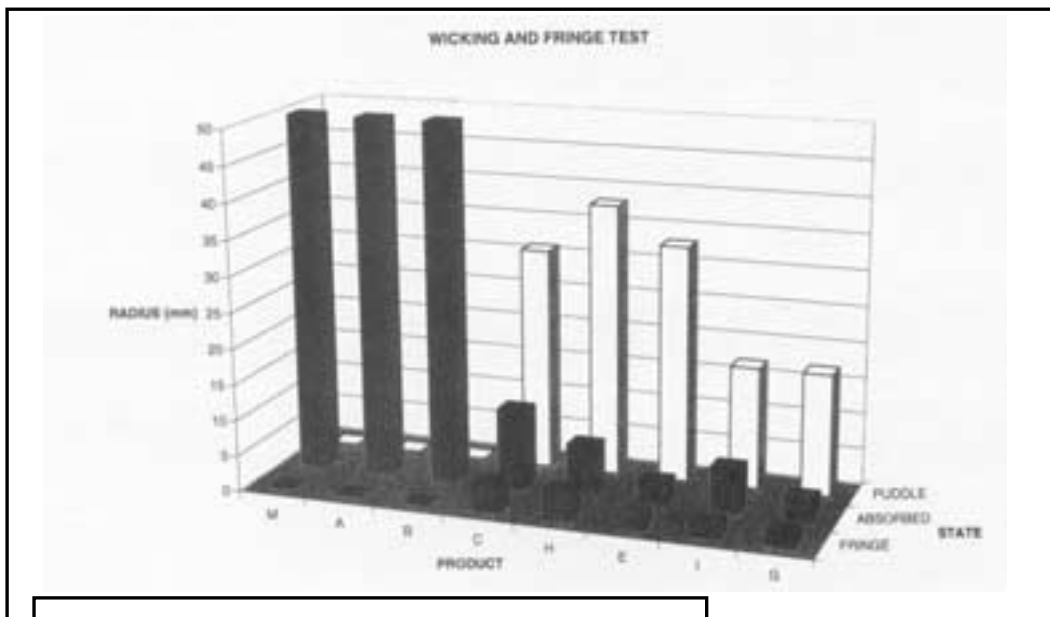
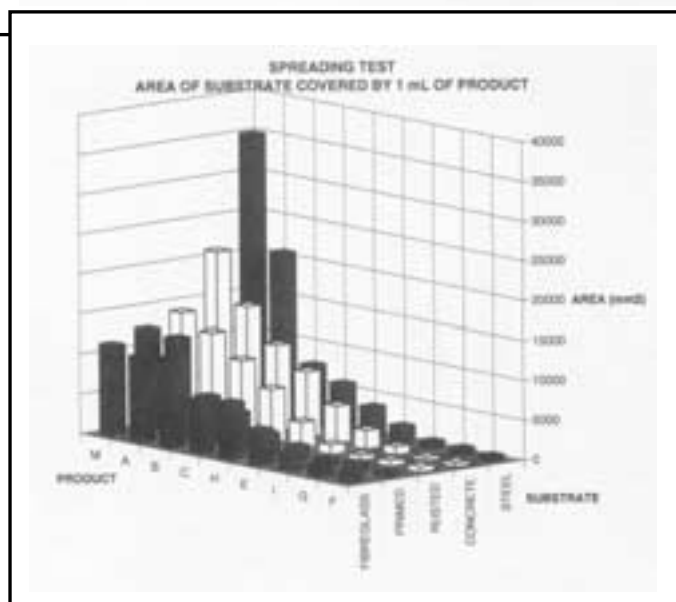


Fig. 8



were lower than for the epoxies. The thinned high-build epoxies had the least penetration into the concretes, possibly because of the higher molecular weight of the polymer, but still had good adhesion. None of the materials penetrated deeply into either the green or mature concrete. The overall depths of penetration into cracks and the paste were similar in both mature and green concrete. Experience in

**Table 6
Data from Photo Micrographs**

| Coating | Substrate | | | |
|---------|---|--|---|---|
| | Rusted Steel | | Inorganic Zinc | |
| | Consolidated Rust | Consolidated Rust Steel Interface | Penetrated Zinc Primer | Reached Primer Steel Interface |
| A | Filled voids to 27 μm, coated larger pores | Good penetration | Penetrated and filled voids to 12 μm | at depth of 150 μm |
| B | Filled voids to 27 μm, coated larger pores | Good penetration but did not saturate as well as A | Penetrated and filled voids to 10 μm | at depth of 150 μm |
| C | Did not consolidate rust or coat pores | Travelled to interface but not as well as A | Penetrated and filled voids to 20 μm | at depth of 150 μm |
| D | Small amount of porosity filled to 20 μm size | No consolidation | Did not fill as many voids as A-C, up to 16 μm | at depth of 150 μm |
| E | Good penetration, filled pores to 67 μm, did not fill small pores < 13 μm | Good consolidation where penetrated | Penetrated at mud cracks, leaves small pores | reached steel at depth of 100 μm |
| F | Little/no penetration | No consolidation | Filled pores larger than 27 μm | did not reach steel |
| G | — | — | Penetrated at mud cracks | not much penetration to steel |
| H | Small amount of porosity filled to 6 μm size | No consolidation | Filler filtered out at surface, small volume of binder penetrated | barely to 150 μm |
| I | No penetration of rust | No consolidation | Good penetration of small pores | penetrated full depth but no accumulation on steel |
| J1 | None | No consolidation | Penetrated to 50 μm | did not reach steel |
| J2 | — | — | Some penetration of surface pores | did not reach steel |
| K1 | Little/no penetration | No consolidation | Penetrated at mud cracks 75 μm | did not reach steel |
| K2 | Little/no penetration | No consolidation | Penetrated only at mud cracks | did not reach steel |
| L1 | No penetration of rust | No consolidation | Penetrated only at some mud cracks | did not reach steel |
| L2 | No penetration of rust | No consolidation | — | — |
| M | Penetration of pores smaller than 7 μm | Good consolidation of interface | Good penetration of small pores | all material absorbed by zinc, little reached interface |

other work by the authors where primers penetrated up to 10 mm into cracks suggests that the primary inhibitor of penetration in this study was moisture in the concrete pores and not the density (maturity) of the matrix or crack sizes.

The large amount of fine, unfilled cracking seen at depth in the green concrete samples was probably due to shrink-

age in the paste during sample preparation for the SEM work. One of the visible indicators of consolidation of concrete porosity was the lack of this fine cracking in zones of penetration in the green concrete.

The surface of the green concrete was not noticeably more damaged by abrasive blasting than the mature concrete.

The epoxies and methacrylate sealer

| Substrate | | | | | | |
|----------------------|----------------|---------------------|----------------------|----------------|---------------------|---------|
| Mature Concrete (mm) | | | Green Concrete (mm) | | | Coating |
| Depth of Penetration | Width of Crack | Depth of Absorption | Depth of Penetration | Width of Crack | Depth of Absorption | |
| no crack seen | 3 | 5 | 600 | 1 | 330 | A |
| 100 | 25 | 1 | — | — | — | B |
| 500 | 5 to 100 | 12 | 1,000 | 5 | 10 | C |
| 1,100 | .08 to 20 | 46 | 600 | <12 | 120 | D |
| — | — | — | 70 | 4 | 170 | E |
| 230 | 10 | none | none seen | 3 | none | F |
| 100 | 2 | 20 | 90 | 7 | 16 | G |
| 70 | none seen | 70 | — | — | — | H |
| 100 | 6 | 60 | — | — | — | I |
| none | — | < 1 | none | — | 20 | J1 |
| none | — | — | 33 | — | 12 | J2 |
| none | — | 15 | none | — | 70 | K1 |
| none | — | 50 | — | — | — | K2 |
| none | — | none | none | — | none | L1 |
| none | — | none | none | — | none | L2 |
| 760 | 0.5 | 1.7 | 660 | 1 | 20 | M |

filled pores of different sizes in the rust test. The epoxies ran into larger pores by preference and left some smaller pores unfilled, while the methacrylate filled only small pores and was barely visible on the inside surfaces of the larger pores. This behavior was reproduced to a certain extent in the wicking and zinc penetration tests. This action is almost certainly due to sur-

face tension and contact angle of the wetting fluid, but these properties were not measured in this work.

The low viscosity penetrant sealers (A, B, C, D, E, and M) soaked into the porous zinc primer without any pinholing or entrapment of air. High-build epoxy coatings can pinhole and crater while they cure on this type of substrate. In pigmented

Table 7
Ranking of Performance in Tests*

| Adhesion to Mature Concrete | Adhesion to Green Concrete | Adhesion to Rusted Steel | Adhesion to Zinc Primer | Viscosity Ford #4 Cup | Wetted Area on Rusted Steel | Penetration into Mature Concrete | Consolidation of Rust |
|-----------------------------|----------------------------|--------------------------|-------------------------|-----------------------|-----------------------------|----------------------------------|-----------------------|
| L2 | L2 | J2 | H | M | M | M | A |
| K1 | J1 | M | E | K2 | A | A | M |
| K2 | D | A | J1 | A | B | D | B |
| M | I | K2 | B | J2 | C | C | C |
| I | B | K1 | L1 | K1 | E | F | H |
| A | G | J1 | J2 | B | H | E | D |
| J2 | K1 | H | K1 | L2 | I | B | E |
| D | M | B | A | I | G | G | F |
| C | K2 | I | D | J1 | F | I | G |
| B | F | D | C | L1 | — | H | I |
| J1 | A | L1 | L2 | E | — | J2 | J1 |
| N | E | L2 | M | D | — | J1 | J2 |
| E | J2 | G | F | H | — | K1 | K2 |
| H | L1 | F | G | G | — | L1 | L1 |
| G | C | — | — | F | — | L2 | L2 |
| F | — | — | — | C | — | — | — |

*Ranked from best to worst, top to bottom

coatings, release of air through a thick film can be impeded by platy pigments that accumulate on top of the zinc film. When these coatings were applied to inorganic zinc, they did bubble at the time of application. However, the authors did not see any significant entrapment or pinholing with inorganic zinc as a primer except for one of the moisture-cured urethanes (F).

Conclusions

The results of this work indicate that good adhesion to green concrete can be achieved by a variety of coatings. Deep penetration into the concrete surface was not required to achieve strong adhesion as is sometimes believed. The behavior of coatings on green and mature concrete seemed much more dependent upon moisture content than maturity of the paste.

This investigation showed that low viscosity, thin-film 100% solids epoxies and methacrylate have generally superior penetration capabilities compared to other pene-

trant primers tested. They were found to have superior penetration into concrete (Tables 4 and 5) and wicking properties (Table 6) compared to thinned high-build epoxies, moisture-cured urethane, and polysiloxane penetrating sealers.

But no primer tested was able to completely impregnate and bind the loose rust surfaces (Table 6 and Fig. 7). This reinforces what is already known to the industry: judicious selection of penetrating sealers and careful preparation of marginal surfaces will reduce coating failures by promoting as good penetration of this critical layer as possible. Loose rust scale must be removed because it will not be saturated by even the thinnest material tested.

The minimum surface preparation of salt-free rusted surfaces should be SSPC-SP 2, Hand Tool Cleaning. Where soluble salts are present, the authors suggest removal with a pressure wash (2,500 psi [14 MPa]) using chemicals that sequester the salts and take them out of the rust to various de-

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Fig. 9A



Fig. 9B

Fig. 9A and B, Spreading of 1 ml of coatings I and M on fiberglass mat and resultant wicking or capillary action

grees. For rust pits and salt nests, a very high pressure wash with the sequestering agent is useful. Test patches should always be carried out where the penetrant sealers are used as primers in an overcoat system.⁹

There is not a marked correlation among the viscosity of a given coating material, its penetration abilities, and its wicking action. Viscosity cannot be used alone as a predictor of penetrating performance since the high-build epoxies thinned to viscosity similar to that of the epoxy primers did not penetrate well.

Using unpigmented primer/sealers provided better wetting and penetration into concrete and rust.

The blocked amine epoxy penetrant sealer (coating A) and a nearly water-thin modified methacrylate sealer (coating M) demonstrated the best penetrating and wicking action. Given that the methacrylate sealer can only be topcoated with itself, the blocked amine epoxy penetrant sealer was identified as the best primer of the 12 coatings, under the conditions tried. These primer/sealers exhibited good penetration and adhesion to damp concrete and on rusted surfaces. □

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