INNOVATIONS IN IMPREGNATION TECHNIQUES
FOR HIGHWAY CONCRETE

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Corrosion of reinforcing steel due to the penetration of deicing salts poses a considerable problem in bridge decks. One approach that has received much attention has been impregnation of the bridge deck with a liquid monomer followed by polymerization to effectively seal the capillary pores against salt intrusion. This approach is technically feasible in the field. The present impregnation techniques, however, are costly in terms of energy, materials, and time, and simplifications and improvement would be desirable. In this paper, results are described of preliminary experiments with sulfur, tar, and mixtures of the two as surface impregnants and with a pressure-mat technique for mechanically assisting monomer or sealant penetration. It is shown that the impregnation of portland cement concrete and mortar by molten sulfur, tar, and an 80:20 mixture of the two yields significant reductions in moisture absorption and increases in strength and that, in the case of concrete slabs, predrying may not be necessary. It is also shown that pressure mechanically applied to patterned rubber mats can effect uniform impregnation with a monomer such as methyl methacrylate or with a sealant such as tar or linseed oil. Such an impregnation could conceivably be effected by using rollers. Thus, initial feasibility of two potential improvements in concrete impregnation processes has been demonstrated.

•EVERY highway department is confronted with the problem of deterioration of concrete bridge slabs. The extensive use of deicing salts induces corrosion of the reinforcing rods and consequent spalling; alternate freezing and thawing compound the problem. Because polymer-impregnated concrete (PIC) (1-20) effectively resists water and salt penetration, and hence freezing and thawing, several research programs have been initiated to apply it to bridge decks in the hope of forestalling or arresting corrosion of the steel by salts and improving strength and abrasion resistance. Other approaches are also under investigation (21). Brookhaven National Laboratory and the U.S. Bureau of Reclamation (1, 2, 3, 5) and the University of Texas (7, 8) showed that surface penetrations [up to 2 in. (5 cm)] of decks could be achieved within a reasonable time by surface-drying and monomer-ponding techniques. The goal of the research at Lehigh and Pennsylvania State Universities (6), however, has been to achieve a 4-in. (10-cm) penetration in sound, salt-contaminated decks that will envelop the steel, seal the pores, and immobilize the salt already present. Such deep penetration has now been demonstrated recently, at least on a small scale (13, 17, 18).

There is considerable room for simplification and improvement of the overall process so that time and cost can be reduced. The purpose of this paper is to describe successful preliminary experiments with two new approaches: (a) a pressure-mat technique for impregnation with organic and other materials and (b) sulfur as an impregnant. This paper also discusses other nonmonomeric materials for hot impregnation.

PRESSURE-MAT TECHNIQUE

Laboratory Experiments

Preliminary impregnation experiments were conducted by using a patterned, flexible rubber pressure mat with methyl methacrylate (MMA), tar (AASHO M-214-65), undiluted boiled linseed oil, and water as impregnants. The technique involves entrapment of the liquid in the cells of the mat and mechanically pressured impregnation of the concrete with the trapped liquid.

Concrete Slab Fabrication

Three 2.0- × 2.0- × 0.5-ft (61- × 61- × 15-cm) concrete slabs were cast from a conventional non-air-entrained mix [water, type 1 portland cement, coarse ≥1-in. (2.5-cm) sand, and limestone aggregate in proportions of 1.00:1.92:6.10:6.75] that had a cement factor of 485 lb/yd^3 (286 kg/m^3) and a 28-day average compressive strength of 4,750 psi (33 MPa). The measured air content was 3 percent, and the slump was 2.5 to 3.0 in. (6.4 to 7.6 cm). The slabs were troweled and cured in a fog room (90 to 100 percent relative humidity) for at least 28 days before they were used. All slabs contained a nominal steel reinforcement at a 4-in. (10-cm) depth. Slabs 1, 2, and 3 were oven dried at 250 to 260 F (121 to 127 C) for 45 hours and cooled overnight before use. Slabs 4, 5, and 6 were dried more rigorously: (a) by a propane torch (17) for 15 hours [maximum surface temperature = 750 F (399 C)] and (b) for 10 hours in an oven at 650 F (343 C).

Impregnation

For the initial experiment, a used, 16- × 20-in. (41- × 51-cm) patterned doormat was used (Figure 1). In subsequent experiments, specially molded neoprene rubber mats (durometer hardness = 70) with similar patterns were used. As shown in Figure 2, a hydraulic tester was used to apply the pressure to a steel loading plate slightly smaller than the mat. A steel frame sealed and clamped to the slab was used as a diked reservoir.

During each load cycle, the load was increased from 0 to 30 kips (133 kN) over a 15.5- × 15.5-in. (39.4-cm × 39.4-cm) area [or from 0 to 125 psi (0 to 0.86 MPa)] within 22 sec and then released within 8 sec. A 30-sec break-in period before reloading was provided to simulate the passage of a roller over a particular cross section of a mat. At peak pressure, liquid squirted out through a small side crack in the slab. This indicated that there was efficient pressure in the diamond-patterned cells. During the pressurization cycle, the liquid level in the framed area rose slowly as the rubber mat was squeezed by the loading head. When the load was released, the liquid level in the dike immediately dropped as the liquid rushed under the mat and filled the cells in readiness for the next load cycle. Figure 3 shows the load cycles used for slabs 1 and 2 (impregnated with MMA and water), and Figure 4 shows the cycle used for slabs 3, 4, 5, and 6 (impregnated with MMA, tar, and linseed oil). Impregnation data are summarized. The rubber mat in slab 1 (MMA) was attacked by the MMA after several hours. In subsequent experiments, however, neoprene mats proved to be quite resistant to MMA.

Polymerization

After impregnation, slabs 1 and 3 were mounted on a pedestal, an insulated steam chamber was placed on top (18), and steam was applied for 6 hours. Before the chamber was mounted, some excess monomer [about 1 lb (2 kg)] was sprinkled over several pounds

Figure 1. Rubber pressure mat.

Figure 2. Experimental setup for pressure-mat impregnation.
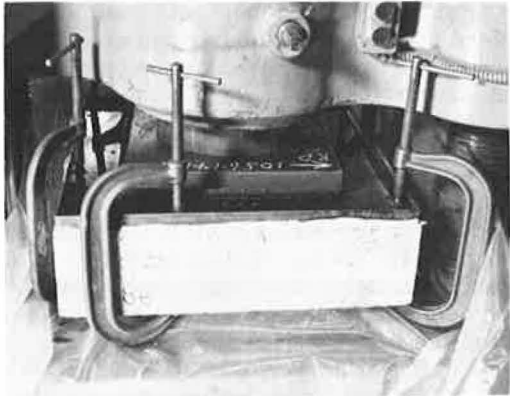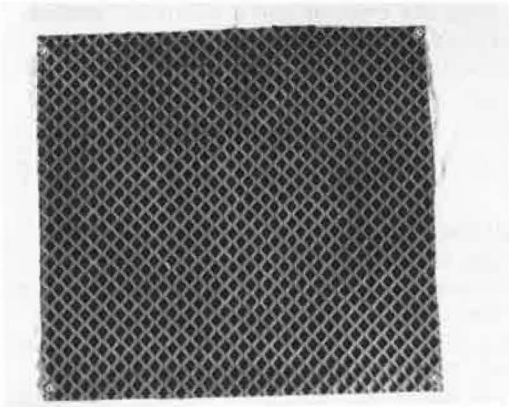

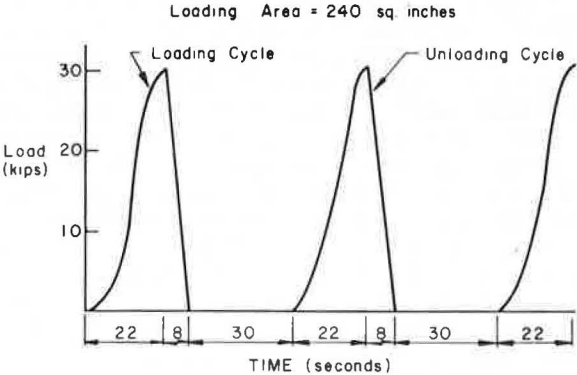**Figure 3. Loading and unloading cycles for slabs 1 and 2.**

Figure 4. Loading and unloading cycles for slabs 3 and 4.
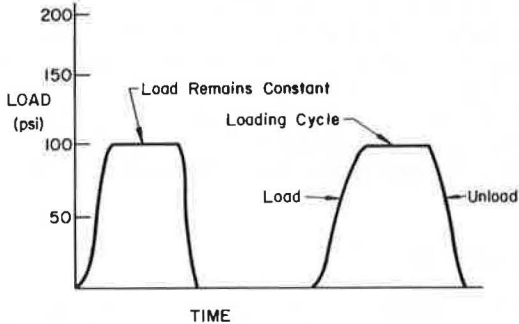

Table 1. Pressure-mat impregnation data.

Slab	Impregnant	Uptake (lb)	Maximum Penetration[a] (in.)	Cycles	Effective Time (hour)	Maximum Pressure (psi)
1	MMA	6	3.5 to 4.0	425	3.2	120
2	Water	7	4.0 to 4.5	710	5.0	100
3	MMA	6	3.5 to 4.0	62[b]	4.0	100
4	Tar	4	2.5 to 3.0	80	5.3	100
5	Tar	3	2.5 to 2.8	160	5.3	100
6	Linseed oil	2	1.0 to 1.5	90	6.0	100

Note: 1 lb = 2.2 kg. 1 in. ≈ 2.5 cm. 1 psi = 6895 Pa.

[a]Corresponds to dense impregnation as observed.

[b]Slabs 3 and 4: 30-sec loading and unloading times, 4-min dwell time. Slabs 5 and 6: 15-sec loading and unloading times, 2-min dwell time.

of cement leveled evenly on the slab area so that loss of monomer by evaporation could
be minimized. Aluminum foil was then placed over the cement and a neoprene gasket
placed between the chamber and slab. The steam pressure was 6 to 10 psi (41 to 69
kPa) at the propane-fired pressure cooker source; the surface temperature of the slab
was 220 F (104 C).

Discussion of Results

As given in Table 1, a 6-in. (15-cm) band of full impregnation was observed in the
central 6 in. (15 cm) of slab 1 (MMA). This depth was greater than that predicted by
using an equation developed previously (18). Around the central region, an impregna-
tion depth of about 2 in. (5 cm) was obtained (Figure 5), and outside the pressure-mat
area, the impregnation depth was ≤1 in. (2.5 m). (The crosshatched area indicates the
limit of dense impregnation.) For slab 2 (water), the predicted depth of penetration
[4.5 in. (11 cm)] was confirmed. A uniform gray color was observed for the top 4.5 in.
(11 cm) of the slab; the bottom 1.5 in. (3.8 cm) of the slab was white. Without the pres-
sure, only about 1.5 in. (3.8 cm) of penetration would have been expected (18).
    For slab 3 (MMA), examination of 3-in.-wide (8-cm) cores revealed dense and uni-
form impregnation to depths of up to 4 in. (10 cm) and evidence of fracture through the
aggregate in the impregnated region (Figure 6). Excellent acid resistance and a major
decrease in water absorption were noted in the impregnated region. Thus, in contrast
to the original slab, which absorbed 7.2 percent of the water by weight (in 24 hours),
the cores absorbed an average of only 0.3 percent of the water: a relative reduction of
96 percent, close to the reductions achieved by using simple hydrostatic pressurization
(17, 18). Only 62 cycles were required to effect this improvement.
    For slabs 4 and 5 (tar), deeper penetrations (Figure 7) were achieved than predicted,
even though the viscosity of 16 cp (0.016 Pa·s) is much higher than the viscosity of
MMA or water, 0.7 cp (0.0007 Pa·s) and 1 cp (0.001 Pa·s) respectively. The treated
depth may be due to the more rigorous drying used. Essentially similar results were
obtained regardless of the differences in load cycling (Table 1). Thus, use of a slow-
moving roller could reduce the number of passes required. For slab 6 (boiled linseed
oil), less penetration was achieved because of the higher viscosity of the oil, 36 cp
(0.036 Pa·s), but the depth was still greater than the few millimeters typically achieved
in the treatment of decks with diluted oil.
    Thus, these experiments confirm our hypothesis that deep and efficient impregnation
with a variety of typical liquids (both monomers and sealants) can be obtained with a
pressure-mat technique. In principle, this technique may eliminate some of the limita-
tions associated with the elaborate pressure impregnation (17, 18) and the slow ponding
technique (6, 13). More experiments are obviously required to refine and quantify this
technique by using different pressure cycles and monomers.

Pressure-Mat Impregnation in the Field

The pressure-mat technique may be expected to be more efficient in the field than in the
simulated laboratory setup. In the field, the loading cycle could be as shown in Figure
4, in which the pressure is applied for a long period of time per cycle (as with a slow-
moving roller). In practice, it should be possible to impregnate a bridge deck (presum-
ably one land width) by using a large mat to cover the required area and a 15- to 20-ton
(14 000- to 10 000-kg) roller to apply the pressure. Conceivably, the new technique
could be used to impregnate large sections or even a whole deck at one time, assuming
that drying would not be a limitation.
    Several concepts of possible field pressure-mat impregnation systems are shown in
Figures 8, 9, and 10. In Figure 8, a closed system with an exterior roller is proposed
to minimize evaporation and toxicity or flammability problems. In Figure 9, the mat is
incorporated in the roller itself. In Figure 10, the area under the roller is sealed ef-

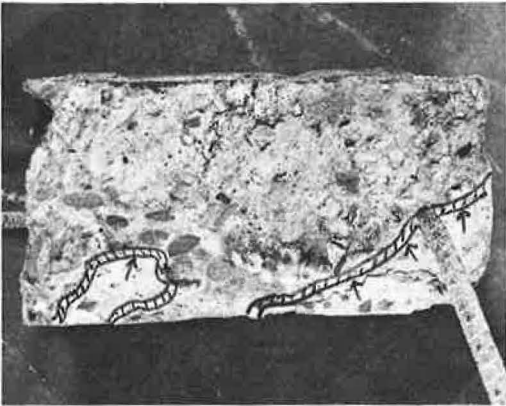Figure 5. Concrete slabs fractured after pressure-mat impregnation with MMA.

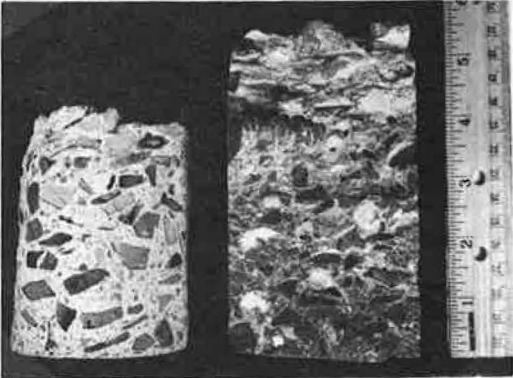Figure 6. Acid-etched (left) and non-acid-etched (right) sections of core from slab 3.

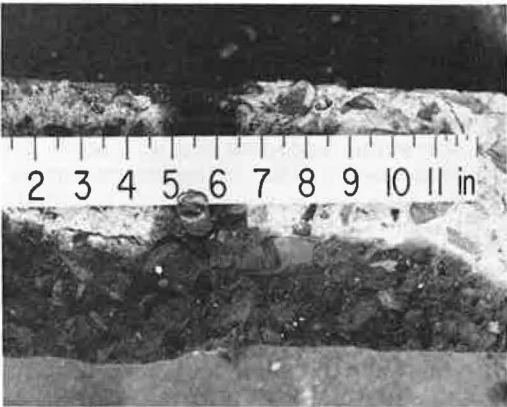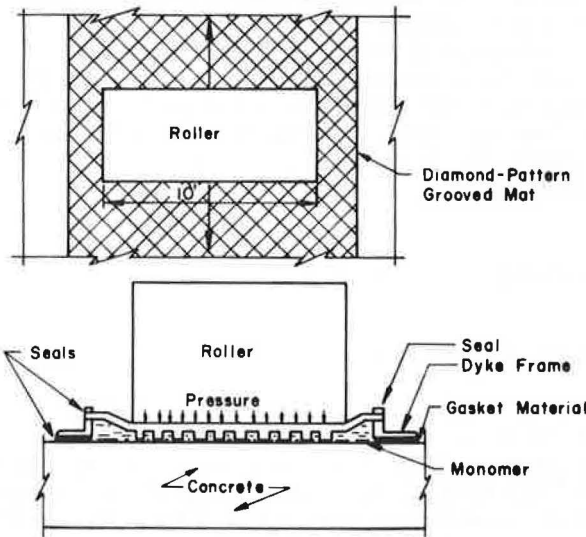Figure 7. Concrete slabs fractured after pressure-mat impregnation with tar.

Figure 8. Roller pressure-mat impregnation in field.

fectively under pressure, and the monomer is fed under pressure through the roller until the interior pressure counterbalances the weight of the roller; ponding could thus be eliminated.

SULFUR IMPREGNATION OF CONCRETE

Sulfur-impregnated portland cement concrete (SIC) (22) yields compressive strengths as high as 25,000 psi (172 MPa), a value higher than for typical PIC, and a loading of only 8 percent of sulfur by weight (9 percent by volume). Because sulfur is about 30 times cheaper than MMA, SIC is of interest as an alternate to PIC. In view of the good freeze-thaw resistance and low water permeability of sulfur concrete (23) and the excellent resistance of sulfur to acids and salts (24), sulfur may have potential applications in bridge decks, provided that no steel corrosion problems arise because of side reactions with water and oxygen. Furthermore, currently available methods for bridge deck impregnation require drying and cooling the deck, impregnating the concrete, and polymerizing the monomer in the pores of concrete. All of the four steps are expensive and time-consuming. For example, drying at a low temperature, say, 250 F (120 C), requires an inordinately long time and, consequently, high costs; drying at a high temperature, say, 600 F (315 C), reduces the time drastically but may, on a large scale, be limited by expansion or cracking of the deck. The use of sulfur may permit use of a simpler process.

Two inherent advantages of using sulfur are that it melts between 235 and 248 F (113 and 120 C), and the viscosity of the molten sulfur remains relatively low, from 12.5 cp (0.0125 Pa·s) at 248 F (120 C) to 6.6 cp (0.0066 Pa·s) at 320 F (160 C). Above this temperature, the sulfur becomes highly viscous because of polymerization (probably undesirable in this case). Also, in the impregnation of a bridge deck with a monomer such as MMA, the high vapor pressure and flammability of the monomer require that the deck be allowed to cool down before impregnation. Thus, an enormous amount of energy is lost: energy that could be used to reduce the viscosity of materials such as sulfur (after drying of the concrete) for easy penetration into the concrete. Moveover, such energy could be conserved if drying could be effected concurrently.

We propose to combine the three steps of drying, cooling, and impregnation into one and to eliminate the polymerization step required for a monomer. The basic idea is to use sulfur to simultaneously dry and impregnate concrete by covering the deck with molten sulfur. The underlying hypothesis is that sulfur will act as an effective heat transfer medium and dry the deck in advance of the penetration front. Of course the portion to be impregnated must be kept at a temperature high enough to avoid solidification in the pores.

When a slab is heated from the surface, the moisture migrates from the hot surface down to the cool underside and condenses in the cooler region (17, 18). As the water migrates downward from a surface heated with molten sulfur, the molten sulfur may follow the water migration because of the suction created in the pores by the moisture migration. If so, the time required for the process should be equivalent only to the drying time.

Laboratory Experiments

Cylinder Fabrication

Experiments were conducted by using 3-in. × 6-in. (8-cm × 15-cm) cylindrical specimens prepared from a conventional non-air-entrained mix [water, type 1 portland cement, coarse ≤1-in. (2.5-cm) sand, and limestone aggregate in proportions of 1:2.0:3.42:4.16] that had a 28-day average compressive strength of 4,000 psi (28 MPa). The measured air content was 3 percent, and the slump was 4.5 in. (11 cm). All cylinders were cured in a fog room (90 to 100 percent relative humidity).

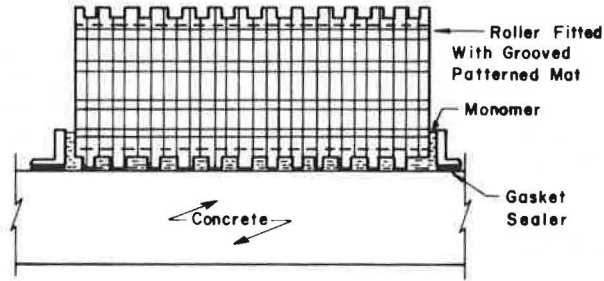Figure 9. Rubber mat fitted on roller for pressure impregnation.

Roller Fitted With Grooved Patterned Mat

Monomer

Gasket Sealer

Concrete

Figure 10. Pressure impregnation by rubber mat fitted on roller.

Gasket

End Steel Plate

End Steel Plate

Treads of Rubber Mat

Roller

Rubber Mat

Pressure

Monomer
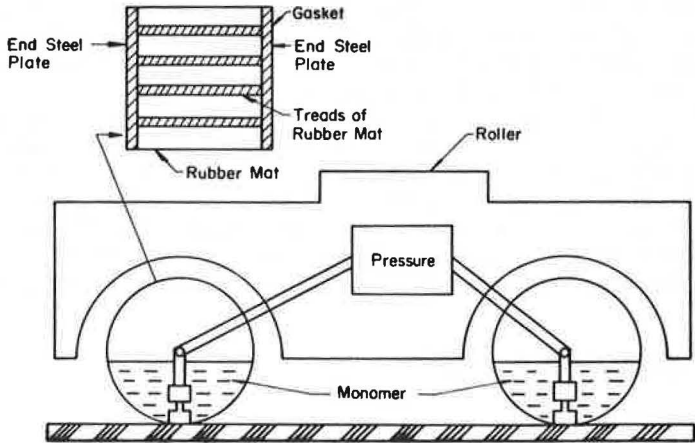
Figure 11. Experimental setup for sulfur impregnation.

Electric Oven 260°F Temp.

3"x6" Conc. Cylinder

Beaker

Moisture Migration
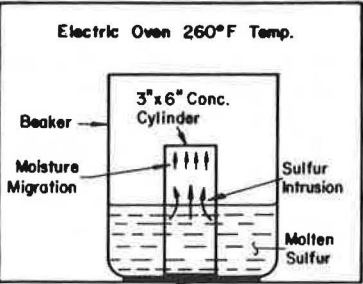
Sulfur Intrusion

Molten Sulfur

Figure 12. Sulfur-impregnated cylinder after compressive failure.
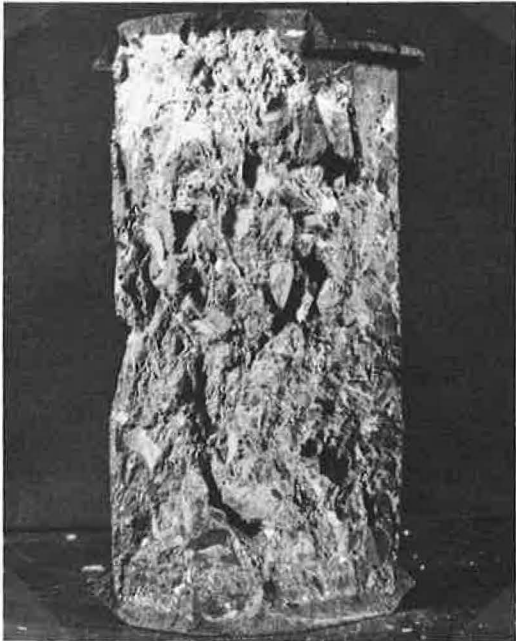
Figure 13. Relative strength of sulfur-impregnated specimens versus sulfur loading.

○ Compression
● Split Tension

RELATIVE STRENGTH

PERCENT LOADING OF SULFUR
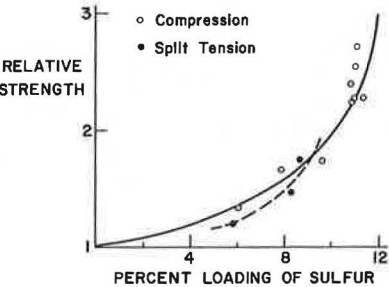
In the first experiment, a cylinder was half-immersed in a molten sulfur bath (Figure 11) and held in an oven for 45 hours at 240 to 260 F (116 to 127 C). Impregnation times for the additional specimens were either 45 or 60 hours. Except for two specimens that were dried beforehand, all specimens were immersed moist in a propane-fired sulfur bath held in the range 250 to 281 F (121 to 138 C).

Slab Impregnation

To simulate the case of an actual bridge deck, the impregnation of two 2.0- × 2.0- × 0.5-ft (61- × 61- × 15-cm) concrete slabs with sulfur was carried out. The experiment with slab 1 was to determine the effect of an applied vacuum on the rate of penetration; the experiment with slab 2 was to determine the effect of a higher porosity. Slab 1 was prepared as it was for monomer impregnation. Impregnation was effected in the moist slab by partial immersion for 45 hours in a propane-fired molten sulfur bath described above. During impregnation, slab 1 was evacuated [25 in. (64 cm) of mercury] from the top surface [effective area, 16 in. (41 cm) wide] with the vacuum chamber described previously (18). After 45 hours, the vacuum was released and the slab was removed and allowed to cool. Slab 2 was prepared similarly except that, to increase porosity, the slab was steam-cured at a low pressure for 24 hours and the air content was increased to 8 percent. Impregnation was effected as with slab 1, except that the vacuum chamber was not used; the top surface was covered with an asbestos sheet.

Discussion of Results

Cylinders

When the cylinders were removed from the bath and cooled in water, a hard, scratch-resistant surface coating of sulfur was observed. When the cylinder from the first experiment was broken open, the sulfur had penetrated completely through the cylinder and had followed the moisture migration path to within 1 in. (2.5 cm) of the upper surface. This is indicated by a change in color of the matrix from white to deep gray (Figure 12). However, examination of a fracture surface indicated that full loading had not been obtained throughout. Although fracture path passed through the aggregate particles in the outer 0.5-in. (12.7-mm) shell, some aggregate pullout, indicating a lower loading of sulfur, was observed in the central region. This was also seen when the cylinder was acid etched; the outer 0.5-in. (12.7-mm) shell was not affected by 15 percent hydrochloric acid, but the core was somewhat affected. It was concluded that full impregnation would require still longer times (but short compared to the usual drying, cooling, and pressurizing times) or applied pressure.

Sulfur loadings for the additional specimens ranged between 6 to 10 percent by weight (at 45 hours) and 11 percent by weight (at 60 hours). Water absorption was reduced by an average of 60 percent for the six 45-hour specimens and by an average of 97 percent for the six 60-hour specimens. As shown in Figure 13, both compressive and tensile strengths increased as sulfur loading increased.

The maximum strength obtained was nearly three times that of the control: an improvement typical of the MMA type of PIC (1-19). The predried specimens exhibited strengths and water absorptions in the same range as the nondried specimens.

A high level of loading and reinforcement was conferred by the sulfur (60-hour impregnation) and was evident in observations of fracture behavior. During compression testing, all such specimens failed explosively. The strong but brittle MMA-impregnated concrete (3, 10, 14, 15) also failed explosively. With the exception of a few regions that exhibited aggregate pullout (such as the cores of less fully impregnated specimens), fracture tended to proceed through, rather than around, the aggregate. The degree of impregnation was also reflected in the improved acid-etched behavior in 15 percent hydrochloric acid (Figures 14 and 15).

Thus, the impregnation of concrete with sulfur yields improvements in strength, water permeability, and acid resistance that are comparable to those obtained with MMA. The strength improvement has been noted previously (20); the lower water permeability and acid resistance have not.

Slabs

For slab 1, examination of cores revealed uniform sulfur penetration to a depth of 0.75 in. (1.9 cm), a depth about one-half the value predicted (18, equation 1 and Figure 7). One reason for the deviation may be ineffective wetting of concrete with sulfur; the use of vacuum in this case did not appear to be beneficial. Better results were obtained with the more porous slab 2, for which cores show a uniform penetration of about 1 in. (2.5 cm) and a 0.5-in. (10-cm) band of partial impregnation (Figure 15). As noted with cylinders, the fracture occurs through the aggregates in the more fully impregnated regions.

Thus, in practice, surface ponding with sulfur may be useful for the shallow impregnations being considered for new bridge decks (or, perhaps, older decks in which salt has not penetrated far). If deeper penetration is required, pressure may be used to facilitate impregnation (18). Tests of freeze-thaw stability and rebar-corrosion resistance remain to be performed.

OTHER NONMONOMERIC MATERIALS FOR HOT IMPREGNATION

Principles developed in this and previous papers should be able to be used for impregnating concrete bridge decks with a wide variety of nonpolymer impregnants (22), such as low-viscosity tar, linseed oil, and inorganic penetrants. Deep-drying temperatures or those high enough to effect concurrent drying, as well as low material costs, could be significant advantages, especially if a suitable pressure mat can be developed. Further, the environmental and fire hazards should be much less important for these materials than for volatile monomers such as MMA. Preliminary experiments with the impregnation of portland cement mortar with tar, tar-sulfur combinations, and barium hydroxide are described in the following.

Laboratory Experiments

Mortar specimens [1.0 × 2.0 in. (2.5 × 5.0 cm)] were prepared by using a mix [water, portland cement (type 1), and Ottawa sand in proportions of 0.5:1.0:2.7] that was cured for 28 days. These specimens were immersed in the impregnant for 24 hours at atmospheric pressure and at a temperature between 230 and 240 F (110 and 115 C). The barium hydroxide impregnant was held at 194 F (90 C).

Discussion of Results

As given in Table 2, all impregnants reduced the water absorption significantly, from 47 percent for the barium hydroxide (22) to 97 percent for tar and to 99 percent for sulfur or an 80:20 sulfur-tar mixture. The most impermeable specimens were about as effective as PIC specimens (18). Compressive strengths were, as expected, significantly increased (Table 3) by the impregnation with sulfur and with a tar mixture with a high sulfur content; they were not increased with the less viscous tar by itself (7). A number of specimens of each category are being tested for salt pickup and corrosion of the embedded steel reinforcement.

In addition to having the advantages of low cost and convenience, such systems would eliminate the need for heating or catalysis to polymerize the materials, as required for a monomer. Further research is needed in the laboratory and the field to evaluate per-

Figure 14. Acid-etched (left) and non-acid-etched (right) sections from specimen partially impregnated with sulfur.
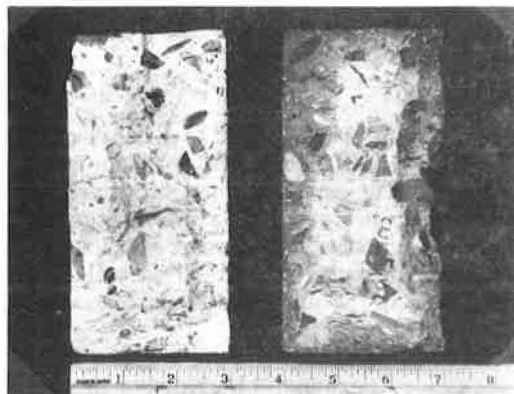

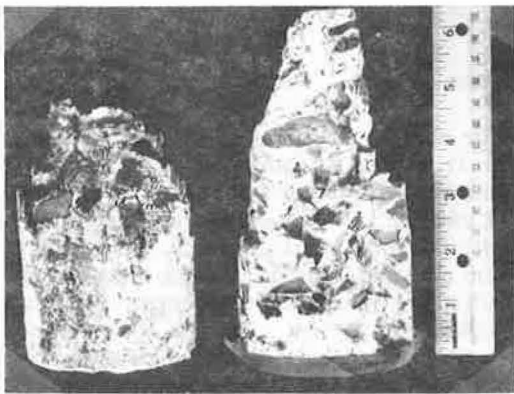Figure 15. Acid-etched (left) and sulfur-impregnated (right) specimens.

Table 2. Water absorption of impregnated mortar specimens.

Impregnant	Specimen Number	Average Loading[a] (percent)	Average Water Absorption (g)	Average Reduction in Water Absorption[b] (percent)
Barium hydroxide[c]	1 to 7	2.3	2.02	47
Molten sulfur[d]	8 to 14	12.5	0.03	99
	29 to 33	10.5	0.06	98
Tar[e]	15 to 21	6.8	0.10	97
Sulfur-tar (80:20)	22 to 28	11.2	0.05	99
Water (control)	34 to 38	7.8	3.84	—

[a]Percent loading = $\left(\dfrac{\text{impregnated weight - dry weight}}{\text{dry weight}} \times 100\right)$; immersion time = 24 hours.

[b]Percent reduction in water absorption = $\left(\dfrac{\text{water required for saturation - water absorbed}}{\text{water required for saturation}} \times 100\right)$.

[c]Barium hydroxide dissolved in its own water of crystallization; surface of concrete specimens severely attacked by solution.

[d]Specimens 8 to 21 and 34 to 38 were immersed in water for 24 hours; the remaining specimens were immersed in water for 72 hours.

[e]Tar used complied with AASHO M-214-65; specific gravity = 1.08.

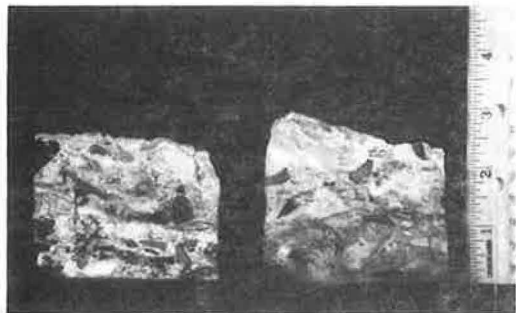Figure 16. Acid-etched (left) and non-acid-etched (right) sections of core from sulfur-impregnated slab.

Table 3. Strength of impregnated mortar specimens.

Impregnant	Specimen Number	Loading (percent)	Compressive Strength (psi)
Sulfur	29	10.2	1,500
	30	10.1	1,480
	31	11.5	1,530
Sulfur-tar (80:20)	39	11.6	1,140
	40	11.9	1,050
	41	10.7	1,180
Tar	43	7.3	640
	44	6.7	640
	45	6.9	640
Control specimens	48	—	650
	49	—	770
	50	—	700

Note: 1 psi = 6895 Pa.

formance with respect to freeze-thaw and rebar-corrosion resistance relative to MMA-based PIC.

CONCLUSIONS

Preliminary experiments with two new approaches to the impregnation of bridge decks, neoprene-pressure-mat impregnation (with MMA, tar, and linseed oil) and sulfur impregnation of moist concrete, show excellent promise for economizing, simplifying, and rationalizing the laboratory field techniques developed so far. Further work is being continued in this area because these techniques show promise of adaptation for the large-scale impregnation of bridge decks, highways, and airport runways.

ACKNOWLEDGMENTS

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