Technical Report Documentation Page

1. REPORT No.
CA-DOT-TL-5299-1-75-34

2. GOVERNMENT ACCESSION No.

3. RECIPIENT'S CATALOG No.

4. TITLE AND SUBTITLE
Field Application Of Polymer Impregnation Of Concrete

5. REPORT DATE
October, 1975

6. PERFORMING ORGANIZATION
19503-762503-635299

7. AUTHOR(S)
Spellman, D.L., Woodstrom, J.H., Bailey, S.N.; Nakao, D.I.; Spring, R.J.

8. PERFORMING ORGANIZATION REPORT No.
CA-DOT-TL-5299-1-75-34

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Transportation Laboratory
5900 Folsom Boulevard
Sacramento, California 95819

10. WORK UNIT No.

11. CONTRACT OR GRANT No.
D-3-53

12. SPONSORING AGENCY NAME AND ADDRESS
California Department of Transportation
Division of Construction and Research
Sacramento, California 95807

13. TYPE OF REPORT & PERIOD COVERED
Final

14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES
Conducted in cooperation with the U.S. Department of Transportation, Federal Highway Administration

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18. No. OF PAGES:
62

19. DRI WEBSITE LINK
http://www.dot.ca.gov/hq/research/researchreports/1974-1975/75-34.pdf

20. FILE NAME
75-34.pdf

DIVISION OF CONSTRUCTION AND RESEARCH
TRANSPORTATION LABORATORY
RESEARCH REPORT

FIELD APPLICATIONS OF POLYMER IMPREGNATION OF CONCRETE

75-34

FINAL REPORT
CA-DOT-TL-5299-1-75-34
OCTOBER 1975

Prepared in Cooperation with the U.S. Department of Transportation,
Federal Highway Administration

Caltrans
CALIFORNIA DEPARTMENT OF TRANSPORTATION

TECHNICAL REPORT STANDARD TITLE PAGE

1. REPORT NO	2. GOVERNMENT ACCESSION NO.	3. RECIPIENT'S CATALOG NO.

4. TITLE AND SUBTITLE	5. REPORT DATE
FIELD APPLICATION OF POLYMER IMPREGNATION OF CONCRETE	October, 1975
	6. PERFORMING ORGANIZATION CODE 19503-762503-635299
7. AUTHOR(S) Spellman, D. L.; Woodstrom, J. H.; Bailey, S. N.; Nakao, D. I.; Spring, R. J.	8. PERFORMING ORGANIZATION REPORT NO CA-DOT-TL-5299-1-75-34
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Portland cement concrete, polymer impregnation, compressive strength, moisture absorption, abrasion resistance, electrical resistivity, permeability, monomers.	Unlimited

19. SECURITY CLASSIF. (OF THIS REPORT)	20. SECURITY CLASSIF. (OF THIS PAGE)	21. NO. OF PAGES	22. PRICE
Unclassified	Unclassified		

IIMRT - 1242 (ORIG. 9/72)

STATE OF CALIFORNIA
DEPARTMENT OF TRANSPORTATION
DIVISION OF CONSTRUCTION AND RESEARCH
TRANSPORTATION LABORATORY

October, 1975

FHWA NO. D-3-53

TL NO. 635299

Mr. R. J. Datel
Chief Engineer

Dear Sir :

I have approved and now submit for your information this final research project report titled:

Field Application of Polymer Impregnation of Concrete

Study made by . . . . . Concrete Branch

Under the Supervision of . . . . . D. L. Spellman

Principal Investigator . . . . . D. L. Spellman

Co-Investigator . . . . . J. H. Woodstrom/
S. N. Bailey

Report Prepared by . . . . . D. I. Nakao/
R. J. Spring

Very truly yours,

George A Hill

GEORGE A. HILL
Chief, Office of Transportation Laboratory

Attachment

ACKNOWLEDGMENT

The authors wish to express their appreciation to Paul E. Mason, Bill J. Chapman, and Thomas F. Tanton for their efforts in relation to this project.

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INTRODUCTION

Since 1965, many agencies have been conducting laboratory investigations of concrete that is impregnated with a monomer and polymerized by either radiation or a thermal-catalytic means. Polymer impregnated concrete (PIC) produced in the laboratory exhibits properties far superior to that of normal portland cement concrete. The lack of a practical system for field application has not allowed full utilization of these superior properties.

This research study was aimed at developing a monomer combination and a practical technique where by concrete could be successfully impregnated in the field. Initially, several different monomer combinations were evaluated in the Laboratory in an attempt to determine which one was best suited. From the initial Laboratory study, the most promising system was selected for additional Laboratory as well as field tests. This resulted in developing a technique to impregnate concrete with the selected monomer combination under field conditions and obtain "in situ" polymerization. In addition means to measure and evaluate the effectiveness of the polymer impregnation treatment were developed.

CONCLUSIONS

The most promising monomer combination of those evaluated was one which contained 90% methyl methacrylate (MMA) and 10% trimethylolpropane trimethacrylate (TMPTMA). To this basic combination was added 2% azobisisobutyronitrile (AIBN). The following conclusions were drawn from the results of both the laboratory and field investigations using the above monomer combination.

1. Mortar strengths increased by 250%.
2. Surface abrasion resistance increased by 35%.
3. Aborption was reduced by 80%.
4. Monomer treated concrete has a low permeability, as indicated by electrical resistance measurements.
5. Technique of drying the concrete with infrared heaters, allowing the monomer to soak through a sand blanket into the concrete, and polymerizing with hot water is applicable to field conditions.
6. Treated concrete must be dry in order to attain significant monomer impregnation.
7. Polymerization of the monomer in impregnated concrete can be achieved by maintaining a minimum concrete temperature of 150°F at the depth of monomer penetration for a minimum of 2 hours.
8. Early heating of the concrete, following monomer soaking, prevents the loss of monomer from the surface of the concrete.
9. No apparent loss in skid resistance results from the polymer impregnation treatment.
10. Existing surface cracks were not filled by the monomer combination.

RECOMMENDATIONS

1. Refinements in the field technique should be made to permit treatment of large areas without requiring them to be divided into small sections.

2. Evaluate other monomer combinations.

3. Develop method for quantitatively measuring moisture and polymer content of the concrete.

4. Investigate the possibility of using a polymer-sand layer system for thin overlays.

IMPLEMENTATION

The findings of this project will be used to develop the necessary specifications to allow for a full scale deck impregnation by a contractor. This will include the type of monomer system best suited for this purpose along with the parameters for drying the concrete prior to impregnation, temperature, as well as time limits for drying.

The specifications will also serve as a guide for future research.

The FHWA has requested the State of California to provide a full-scale field evaluation utilizing the deck impregnation process they have developed.

BACKGROUND

Much laboratory research has been done by others[1-13,15]* toward developing a method of impregnating concrete with monomers which are later polymerized. However, before similar results can be achieved under field conditions, a practical field technique would have to be developed. The goal of this research project, therefore, was to develop a field technique for the impregnation and polymerization of a monomer in portland cement concrete.

Laboratory work clearly indicates that the properties of concrete can be greatly improved by impregnating concrete surfaces with a monomer, which is later polymerized [1-8]. Successful field application of this process could provide the solution to many of the present day problems related to the performance of portland cement concrete structures, such as (1) produce a more durable wearing surface that will maintain a high degree of skid resistance; (2) increase resistance to penetration by deicing chemicals and thereby significantly decrease corrosive damage to concrete and reinforcing steel; and (3) increase the compressive strengths of concrete to as much as 20,000 pounds per square inch, increasing its ability to resist damaging loads.

Much of the basic information about polymer concrete in the United States is a result of the work done by the Brookhaven National Laboratory[10] and U. S. Bureau of Reclamation[1,5]. The results of their laboratory research has stimulated much interest in development of this new idea into a practical process. From the work done at Brookhaven National Laboratory[4], the following terms related to polymer systems have been defined:

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*Numbers refer to references listed at the end of this report.

1. Polymer Impregnated Concrete (PIC)
Hardened portland cement concrete which is later impregnated with low viscosity monomer. The monomer is then polymerized in place.

2. Polymer-Cement Concrete (PCC)
A mixture of portland cement, water, aggregate, and a liquid monomer. After the mixture is placed, the monomer is polymerized.

3. Polymer Concrete (PC)
A mixture of liquid monomer and aggregate without the addition of either portland cement or water. After the mixture is placed, the monomer is polymerized.

The PIC system is considered to have the greatest application to highway works at the present time. The most promising monomers for possible field application appear to be the styrenes and methacrylates. Polymerization of these materials in concrete in earlier research was achieved with the use of radiation from Cobalt 60, which is not practical for use in field construction. Catalyst-promoter combinations can cause polymerization to take place at room temperature, but are very hard to control as far as polymerization time is concerned. It appears that the best method for field use is to polymerize the monomer using a thermal catalyst and the application of heat.

Research into field applications of polymer concrete is being conducted by various states, agencies, and foreign countries. Much work has also been done in Japan and Russia. Recently under a Federal Highway Administration Research Contract, the Bureau of Reclamation impregnated a highway bridge deck in Denver, Colorado.

The "State of the Art" for polymer impregnated concrete is still so new that optimization is needed of the application techniques that could be employed in the field. California's Transportation Laboratory, has on a limited basis, experimented with impregnating the surface of small concrete specimens[6]. The method used was to oven dry the specimens and then submerge them in a liquid monomer for a minimum of 12 hours. This limited experimentation indicated that surface impregnation by ponding was possible.

OBJECTIVES

The main objectives of this research project were as follows:

1. Select the most promising monomer combination for impregnating concrete.

2. Develop a technique for applying the monomer to a concrete surface in the field and obtain in-situ polymerization.

3. Measure and evaluate the results of the field experiment.

While some techniques for measuring properties of concrete are "standard", others needed for this project had to be developed.

DISCUSSION OF TESTING PROGRAM

Selection of Monomer System

There are many monomers on the market that could have been evaluated during this research study with characteristics suitable for Polymer Impregnated Concrete (PIC). Of these, methyl methacrylate has been the one most commonly used by other researchers. Most experimentation reported was done using some system of methyl methacrylate (MMA), trimethylolpropane trimethacrylate (TMPTMA), which is a crosslinking agent, and benzoyl peroxide (BPx), which is a catalyst. Crosslinking agents are used to aid polymerization and prevent dissolvement of the polymerized plastic by solvents. Catalysts are used to overcome inhibitors added to the monomer for stability during storage.

The usefulness of a free radical catalyst is determined by its half-life, or rate of forming free radicals, and efficiency or ability of the free radicals to initiate polymerization. The peroxides generally have low efficiencies because of the free radicals reacting with other molecules of the peroxide. The Azo catalysts, such as AIBN, generally have higher efficiencies because they are not susceptible to this type of inner reaction. Table 1 shows the half-lives of three catalysts.

Table 1

Half-life at 50-100°C[15]

Name	Half-life in Hours, at 50-100°C				
	50°	60°	75°	85°	100°
Benzoyl Peroxide	190	44	6.7	2.1	0.4
Azobisisobutyronitrile	83	13.2	2.4	0.53	0.12
Lauroyl Peroxide	54	12	1.7	0.45	0.1

Note: High values of half-life result in lower efficiency.

Laboratory Testing

To select the most promising monomer system, several rounds of tests were performed within the laboratory utilizing concrete and mortar specimens. (See Tables 2 and 3 for mortar and concrete mix designs.) After fabrication, all specimens were cured at 100% relative humidity for 14 days, dried to a constant weight at 230°F, and sealed in plastic bags until used. Some additional tests were conducted outside the laboratory on slabs no longer being used for another project.

Tests performed on the mortar samples included:

1. Compressive strength

2. Surface abrasion resistance. Tests were based on Test Method No. Calif. 360, modified to use steel balls[14].

Table 2

Mortar Mix Design

Aggregate	Percent	Weight, Lbs.
No. 4x8	26	18.4
8x16	15	10.6
16x30	16	11.3
30x50	26	18.4
50x100	17	12.1
Net Water		12.5
Cement, Type II		20.8
Water-Cement Ratio	0.60	

Table 3

Concrete Mix Design

Aggregate*	Percent	Weight, Lbs.
1 x 3/4 in	23	24.3
3/4 x 3/8	52	55.0
3/8 x 4	25	26.4
Total rock	100%	105.7
Concrete Sand		91.8
Net water		22.0
Cement, Type II		43.9
Water-Cement Ratio	.50	

*Mortar and concrete made with local aggregates from the American River.

3. Permeability Tests

In this test, mortar or concrete disks are placed in salt water just deep enough to keep the bottom saturated. Fresh water is ponded on the top surface of the disks within a waterproof dyke. If the mortar is permeable, water, in time, will penetrate the disk to provide a continuous electrical path which can be measured with an ohmmeter. Strictly speaking, this is an indirect measurement of permeability as only the effect of water penetration is measured (see Figure 1).

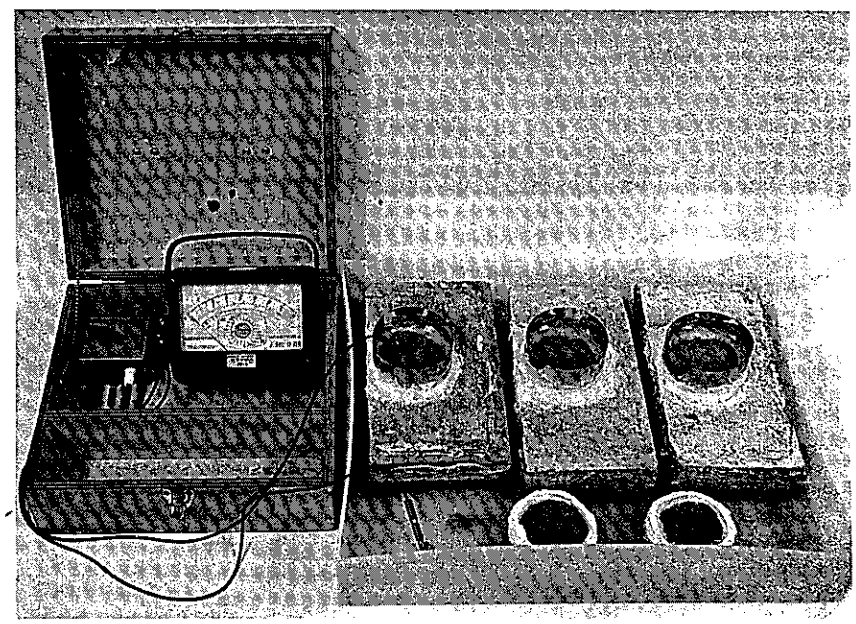

Figure 1. Test setup for measuring "permeability" indirectly by electrical resistivity. Test specimens and the ohmmeter.

Dry mortar or concrete specimens normally have high electrical resistance. The penetration of salt water and accompanying resistance drop becomes apparent within 24 hours for untreated concrete. Impermeable mortar or concrete retain a high resistance. Thus, change in resistivity provides a quantitative measurement for the effectiveness of the impregnation treatment.

A series of test programs was conducted for the purpose of evaluating various properties and procedures for development of impregnating techniques.

Rounds A, B, and C

Rounds A, B, and C were performed to evaluate the impregnation and polymerization characteristics of the MMA and TMPTMA combination with three free radical catalysts: Benzoyl peroxide (BPx); Azobisisobutyronitrile (AIBN*); and lauroyl peroxide (LPx).

The three monomer combinations selected were:

A. 95% MMA and 5% TMPTMA, with 1% BPx added
B. 95% MMA and 5% TMPTMA, with 1% AIBN added
C. 90%MMA and 10% TMPTMA, with 1% LPx added

Impregnation was accomplished by submerging specimens in the three different monomer combinations. To determine the depth of impregnation as related to the soak time, half of the specimens were submerged for 2 hours, and the remaining half for 24 hours. Once impregnated, the specimens were wrapped with plastic to reduce monomer evaporation, then placed in a 125°F oven for polymerization. Since at 120°F the time required for polymerization to take place was unknown, heating durations of 2 and 8 hours were incorporated for each soak time.

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*In this report, AIBN refers to Vazo 64 (trade name), which initiates polymerization at 64°C.

Monomer loading for all specimens was designed to average 6 to 7% by weight. However, only the AIBN catalyzed specimens in conjunction with the 8-hour heating period achieved significant polymerization (see Table 4).

Rounds D, E, and F

Rounds D, E, and F were performed to further evaluate monomer combinations. The combinations were the basic 90% MMA and 10% TMPTMA used in Rounds A, B, and C except a higher dosage of catalyst was used. This resulted in the following:

D. 90% MMA and 10% TMPTMA, with 2% BPx added
E. 90% MMA and 10% TMPTMA, with 2% AIBN added
F. 90% MMA and 10% TMPTMA, with 2% LPx added

A 2x4-inch disk, a 2x2-inch cube, and a 12x12x3-inch concrete slab were impregnated with each monomer combination. The mortar cubes and disks were submersed in monomer for 2 hours, then wrapped and placed in a 140°F oven for 16 hours. The slabs were impregnated by ponding the monomer on the surface for 2 hours. A 3-inch high wooden form was used to contain the monomer and a 1/4-inch layer of sand was applied to the concrete surface to aid in impregnation. Enough monomer was added to keep the sand saturated. A polyethylene sheet was placed directly on the sand to reduce monomer evaporation. To produce polymerization, 3 inches of water was ponded on the polyethylene sheet and the water temperature gradually increased to 150°F. This water temperature was maintained for one hour, then allowed to cool overnight.

Table 4

Test Results of Rounds A, B, and C

	Soak Time, Hours	Heat Time, Hours	Percent Loadings by weight	Compr. Str., psi	Surface Abrasion Resistance, Grams	Electrical Resistance Ohms	
						Initial	8 days
Mortar Cubes, 2x2x2"							
Control	Dry	----	-------	5460	--------	-------	-------
95% MMA-5% TMPTMA+1% BPx	2	2	1.0	4880	--------	-------	-------
	2	8	1.0	5110	--------	-------	-------
	24	2	1.0	4900	--------	-------	-------
	24	8	1.0	4980	--------	-------	-------
95% MMA-5% TMPTMA+1% AIBN	2	2	1.5	4980	--------	-------	-------
	2	8	5.8	11470	-------	-------	-------
	24	2	1.7	5050	--------	-------	-------
	24	8	6.4	13150	--------	-------	-------
90% MMA-10% TMPTMA+1% LPx	2	2	1.6	4390	--------	-------	-------
	2	8	1.6	4840	--------	-------	-------
	24	2	1.7	5000	--------	-------	-------
	24	8	1.8	4740	--------	-------	-------
Mortar Disks, 2" high x 4" diameter							
Control	Dry	---	------	-----	13.5	3 megohms	9 K ohms
95% MMA-5% TMPTMA+1% BPx	2	2	2.0	-----	15.5	17 megohms	12 K ohms
	24	8	2.0	-----	15.5	100 megohms	13 K ohms
95% MMA-5% TMPTMA+1% AIBN	2	2	1.3	-----	15.0	25 megohms	2 K ohms
	24	8	7.4	-----	9.0	200 megohms	67 megohms
95% MMA-10% TMPTMA+1% LPx	2	2	2.8	-----	----	-------	-------
	24	8	2.9	-----	----	-------	-------

Note: All results are average of 3 tests with the exception of the polymer loadings of the disks for the 90% MMA and 10% TMPTMA with 1% LPx combination which contain the results for one sample only.

Conclusions From Rounds A, B, C, and D, E, F

From the test results obtained by using the three different catalysts with the 90% MMA and 10% TMPTMA monomer combination, AIBN resulted in the best polymer loading and strength characteristics. (See Table 5.) As a result, all following rounds were made with the monomer combination containing 90% MMA and 10% TMPTMA with 2% AIBN added.

Round G

Round G consisted of evaluating the polymerization of the monomer by ponding hot water directly on the concrete surface. Test specimens consisted of six 12x12x3-inch concrete slabs, of which two served as control slabs, while the remaining four were impregnated. Impregnation was accomplished by the same method as for slabs in Rounds D, E, and F. Two of the slabs were then polymerized by applying hot water directly to the slab, while the other two were polymerized by applying hot water to the polyethylene sheeting as in Rounds D, E, and F (see Figures 2 and 3).

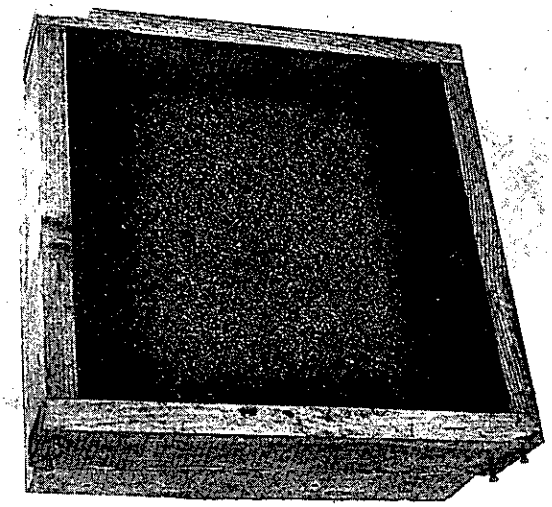


Figure 2
12x12x3 - inch concrete slab ready for impregnation.

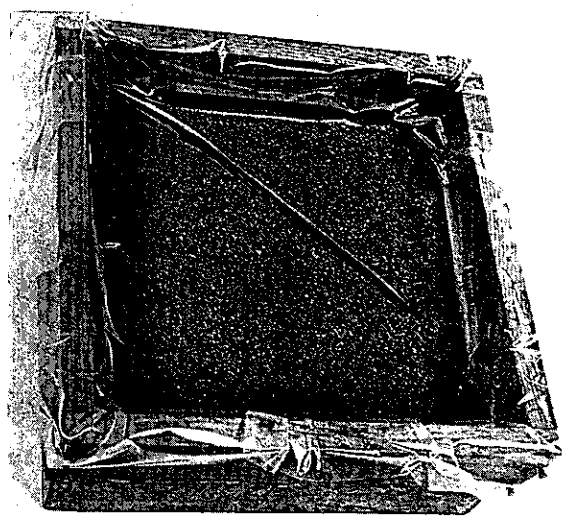


Figure 3
Polymerization by ponding hot water over polyethylene sheeting.

Table 5

Polymerization and Test Results

	Round D (90% MMA-10% TMPTMA + 2% BPx)	Round E (90% MMA-10% TMPTMA + 2% AIBN)	Round F (90% MMA-10% TMPTMA + 2% LPx)
Cubes & Disks			
% Wt. gain, cubes	1.0%	6.2%	1.0%
% Wt. gain, disks	2.0%	7.0%	2.0%
Cube compressive Strength, psi*	4850	9120	4970
Monomer odor when unwrapped	Strong	Slight	Strong
Slabs			
Penetration	1/2" - 3/4"	1/2"	1/2" - 3/4"
Odor when broken open	Strong monomer odor	Strong monomer odor	Strong monomer odor
Polymerization	Very little	1/2" dark (splotchy)	All evaporated

*Control cube broke at 5350 psi.

Conclusions: Best results with 2% AIBN catalyst.

Thermocouples were inserted at depths of 1/2-inch, 1-inch, and 2-inches below the surface of the impregnated slabs to determine the temperature gradients while polymerizing. (See Figures 4 and 5.)

Conclusions from Round G

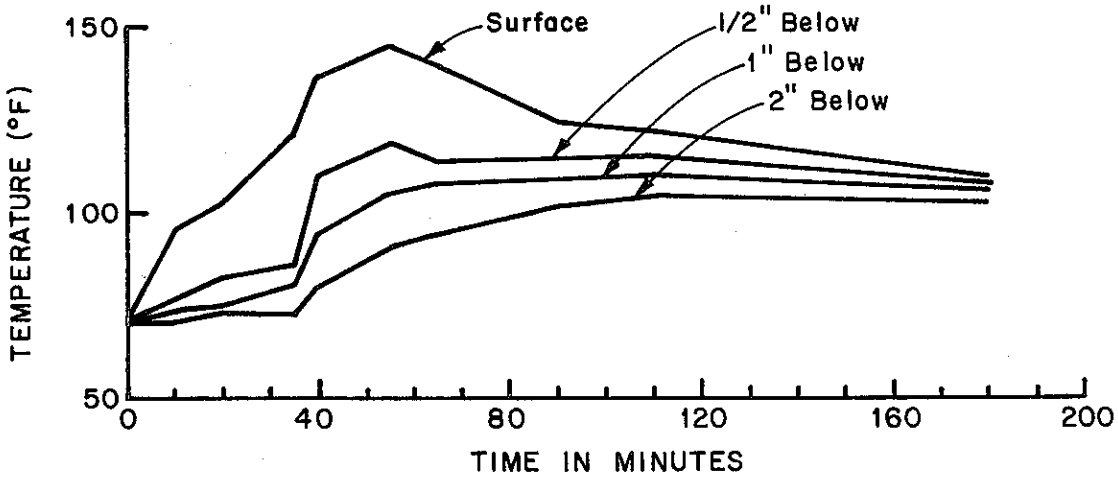
1. During polymerization, concrete temperatures in the slabs polymerized with water directly in contact with the concrete averaged 10°F higher as compared to those where the water was separated from the concrete surface with a polyethylene sheet.

2. Inspection of the slabs showed the presence of monomer to a depth of 1 inch, but only the top 1/4-inch was polymerized. This indicates that in order to achieve total polymerization, an increase in water temperature or polymerization time, or both, might be necessary.

Round H

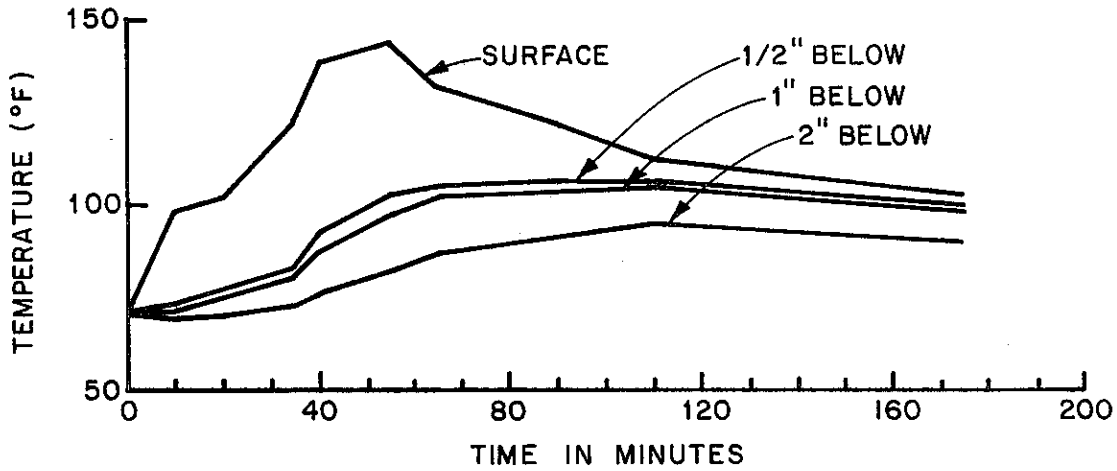
Four 12x12x3-inch slabs were impregnated with the 90% MMA - 10% TMPTMA + 2% AIBN monomer combination. The slabs were impregnated by the method previously described. The slabs were polymerized by ponding 3 inches of 135°F water for 2 hours, two slabs by applying the water directly to the surface, and two by applying the water over the polyethylene sheet. The plan was to polymerize with 160°F (tap water), but the water heater did not have the capacity to keep the water at that temperature.

A strong monomer odor was present when the slabs were broken, indicating the presence of unpolymerized monomer. The depth of polymer in all the slabs was about 1/4 to 1/2-inch and appeared spotty. Electrical resistivity measurements indicated very little polymerization.



TEMPERATURE GRADIENT OF SLABES POLYMERIZED BY WATER APPLIED DIRECTLY TO THE SURFACE OF THE SLAB

Figure 4



TEMPERATURE GRADIENT OF SLABS POLYMERIZED BY WATER APPLIED ON TOP OF A POLYETHYLENE SHEET

Figure 5

Conclusion from Round H

1. Higher temperatures and/or longer curing are necessary for successful polymerization of the 90% MMA - 10% TMPTMA type monomer used.

Round I

Round I was conducted to evaluate the electrical resistance of polymer impregnated slabs polymerized by oven heating and water ponding. Three 12x12x3-inch test slabs were impregnated for 2 hours using the monomer combination of 90% MMA and 10% TMPTMA with 2% AIBN added. The method of impregnation was the same as that used for the previous slabs. Slab I-1 was polymerized by placing in a 180°F oven for 2 hours. Slabs I-2 and I-3 were polymerized by ponding 3 inches of hot water (175°-200°F) for 2 hours. Slab I-2 received water by ponding on a polyethylene sheet while the water for Slab I-3 was ponded directly on the concrete. In addition to the polymerized slabs, a corresponding control slab was also evaluated. The 1/4-inch layer of sand was left on the slab surface during polymerization. As a result some of the sand adhered very tightly to those slabs that were polymerized by ponding water on top of the polyethylene sheeting, but did not adhere to the slabs that were polymerized by ponding water directly on the surface. With this in mind, a special effort was made in all future tests to remove as much as possible of the sand layer, along with the excess monomer by adding additional cool water prior to polymerization. Following polymerization, each slab was fractured into as many as four parts and electrical resistance measurements made.

Conclusions from Round I

Inspection of the slabs revealed a well polymerized polymer penetration of 3/4-1-inch for Slab I-1, and 1/2 - 3/4-inch for Slabs I-2 and I-3 (see Figure 6). As can be seen from the results in

Table 6

Electrical Resistance Measurements on Portions of 12x12x3-inch Slabs

(Round 1)

Slab No.	Specimen	Initial	24 Hours	7 Days	1 Month	4 Months
I-8	A	11 megohms	340 K ohms	40 K ohms	70 K ohms	85 K ohms
	B	19 megohms	9 megohms	85 K ohms	45 K ohms	60 K ohms
I-2	A	1 megohm	1.6 megohms	800 K ohms	110 K ohms	70 K ohms
	B	2.6 megohms	73 K ohms	55 K ohms	90 K ohms	70 K ohms
I-3	A	1.7 megohms	1.8 megohms	1.3 megohms	520 K ohms	120 K ohms
	B	1.3 megohms	2.2 megohms	1.2 megohms	390 K ohms	95 K ohms
Control	A	100 K ohms	2 K ohms	3 K ohms	10 K ohms	4 K ohms
	B	300 K ohms	2 K ohms	3 K ohms	10 K ohms	4 K ohms

Table 6, the electrical resistance of the concrete was increased considerably.

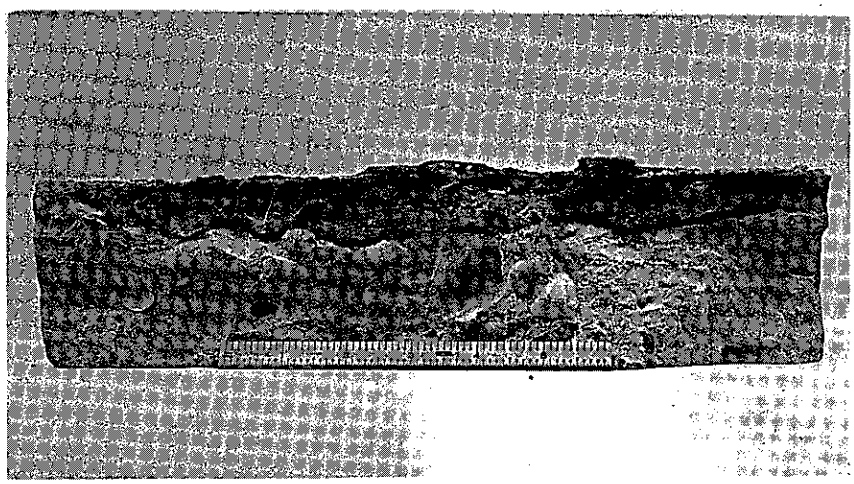


Figure 6. Round I slab, polymerized in oven and split open for visual inspection (12x12x3-inch slab)

Round J

Round J was performed in order to determine what effect a concrete curing compound might have on the depth of monomer penetration. For this experiment, six 12x12x3-inch concrete slabs were used. Two of the slabs served as control specimens, two were surface sprayed with a resin base compound at a spread rate of 250 sq.ft./gallon and two

with a chlorinated rubber compound at a spread rate of 300 sq.ft./gallon. The compounds were allowed to dry for 24 hours before monomer impregnation. All slabs, including the controls, were impregnated with the monomer combination consisting of 90% MMA and 10% TMPTMA with 2% AIBN added. Impregnation was accomplished by the same method used in Rounds D, E, and F with the exception that two soaking times were used, 2 and 18 hours.

After impregnation, the sand was washed loose from the surface and removed. The slabs were then ponded immediately with hot water in order to achieve polymerization. The temperature of the hot water was maintained between 175° and 200°F for a period of 2 hours by the use of steam generated by a small steam cleaning unit.

Conclusions from Round J

Visual inspection of the slabs (see Figures 7 and 8), indicated the resin and chlorinated rubber curing compounds had little effect on the depth of impregnation.

Electrical resistance measurements (Table 7) for some unknown reason were not consistent and may have been affected by the presence of the curing compounds even though they apparently did not affect penetration of the monomer.

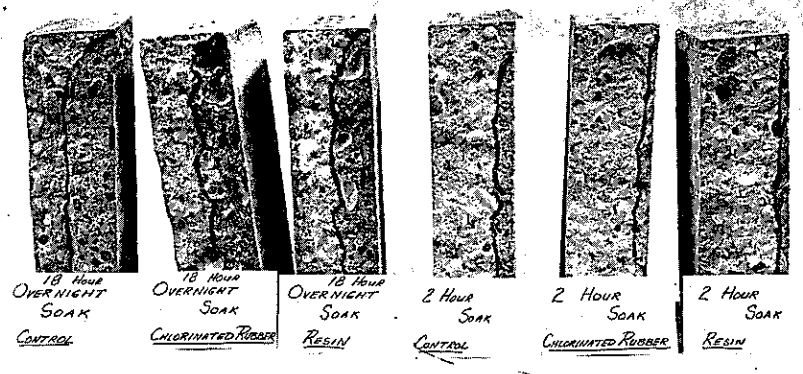


Figure 7. Round J Slabs split open for visual inspection. (12x12x3-inch slabs.)

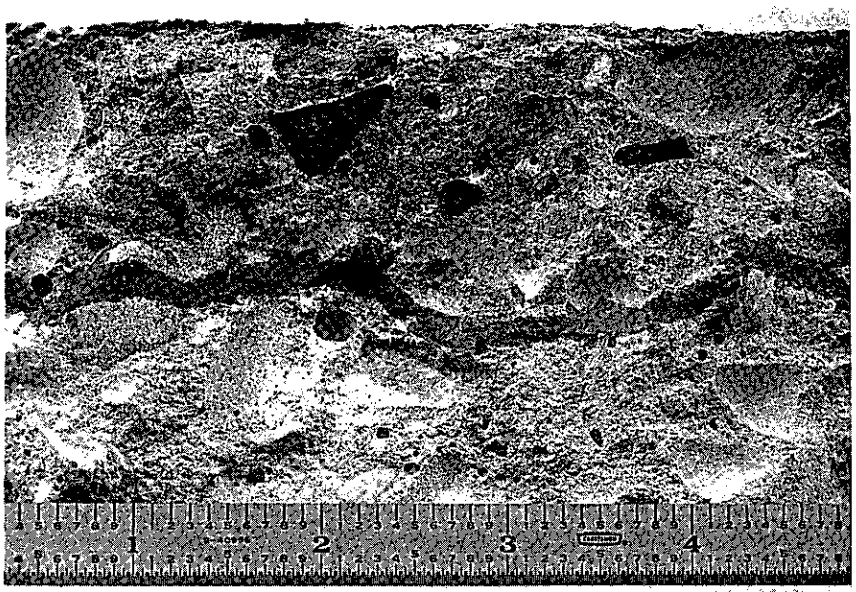


Figure 8. Close-up shot of chlorinated rubber slab soaked with monomer for 18 hours (Round J). Notice aggregate failure in impregnated protion of slab.

Table 7

Depth of Impregnation and Electrical Resistance Measurements

	Depth of Impregnation, Inches	Electrical Resistance, Megohms		
		Initial	23 days	10 Weeks
Control				
2-hour soak	3/4	-------	-------	--------
18 hour soak	1-1/2	4	2.3	1.4
Resin Base				
2-hour	1/2	-------	-------	--------
18 hour soak	1-1/4 to 1-1/2	30	20	10.5
Chlorinated Rubber				
2 hour soak	1/4 to 1/2*	-------	-------	--------
18-hour soak	1-3/8 to 1-5/8	32	20	10.5

*Low polymer density.

Round K

Round K was conducted in order to determine what effect a delay in applying heat would have on polymerizing the monomer.

Six 12x12x3-inch concrete slabs were treated with the monomer combination containing 90% MMA and 10% TMPTMA, +2% AIBN added. The slabs were allowed to soak for approximately 18 hours before the normal sand bainket was removed by washing with water. All slabs were then covered with approximately 2 inches of water to prevent evaporation of the monomer from the surface.

For all slabs, heat was applied in the form of steam to bring the water up to 200°F and maintain that temperature for a period of 2 hours. However, the time at which this heating process began varied for the different slabs.

Heat was applied to Slab No. 1 within 30 minutes following the removal of the sand blanket. Slab No. 2 was heated at 2 hours, No. 3 at 3 hours, No. 4 at 4 hours, No. 5 at 5 hours, and No. 6 at 24 hours.

Following the polymerization of the six slabs, each slab was allowed to cool and then broken open to determine what effect the time delay had on the penetration and polymerization. The results showed that each slab had a thin layer along the top surface which appeared to be void of monomer. This layer was smallest on Slab No. 1 (approximately 1/16-inch) and greatest on Slab No. 6, (approximately 1/4-inch).

It appears that one of two factors may have been responsible for this condition:

1. Monomer may have been lost from the surface upward through the water cover during the washing action.

2. Monomer may have continued to migrate downward during the polymerization period and was replaced by water.

Electrical resistivity test results are shown in Table 8.

Table 8

Electrical Resistance Measurements of Round K

Slab No.	Time Delay in Applying Heat	Electrical Resistance, Megohms		
		Initial*	7 days	5 weeks
1	30 min.	20.0	12.0	7.0
2	2 hrs.	16.5	8.5	4.5
3	3 hrs.	6.5	2.5	1.5
4	4 hrs.	26.0	15.0	8.0
5	5 hrs.	17.5	10.0	5.0
6	24 hrs.	2.5	2.0	1.5

*Initial readings for all slabs were taken following the heating of Slab No. 6

Conclusions from Round K

In order to achieve a good polymerization along the top surface, it appears that the heating process should be conducted as soon as possible following the soak time.

LABORATORY TESTING - OUTDOOR

It was next decided to impregnate concrete in an outdoor environment. This would provide the experience and the opportunity to check out the process that would be ultimately used on a bridge deck. An existing concrete slab located on the laboratory grounds was selected as a test site for this work. The slabs had previously been used as a bed for prestressing operations and was constructed of high quality structural concrete.

Slab Section No. 1

A 5-foot square section was selected as a test area to receive the monomer impregnation. Within the 5-foot square section, three sets of thermocouples were imbedded at depths of 1/2-inch and 1-inch below the surface to monitor temperature during the drying and impregnation cycles. These thermocouples were also used experimentally as electrodes to measure the change in electrical resistance of the concrete during the drying operation.

The slab surface was ponded with water for 4 days in order to saturate the concrete and simulate the worst possible condition which might exist in the field. Following this saturation period, the water was removed and three small portable propane infrared heaters were used to dry the concrete. The majority of the 5x5-ft. test area was subjected to drying for a period of 50 minutes. During this time, temperatures rose to 200°F at a depth of 1-inch below the surface, and 250°F at a depth of 1/2-inch below the surface. A small additional portion of the slab was dried for only 20 minutes to determine the variation in monomer impregnation with relation to drying time. Once the drying cycle had been completed, the slab was left to cool over night.

The following morning the slab was impregnated with the monomer combination containing 90% MMA and 10% TMPTMA with 2% AIBN added. Impregnation procedure was the same procedure discussed for Rounds D, E, and F. Soak time was 2 hours. Polyethylene was used to cover the sand to reduce evaporation of the monomer during the soak period. Polymerization was accomplished by first ponding 1 inch of cool water on the slab, and then increasing the water temperature by inserting the nozzle of a steam generator into the ponded water. After 40 minutes, the water temperature reached 200°F and was held at that temperature during the rest of the 2-hour polymerization period. The maximum concrete temperatures achieved during

polymerization were 177°F at a depth of 1/2-inch, and 165°F at a depth of 1-inch below the surface. Upon completion of the polymerization cycle, the water was left ponded on the slab to cool over night.

On the day following polymerization cores were taken from the treated areas. Evaluation of the cores from the 20-minute drying area revealed very little polymer penetration, however, the cores from the 50-minute drying area revealed 1/2-inch to 3/4-inch polymer penetration. Electrical resistance measurements of the cores, after drying at 230°F for 72 hours, then wetted in accordance with the resistivity test procedure indicated water had penetrated through the treated concrete (see Figure 9).

Nuclear gage readings were taken on the slab before and after polymerization in an attempt to evaluate this type of test for field measurements of impregnation (see Table 10). Since there was no established relationship between the number of counts on the gage and density of the concrete, the counts were obtained for comparison only.

Resistivity readings between the thermocouples showed a trend of increased resistance with drying; however, these data were not consistent and thus could not be viewed as meaningful information.

Table 9

Electrical Resistance Measurements

	Initial	48 Hours
50-minute drying core	400 K ohms	16 K ohms
20-minute drying core	250 K ohms	2 K ohms

Table 10

Nuclear Gage Readings

	5x5-ft. Test Section (Water Saturated, 4 days) Dried Area	
	(50 Minutes)	(20 Minutes)
After Drying	639 counts	742 counts
After polymerization	722 counts	764 counts

The count on an adjacent part of the concrete slab not receiving any wetting, drying, or impregnation varied from 652 to 662 counts.

Slab Section No. 2

A 5-foot square section adjacent to test area No. 1, was dried with infrared heaters and impregnated with the same monomer combination as Test Area No. 1. The slab had been subjected to drying by the sun for several months and was assumed to be fairly dry. The average drying time with the infrared heaters was 30 minutes. During this time, the surface temperature reached 320°F, while the temperature 1 inch below the surface was recorded at 260°F. Ninety minutes after the heaters were removed, the temperature at 1-inch depth

dropped to 170°F (ambient temperature was 95°F). In addition to this 5x5-ft. section, two adjacent 12x24-inch sections were also impregnated. One section was not dried and the other was dried for 70 minutes (surface temperature up to 330°F). This provided three test areas; one not dried (artifically), one dried 30 minutes, and the third dried 70 minutes.

All three of these sections were impregnated for 2 hours by the same method used for Test Area No. 1. After impregnation, water from a hose was used to wash the excess monomer from the sand in an attempt to keep it from sticking to the concrete surface. The polyethylene sheet was then placed directly on the sand and hot water ponded on the sheet. Heat was applied for 2 hours by the steam generator and once completed, the water was left ponded over night. Water and slab temperatures recorded are shown in Table 11.

Table 11

Water and Slab Temperatures During Polymerization

	In Area dried 30 min.				
				Water Temperature on 12x24-inch sections	
				Temperature, °F	
Time, Minutes	Water Temp., °F	1/2-inch below Surface, Temp., °F	1-inch below Surface, Temp., °F	Dried 70 min.	Undried
0	70	93	93	70	70
15	160	102	102	180	180
30	190	120	113	195	195
60	200	150	135	185	185
80	212	158	149	180	180
120	210	167	156	180	180

Examination of cores indicated no visible polymer in the undried area and about 3/8-inch polymer penetration in the two dried sections. Electrical resistance measurements on the cores are shown in Table 12 and nuclear gage readings in Table 13. Measurements indicate little sealing effect was achieved by this procedure.

Table 12

Electrical Resistance Measurements

	Initial	48 Hours
Cores from:		
5x5-ft, Area (1)*, Dried 30 minutes	2 megohms	8 K ohms
5x5-ft, Area (2)**, Dried 30 minutes	500 K ohms	4 K ohms
12x24-in. Area, not dried	750 K ohms	2 K ohms
12x24-in. Area, Dried for 70 minutes	2.1 megohms	3 K ohms

*Area 1 cores represent the north half of slab
**Area 2 cores represent the south half of slab

Results are average of two cores taken from each area.

Table 13

Nuclear Gage Counts

	5x5-ft. Area Dried 30 Min.	12x24-inch Areas Undried	12x24-inch Areas Dried 70 Min.
Initial	675	698	688
After drying	657	No drying	625
After polymerization	710	693	671

Conclusions from the experiment are:

1. Sand layer was polymerized to the concrete in some areas.

2. The measured electrical resistance is an erratic indication of the amount of polymer impregnated into the concrete.

3. Nuclear gage appears to have some potential in determining moisture and polymer contents.

4. A longer drying period may result in a higher polymer loading concrete.

FIELD APPLICATION OF POLYMER IMPREGNATED CONCRETE

Preparation

In mid-October, 1974, a 90-ft by 8-ft test section was established on a portion of the Grand Avenue Overhead Structure located on future Route 80 near Sacramento (see Figure 9). The overhead is a highway bridge structure that is several years old but has not as yet been open to traffic. The section selected for a treatment had a longitudinal grade of approximately 1% and a transverse grade of 1-1/2% (see Figure 10). The test section

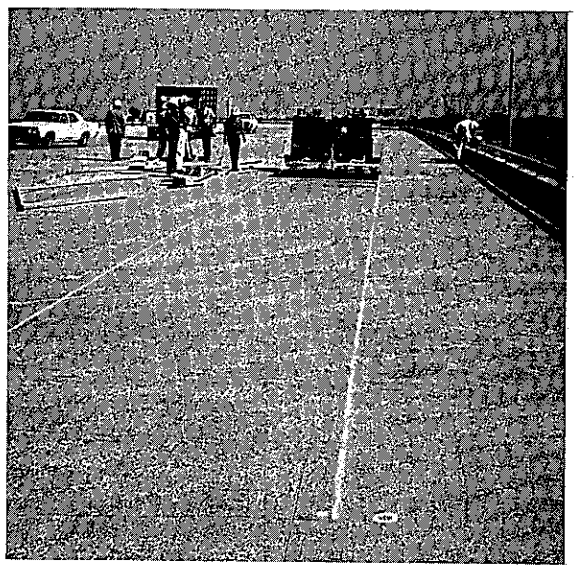
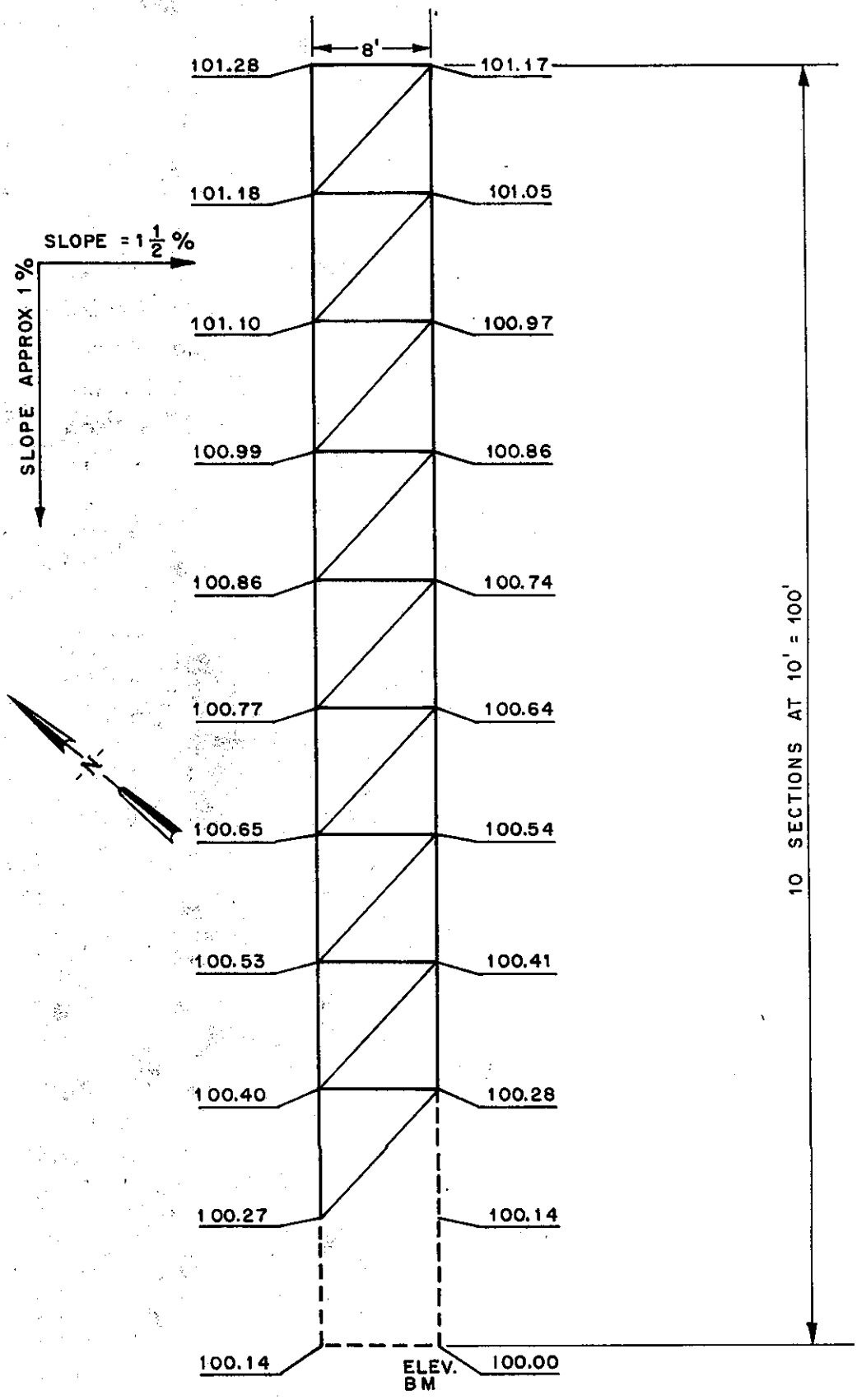


Figure 9. Test Section on Grand Avenue Overhead near Sacramento



PLAN VIEW OF TEST SECTION

Figure 10

was divided into nine 8-ft wide by 10-ft long areas. Thermocouples were placed at 1-inch depths in each area to monitor temperatures during the drying, cooling, and polymerization cycles.

Drying of Test Section

Beginning at 0900, two 4-ft by 5-ft infrared heaters mounted side by side were used to dry the concrete (see Figures 11 and 12).

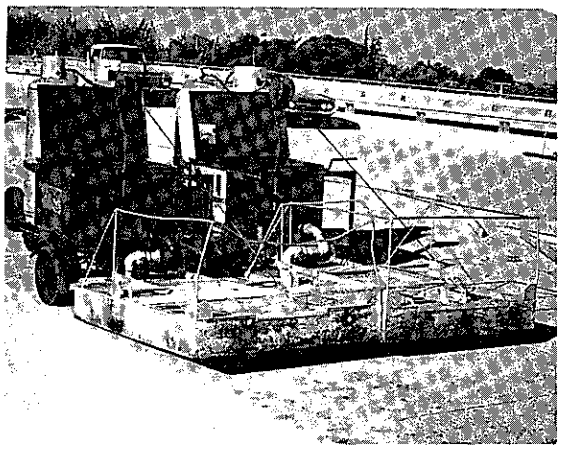


Figure 11. Infrared heaters

Figure 12. Infrared Heaters

The criteria selected for adequate drying was to raise the concrete temperature 1-inch depth below the surface to 200°-250°F and maintain that temperature for a period of 2 hours without increasing the

surface temperature above 350°F. In order to maintain the surface temperature below 350°F, the heaters had to be moved every few minutes. The drying procedure used followed one of applying heat to half of the first section for a few minutes, then moving to the second half, followed by a return to the first half, and so on until the temperature of the entire first section, 1 inch below the surface, was above 200°F. Once that temperature was achieved, it was fairly easy to maintain while another section was added to the process. A total area 8-ft by 60-ft was dried by the above process. In addition, one 8-ft by 10-ft section was heated to a temperature of 200°F at 1-inch below the surface, then immediately allowed to cool with no more heat applied. Two 8-ft by 10-ft sections to be used as controls were not dried at all. The heaters were removed from the last fully dried section at 1700 hours.

Application of Monomer

To confine the monomer and retain the hot water during polymerization, wooden forms were placed on the 8-ft by 10-ft areas using 2x4's on edge sealed to the deck surface using silicone sealant. (See Figure 13.) This work, as well as the spreading of the 1/4 inch sand layer, was done during the required cooling period. The

Figure 13. Sealing 2x4 form to concrete deck.

application of monomer could not procede until the temperature 1-inch below the surface cooled to 100°F, which was considered the maximum safe temperature at which the monomer could be applied. At 0100 hours on the second day, monomer was applied to the concrete. Theoretically at least, applying the monomer during a period of falling temperature should also promote impregnation. Outgassing which occurs during a rise in temperature creates an undesirable condition.

The application procedure consisted of pumping the monomer from a 55-gallon drum into smaller sprinkling containers. These smaller containers were then used to apply and spread the monomer by hand over the entire surface of the sand blanket (see Figure 14). Once the sand became saturated with monomer, the entire test section was covered with a clear polyethylene sheet to reduce the loss of monomer from the surface through evaporation (see Figure 15).

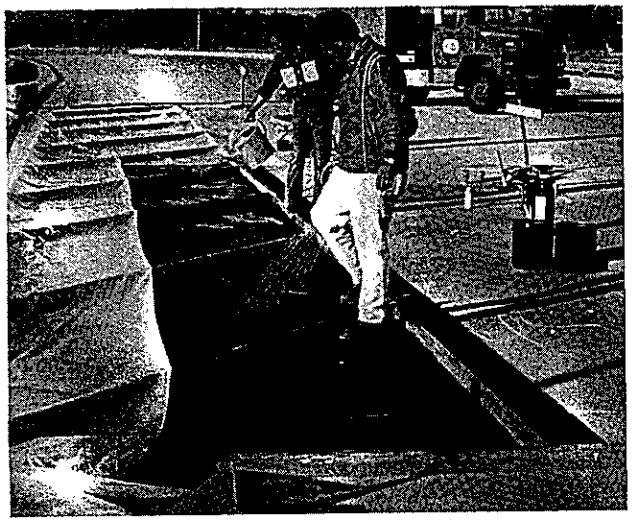


Figure 14. Applying monomer to deck.

Figure 15. Polyethylene covering over monomer.

After 2 hours of soaking, a check was made to determine if the sand blanket was still saturated. Upon visual observation, it was decided that additional monomer should be applied to the surface. The polyethylene sheet was removed and additional monomer was applied using the same procedure as before. Once again the polyethylene sheet was used to cover the test section following the monomer application. A third and final application of monomer was applied to the surface 4 hours after the initial application (about 0500). This application was not as heavy as the first two.

The procedure used to apply the monomer presented no problems other than the hand labor which was time consuming. There was considerable odor from the monomer while the covering was removed; however, once the covering was replaced following the monomer application, the odor quickly dissipated. There is a potential fire hazard and use of matches or other flammable material around the operation must be rigidly controlled.

The total amount of monomer applied to the test section was approximately 95 gallons (1.1 lbs/sq.ft.).

It was originally planned to allow the monomer to soak for a minimum of 12 hours. Shortly after sun-up, some of the areas which had an excess amount of monomer began to show signs of jelling. In an attempt to prevent further monomer jelling, the area was shaded with black polyethylene sheeting.

At 1200 hours, the polyethylene sheeting was removed and cool water poured into the forms. This water was used to aid in the removal of the sand as well as to prevent loss of the impregnated monomer in the concrete by evaporation. The sand was removed by the use of broom and shovels prior to the polymerization.

Polymerization

Heat application began at 1230, with three portable steam generators being used to heat the water. Criteria for polymerization was 150°F at 1-inch concrete depth for 2 hours. Initially each steam cleaner was intended to supply heat to three of the 8x10-foot sections, but the capacity of the steam cleaners was not sufficient to do so. As the process continued it became clear that the best method would be to heat a section to the required 150°F using two steam cleaners, then move both cleaners to heat an adjacent section and return to reheat the first section with a single cleaner. Using this procedure, it was 1430 hours before the temperature of the first section had increased to 150°F at 1-inch and 2200 hours before the last section was polymerized (see Figure 16).

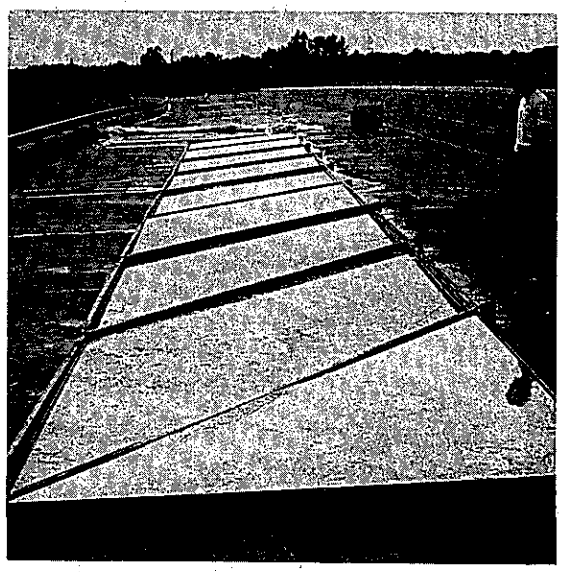


Figure 16. Polymerized section following heating before water was removed.

Evaluation of Field Application

Nuclear gage readings were taken initially after the drying process but before impregnation, and six days following impregnation and polymerization. The readings indicate (1) initially, the concrete was fairly dry; (2) the sun dries out the concrete during the day; and (3) after polymerizing with water, it is impossible to determine the counts due to polymer without once again drying out the concrete.

Four-inch diameter cores were taken from the test sections. Visual inspection of the cores, after being cut and polished, shows about a 3/4-inch polymer penetration. The polymer seems to be more dense in cores taken from areas that were dried by the heaters. The top 1/16 to 1/4-inch of all the cores were low in polymer content. It is possible that during the long period of time between removing the sand and polymerizing the monomer, the monomer continued to diffuse downward, drawing some air and/or water behind it (see Figures 17 and 18).

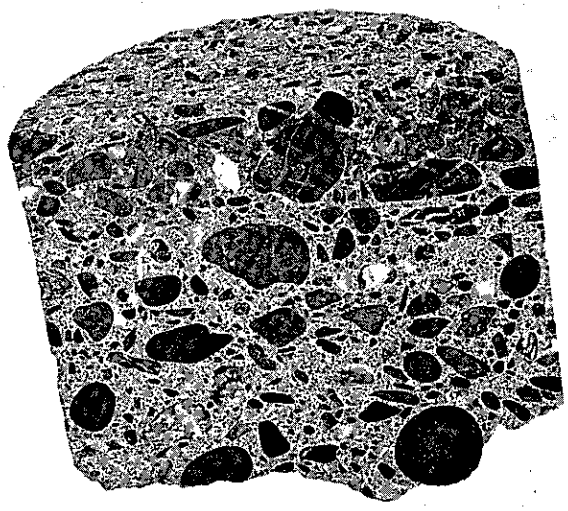


Figure 17. 4" diameter core from the Grand Avenue Overhead. This core is from an area that was not dried and was impregnated with monomer.

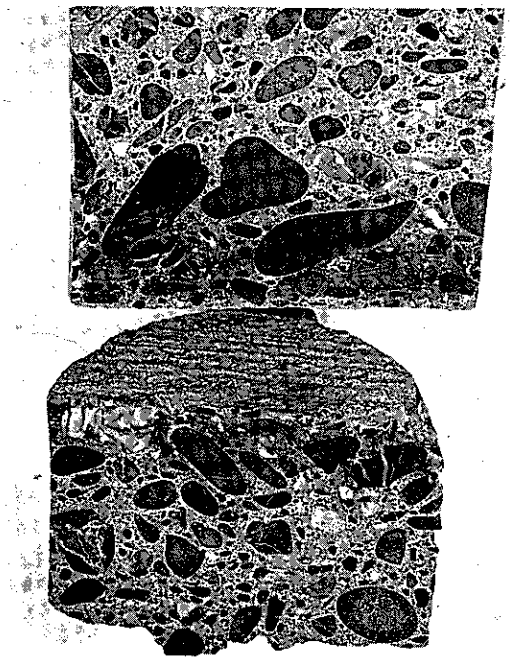


Figure 18. 4" Diameter cores from Grand Ave. OH. The area was dried for 2 hrs. before impregnation.

One significant observation made during the inspection of the cores was that the polymerized monomer did not appear to fill the cracks in the concrete. This was quite evident in one particular core where paint, used to mark the core locations on the bridge deck had penetrated into a crack and was visible to a depth of approximately 1 to 1-1/2 inches. Further inspection of other cracks showed indications that the monomer had travelled down the cracks and penetrated into the surrounding concrete. However, the polymerized monomer did not actually fill the cracks. If a deck is to be sealed against entry of salt water that causes corrosion of imbedded steel, then cracks too must be sealed.

Electrical resistance measurements taken on these cores are listed in Table 14. The results do show that some significant reduction in conductivity was achieved by polymerization.

Using Test Method No. Calif. 342-D, a series of 10 skid measurements were made on the treated area and compared to the adjacent untreated area. The average result of the skid number measurements was 45 in the test area and 44 in the adjacent control area. These measurements indicate that the impregnation treatment as carried out had no significant effect on the skid resistance of the concrete surface.

Several observations were made during the actual polymer impregnation process on this bridge structure. Some of these were:

1. The infrared heaters worked well in drying out the concrete.

2. Once the concrete was heated, it required a long cooling period before monomer could be applied. It took about 8 hours for the concrete temperature to drop from 250-300°F to about 100°F.

Table 14

Electrical Resistance Measurements on

Grand Avenue Cores

(See text)

	Resistivity, K-ohms			
	Initial	16 Hrs.	10 Days	6 Weeks
No Treatment				
Control 1	15	4	3	3
Control 2	16	4	3	3
No Drying, Treated with Monomer				
No. 2	160	65	38	25
No. 3	250	95	42	29
Heated to 200°F and Cooled				
No. 4	115	55	36	31
No. 6	90	33	23	24
Normal Treatment				
No. 8	660	130	95	70
9	480	95	35	25
11	410	80	38	30
13	60	25	19	18
14	60	35	24	20
20	160	60	41	34
23	55	20	20	24
Polymerized at 170°F Concrete Temperature				
No. 15	80	45	29	24
No. 17	85	45	29	26
Polymerized at Max. Water Temperature of 150°F				
No. 18	80	25	17	13

3. The hand method of removing the sand prior to polymerization was very time consuming.

4. An adequate water supply is necessary to supply the steam generators. They consumed more water than expected, creating a supply problem.

5. Polymerization could have been achieved more rapidly if additional and higher capacity steam generators had been available to apply heat to all the test areas simultaneously.

6. The application of the monomer should be made during the evening hours because the ultraviolet radiation of the sun can produce premature jelling of the monomers.

7. The technique developed for surface impregnating of the test bridge deck should be applicable to other similar type structures, though some operations are inefficient. Ponding of the monomer and hot water needed for polymerization presents a real problem on decks that are not level.

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