# Report No. FHWA-RD-74-18 NONMETALLIC COATINGS FOR CONCRETE REINFORCING BARS

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TECHNICAL FILES D-10 RESEARCH

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# February 1974 Final Report

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This work was undertaken to ascertain the feasibility of using organic coatings, especially epoxies, to protect the steel reinforcing bars embedded in concrete of bridge decks from rapid corrosion.] This corrosion is caused by the chloride ions from the most commonly applied deicing salts, sodium chloride and calcium chloride Altogether, 47 different coating materials were evaluated to some extent, consisti of: 21 liquid and 15 powder epoxies; 5 polyvinyl chlorides; 3 polyurethanes; 1 polypropylene; 1 phenolic nitrile, and one zinc rich coating. The chemical and physical durabilities, chloride permeabilities, and protective qualities of coatir were assessed. The bonds between coated and uncoated bars and concrete were measu by both pullout and creep tests.						
The results indicate that both epoxy and polyvinyl chloride coatings, if properly applied, should adequately protect steel reinforcing bars from corrosion. However, only the epoxy coated bars had acceptable bond and creep characteristics when embedded in concrete. The powder epoxy coatings overall performed better than the liquid epoxies, and four powder epoxy coatings have been identified as promising materials to be used on reinforcing bars embedded in concrete decks of experimental bridges.						
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## CONTENTS

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			Page
LIS	r of	FIGURES	v
LIS	r of	TABLES	vi
SUM	MARY.		vii
1.	INTR	ODUCTION	1
	1.1	Project Objective	1
	1.2	Background Information	1
	1.3	Project Program	1
2.	COAT	ING MATERIALS	2
	2.1	Selection of Coating Materials	2
	2.2	Curing Methods and Specimen Preparation	6
		2.2.1 Two-Component Liquid Systems	6
		2.2.2 One-Component Liquid Systems	6
		2.2.3 Powder Systems	6
		2.2.4 Reinforcing Bars Coated by Applicators	7
3.	REIN	FORCEMENT AND CONCRETE SPECIMENS	7
	3.1	Reinforcement	7
	3.2	Concrete	7
	3.3	Test Specimens	12
		3.3.1 Pullout Specimens	12
		3.3.2 Creep Specimens	12
		3.3.3 Corrosion-Test Specimens	12
		3.3.4 Fabrication and Curing of Specimens	12
4.	EVAL	UATION OF COATING MATERIALS, EXPERIMENTAL METHODS AND RESULTS	12
	4.1	Chemical Resistance	13
		4.1.1 Epoxy Disc Specimens	13
		4.1.2 Immersion Studies of Coatings on Reinforcing Bars	13
		4.1.3 Chloride Permeability	16
	4.2	Impact and Abrasion Resistance of Epoxy Coatings on Steel Plates	16
	4.3	Inspection of Coatings on Steel Reinforcing Bars	22

' |

	4.4	Physic	al Testing of Coatings on Reinforcing Bars	Page 22
		4.4.1	Bend Tests	22
		4.4.2	Impact Tests	29
			4.4.2.1 Dropping Coated Bars on Concrete	29
			4.4.2.2 Falling Weight Method	29
		4.4.3	Hardness Determination	32
			4.4.3.1 Pencil Hardness	32
			4.4.3.2 Indentation Method	32
	4.5	Electr	ochemical Tests	32
		4.5.1	Applied Voltage Studies	32
		4.5.2	Electrical Potential and Electrical Resistance Measurements	34
			4.5.2.1 Coated Reinforcing Bars in 3.5 percent Solution of Sodium Chloride	34
			4.5.2.2 Coated Reinforcing Bars Embedded in Concrete	44
5.	BOND	STRENG	TH AND CREEP DETERMINATIONS	44
	5.1	Pullou	t Studies	44
		5.1.1	Pullout Test Procedures	49
		5.1.2	Results of Pullout Studies	49
	5.2	Creep	Studies	58
		5.2.1	Creep Test Procedures	58
		5.2.2	Results of Creep Studies	64
			5.2.2.1 Slip-Time Relationships	64
			5.2.2.2 Slip Values at 45 Days	64
6.	DISC	USSION	•••••••••••••••••••••••••••••••••••••••	67
	6.1	Evalua	tion of Coating Materials	67
		6.1.1	Chemical Resistance of Coating Materials	68
		6.1.2	Physical Durabilities of Coatings	68
		6.1.3	Corrosion Protective Qualities of Coating	69
		6.1.4	Bond Strengths and Creep Characteristics of Coated Bars in Concrete	69

		rage
	6.2 Proposed Qualification Criteria for Coating Materials	70
	6.3 Implementation of Epoxy-Coated Reinforcing Bars	70
7.	CONCLUSIONS AND RECOMMENDATIONS	72
	7.1 Conclusions and Recommendations	72
	7.2 Recommended Further Studies	73
8.	REFERENCES	74
9.	APPENDIX A. FIRMS SUBMITTING COATING MATERIALS FOR EVALUATION	77
10.	APPENDIX B. CONVERSION FACTORS OF U.S. UNITS TO SI UNITS	78

Pa	σ	e	
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1.	View of reinforcing bars	8
2.	Typical stress-strain characteristics of reinforcing bars	9
3.	Weight stabilities of cured specimen discs	15
4.	Permeability cell	19
5.	Concentration of chloride ions passing through epoxy films	21
6.A	. Applied voltage test at beginning of test	35
6.B	. Results of application of 2 volts for 30 minutes	36
7.	Measurement of electrical potential of coated reinforcing bars	37
8.	Measurement of electrical resistance of coatings on reinforcing bars	40
9.	Electrical potential and resistance measurements of reinforcing bar coated with material No. 1 immersed in 3 1/2 percent NaCl	41
10.	Electrical potential and resistance measurements of reinforcing bar coated with material No. 25 immersed in 3 1/2 percent NaCl	42
11.	Electrical potential and resistance measurements of reinforcing bar coated with material No. 31 immersed in 3 1/2 percent NaCl	43
12.	Corrosion-test specimen with coated reinforcing bar embedded in concrete block	45
13.	Corrosion-test specimens immersed in 3.5 percent solution of NaCl	46
14.	Pullout specimen on electromechanical testing machine	51
15.	Schematic of pullout specimen	52
16.	Applied load to reinforcing bar in pullout specimens versus free-end and loaded-end slip	53
17.	Schematic of creep specimen	59
18.	Creep specimen loaded to a tensile stress level of 30,000 psi	60
19.	Schematic of creep test frame	61
20.	Apparatus for calibrating steel springs for the creep study	62
21.	Range of displacement response of 24 steel springs used in the creep study	63
22.	Free-end and loaded-end slips in creep specimens	65

v

## LIST OF TABLES

,

\_

		Page
1.	Description of Coating Materials	3
2.	Properties of Reinforcing Bars	10
3.	Properties of Deformations	11
4.	Weight Changes of Cured Epoxy Discs Immersed in Aqueous Solutions	14
5.	Immersion Studies of Coatings on Reinforcing Bars	17
6.	Permeability of Chloride Ions through Epoxy Films	20
7.	Abrasion and Impact Resistance of Cured Epoxy Coatings on Steel Panels	23
8.	Inspection of Coated Steel Reinforcing Bars	24
9.	Results of Bending Coated Reinforcing Bars	27
10.	Drop Resistance and Impact Resistance of Coatings on Rebars	30
11.	Indentation Hardness of Coatings on Reinforcing Bars	33
12.	Electrochemical Studies of Coated Reinforcing Bars	38
13.	Electrical Potential and Resistance Measurements of Corrosion-Test Specimens in Aqueous Solution of 3.5 Percent NaCl	47
14.	Pullout Data	54
15.	Creep of Coated and Uncoated bars in Concrete Prisms (at 45 days)	66
16.	Proposed Criteria for Qualifying Coating Materials	71

#### SUMMARY

The objective of this project was to determine the feasibility of using organic coatings, especially epoxies, to protect steel reinforcing bars embedded in concrete of bridge decks from corrosion accelerated by chloride ions. The study was to include the selection and procurement of promising coating materials; evaluation of the physicochemical durabilities of coatings as well as their protective qualities; the determination of whether coated reinforcing bars would adversely affect the structural integrity of concrete bridge decks; and the development of performance criteria for the evaluation of new coating systems.

The coating materials were restricted to organic formulations, with the exception that the pigments could be of inorganic composition, and the selection was done on a generic basis. The most important criteria for selection were: inertness towards the constituents of cement paste and also chloride ions; creep characteristics; film integrity and protective qualities; and bond to steel. Altogether 47 different commercially available materials, of which 36 were epoxies, were evaluated to some extent.

Most of the epoxy coatings studied in this project had satisfactory chemical resistance to test solutions chosen to simulate the aggressive materials likely to be present in concrete bridge decks. Exceptions were some solvent-containing liquid epoxy systems which experienced large weight changes when immersed in the test solutions. Many of the epoxy coatings even as thin films, were found to be essentially impervious to chloride ions.

The abrasion resistances of all but two epoxy coatings were judged to be acceptable. Good correlation between the impact resistance (determined by the falling weight method) and the bend test was obtained. Brittle materials failed in both tests and conversely flexible materials had acceptable impact resistances and experienced minimal damage in the bend test. A large variation was observed between the relative flexibilities of epoxy coatings. However, in general, the powder epoxy system had better flexibilities than the liquid epoxy systems. Polyvinyl chloride coatings had excellent flexibilities even in film thicknesses up to 35 mils. The hardness determinations indicated that epoxies are tougher materials than the few polyvinyl chlorides that were submitted for the test and, therefore, should be more resistant to the abuse reinforcing bars normally experience.

Powder epoxy coatings were observed to provide more uniform coatings with fewer holidays than the liquid epoxy materials. Liquid epoxies tended to flow-off of the tops of the deformations of reinforcing bars during curing and accumulated in the low-lying regions between deformations, thus leaving the deformations either bare or thinly covered. In most corrosion studies carried out in the project, corrosion was observed to initiate at the deformations. The powder epoxy coatings, when properly applied and having a film thickness greater than 4 mils, adequately protected reinforcing bars from corrosion.

The effect of coated reinforcing bars on the structural integrity of bridge decks was assessed by pullout and creep studies. Epoxy coated reinforcing bars, with average film thicknesses between 5 and 11 mils, had acceptable bond strengths to concrete as measured in the pullout tests. All but two of the nine epoxy coatings that were included in the creep studies, had acceptable creep rates, i.e. creep rates comparable to those of uncoated bars. The polyvinyl chloride coated bars had unacceptable bond and creep characteristics.

Considering flexibility, bond strength, creep characteristics, and minimum corrosion protective requirements, it is concluded that the optimum film thickness of epoxy films on steel reinforcing bars is about 7  $\pm$  2 mils.

Of the materials evaluated under this research study, four powder epoxy coatings were found to be the best candidates for protecting steel reinforcing bars from corrosion. It is recommended that these four coatings be further evaluated in experimental bridge construction. Performance criteria have been suggested for the evaluation of similar coating.

#### 1. INTRODUCTION

#### 1.1 Project Objective

This project was undertaken to determine if organic coatings, especially epoxies, could be used to protect steel reinforcing bars in concrete of bridge decks from rapid corrosion.

#### 1.2 Background Information

The deterioration of concrete bridge decks, in 5-10 years, has become a major problem during the past decade  $[1-7]\underline{1}^{\prime}$ . The annual cost of such repairs on interstate highways has been estimated to be more than \$70 million in 1972. Often, this early deterioration has been attributed to accelerated corrosion of the steel reinforcing bars induced by chloride ions from deicing materials [8, 9]. Use of the two most commonly applied deicing materials, calcium chloride and sodium chloride, has increased substantially since the early 1960s [10]. Normally, steel is passive towards corrosion when in an environment of high basicity (pH of about 13) as in portland cement concrete [11]; chloride ions, however, are able to depassivate steel and thereby promote its active corrosion [11-12]. The resulting insoluble corrosion products occupy a substantially larger volume (possibly more than a tenfold increase) than the steel from which they were formed; and as a consequence large pressures may be exerted within the concrete which eventually cause the cracking and spalling of the concrete. Spellman and Stratfull reported [14] that as little as 1 mil of steel being converted into its characteristic corrosion products can cause the cracking of a 7/8 inch thick concrete layer.

Coating reinforcing steel with protective materials has been considered previously as a practical means of reducing the rapid corrosion of the bars. Much attention has been given to protecting reinforcing bars with metallic zinc with satisfactory results [15-16]. However, studies [17-18] suggest that zinc may be susceptible to rapid corrosion by chloride ions in basic environments. Cadmium [19] and nickel [20] have been reported to be satisfactory coatings for reinforcing steel. Their cost, however, may be prohibitively high. The use of organic types of barrier coatings for protecting reinforcing bars has been recommended [21-22]. Tripler and co-workers evaluated a few nonmetallic coatings and suggested that an epoxy-coal tar type of coating could have potential as a protective coating for reinforcing steel [20].

#### 1.3 Project Program

The program of this project was established to include: the selection and procurement of promising coating materials; evaluation of the physicochemical durabilities of coatings as well as their protective qualities; and the determination of whether coated reinforcing bars would affect the structural integrity of concrete bridge decks. The coating materials were evaluated according to the five-part scheme outlined below.

The first part, chemical resistance of coatings, consisted of preliminary screening tests performed to choose the most promising materials. Then these selected materials were subjected to the more exhaustive testing phase, parts 2 to 5. The tests in parts 2 to 5 were performed on coated reinforcing bars which had been coated in the factory by the applicators or manufacturers handling the respective materials.

- 1. Chemical Resistance of Coating
  - A. Resistance to water, calcium chloride, calcium hydroxide, calcium sulfate and fresh portland cement paste.
  - B. Chloride permeability.

1/ Figures in brackets indicate the literature references at the end of this paper.

- 2. Film Integrity of Cured Coatings
  - A. Coverage characteristics; variations in film thickness, etc.
  - B. Application methods.
  - C. Preparation of steel substrate prior to coating application.
- 3. Physical Durabilities of Coatings on Rebars
  - A. Abrasion and impact resistances.
  - B. Flexibilities determined by bending coated rebars.
- 4. Electrochemical Measurements of Coated Rebars Immersed in Sodium Chloride Solutions
  - A. Applied voltage studies.
  - B. Electrical potential of coated bar.
  - C. Electrical resistance of coating film.
- 5. Bond Tests of Coated Rebars Embedded in Concrete
  - A. Pull out.
  - B. Creep.

#### 2. COATING MATERIALS

#### 2.1 Selection of Coating Materials

The coating materials to be evaluated were restricted to organic formulations, with the exception that the pigments could be of inorganic composition, and the selection was done on a generic basis. The most important criteria for selection were: inertness towards the constituents of cement paste and also chloride ions, creep characteristics; film integrity and protective qualities; and bond to steel and concrete.

The coatings selected for evaluation are listed in table 1 (also included are a few unsolicited materials submitted by the respective firms who handle them), and all are of commercial origin. The coating materials have been assigned code numbers for identification purposes. The code number sequence has no significance other than indicating the chronological order in which the materials were received.

The polyurethanes and epoxies each consist of two components, a resin and a curing component, and are classified as thermosetting materials because their cure (polymerization) is accelerated by the application of heat. Once cured, thermosetting materials normally retain their shapes up to their decomposition temperatures. The other coatings in table 1 are classified as thermoplastics as they soften and change shape far below their decomposition temperatures when heated. Thermoplastics usually are one component systems.

Emphasis in this study has been given to the thermosetting materials and especially epoxies because these materials seem to best satisfy the established criteria. Altogether 36 epoxy coatings, both powder and liquid systems, have been evaluated to some extent. The terms powder and liquid refer to the uncured state of epoxy coatings; when cured, i.e. polymerized, they form hard solids.

Some materials, especially powders, were submitted only in the form of cured films on steel reinforcing bars.

Code			Percent $\frac{1}{}$	
Number	Туре	Uncured State	Solvent	Comments
1	Epoxy-Polyamide <sup>2/</sup>	Liquid - 100	0	Used for concrete overlays.
2	Epoxy-Polyamide	Liquid - 100	0	Has been previously tested as coating for steel rebars,
3	Epoxy-Polyamide	Liquid - 100	0	Primer.
4	Epoxy-Modified amine	Liquid - 100	0	Primer.
5	Epoxy-Polysulfide	Liquid - 100	0	Used to bond fresh concrete to old concrete.
6	Epoxy-Modified amine	Liquid - 100	0	Topcoat.
7	Epoxy	Liquid - 50	50	Water emulsion activated system.
8	Epoxy-Polyamide	Liquid - 60	40	
9	Epoxy-Polyamide	Liquid - 50	50	Primer.
10	Epoxy-Ketamine	Liquid - 100	0	High viscosity.
11	Epoxy-Ketamine	Liquid - 100	0	3
12	Ероху	Liquid - ca. 100	nil	Cures at relative humidity above 50 percent.
13	Ероху	Liquid - ca. 100	nil	Limited flexibility.
14	Vinyl	Liquid - 20	80	Primer paint.
15	Epoxy-Polyamide	Liquid - 50	50	Epoxy paint.
16	Epoxy-Polyamide	Liquid - 50	50	Epoxy paint.
17	Epoxy	Liquid - 100	0	Low viscosity.
18	Coal-tar epoxy	Liquid - 100	0	Protective overlay on concrete pavements.
19	Ероху	Liquid - 46	54	One component; cures by heating.
20	Ероху	Powder		Only coated rebars have been received.
21	Ероху	Powder		Only coated rebars have been received.

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# TABLE 1. DESCRIPTION OF COATING MATERIALS

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Code			Percent $\frac{1}{}$	
Number	Туре	Uncured State	Solvent	Comments
22	Ероху	Powder		Only coated rebars have been received.
23	Polyvinylchloride	Powder		Only coated rebars have been received.
24	Polyvinylchloride- Plastisol	Powder		Only coated rebars have been received.
25	Epoxy	Powder		
26	Polyvinylchloride	Powder		Only coated rebars have been received.
27	Ероху	Powder		
28	Ероху	Powder		
29	Ероху	Powder		
30	Polyvinylchloride	Powder		Only coated rebars have been received.
31	Ероху	Powder		Same material as No. 22 but coated rebars submitted from a different applicator and also the pure powder was submitted.
32	Ероху	Powder		
33	Polyurethane	Liquid	0	Only coated rebars have been received.
34	Phenolic nitrile	Liquid - 10	90	Adhesive for metals.
35	Polyurethane	Liquid - 100	0	Elastomer, elongation - 600%.
36	Polyurethane	Liquid - 100	0	Elastomer, elongation - 550%.
37	Ероху	Liquid - 100	0	Adhesive for metals.
38	Ероху	Powder	P.	
39	Ероху	Powder		
40	Ероху	Powder		
41	Epoxy	Powder		Only coated rebars have been

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Unly coated rebars have been received.

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Code			$Percent^{1/2}$	
Number	Туре	Uncured State	Solvent	Comments
42	Ероху	Powder		Only coated rebars have been received, same materials as No. 41, but different application procedure.
43	Ероху	Powder		
44	Zinc in a zinc silicate binder	Liquid - 80	20	Metallic zinc filler and liquid base are mixed, hardens by solvent evaporation.
45	Coal-tar epoxy	Liquid	Unknown	One component, epoxy resin and coal tar, no curing components.
46	Epoxy-Polysulfide	Liquid		
47	Polypropylene	Powder		Only coated rebars have been received. Coating very brittle.

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1/ Solvents comprised the listed percent of liquid materials.

2/ Materials identified as 1-1 and 1-S in tables 13 and 14 denote a different pigmentation of material No. 1 and material No. 1 mixed with small amount of sand, respectively.

#### 2.2 Curing Methods and Specimen Preparation

#### 2.2.1 Two-Component Liquid Systems

The epoxy liquid systems consist of two components which must be mixed in the proper ratio to attain the optimum degree of polymerization. In the present work, the ratio of curing component to epoxy resin, and the mixing and curing times, were closely controlled and were the same as those specified by the manufacturers' accompanying instructions. The epoxy resins and curing components were mixed at room temperature, ca. 75°F, relative humidity of ca. 50 percent, using either an electric stirrer for solvent-free systems or a metal spatula for solvent-contining systems. The two component urethane materials and the zinc-filled coatings were similarly mixed.

Test specimens of the coating materials were cast immediately after mixing was completed and, in addition, steel plates and steel reinforcing bars were coated with thin films. Specimen discs of 2 1/4 inches in diameter with thickness of ca. 3/8 inches (thicknesses of solvent containing systems were reduced to 3/16 inches) were cast using aluminum weighing dishes as molds. The discs were stripped from the molds after a curing period of seven days.

Wet films of 3-7 mils<sup>1</sup>/ thickness were formed by applying the coatings with a Baker roller film applicator to the gel side of photographic paper or to sheets of Teflon<sup>2</sup>/. The cured films were stripped from the photographic paper after being immersed in water at room temperature for 16 hours. Cured films were easily stripped from Teflon sheets using a thin-bladed spatula. Coating materials were applied to 4 x 4 x 0.050 inch cold-rolled steel plates and to No. 6 steel reinforcing bars<sup>3</sup>/ using a paint brush. The steel plates had been degreased previously using mineral spirits, and the reinforcing bars had been sand blasted to a near white surface [23].

#### 2.2.2 One-Component Liquid Systems

The two one-component liquid systems, a polyvinyl chloride (PVC) and a phenolic nitrile, were hardened by the evaporation of solvents. Test specimens were formed as previously described for the two-component systems.

#### 2.2.3 Powder System

No mixing of the epoxy powders was necessary since the two components are contained within each powder particle. The powders were applied to steel and Teflon substrates preheated to ca. 400°F in an electric oven by immersing the substrates into a fluidized bed [24] of the powders.

Then the coatings were cured in the electric oven under the conditions specified by the manufacturers. When allowed to cool to room temperature, the cured epoxy films were easily removed from the Teflon substrate by using a thin-bladed spatula. Both No. 6 steel reinforcing bars and  $4 \ge 4 \ge 0.050$  inch cold-rolled steel plates were coated with the powders.

Specimen discs could not be satisfactorily fabricated from the epoxy powders because, when sufficient masses to make 3/16 inch thick discs were heated to their specified curing temperatures, porous solids that had expanded over 100 percent were produced. Even fourfold reduction of the masses did not yield satisfactory specimens. Only one powder epoxy

#### 1/ One mil equals 0.001 inch, exactly.

<u>2</u>/ Certain instruments and materials are identified in this paper in order to adequately specify the experimental conditions. In no case does such identification imply recommendations or endorsement by the National Bureau of Standards or the Federal Highway Administration, nor does it imply that the material or instruments are necessarily the best available for the purpose.

3/ No. 6 steel reinforcing bars have a nominal diameter of 3/4 inch.

did not exhibit this expansion phenomenon. Possibly, some of the curing components are vaporized at the curing temperatures causing the formation of porous structures in the thick castings. However, no difficulties were encountered when films that were less than 20 mils thick were formed from the powder epoxies.

#### 2.2.4 Reinforcing Bars Coated by Applicators

The coatings materials that were judged (on the basis of preliminary screening tests which included: resistance to chemicals; cure time; film integrity; and evaluation of relative brittleness) to have the most promise as potential protective coatings for steel reinforcing bars were subjected to further testing using bars coated by the applicators or manufacturers submitting the respective coatings. No. 6 steel reinforcing bars, grade 60, four feet in length having two different deformation patterns, were supplied to each applicator. The surfaces of the bars were usually cleaned, often by sandblasting; coating applied and cured; and the bars returned to the National Bureau of Standards for evaluation.

#### 3. REINFORCEMENT AND CONCRETE SPECIMENS

#### 3.1 Reinforcement

The tensile reinforcement in the pullout, creep, and corrosion tests consisted of No. 6 deformed bars having either a barrel (B) or diamond shaped (D) deformation pattern as shown in figure 1. The bars were randomly selected and may not all have been the same heat. A 4 foot length of each type of bar was tested to rupture in tension. The yield strengths determined by the "0.2 percent offset" method were 67,600 psi for No. 6 rebars (D) and 62,500 psi for No. 6 rebars (B). These bars did not exhibit a well-defined yield point, however, their stress-strain relationships (figure 2) were linear up to a stress of about 64,000 psi for the (D) rebars and approximately 62,000 psi for the (B) rebars. Tensile properties of the bars are listed in table 2. The yield, tensile strengths and deformations of the bars met the requirements of ASTM A 615-72 [25] for Grade 60 bars. The properties of deformations were determined from three coupons of each type of bar and are given in table 3.

#### 3.2 Concrete

The concrete was procured from a transit-mix concrete company. The mix proportions of portland cement (type I), sand, and coarse aggregate were approximately 1:1.7:2.5, by weight. The sand was a silicious aggregate and the coarse aggregate was crushed stone. Maximum size of the coarse aggregate was 3/4 in. Water content of the concrete was about 5 1/2 gallon per sack of cement and the slump ranged from 3 to 5 in. Although the concrete contained an air entraining admixture, the air content ranged from only 1 to 3 percent. Three batches of concrete were used to cast the pullout specimens and the corrosion test specimens.

Six standard 6 x 12 in. cylinders were cast from each batch of concrete along with the pullout specimens. The cylinders were stored and cured in the same manner as the pullout specimens, and their compressive strengths were measured at the same time the specimens were tested. The compressive strength was determined in accordance with ASTM C39-66 [26]. The average compressive strengths at 27 to 29 days were 6160 psi for concrete batch No. 1, 6620 psi for batch No. 2 and 5730 psi for batch No. 3. The ranges and coefficients of variation [27] of the strength of the concrete cylinders were 226 psi and 1.5 percent, 136 psi and 0.8 percent, 355 psi and 2.3 percent for concrete batches Nos. 1, 2, and 3, respectively. The creep specimens were cast from two additional batches, batch No. 4 and 5 of concrete, which had average compressive strengths at 28 days of 5494 psi and 5665 psi. The range of the strength of the concrete cylinders and coefficient of variation were 442 psi and 4.1 percent and 285 psi and 2.5 percent, respectively.



FIGURE 1. View of No. 6D (upper bar) and No. 6B (lower bar) reinforcing bars. D denotes diamond shaped deformation pattern and B indicates barrel pattern.





Bar Size and Type	Area, <sup>A</sup> s2 (in <sup>2</sup> )	Perimeter, <sup>∑</sup> 0 (in)	Yield <sup>1/</sup> Strength f <sub>y</sub> (psi)	Proportional Limit (psi)	Tensile Strength (psi)	Modulus of Elasticity, E <sub>s</sub> (10 <sup>6</sup> psi)	Elongation in 10 in (percent)
No. $6D^{2/}$	0.441	2.35	67,600	63,900	95,700	30.7	11.2
No. 6B <sup>3/</sup>	0.434	2.34	62,500	61,800	95,200	28.4	8.2

### TABLE 2. PROPERTIES OF REINFORCING BARS

1/ Yield strength was determined by the "0.2 percent offset" method.

2/ D denotes diamond deformation pattern.

3/ B denotes barrel deformation pattern.



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TABLE	3.	PROPERTIES	OF	DEFORMATIONS-1

Bar and	Size Type	Width of gap (in.)	Average Spacing (in.)	Average Height (in.)	Average Projected Length (in.)	Bearing Area ( (in. <sup>2</sup> /in.)
No.	6D <u>1</u> /	0.064	0.300	0.040	2.22	0.296
No.	6B <sup>2/</sup>	0.047	0.402	0.038	2.25	0.212

 $\underline{1}$  / Methods of measuring properties of deformation and definition of terms are given in reference 25.

2/ D denotes diamond deformation pattern.

3/ B denotes barrel deformation pattern.

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#### 3.3 Test Specimens

#### 3.3.1 Pullout Specimens

The pullout specimens were  $10 \ge 10 \ge 12$  in. concrete prisms with the reinforcing bar concentric with the longitudinal axis of the specimens, so that the length of embedment of the bar in concrete was 12 inches. This development length of the deformed bar was selected based on previous studies at NBS [28] and because the current ACI Standard 318-71, "Building Code Requirements for Reinforced Concrete" states that the development length should not be less than 12 inches [29]. The pullout specimen was designed so that the loaded-end slip reached a value of 0.01 inch corresponding to a steel stress of approximately one half of its tensile strength when uncoated bars were embedded in the specimen. Splitting of the concrete was minimized by reinforcing the specimen with a cylindrical cage of  $2 \ge 2 - 12/12$  welded wire fabric. The cages had a diameter of 8 in., extending the length of the specimen and were concentric with the reinforcing bar.

#### 3.3.2 Creep Specimens

The creep specimens were the same as the pullout specimens except that the lower 23 inches of the bars were threaded, thread size of 5/8 inch diameter with 18 threads per inch, to permit loading of the specimen assembly. Altogether 24 creep specimens were cast from concrete batches Nos. 4 and 5 in the same wooden forms used to cast the pullout specimens.

#### 3.3.3 Corrosion-Test Specimens

The corrosion test specimens were concrete blocks  $2 7/8 \ge 4 7/8 \ge 15$  inches in which were embedded a 24 inch length of No. 6 reinforcing bar concentric with the longitudinal axis of the block. The bars protruded out of the concrete blocks with exposed ends of 1 inch and 8 inches. The end of the block with the 1 inch of bar protruding which was to be immersed in the salt solution, was first coated with an "underwater" type of epoxy which bonded firmly to the concrete. Then the 1 inch exposed end of the bar and the epoxy were coated completely with a heavy layer of silicone rubber. The bond of the rubber to steel and to the epoxy was very good, although its bond to concrete in a salt solution is poor. A terminal was inserted into the long end of each reinforcing bar so that electrical connections could be made to a voltmeter or conductivity bridge.

#### 3.3.4 Fabrication and Curing of Specimens

All concrete pullout specimens were cast with the reinforcing bar in horizontal position in wooden forms which were lined with stripping oil. The specimens were removed from the forms after 2 days and moist cured for 14 days with wet burlap and then room cured at 73°F and 50 percent relative humidity until tested.

Two pullout specimens with uncoated reinforcing bars were fabricated from each of concrete batch Nos. 1 and 2 and one such specimen was cast from batch No. 3. Duplicate specimens were fabricated for each coating material from the same batch of concrete with the exception that only one pullout specimen was fabricated that contained coating No. 1-S.

The corrosion test specimens were fabricated from the same batches of concrete as the pullout specimens. Coated bars selected for corrosion testing coincided (when sufficient specimens were available) with those used in the pullout test.

#### 4. EVALUATION OF COATING MATERIALS -EXPERIMENTAL METHODS AND RESULTS

Descriptions of the test methods used to evaluate coating materials are given in this section along with the experimental results.

Not all of the 47 coating materials which were evaluated were subjected to the same degree of testing; some were quickly judged not to be acceptable for such reasons as: gel times for two component systems longer than eight hours, coatings Nos. 10 and 11;

poor film integrity and excessive entrapped air in the cured state, Nos. 12 and 13; the tendency of some thermoplastics to soften at 140°F, Nos. 14 and 47; rubber-like expansion qualities (500 to 600 percent elongation) of two urethanes, Nos. 35 and 36 (obviously these two coatings would not pass the pull-out and creep requirements); and extreme brittleness of some epoxy systems, Nos. 5 and 13.

#### 4.1 Chemical Resistance

#### 4.1.1 Epoxy Disc Specimens

Disc shape castings of cured epoxy specimens (these epoxies are liquids in the uncured state) were immersed in the following: water; an aqueous solution of 3M CaCl; an aqueous solution of 3M NaOH; and a solution saturated with both Ca(OH) and CaSO<sub>4</sub>.2H<sub>2</sub>O, and containing 0.5M CaCl<sub>2</sub>. These test chemicals were selected because they are with the exception of NaOH, present in concrete of bridge deckings and are probably the major chemicals most potentially deleterious to epoxy coatings. Ca(OH) is a reaction product of portland cement and water; it stabilizes the silicate gels which are important constituents of durable conretes. CaSO<sub>4</sub>.2H<sub>2</sub>O is often added as a set-regulator to portland cement and also is frequently present in soil drainage water. CaCl<sub>2</sub> is one of the two most commonly used deicing materials. Since the solubility of Ca(OH)<sup>2</sup> is low (0.2M at 25°C), 3M NaOH was used in an accelerated-type of test to determine if<sup>2</sup> hydroxide ions are detrimental to the long-term embedment of epoxy coatings in concrete.

Water, in itself, can have a deleterious effect on coating materials. It was felt that these test solutions are probably as aggressive or even more aggressive than those encountered in concrete. Therefore, materials performing well in the immersion tests will probably not be degraded by long-term embedment in concrete. The specimens were immersed in water for one or two minutes and wiped dry before measuring the original weights prior to the immersion studies. Original weights of the discs varied from ca. 20 grams for solvent containing systems to ca. 50 grams for the solventless epoxy systems. The temperatures of the test solutions were  $75 \pm 1^{\circ}F$ .

The immersion data are listed in table 4. In some cases two separate castings were made, indicated by two sets of data with different immersion times. In general, the specimens had average weight changes in the four test solutions that were under 4 percent. The three materials experiencing the largest weight changes, Nos. 7, 8, and 9, contained solvents in their uncured states. The surfaces of both No. 7 and No. 9 changed from smooth to rough textures during the immersion period. No visually apparent surface deterioration was observed with the other epoxy specimens.

The weight changes versus immersion times are plotted in figure 3 for epoxies Nos. 3, 4, 7, and 16, which represent the different behaviors of the epoxy specimens. No. 4 is typical of most of the specimens, with a modest weight increase irrespective of the test solution; in contrast No. 16 has a gradual loss of weight in all four test solutions. No. 7 had the largest weight change of all the epoxies tested with weight increases ranging from 13 to 19 percent; which essentially occurred during the first month of immersion. No. 3 experienced a gradual weight loss except when immersed in the NaOH solution.

A weight increase probably can be attributed to absorption of the test solution by the epoxy specimen; while loss in weight can be attributed to dissolution of the specimen in the test solution, or in the case of solvent-containing systems, the loss of entrapped solvent. It is felt that epoxies exhibiting minor weight changes, i.e. average under 4 percent, will not be degraded by long-term embedment in concrete.

#### 4.1.2 Immersion Studies of Coatings on Reinforcing Bars

The chemical resistances of the powder epoxy and polyvinyl chloride systems were investigated by immersing coated reinforcing bars, supplied by applicators, in aqueous solutions of the following: 3M NaOH; saturated Ca(OH); and 3.5 percent (0.7M) NaCl. Many of the liquid epoxies, usually applied by the staff of the National Bureau of Standards using brushes, were also included in these immersion studies. The coated bars were •

	TERCENT WEIGHT CHANGES					
Immersion Time (weeks)	Water	3M CaCl <sub>2</sub>	3M NaOH	Saturated Ca(OH) $_{2}$ Saturated CaSO $_{4}$ $_{2}$ $_{2}$ $_{2}$ and 0.5M CaCl $_{2}$		
66 53	2.3 4.2	1.7 1.2	2.5 3.9	3.5 4.6		
66 53	4.2 3.9	2.5 2.7	3.6 3.8	5.2 3.2		
66 53	-2.3 -3.2	-2.4 -3.5	1.8 3.7	-3.0 -3.9		
66 53	2.9 2.5	1.5	2.1 2.2	2.6 2.4		
66 53	1.5 1.8	1.3 0.8	2.1 0.7	2.1 1.7		
66 53	3.7 3.0	1.8 1.5	2.8 2.5	3.6 3.2		
66	16	19	13	18		
66	-9.1	-6.3	-2.7	-5.5		
66	-6.8	- 10	5.3	-13		
64	1.5	1.0	2.1	2.0		
64	3.3	1.0	2.5	2.5		
57 53	-0.8 -3.6	-2.3 -4.2	0.4 0.9	-2.0 -4.5		
59 53	3.3 2.6	1.0 1.2	3.0 2.7	3.9 2.6		
59 53	0.8 1.0	0.2	0.9 1.0	0.9		
	Immersion Time (weeks) 66 53 66 53 66 53 66 53 66 53 66 53 66 66 66 66 66 66 66 64 64 64 57 53 59 53 59 53	$\begin{array}{c c} \mbox{Immersion}\\ \mbox{Yater}\\ \mbox{(weeks)} \end{array} & \begin{tabular}{ c c c } \hline Water\\ \hline & & & & & & & & & & & & & & & & & & $	Immersion Time (weeks)Water $3M \operatorname{CaCl}_2$ $66$ $2.3$ $1.7$ $53$ $4.2$ $1.2$ $66$ $4.2$ $2.5$ $53$ $3.9$ $2.7$ $66$ $-2.3$ $-2.4$ $53$ $-3.2$ $-3.5$ $66$ $2.9$ $1.5$ $53$ $2.5$ $1.2$ $66$ $1.5$ $1.3$ $53$ $2.5$ $1.2$ $66$ $1.5$ $1.3$ $53$ $1.8$ $0.8$ $66$ $3.7$ $1.8$ $53$ $3.0$ $1.5$ $66$ $16$ $19$ $66$ $-9.1$ $-6.3$ $66$ $-6.8$ $-10$ $64$ $1.5$ $1.0$ $64$ $3.3$ $1.0$ $57$ $-0.8$ $-2.3$ $53$ $-3.6$ $-4.2$ $59$ $3.3$ $1.0$ $53$ $2.6$ $1.2$ $59$ $0.8$ $0.2$ $53$ $1.0$ $0.5$	Immersion Time (weeks)Water $3M \operatorname{CaCl}_2$ $3M \operatorname{NaOH}$ 662.31.72.5534.21.23.9664.22.5533.92.73.866-2.3-2.453-3.2-3.5532.51.2662.91.5532.51.2661.51.32.52.1532.51.2661.51.3531.80.80.766663.7662.91.52.1531.80.80.7663.766-9.1-6.3-2.766-6.8-105.3641.51.02.1643.31.053-3.6-4.20.9593.31.03.0532.61.22.7590.80.20.90.5531.0		

PERCENT WEIGHT CHANGES



FIGURE 3. Weight stabilities of cured specimen discs, fabricated from four liquid epoxies, immersed in aqueous solutions.

visually inspected for evidence of softening and color changes of the coating, disbonding between the coating and steel surface, and for number and size of corrosion sites. Temperatures of the test solutions were  $75 \pm 1^{\circ}F$ . The results after one year of immersion are described in table 5.

The NaCl solution had a more deleterious effect on the coatings than the  $Ca(OH)_2$  and NaOH solutions, with the NaOH solution being the least aggressive. Only one coating, No. 11, gave any indication of being degraded by the long-term immersion in 3M NaOH. The greater discriminating effect of  $Ca(OH)_2$  versus NaOH is an interesting phenomenon since the pH of both solutions (pH of saturated  $Ca(OH)_2$  is 12.6 and of 3M NaOH is 14.5) should be sufficient to passivate steel. The causes of this phenomenon are not presently obvious and this is an area worthy of additional studies.

Coatings with ratings of 1 or 2 (Nos. 22, 23, 24, 25, 30, 31, and 41) adequately protected the reinforcing bars from corrosion when immersed in any of the test solutions. Four of the coatings with ratings of 1 or 2, Nos. 22, 25, 31, and 41 were powder epoxies; other coatings with high ratings, Nos. 23, 24, and 30, were polyvinyl chlorides with heavy film thicknesses ranging from 15 to 35 mils. The poor performance of some bars in this test may be not because of some undesirable property of the coating itself, but rather because of the improper coating of the individual bars such as insufficient thickness of the coating, preexisting holidays, etc.

#### 4.1.3 Chloride Permeability

If coatings are to be effective in protecting steel reinforcing bars, the intrinsic chloride permeabilities of the coatings must be low. Little if any data on the permeability characteristics of epoxy films to chloride ions have been reported previously.

The chloride permeability characteristics of thin films (3-7 mils) of cured epoxies were determined using permeability cells of the type shown in figure 4. Films selected for study were carefully handled, and examined for any defects prior to installation in the cell. The cell consisted of two glass compartments separated by an epoxy film sandwiched between two glass plates, each having centered 1 inch diameter holes. One compartment contained 175 ml of 3M NaCl and the other 115 ml of distilled water. The activity of chloride ions passing through the epoxy membrane was measured using a Model 401 Orion Specific Ion Meter, along with a Model 94-17 Orion Chloride Electrode, and a Model 90-02 Orion Double Junction Reference Electrode. Activity measurements were converted into concentration values of mole per liter with a conversion diagram, constructed by plotting measured chloride ion activities versus known chloride ion concentrations.

The data for 15 different epoxies are listed in table 6. Many of the epoxy films, Nos. 1, 3, 17, 18, 31, and 39, appear to be essentially impervious to chloride ions (at least during the listed exposure times). The accumulative concentrations of chloride ions passing through epoxy films are plotted versus time in figure 5 for six different epoxy films. Generally, the chloride permeability rates were highest during the first six weeks of exposure. An exception is the film of No. 38 which initially seemed to be impervious to chloride ions, however, after about six weeks its permeability rate began to sharply increase and after 39 weeks the accumulative concentration of chloride passing through the film was about 3 x  $10^{-3}$ M. Two other films, Nos. 13 and 16, also permitted sufficient chloride ions to migrate through so that the concentration in the compartment originally containing distilled water approached or reached the chloride ion threshold concentration of 0.02M which has been reported [30] to induce the corrosion of steel embedded in portland cement-concrete.

4.2 Impact and Abrasion Resistance of Epoxy Coatings on Steel Plates

In the preliminary screening phase of the study, the impact and abrasion resistances of coatings on steel plates were determined to assess the ability of coatings to withstand harsh treatment.

Both direct and reverse impact resistances of cured epoxy coatings on  $4 \ge 4 \ge 0.050$  in. cold-rolled steel plates were determined by the falling weight method outlined in ASTM Designation G14-69T [31]. A Gardner Laboratory impact tester was used along with a four pound hammer.

			AQUEOUS IESI SOLUII	0105	
Code Number	Applied by 1/	3M NaOH	Saturated Ca(OH) 2	3.5 Percent (0.5M) NaCl	Rating <sup>2/</sup>
1	MFR	No change	Few rust spots	8 rust spots	3
1-1	MFR	No change	Few rust spots	10 rust spots	3
1-2	MFR	No change	Few rust spots	7 rust spots	3
2	NBS	No ch <b>an</b> ge	Few rust spots	6 rust spots	3
3	NBS	No change	Few rust spots	7 rust spots	4
2 & 3	NBS	No change	Few rust spots	3 rust spots	2
4	NBS	No change	Few rust spots	Severely rusted on ridges	3
5	NBS	No change	Few rust spots	Severely rusted on ridges	4
6	NBS	No change	No change	Severely rusted on ridges	4
10	MFR	No change	No change	18 rust spots	5
11	MFR	Coating cracked & disbonded. Color leached from epoxy.	No change	Severely rusted	5
14	MFR			Severely rusted	4
15	MFR			3 rust spots; poor bond	3
16	MFR			No change	3
17	NBS		No change	20 rust spots	4
18	NBS	No change	6 rust spots	Badly rusted; coating softened	5
19	MFR	No change	Badly rusted	Very badly rusted; most of coating gone	5
20	MFR			Light rust spots under coating; bond very poor	3
21	MFR			12 rust spots	3
22	MFR	No change	No change	No change	1
23	MFR	No change	No change	No change	1
24	MFR	No change	No change	No change	1
25	MFR	No change	No change	No change except l rust spot and small blisters	2

AQUEOUS TEST SOLUTIONS

17

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			HOUGOD TEDT DODOTT			
Code Number	Applied by $\frac{1}{}$	3M NaOH	Saturated Ca(OH) <sub>2</sub>	3.5 Percent (Q.5M) NaCl	Rating <sup>2/</sup>	
26	MFR			Rusted badly	5	
27	MFR	No change	No change	No change except few rust spots	3	
28	MFR	No ch <b>an</b> ge	Badly rusted	Rusted badly; coating softened	4	
29	MFR	No change	Few rust spots	Very badly rusted	4	
30	MFR	No change	No change	No change except 5 small rust spots	2	
31	MFR	No change	No change	No change except l rust spot	1	
32	MFR	No change	No change	Rusted badly	3	
33	MFR	No change	No change	Rusted severely. Tubercles 1/4" high	3	
34	NBS			Rusted badly	5	
35	NBS			Large rusted areas	5	
36	NBS			Large rusted areas	5	
37	NBS			Large rusted areas	5	
38-Blast	MFR	No change	Rusted	Badly rusted	4	
38-Phosp	MFR	No change	Rusted badly	Rusted badly; coating softened	5	
39-Blast	MFR	No change	No change except for few rust spots	Rusted	3	
39-Phosp	MFR	No change	No change except slight softening	Rusted	4	
40-Blast	MFR	No change	Rust spots	Badly rusted	3	
40-Phosp	MFR	No change	Rust spots; coating softened	Badly rusted; coating softened	4	
41	MFR	No change	No change	15 rust spots	2	
42	MFR	No change	No change	10 rust spots	3	
42-Un- coated	MFR	Slightly rusted	Badly rusted	Badly rusted	5	

AQUEOUS TEST SOLUTIONS

<u>1</u>/ MFR denotes the firm handling the material applied the coating to reinforcing bars and submitted the coated bars to NBS for evaluation. NBS denotes coating applied by the staff of the National Bureau of Standards.

 $\underline{2}$ / Rating sequence in order of decreasing protective qualities 1<2<3<4<5.



FIGURE 4. Permeability Cell

19

Components: A. Compartment containing distilled water;

- B. Epoxy film sandwich between two glass plates each having centered one-inch diameter hole;
- C. Component containing 3M NaCl

Code Number	Thickness Thickness (mils)	Exposure Time (weeks)	Concentrations <sup>1/</sup> (Moles per liter)	Permeability <u>Units<sup>2</sup></u> /
1	3	50	$<5 \times 10^{-5} \frac{3}{2}$	$<2.5 \times 10^{-6}$
2	3	23	$1 \times 10^{-4}$	$9.7 \times 10^{-6}$
3	3	16	$(5 \times 10^{-5})$	$<8.5 \times 10^{-6}$
4	3	23	$1 \times 10^{-4}$	$9.7 \times 10^{-6}$
6	3	23	$1 \times 10^{-4}$	9.7 x $10^{-6}$
11	3	12	$4 \times 10^{-3}$	$7.5 \times 10^{-4}$
13	3	21	$1 \times 10^{-2}$	5.0 x $10^{-4}$
16	7	23	$2 \times 10^{-3}$	$6.2 \times 10^{-4}$
	3	10	$8 \times 10^{-1}$	$2.3 \times 10^{-1}$
17	3	50	$<5 \times 10^{-5}$	$<2.5 \times 10^{-6}$
19	7	37	N. C. <u>4</u> /	
29	10	37	N. C.	
31	10	37	N. C.	
38	2.5	39	$3 \times 10^{-3}$	$1.8 \times 10^{-5}$
39	2.5	39	N. C.	
40	2.5	39	$6 \times 10^{-2}$	$3.2 \times 10^{-4}$

 $\underline{1}/$  Concentration of chloride ions in the chamber originally containing only distilled water.

2/ Permeability units are: (grams per day)/exposed area (in.<sup>2</sup>)/film thickness (mils); i.e. theoretically square inch and a film thickness of one mil.

3/ Millivolt readings were near the region of distilled water and the lower limit of the chloride ion concentration was estimated.

4/ N. C. denotes that no changes from the original millivolt values were measured.



FIGURE 5. Concentration of chloride ions passing through epoxy films of materials Nos. 2, 13, 16, 31, 38 and 39. Film thicknesses were 3 mils.

The impact data are presented in table 7. The reverse impact was found to be more severe than the direct impact and probably gives a better indication of the flexibility of a coating. Reverse impact values lower than 40 in. lb. are judged to be indicative of brittle materials. It is felt that the impact values for some powder materials (Nos. 25, 28, 29, 31, and 38-40) are too low and are not reliable indicators of their properties. Possibly, the low values can be attributed to poor adherence to the surface of the steel plates used in this impact test. The bend testing of coated reinforcing bars, and also impact tests on coated bars, discussed later, are considered to yield more reliable results.

The abrasion resistances of epoxy coatings on similar steel panels were determined in accordance with ASTM Designation D1044-56 [32] by using a Taber Abraser and Taber CS-10 wheels with 1000g load per wheel. After each 200 cycles the wheels and specimens were gently cleaned with a soft bristle brush. The abrasion data are given in table 7 in units of weight loss in mg per 1000 cycles. Two of the solvent containing materials, Nos. 3 and 16, had weight losses over 100 mg indicative of poorer abrasion resistances than the other epoxy coatings tested.

#### 4.3 Inspection of Coatings on Steel Reinforcing Bars

The coatings applied to reinforcing bars by the applicators were inspected immediately after receipt for the following: film thickness, number of holidays  $\frac{4}{2}$  per unit bar length (4 ft.), and for their general appearance. The film thicknesses were measured with a Mikrotest Model 790000 Magnetic Gage, and the number of holidays was determined with a 67 1/2 volt holiday detector.

The results of the inspection are listed in table 8. In general, the powder epoxy coatings yielded films of more uniform thicknesses and with fewer holidays than the liquid epoxy coatings. However, the films of both powders and liquid epoxies have excessive numbers of holidays (more than 10) when their film thicknesses are in the range of 1-4 mils.

#### 4.4 Physical Testing of Coatings on Reinforcing Bars

The abilities of coatings on reinforcing bars to withstand rough handling were assessed on the basis of bend tests, impact tests, and hardness measurements. These tests were carried out either on the same specimens listed in table 8 or on companion specimens.

#### 4.4.1 Bend Tests

No. 6 bars coated by applicators were bent at a  $120^{\circ}$  angle with a radius of curvature of ca. 3 in. using a Green Lee Tool Company Model 770 Bar Bender. Portions of the bars coming in contact with the bending machine were protected with rubber tubing of 1 1/2 in. o.d. and 3/4 i.d. to avoid mechanical damage to the coating, so that any cracking in a coating occurring during the bend test could be attributed to strain failure of the coating. The tests were performed at  $72 \pm 1^{\circ}F$ .

Cracking and disbonding took place on the area of some bars that was under tension during the bending (table 9). The four different polyvinyl chloride coated rebars (Nos. 23, 24, 26, and 30) gave excellent performances even though their film thicknesses ranged from 2 to 35 mils. A greater variation was observed for the epoxy coated rebars as some performed well while a few were classified as failing. Generally, the epoxy coatings which did not perform well were either the most brittle epoxies or their cured film thicknesses were over 10 mils. The effect of the film thickness is well illustrated by comparing the coated reinforcing bars Nos. 22 and 31, which were both coated with the same material applied by different application methods. No. 22 has a film thickness of ca. 25 mils (applied by the fluidized bed technique) while the film thickness of No. 31

<sup>4/</sup> Holidays are defined as pinholes normally not visually discernible.

TABLE 7. ABRASION AND IMPACT RESISTANCE OF CURED EPOXY COATINGS ON STEEL PANELS 1/

		IMPACT RES	ISTANCE <sup>2/</sup>	ABRASION RESISTANCE $\frac{3}{}$	
Code Number	Film Thickness (mils)	Reverse Impact (in.1b.)	Direct Impact (in.1b.)	Weight loss per 1000 cycles (mg)	
1	7	20	90	71	
2	7	4	1204/	56	
3	8	40	160	107	
4	5	4	40	70	
5	7	8	20	58	
6	6	4	20	71	
11	6			89	
16	5	8	50	148	
17	6	7	50	58	
18	7	12	110	52	
19	1	>160	>160	51	
	4	160	160		
25	7	4	60	70	
28	8	40	60	88	
29	8	50	60	57	
31	6	20	80	35	
38	7	20	80		
39	8	20	40	90	
40	11	20	5/		
41	7			85	

1/ Epoxy coatings applied to 4 x 4 x .050 inch steel plates (cold rolled).

2/ Four pound hammer dropped from increasing heights until coating was ruptured.

3/ Taber CS-10 wheels, with 1000 g load per wheel.

4/ Bond at the steel-epoxy interface severed at 10 in-1b.

5/ Coating shattered off of steel panel.

Code Number	Application Method	Film Thickness (mils)	No. of Holidays per 4 foot bar	Visual Inspection and Comments
1	Brush	4-5	40	Deformation not well coated and slightly exposed. Accumulation of epoxy in low lying regions.
2	Brush	5-15	ca. 10	Deformations not well covered - long uncovered regions. Material is brittle.
3	Brush	2-5	ca. 30-40	Deformations appear to be well covered.
4	Brush	10-20	none	Deformation not well defined as the epoxy is concentrated in the low lying region between the deformations.
5	Brush	10-15	too many to estimate	Longitudinal deformations are not well covered. Excess epoxy in regions between the deformations.
10	Brush	10	ca. 10	Susceptible to abrasion; coating easily comes off of rebars. Rough texture. Epoxy accumulated between deformations.
11	Brush	10-12	none	Coating easily chips off of rebar. Regions where defor- mations are not well covered. Evidence of epoxy dripping from bar.
16	Electro- static spray gun	2-4	40	Deformations not well covered; accumulation of epoxy in low lying areas.
17	Brush	4	too many to estimate	Tops of deformations not well covered. Brittle material that easily chips off steel substrate.
18	Brush	4	too many to estimate	Bad adhesion to steel. Abrades easily off of rebar.
19	Single dipping	1	40	Good coverage; well defined deformation pattern. No bad dipping regions observed.
22	Fluidized bed	25	none except at ends	Uniform built-up film. Large thickness characteristic of fluidized bed application.
23	Fluidized bed	25	none except at ends	Uniform build-up film. Thermoplastic coating.
24	Fluidized bed	35	none	Deformation pattern is hidden. Thermoplastic.

# table 8. Inspection of coated steel reinforcing ${\tt bars}^{\underline{1}/}$

24

# TABLE 8. (continued)

Code Number	Application Method	Film Thickness (mils)	No. of Holidays per 4 foot bar	Visual Inspection and Comments
25	Electro- static spray gun	6-11	none	Good coating material. Some bad spraying techniques; bars were sprayed from one direction giving the underside of the deformations a thin coating.
26	Electro- static spray gun	2-3	too many to estimate	Top of deformations not covered. When received, coated rebars were sticky. Thermoplastic
27	Electro- static spray gun	8	ca. l	High gloss coating. Even build-up. Very tough coating that did not chip off.
28	Electro- static spray gun	1-2	cannot estimate	Either very high number of holidays or electrical conducting pigments. Rough texture.
29	Electro- static spray gun	1-2	cannot estimate	Either very high number of holidays or electrical conducting pigment. Deformations do not appear to be well coated. Tough coating that is not susceptible to chipping.
30	Fluidized bed	15-18	none	Thermoplastic coating. Deformation pattern hidden, possibly due to tendency of material to flow when cured at elevated temperatures.
31	Electro- static spray gun	8-9	none	Good even built-up film. Deformations well covered. Tough coating that is not susceptible to chipping.
32	Electro- static spray gun	4-6	40	Mill scale was not removed and coating chips came off easily due to disbonding between the mill scale and the steel rebars.
33	Brush	3-4	too many to estimate	Coating easily damaged and chips off of rebar.
38	Electro- static spray gun	2-4	ca. 30-40	Deformations not well covered. Some bars were phosphatized.
39	Electro- static spray gun	2-4	ca. 30-40	Holidays located on deformations. Some bars were phosphatized.
40	Electro- static spray gun	2-4	10	Tough coating that is not susceptible to chipping; some bars were phosphatized.

TABLE 8. (c	continued)
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Code Number	Application Method	Film Thickness (mils)	No. of Holidays per 4 foot bar	Visual Inspection and Comments
41	Electro- static spray gun	3-7	1	Good coverage. Bar heated prior to application of powder coating.
42	Electro- static spray gun	3-4	too many to estimate	Same material as No. 41, but powder applied to cold rebars.
43	Electro- static spray gun	3-4	too many to estimate	Material readily abrades off of rebar.

1/ Number 6 steel reinforcement bars coated by applicators or coating producers. Unless otherwise stated, the mill scale was removed by sandblasting.

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# table 9. results of bending coated reinforcing $\text{bars}^{\underline{1}/}$

Code Number	Film Thickness (mils)	Results of $120^{\circ}$ bend <sup>2/</sup>
1	4 <b>-</b> 5	Slight cracking near edge of deformation, length of cracks were ca. 1/8 inch.
2	5-15	Complete failure in bend area. Almost complete disbonding.
3	2-5	Few small cracks ca. 1/8 inch long. Good performance.
4	20-30	Severe cracking at almost every transverse deformation in bend area. Lengths of cracks were 1/2 to 3/4 inch.
5	40-50	Severe cracking at deformations. Cracks were ca. 1/8 inch wide and undercutting disbondment between the films and steel took place.
10	10	Severe cracking which extended from longitudinal deformation. Disbonding between the coating and steel was observed.
11	10-12	Same as No. 10.
16	2-4	Very fine cracks, good performance.
17	4	Cracking started at 20° bend. Total disbondment in area under tension. Complete failure.
18	4	No cracking, excellent performance.
19	4	No cracks, excellent performance.
22	25	Substantial cracking extending from longitudinal to longitudinal deformation, some disbonding between the coating and steel was observed.
23	25	No cracks, excellent performance.
24	35	No cracks, excellent performance.
25	6-11	Many small (ca. 1/8 inch long) thin cracks, considered as moderate cracking.
26	2-3	No cracks, excellent performance.
27	8	Substantial cracking extending from longitudinal to longitudinal deformation. Some disbonding was observed.
28	1-2	<ul> <li>A. Slight cracking, good performance.</li> <li>B. Substantial cracking and disbonding observed<u>3</u>/.</li> </ul>
29	3-4	No cracks, excellent performance.
30	15-18	No cracks, excellent performance.
31	8-9	No cracks, excellent performance.
32 <u>3</u> /	4-6	Complete failure as total disbonding in bend area; probably attributable to disbonding between mill scale and steel.
33	3-4	No cracks, excellent performance.

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TABLE	9.	(Continued)
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Code	Film	2/
Number	Thickness (mils)	Results of 120° bend <sup>27</sup>
$38 \frac{A\frac{4}{4}}{B^{\frac{4}{2}}}$	2-4 2-4	No cracks, excellent performance. Severe cracking on every deformation in area under tension during bending.
$39 \frac{A^4}{B^4}$	2-4 2-4	No cracks, excellent performance. Two or three small cracks. Good performance.
40 $A^{4/}$	2-4	Excellent performance; no cracking.
40 в <sup><u>4</u>/</sup>	2-4	Failure. Phosphate coating adhered poorly to the steel substrate. Cracking of epoxy coating in complete area under tension during bending.
41 <u>5/</u>	3-7	Excellent performance, no cracking
42 <sup>6</sup> /	3-4	Substantial cracking.
43	3-4	Slight "popping off" of coating. Good performance.

- 1/ Number 6 steel reinforcement bars coated by applicators or coating producers. Unless otherwise stated the mill scale was removed by sandblasting.
- <u>2</u>/ Crack rating in order of decreasing performance: Excellent>Good>Moderate>Substantial> Severe>Complete failure.
- 3/ Mill scale was not removed.
- <u>4</u>/ Surface of A sandblasted prior to application of coating material. Surface of B sandblasted and phosphatized prior to coating application.
- 5/ Bars heated to 190°C prior to applying powder coating.
- $\underline{6}$  / Same coating material as 41, but applied to cold reinforcing bars.

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was ca. 8-9 mils (applied using an electrostatic spray gun). When bent, substantial cracking was observed in the film of No. 22, while No. 31 was completely free of cracking.

Another factor affecting the bending characteristics of coated reinforcing bars is the type of surface preparation of the substrate prior to application of the coatings. In two series of coated reinforcing bars, No. 28B and No. 32, epoxy coatings were applied to unprepared surfaces which were still covered with mill scale. Almost total disbondment was observed when each series of bars was bent; while the epoxy adhered tenaciously to the mill scale, the mill scale was disbonded from the steel substrate. A portion of the coated rebars Nos. 38, 39 and 40 were both sand blasted and phosphatized prior to being coated while the remainder were just sand blasted. The sand blasted coated rebars gave no indications of coating failures when bent while the phosphatized bars were susceptible to varying amounts of failure in the coatings.

The temperature of the steel substrate, when being coated, can affect the flexibility of the cured epoxy coating. For example, Nos. 41 and 42 rebars were coated with the same material. However, No. 41 rebars were heated to  $342^{\circ}F$  and immediately coated while No. 42 rebars were at ambient temperature when coated. The epoxy coating was then cured at  $319^{\circ}F$ on both sets of rebars. Excellent flexibility was exhibited by No. 41, whereas No. 42 cracked badly when bent.

#### 4.4.2 Impact Tests

The resistances of coatings on reinforcing bars to impact were determined by dropping bars on concrete and by the falling weight method.

### 4.4.2.1 Dropping Coated Bars on Concrete

An 18 in. length of coated No. 6 reinforcing bar was dropped on a slab of concrete so that impact occurred lengthwise as follows:

- 1. A single bar was dropped one meter from a horizontal position to the concrete.
- 2. The same bar was dropped from a height of two meters.
- 3. A companion specimen was taped loosely between two bare No. 7 bars of the same length and the assembly was dropped from a height of 2 meters to the concrete slab.
- 4. The bars were inspected after each drop for the following types of damages:
  - A. Shattering of the coating to expose bare metal.
  - B. Cutting of the coating to expose bare metal.
  - C. Cracking of the coating.
  - D. Disbonding of the coating from the steel substrate.

The coatings were rated on a relative basis and the results are given in table 10.

### 4.4.2.2 Falling Weight Method

The test methods outlined in ASTM Designation G14-69T [31] were followed. A Gardner Laboratory impact tester was used along with a four pound hammer. Impact occurred on the low-lying areas between the deformations.

The type and extent of damage to the coating caused by an impact of 120 in-1b was visually assessed and also the area of damage was measured (table 10). When compared with other tests in this study, it is felt that with an impact of 120 in-1b the area of damage should not exceed 0.15 in<sup>2</sup> for an acceptable coating.

## TABLE 10. DROP RESISTANCE<sup>1/</sup> AND IMPACT RESISTANCE<sup>2/</sup> OF COATINGS ON REBARS

			IMPACT RESISTANCE	
Code Number1/	Film Thịckness (mils)	Damaged Area (in. <sup>2</sup> )	Type and Severity of Damage	Drop Resistance <u>3</u> /
1			Not tested.	Good
2	5-15	0.110	Shattering and disbonding of coating propagating from area of impact.	Poor
3	2-5	.028	Only indentation in coating and rebar at impact area.	Good
4	10-20	.082	Shattering and disbonding of coating propagating from area of impact.	Poor
5	10-15	.383	Large amount of shattering and disbonding of coating surrounding area of impact.	Poor
10	10	.079	Shattering and disbonding of coating ' at impact area.	Poor
11	10-12	0.188	Shattering and disbonding of coating propagating from area of impact.	Poor
16	2-4	.038	Slight shattering and disbonding of coating at impact area.	Fair
17	4	.028	Slight shattering and disbonding of coating at impact area.	Fair
18	4	.038	Slight shattering and disbonding of coating at impact area.	Fair
19	1	.028	Only indentation in coating and rebar at impact area.	Fair
22	25	0.234	Large amount of shattering and disbo disbonding of coating surrounding area of impact.	Excellent
23	25	.077	Large indentation in coating.	Excellent
24	35	.110	Large indentation in coating.	Excellent
25	6-11	.049	Shattering and disbonding of coating at impact area.	Good
27	8	.077	Coating shattered at area of impact with slight propagating of shattering from impact region.	Good
28	1-2	0.038	Slight shattering and disbonding of coating of impact area.	Fair
29	1-2	.028	Slight shattering and disbonding of coating at impact area.	Good
30	15-18	.110	Large indentation in coating accompanied by slight cracking at impact area.	Poor
31	8-9	.110	Shattering and some disbonding of coating at impact area.	Excellent
32	4-6	.049	Cracking in coating at impact area, slight cracking extending from impact region.	Fair

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TABLE 10. (Continued)

Code Number1/	Film Thickness (mils)	Damăged Area (in. <sup>2</sup> )	Type and Severity of Damage	Drop Resistance <sup>3/</sup>
33	3-4	.028	Shattering of coating at impact area.	Good
38	2-4	0.038	Shattering of coating at impact area, slight cracking extending from impact region.	Excellent
39	2-4	.028	Only indentation in coating and rebar at impact area.	Excellent
40	2-4	.079	Shattering of coating at impact area, slight disbonding extending from impact region.	Excellent
41	3-7	.038	Shattering of coating at impact area, slight cracking extending from impact area.	Good
42	3-4	.028	Only indentation in coating and rebar at impact area.	Good
43	3-4	.038	Smashing of coating at impact area, slight cracking extending from impact region.	Good

### IMPACT RESISTANCE

 $\underline{1}/$  Coated reinforcing bars dropped from heights of 1 and 2 meters on concrete.

 $\underline{2}$ / Falling weight method ASTM Designation G-14 with impact of 120 in-1b.

3/ Relative rating: Excellent>Good>Fair>Poor.

The criteria of 0.15 in<sup>2</sup> permissible area of damage was only exceeded in three cases, namely, by coatings Nos. 5, 11, and 22. Both No. 5 and No. 11 were previously classified as brittle materials on the basis of their performance in the bend test. The poor performance of No. 22 probably can be attributed to its large film thickness, ca 25 mils; the same material with a film thickness of 8-9 mils, No. 31, had acceptable impact resistance.

There is a fairly direct correlation between the results of the drop and impact tests with a few exceptions such as coating No. 22. Although the results of the impact test are easier to quantitatively evaluate, the drop test more closely simulates the rough handling coated reinforcing bars will probably experience.

### 4.4.3 Hardness Determination

The hardness value of a coating gives an indication of the relative resistance of the coating to the type of mechanical damage which results in scratching, cutting, indentation, etc. of the film. The hardness of coatings on reinforcing bars was determined by the pencil method and the indentation method.

### 4.4.3.1 Pencil Hardness

Pencil hardness values were determined using a series of lead pencils that covers the range of H to 8H, with steps of one hardness increment. The hardness is designated as the softest lead that imparts a scratch in the coating. All of the epoxy coatings had ratings above 8H; while the polyvinyl chloride coatings were softer, with hardness values of H for polyvinyl chloride coatings Nos. 24 and 30, and a value of 8H for coating No. 23.

### 4.4.3.2 Indentation Method

The microhardnesses of coatings on steel reinforcing bars were also measured by the indentation method to determine the Knoop Hardness number, which is more quantitative and reproducible than the pencil hardness. An apparatus of the type described in ASTM Designation D1474-68 [33], following the methods outlined therein, with a 10 gram load, was used for this determination.

The Knoop Hardness Number (KHN) is calculated with the equation:

$$KHN = \frac{L}{Ap} = \frac{L}{1^2Cp}$$

where L is the load applied to the indenter in kilograms; l is the measured length of the long diagonal of the indentation in the coating in millimeters; Cp is a constant with the value of  $7.028 \times 10^{-2}$ ; Ap is the projected area of the indentation.

The results for five coatings on rebars are given in table 11. No. 30 is a polyvinyl chloride and has a relatively low hardness of 6.7 KHN, while the other four coatings are powder epoxies having hardnesses above 18 KHN. As previously discussed, No. 22 and No. 31 are rebars coated with the same epoxy material applied by different methods which yielded different film thickness. The film thickness of No. 22 is ca. 25 mils and the film thickness of No. 31 is ca. 8 mils. The microhardness was determined to be 20.7 KHN for both coating films, therefore, it seems that the microhardness of the coating film alone was being measured and not the composite hardness of the coating and the steel substrate.

### 4.5 Electrochemical Tests

Electrochemical tests were undertaken to quantitatively rate the relative performance of coatings exposed to solutions corrosive to steel embedded in concrete.

#### 4.5.1 Applied Voltage Studies

The effects of electrochemical stresses on coatings on reinforcing bars were assessed by applied voltage studies. Such stresses can be induced in the field by cathodic protection

(1)

### Table 11. Indentation Hardness of Coatings on Reinforcing Bars

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Code Number	· · · · · · · · · · · · · · · · · · ·	Hardness <u>KHN1</u> /
22		20.7
29	· · · ·	19.8
30		6.7
31		20.7
39		21.2

1/ Knoop Hardness Number

devices, stray currents, or by corrosion processes. The cathode and anode were No. 6 reinforcing bars, 6 inches long, both coated with the same material. The electrolyte was an aqueous solution of 7 percent NaCl. A potential of two volts was applied and the electrodes were visually observed periodically for evolution of hydrogen gas at the cathode and for evidence of corrosion products of iron at the anode. Before immersion, any bare ends or obvious mechanically damaged areas on the electrodes were covered with a film of silicone rubber, and no intentional holidays were induced. The sources of any corrosion, therefore, were holidays in the films. The applied voltage method also serves as a sensitive holiday detector and can be used to ascertain whether holidays are developing in a film because of degradation of the coating. A typical experimental set-up is shown in figure 6A. In this photograph, taken at the beginning of the test, the bars are coated with material No. 16. After 30 minutes of application of 2 volts copious amounts of corrosion products were observed (figure 6B).

The results of these voltage studies are presented in table 12. Altogether 31 coatings were investigated and 19 of these permitted the evolution of hydrogen gas within 15 minutes. No holidays developed in three of the polyvinyl chloride coated specimens, Nos. 23, 24, and 30, nor in two of the epoxy coated specimens, Nos. 22 and 31, during a test period of over 90 hours.

### 4.5.2 Electrical Potential and Electrical Resistance

Alternate means of assessing the protective qualities of barrier coatings are electrical potential measurements and the electrical resistance of the coating films. Such measurements were made on both coated reinforcing bars partially immersed in 3.5 percent aqueous solutions of NaCl and on coated bars embedded in concrete specimens partially immersed in 3.5 percent NaCl solution. The electrode potentials were measured using a Coleman Model 37A pH meter with a saturated calomel electrode (S.C.E.) as the reference electrode, as illustrated in figure 7. Measurements of electrical resistances were taken using a Yellow Springs Instrument Company Model 31 Conductivity Bridge along with a platinum electrode, figure 8.

### 4.5.2.1 Coated Reinforcing Bars in 3.5 Percent Solution of Sodium Chloride

The coated bars were partially immersed in aqueous solutions of 3.5 percent NaCl in 3 liter polyethylene buckets fitted with lids. Two holes were cut in each lid, one for the reinforcing bar, the other for the reference electrode.

The electrical potential and electrical resistance data are presented in table 12. Low resistance values, below 500 ohms, are indicative of coating films which either have many holidays or are permeable to water and/or chloride ions. The measured corrosion potential of uncoated steel reinforcing bars was -634 mv vs S.C.E. after 1000 hours. All of the specimens had electrical potentials below these values, even though numerous corrosion sites were visually observed on all of the specimens which had resistances below 500 ohms. The electrical resistance values appear to be more reliable indicators of the corrosion state of the coated reinforcing bars than the electrical potential values.

The thicker films (above 15 mils), Nos. 22, 23, 24, and 30, were free of holidays and had resistances above  $24 \times 10^5$  ohms, which are beyond the range of the measuring device. These films were in effect perfect insulators preventing the flow of current, therefore, the electrical potential of the protected bars could not be measured.

Potential and resistance data for three sets of reinforcing bars, each set coated with a different epoxy (Nos. 1, 25 and 31) are plotted versus test time in figures 9, 10 and 11 (initial plotted values measured after 24 hours of immersion of coated bars). The wide variance in the initial millivolt and ohmic readings of duplicate and triplicate specimens decreased rapidly during the first 200 hours of testing and after 1000 hours good agreements were obtained for companion specimens. A rapid decrease in the resistance of a coating probably can be attributed to the emergence of holidays, while an increase in resistance is probably indicative of some type of healing mechanism.

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FIGURE 6.A. Applied voltage test at beginning of test. Anode and cathode coated with material No. 16.



FIGURE 6.B. Results of application of 2 volts for 30 minutes.



FIGURE 7. Measurement of electrical potentials of coated reinforcing bars immersed in 3.5 percent solution of NaCl.



FIGURE 7. Measurement of electrical potentials of coated reinforcing bars immersed in 3.5 percent solution of NaCl.

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Code	Time to 1,	Resistance	$(ohms)^{2/2}$	Volt	age (millivol	$(ts)^{2/2}$
Number	Evolve H <sub>2</sub> (g) <sup>1/</sup> at Cathode (hours)	1 hour	1000 hours	1 hour	1000 hours	Ratings 7
1	<1/4	537	200	-340	-570	3
2	<1/4	250	190	-588	-576	3
3	<1/4	400	180	-615	-604	3
4	<1/2	700	260	-470	-555	3
5	<1/4	$1.1 \times 10^3$	425	-513	-617	3
10	<1/4					3
11	<1/4	9.6 x $10^3$	800	-497	-560	3
16	1	400	250	-516	-593	2
17	<1/4	$1.5 \times 10^3$	700	-430	-545	3
18	<1/4	98	240	-516	-604	3
19	24	435	225	-503	-568	2
22	<648 <u>4</u> /	25 x 10 <sup>5</sup> <u>4</u> /	$25 \times 10^5 \frac{4}{-100}$	N. R. <u>5</u> /	N. R. <u>5</u> /	1
23	<1204/	25 x 10 <sup>5</sup> <u>4</u> /	$25 \times 10^5 \frac{4}{4}$	N. R. <u>5</u> /	N. R. <u>5</u> /	1
24	<480 <u>4</u> /	$13 \times 10^5 \frac{4}{4}$	$25 \times 10^5 \frac{4}{}$	N. R. <u>5</u> /	N. R. <u>5</u> /	1
25	6	$2 \times 10^{5}$	$25 \times 10^5 \frac{4}{4}$	-613	-541	1
26	<1/4					3
27	<1/4					3
28	<1/4	250	240	-640	-606	3
29	<1/4	475	300	-518	-565	2
30	>168 <u>4</u> /	$25 \times 10^5 \frac{4}{}$	$25 \times 10^5 \frac{4}{4}$	N. R. <u>5</u> /	n. r. <u>5</u> /	1
31	> 964/	$25 \times 10^5 \frac{4}{}$	1500	-532	-588	1
32	<1/4	800	540	-617	-573	3
33	<1/4	550	400	-516	-565	3
38 в <u>-</u> /	<1/4	360 <u>7</u> /	210 <u>8</u> /	-514-7/	-589 <sup>8/</sup>	3
38 p <sup>6/</sup>	<1/4	380 <u>7</u> /	220 <u>8</u> /	-481 <u>7</u> /	-606 <u>8</u> /	3
39 B <sup>6</sup> /	<1/4	380 <u>7</u> /	240 <u>8</u> /	-557 <sup>7/</sup>	-6108/	2
$39 p^{6/}$	3	4107/	230 <u>8</u> /	-557 <sup>7/</sup>	-643 <sup>8/</sup>	2

### TABLE 12. ELECTROCHEMICAL STUDIES OF COATED REINFORCING BARS

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### TABLE 12. (continued)

Code	Time to 1/	Resistance	$(ohms)^{2/2}$	Vol	tage (millivol	$(ts)^{\frac{2}{2}}$
Number	Evolve H <sub>2</sub> (g) <sup>1</sup> at Cathode (hours)	1 hour	1000 hours	1 hour	1000 hours	Ratings"
40 B <sup>6</sup> /	<1/4	2807/	200 <u>8</u> /	-513 <sup>7/</sup>	-608 <u>8</u> /	3
40 p <sup>6/</sup>	1/2	290 <u>7</u> /	240 <u>8</u> /	-481 <u>7</u> /	-606 <sup>8/</sup>	2
41	1/2	$3.8 \times 10^5$	$3.0 \times 10^3$	-451	-570	2
42 <u>9</u> /	<1/4	400	370	-526	-579	3
Uncoated bar	immediate	200	370	-648	-634	4

1/ Potential of 2 volts was applied to coated bar. Bars partially immersed in 7 percent NaCl.

2/ Bars partially immersed in 3 1/2 percent NaC1. Listed data are average values.

3/ Ratings sequence in order of decreasing corrosion protection: 1>2>3>4.

4/ Resistance values beyond capacity of measuring device.

5/ N. R. denotes no reading possible, i.e. no current flow because of holiday-free film.

6/ B represents sandblasted surfaces, only, while p indicates that the surfaces were phosphatized before applying the coatings.

7/ Initial measurement taken after immersion time of 120 hours.

8/ Measured after 696 hours.

9/ Same coating material as No. 41, however, applied to cold bars: coating applied to bars heated to 190°C in the case of No. 4.



FIGURE 8. Measurement of electrical resistance of coatings on reinforcing bars immersed in 3.5 percent solution of NaCl.



FIGURE 9. Electrical potential and resistance measurements of reinforcing bar coated with material No. 1 immersed in 3 1/2 percent NaCl. In the ordinate caption, M.V. vs. S.C.E. denotes millivolt reading taken using a saturated calomel electrode as the reference.



FIGURE 10. Electrical potential and resistance measurements of reinforcing bar coated with material No. 25 immersed in 3 1/2 percent NaCl. In the ordinate caption, M.V. vs. S.C.E. denotes millivolt reading taken using a saturated calomel electrode as the reference.



FIGURE 11. Electrical potential and resistance measurements of reinforcing bar coated with material No. 31 immersed in 3 1/2 percent NaCl. In the ordinate caption, M.V. vs. S.C.E. denotes millivolt reading taken using a saturated calomel electrode as the reference.

### 4.5.2.2 Coated Reinforcing Bars Embedded in Concrete

The corrosion-test specimens, figure 12, consisting of reinforcing bars embedded in 2 7/8 x 4 7/8 x 15 inch concrete blocks were partially immersed in an aqueous solution of 3.5 percent NaCl. The solution was contained in a 105 gallon polyethylene-lined, fiberglass reinforced, polyester tank,  $4 \times 2 \times 2$  ft. The bars were in a vertical position with their lower ends, protected by silicone sealant, fitted into 1 inch diameter holes drilled in 1 ft x 1 ft spacing racks of 3/4 in. thick marine plywood. The racks rested on a sheet of marine plywood laid on the floor of the tank, and spacing racks were placed on top of the specimens (figure 13). The lower 13 inches of each specimen was immersed in the NaCl solution, with the upper 2 inches of the concrete being above the level of the solution.

The electrical potentials and electrical resistances of the corrosion-test specimens are listed in table 13. The results are in general agreement with the protective ratings given in table 12. No evidence of cracking in the concrete cover nor of rust stains were observed.

In Section 4.5.2.1 it was noted that electrical potential measurements do not seem to accurately indicate the corrosion state of the coated reinforcing bars. The uncoated bars have potentials of -180 and -207 mv after 3480 hours (table 13), which are considered to be in the passive region [34], while many of the coated bars have much more active potentials.

Resistance measurements are probably more reliable indicators than potential measurements, since the resistance values are primarily dependent on the integrity of the coating films. The resistance of a film will sharply decrease if holidays develop or decrease more slowly if the film is gradually overall deteriorating. The resistance of the protective layer of water-soaked concrete is low [35], certainly much lower than the resistance of a good protective coating on a bar. The corrosion-test specimens with the uncoated bars had the lowest resistance values measured, 220 and 230 ohms; while the specimens with bars protected with coating No. 30, assigned a protective rating of 1, gave the highest resistance values of  $2.1 \times 10^5$  and  $1.6 \times 10^5$  ohms. However, it is difficult to understand why many of the bars embedded in concrete had lower measured resistances than the unembedded bars (Section 4.5.2.1).

#### 5. BOND STRENGTH AND CREEP DETERMINATIONS

An important phase of the project was the determination of the bond between coated reinforcing bars and concrete and of the creep characteristics of coated bars in concrete. Probably, the main reason that little consideration was previously given to epoxy materials as protective coatings for reinforcing bars was the supposition that the coated reinforcing bars would have unacceptable bond strengths to concrete [22]. Few, if any, reports have been published of any type of structural testing performed on epoxy coated bars embedded in concrete. The bond with coated bars should not be significantly less than that between uncoated bars and concrete if coated bars are to be used in established bridge deck design. The structural characteristics of coated bars in concrete have been compared with the properties of uncoated bars by pullout tests and creep tests.

### 5.1 Pullout Studies

The pullout tests are tests in which increasingly higher loads are applied in equal increments to the reinforcing bar until either the bar yields or the bond strength between the reinforcing bar and concrete is greatly exceeded (estimated by measuring the slip of the reinforcing bar relative to the concrete prism).

Altogether 34 pullout specimens were tested consisting of 5 specimens with uncoated reinforcing bars, 23 specimens with epoxy-coated bars and 6 specimens with polyvinyl chloride-coated bars.



FIGURE 12. Corrosion-test specimen with coated reinforcing bar embedded in concrete block of dimensions 2 7/8 x 4 7/8 x 15 inches.



FIGURE 13. Corrosion-test specimens immersed in 3.5 percent solution of NaCl.

### Table 13. Electrical Potential and Resistance Measurements of Corrosion-Test Specimens in Aqueous Solution of 3.5 Percent NaCl

### Exposure Time (Hours)

6

		24		34		
Coating		Electrical Potential <u>2</u> /	Resistance	Electrical Potential <u>2</u> /	Resistance	Protective
Code No.	· <u>1</u> /	(MV)	(ohms)	(MV)	(Ohms)	Rating 2/
1	A B	-345.0 -408.8	$3.8 \times 10^2$ 7.0 x 10 <sup>2</sup>	-283.0 -362.4	$3.9 \times 10^2$ 8.2 x 10 <sup>2</sup>	3
1-1		-337.0	$2.5 \times 10^2$	-215.0	$2.5 \times 10^2$	-
1 <b>-</b> S		-484.5	$4.8 \times 10^2$	-371.5	$4.2 \times 10^2$	-
3	A B	-285.6 -260.3	$3.1 \times 10^2$ 2.7 x 10 <sup>2</sup>	-432.4 -365.5	$2.2 \times 10^2$ 2.4 x 10 <sup>2</sup>	3
4	A B	-339.2 -130.0	$2.4 \times 10^{4}$ 1.0 x 10 <sup>5</sup>	-142.3 -115.5	$1.1 \times 10^{5}$ $1.4 \times 10^{4}$	3
18		-575.6	$6.0 \times 10^{3}$	-003.0 4/	$1.0 \times 10^4$	3
19	A B	-484.0 -438.0	$5.6 \times 10^2$ $6.1 \times 10^2$	-399.5 -282.0	$5.4 \times 10^2$ $6.0 \times 10^2$	2
25		-542.7	$4.1 \times 10^2$	-271.4 <u>4</u> /	$5.1 \times 10^2$	1
27	A B	-654.6 -571.5	$1.3 \times 10^4$ 6.8 x 10 <sup>3</sup>	-167.0 <u>4</u> / -542.0	$7.2 \times 10^4_4$ $1.1 \times 10^4$	3
28		-461.5	5.2 x $10^2$	-262.8 4/	5.4 x $10^2$	3
29	A B	-376.3 -403.4	$6.4 \times 10^2$ $6.6 \times 10^2$	$-163.0 \frac{4}{-360.5}$	$7.8 \times 10^2$ 5.4 x 10 <sup>2</sup>	2
30	A B	-058.0 -448.2	$1.0 \times 10^{5}$ $1.5 \times 10^{5}$	$-127.4 \frac{5}{4}$	$2.1 \times 10^{5}$ $1.6 \times 10^{5}$	1
31	A B	-359.8 -092.2	$1.5 \times 10^{3}$ 9.8 x 10 <sup>3</sup>	-038.5 <u>4</u> / -013.5	$9.8 \times 10^4$ $6.2 \times 10^4$	1
38		-392.7	$3.2 \times 10^2$	-165.7	$4.1 \times 10^2$	3
39 <b>-</b> Phos	A B	-513.0 -536.2	$4.9 \times 10^2$ 5.0 x 10 <sup>2</sup>	-348.0 -402.0	$4.7 \times 10^2$ $4.8 \times 10^2$	3
40-Phos 40-Phos	A B	-282.2 -382.5	$2.5 \times 10^2$ 3.4 x 10 <sup>2</sup>	-256.6 -325.5	$2.2 \times 10^2$ 2.7 x 10 <sup>2</sup>	2
40	A B	-431.8 -377.0	$2.9 \times 10^2$ $2.8 \times 10^2$	-398.0 -316.9	$3.1 \times 10^2$ 2.3 x 10 <sup>2</sup>	3
41	A B	-540.5 -575.9	$6.0 \times 10^3$ 5.4 x 10 <sup>2</sup>	-432.2 -324.4	$1.3 \times 10^4$ 2.5 x 10^4	2

		2	.4	34		
Coating Code No.	<u>1</u> /	Electrical Potential <u>2</u> / Resistance (MV) (ohms)		Electrical Potential <u>2</u> / (MV)	Resistance Protect (ohms) Rating	
Uncoated	A B	-334.2 -264.0	$2.7 \times 10^2$ 2.6 x 10 <sup>2</sup>	-206.6 -180.3	$2.3 \times 10^2$ 2.2 x 10 <sup>2</sup>	4

 $\underline{1}$  / A and B denote duplicate specimens.

2/ Electrical potential vs. S.C.E.

3/ Ratings from Table 12.

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<u>4</u>/ Large shifts in electrical potential attributed to sealing small holes in the a silicone seal.

5/ Not possible to measure.

### 5.1.1 Pullout Test Procedures

Pullout specimens were tested in a 200,000 lb. capacity universal electromechanical testing machine 27 to 29 days after fabrication. A pullout specimen positioned on the testing machine is shown in figure 14. The pullout specimen shown in figure 15 is seated on leather cushions on two segments of a 2 in. base plate attached to a spherical bearing block. Free- and loaded-end slips of the reinforcing bar were measured with  $1 \times 10^{-4}$  in. micrometer dial gages and estimated to  $1 \times 10^{-5}$  in. At the loaded end of the specimen, two dial gages were attached to a steel bar fastened to the face of the concrete by bolts secured into inserts cast in the concrete. The gages bore on a steel yoke fastened to the reinforcing bar about 1 in. below the face of the concrete. The bar supporting the dial gages measurements gave the displacement of the point on the reinforcing bar where the yoke was attached, with reference to the face of the concrete. Slip at the free end was measured with a gage that bore on the exposed end of the reinforcing bar (any coating material on the exposed end of the reinforcing bar was removed prior to testing). The gage was mounted on a support attached to the top face of the concrete by bolts secured into inserts cast in the concrete.

Loads were applied in increments of 2,000 pounds to the reinforcing bars in the pullout tests until failure occurred either by yielding of the steel or excessive slip between the bar and concrete.

### 5.1.2 Results of Pullout Studies

The relationships between applied load and the free-end and loaded-end slip are plotted in figure 16 for the 34 pullout specimens tested. Roman numerals denote the concrete batch number while the Arabic numbers next to the plots identify the coating materials (table 1). The loaded-end slip was larger than the free-end slip for all specimens tested primarily because slipping initiates at the loaded-end and extends toward the free-end as the load is increased or loaded.

Bond failure in a reinforced concrete member is defined as excessive slip, or movement, of the free or loaded end of a bar stressed in tension caused by only a slight increase in the applied load [28]. Therefore, the large slips shown in figure 16, occurring at about 28000 pounds, are indicative of bond failures.

The mode of failure, critical bond strengths, and critical bond stresses are given in table 14. The critical bond strengths and critical bond stresses are defined as the values corresponding to either a loaded-end slip of 0.01 in. or a free-end slip of 0.002 in., whichever is lower [28].

The critical bond strengths corresponded to applied loads ranging from 17,000 to 21,600 lb. for uncoated bars and for coated bars, except those coated with materials Nos. 22, 23, 24, and 30. The average applied loads corresponding to the critical bond strengths for bars coated with the latter materials were 9,000, 1,100, 60 and 5,700 lb. respectively. Note that the mode of failure was yielding of the reinforcement for all pullout specimens except those containing bars with coatings Nos. 22, 23, 24, and 30.

Bond stresses were computed from the formula [28]

$$u = \frac{f_s A_s}{\sum_{c} L}$$

where f is the stress in the reinforcing bar, A is the nominal cross sectional area of the bar,  $\Sigma_{\circ}$  is the nominal perimeter of the bar and L is the length of embedment of the reinforcing bar in the pullout specimen. Values of A and  $\Sigma_{\circ}$  for each of the two types of rebars are given in table 2. The value of f is given by

$$f_s = \frac{P}{A_s}$$

where P is the load or tensile force applied to the reinforcing bar in pounds. Therefore, equation (2) can be reduced to

 $u = \frac{P}{\Sigma_0 L}$ 

which was used to calculate u from the pullout tests.

Values of bond stress developed in the pullout specimens were compared with allowable values given in codes and specifications. The American Concrete Institute Building Code 318-63 [36] allowed a working bond stress design for deformed bars (other than top bars) conforming to ASTM A 305 [37] calculated from, but not greater than, 500 psi

where f ' is the strength of the concrete and D is the nominal diameter of the bar in inches. Using the value of f ' as 6170 psi (average of the strength of the three batches of concrete used in the pullout studies) the bond stress, u, is 490 or about 500 psi.

The Standard Specification for Highway Bridges Adopted by the American Association of State Highway Officials [38] states that slabs (decks) designed for bending moment in accordance with the given provisions shall be considered satisfactory in bond and shear. In another section of this Standard Specification on concrete design, the allowable bond stress for tension bars conforming to AASHO M31 [38] and ASTM A615-72 [25] is

$$4.8\sqrt{f_c}$$
, 500 psi maximum

and is the same as that given by the ACI 318-63 Code [36].

The critical bond stresses and bond stresses corresponding to one half the maximum applied load,  $U_m/2$ , were greater than 600 psi (table 14) for all pullout specimens except those having bars coated with materials Nos. 22, 23, 24, and 30.

An evaluation of the pullout tests results indicates that epoxy-coated reinforcing bars have bond strengths essentially equal to uncoated bars when the film thicknesses are approximately 10 mils or less. Both liquid and powder epoxies performed equally well, and the application method did not significantly affect the bond strength of coated bars. The polyvinyl chloride coated bars had bond strengths considerably less than that for uncoated bars and bars with these coatings are not recommended for structural use. The

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FIGURE 14. Pullout specimen on electromechanical testing machine being prepared for testing.



FIGURE 15. Schematic of pullout specimen. Size of concrete prism is 10 x 10 x 12 inches.



FIGURE 16. Applied load to reinforcing bar in pullout specimens versus free-end and loaded-end slip. Roman numbers indicate concrete batch number, while Arabic numbers identify the coating materials (table 1).

Pullout	Load Corresponding	Maximum Slip	Bond Stress C	orresponding to	u	Mode of Failure
No.	to Critical Bond Strength P <sub>cr</sub> (1b.)	critical Bond     Observed at       trength     Free End       P     (1b.)       cr     (in.)	Loaded-End Slip of 0.01 in. u <sub>1</sub> (psi) 1	Free-End Slip of 0.002 in. u <sub>2</sub> (psi)	 (psi)	
U-B	20,300	.006	723	978	712	Yielding of reinforcement; no cracks.
U-B	18,000	.007	641	889	629	Yielding of reinforcement; Small longitudinal crack extending one third length of one face. Small trans- verse crack at loaded end.
U-B	20,000	.006	712	1157	729	Yielding of reinforcement. Small longitudinal crack extending one half length of specimen on one face.
U-D	21,600	.006	764	1037	628	Yielding of reinforcement; no cracks.
U-D	21,400	.002	755	<u>1</u> /	664	Yielding of reinforcement; no cracks.
1-В	21,200	.003	751	1185	727	Yielding of reinforcement; no cracks.
1-D	18,000	.003	638	1060	645	Yielding of reinforcement. Small longitudinal crack extending one half length of specimen on two opposite faces.
1-B-S	17,100	.01	609	925	727	Yielding of reinforcement; no cracks.
3-B	20,000	.006	712	1210 ~	727	Yielding of reinforcement; no cracks.
3-D	21,000	.002	745	1199	646	Yielding of reinforcement. Small longitudinal crack extending one sixth of length of specimen on one face.

### TABLE 14. PULLOUT DATA

Pullout	Load Corresponding	Maximum Slip	Bond Stress C	orresponding to	u m	Mode of Failure
No.	to Critical Bond Strength P (1b.)	Observed at Free End (in.)	Loaded-End Slip of 0.01 in. u <sub>1</sub> (psi)	Free-End Slip of 0.002 in. u <sub>2</sub> (psi)	(psi)	
18-в	21,500	.002	766	1352	675	Yielding of reinforcement; no cracks.
18-D	18,800	.003	656	1197	673	Yielding of reinforcement; no cracks.
19-B	19,000	.004	677	1089	727	Yielding of reinforcement. Small longitudinal crack extending one sixth length of specimen on two faces.
19-D	21,400	.003	759	1277	726	Yielding of reinforcement; no cracks.
22-B-1	11,600	.01	445	413	497	Bond failure. Small trans- verse crack extending one half length of loaded end.
22-в-2	6,500	.007	363	231		Test stopped before concrete cracked.
23-в	700	<u>4</u> /	107	25	447	Bond failure. Specimen badly cracked.
23-D	1,400	.03	167	50	395	Bond failure. Specimen badly cracked.
24-в	100	.05	18	5	133	Bond failure. Excessive free-end slip.
24-D	30	.06	18	1	165	Bond failure. Excessive free-end slip.
25-D-1	18,500	.003	656	1050	638	Yielding of reinforcement. Small longitudinal crack extending one half of length of specimen on two opposite faces.

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TABLE 14. (continued)

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Pullout No.	Load Corresponding to Critical Bond Strength P (1b.) cr	Maximum Slip Observed at Free End (in.)	Bond Stress C Loaded-End Slip of 0.01 in. u <sub>1</sub> (psi)	orresponding to Free-End Slip of 0.002 in. u <sub>2</sub> (psi)	um 2 (psi)	Mode of Failure -
25-D-2	17,800	.005	631	922	628	Yielding of reinforcement. Small longitudinal crack extending one half of length of specimen on two opposite faces.
29-в-1	17,000	. 004	605	979	673	Yielding of reinforcement. Small longitudinal crack extending one third of length of specimen on two opposite faces.
29-в-2	18,200	. 004	648	1033	638	Yielding of reinforcement. Small longitudinal crack extending one third of length of specimen on two opposite faces.
30-в	6,000	.03	410	214	605	Bond failure. Small longi- tudinal crack extending entire length of specimen on two opposite faces.
30-D	5,400	.02	348	191	569	Bond failure. Numerous small longitudinal cracks on all faces.
31-D	19,500	.012	670	1056	675	Yielding of reinforcement. Small longitudinal cracks extending one third of length of specimen on two opposite faces.
31-B	18,700	.006	646	956	674	Yielding of reinforcement. Small longitudinal cracks extending one third of length of specimen on two

TABLE 14. (continued)

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opposite faces.

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Pullout No.	Load Corresponding to Critical Bond Strength P <sub>cr</sub> (1b.)	Maximum Slip Observed at Free End (in.)	Bond Stress C Loaded-End Slip of 0.01 in. u <sub>1</sub> (psi)	orresponding to Free-End Slip of 0.002 in. u <sub>2</sub> (psi)	<u>"m</u> (psi)	Mode of Failure
38-в	19,700	.008	702	1129	766	Yielding of reinforcement; no cracks.
38-D-Ph	21,500	.003	762	1032	726	Yielding of reinforcement. Small longitudinal crack extending one third length of specimen on two opposite faces.
39-d	20,000	.004	709	1177	638	Yielding of reinforcement. Small longitudinal crack extending one sixth of specimen on two opposite faces.
39-в	17,500	.004	623	1122	628	Yielding of reinforcement. Small longitudinal crack extending one sixth of specimen on two opposite faces.
41-D	18,500	.004	656	1046	675	Yielding of reinforcement. Small longitudinal crack extending entire length of two opposite faces.
41-B	17,000	.004	605	1068	673	Yielding of reinforcement; no cracks.

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TABLE 14. (continued)

 $\underline{1}/$  Unreliable data due to sticking gage.

2/ Failure took place before free-end slip of 0.005 in. was reached.

 $\underline{3}/$  Test stopped at  $\boldsymbol{f}_{s}$  of 36,300 with free-end slip of 0.007.

 $\underline{4}$  Not recorded, greater than .02 inch.

lower bond strengths for polyvinyl coated bars are attributed in part to the thermoplastic nature of the polyvinyl chloride. The thickness of the polyvinyl chloride films was greater than most of the epoxy films but thicker films are normal for thermoplastics [39].

#### 5.2 Creep Studies

In contrast to the pullout tests, the creep tests were performed with two specific stress levels in the reinforcing bars. Twenty-four specimens were studied which consisted of 18 reinforcing bars coated in duplicate with 9 different epoxy materials; 2 reinforcing bars coated with a polyvinyl chloride material; and 4 uncoated reinforcing bars. The two levels of tensile stresses were 15,000 and 30,000 psi (in the steel reinforcing bars). These stress levels were selected because the lower value represents the stress to which rebars in bridge decks are normally subjected and the higher value represents a stress which may be included in future bridge deck designs using high strength steel.

### 5.2.1 Creep Test Procedures

A creep specimen with the loading assembly attached is illustrated in figure 17. Tensile stresses of either 15,000 or 30,000 psi (in the steel reinforcing bar) were attained by compressing the spring with a 30 ton hydraulic ram. The stress level in the reinforcing bar was monitored with both the load cell and the strain gages attached to the reinforcing bar. When the desired tensile level was reached, the upper nut on the threaded reinforcing bar was firmly tightened against the steel bearing plate holding the spring in a compressed position. Subsequently, the lower nut was released and the hydraulic ram, load cell and spacer assembly were removed. Releasing the loading apparatus caused a negligible decrease in the tensile stress in the reinforcing bar. Shown in figure 18 is a creep specimen under test at a tensile stress of 30,000 psi.

The creep specimen in figure 18 was seated on leather cushions on two segments of the test frame (figure 19). The dial gages were of the same type and attached in the same way as described for the pullout specimens. The free-end and loaded-end slip were also measured.

The dimensions of the steel spring used to exert the tensile loads in the reinforcing bar were: height of 8 inches; outside diameter of 5 1/4 inches; and the steel coils had a diameter of 1 3/16 inches. The springs were calibrated (load vs displacement) using the device shown in figure 20. The compressive displacement of the springs was measured with  $1 \times 10^{-4}$  in. micrometer dial gages. The load was exerted on the springs with a 60,000 lb. capacity hydraulic universal testing machine. Loads were in the range of 0 to 14,000 lb. (14 kips) and dial gage readings of spring displacement were taken at intervals of 1 kip between 0 and 5 kips and 8 to 12 kips, while between 5 to 8 and 12 to 14 kips the intervals were 0.5 kips. Amounts of displacement were determined from the average values of the two dial gages located diametrically opposite on the calibration device. The 24 springs had nearly the same displacement response to loading as indicated by the plot in figure 21, which gives the range in displacement for corresponding load application.

The strain gages, to monitor the tensile stresses, were attached to the reinforcing bars approximately 3 inches from the concrete prism with an epoxy adhesive. The gages were covered with a protective coating of wax followed by a coating of an epoxy material. Two strain gages, electrically connected in series, were attached diametrically opposite on each bar. These gages when attached as recommended by the manufacturer are claimed by the manufacturer to have only a small intrinsic creep of 10  $\mu\epsilon$  (microstrain units)/year. The strain values were measured with a Vishay Instruments Strain Indicator Model P 350 A. Strain measurements were converted to stress values in psi using a calibration diagram obtained from tensile tests of reinforcing bars instrumented the same as the bar in the creep specimens.

The tensile stresses in the reinforcing bar were also monitored periodically by comparing the heights of the compressed springs with the heights of the springs immediately after the application of load. These data were compared to the dial gage readings which indicate the slip of the reinforcing bar relative to the concrete prism. Based on the



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Specimen Loading Assembly

FIGURE 17. Schematic of creep specimen. Size of specimen is the same as the pullout specimen.



FIGURE 18. Creep specimen loaded to a tensile stress level of 30,000 psi (in the steel reinforcing bar).



## Test Frame

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FIGURE 19. Schematic of creep test frame.


## SPRING CALIBRATION APPARATUS

FIGURE 20. Apparatus for calibrating steel springs used to exert tensile stresses in the creep study.



FIGURE 21. Range of displacement response of 24 steel springs used in the creep study.

calibration of the displacement response of springs to loading, any increase in the height of the compressed springs could be directly related to the amount of loss of tensile stress.

After 45 days of testing, the average relaxation in tensile stress was 205 psi and 701 psi for specimens having tensile stress of 15,000 psi and 30,000 psi, respectively.

#### 5.2.2 Results of Creep Studies

The creep tests simulate more closely the structural rigors an acceptable coating must endure over a long period of time, than do the pullout studies. In the creep test, tensile stresses were maintained whereas in the pullout test increasing increments of load were applied until failure occurred. Creep properties of reinforcing bars embedded in concrete have not been well characterized. The performance of coated bars in the creep tests has been assessed, therefore, by comparing their slip-time relationships with those of uncoated bars. It is the opinion of the authors that the slip-time relationship for coated bars should not vary significantly from the slip-time relationship measured for uncoated bars for normally expected steel stresses. Furthermore, there should be no significant increase in the magnitude of either free-end or loaded-end slip of the coated bar as compared to the uncoated bar. These criteria will be more quantitatively developed later in this section.

#### 5.2.2.1 Slip-Time Relationships

Both the free and loaded-end slip of coated and uncoated bars, at tensile stresses of 15,000 and 30,000 in the bars, are plotted versus time in figure 22. Rates of slip (analogous to creep) usually were highest during the first two days after loading the specimens, and thereafter, the rates gradually decreased. However, even after 45 days, measurable slip was still detected for all specimens. Similar to the behavior of the pullout specimens, the loaded-end slip-time relationships were significantly larger than the free-end slip relationships for all creep specimens, with the exception of the bars coated with material No. 30, primarily because slipping initiates at the loaded-end and slip propagation towards the free-end slip time curves, at tensile stresses of both 15,000 and 30,000 psi, with the bars coated with material No. 30 (a polyvinyl chloride coating) were essentially identical to the respective loaded-end slip-time curves; therefore, these coated bars were not interlocking in the concrete and probably would not have acceptable reinforcing properties if embedded in concrete.

A comparison of the slip-time curves in figure 22 indicates that with the possible exceptions of materials Nos. 1 and 18, the epoxy coatings did not have a detrimental effect on the magnitude of the slip-time relationships developed with uncoated bars. In contrast, obviously the bars coated with the polyvinyl chloride material, No. 30, developed unacceptable slip-time relationships.

#### 5.2.2.2 Slip Values at 45 Days

Free- and loaded-end slip data, at 45 days, of coated and uncoated bars are listed in table 15. The slip data obtained at the tensile stress level of 30,000 psi will be emphasized. The respective slip values for both the coated and uncoated bars attained at the tensile stress of 15,000 psi were about 10 to 50 percent of the values obtained at 30,000 psi stress level. Furthermore, the same conclusions are derived by analyzing either set of data.

Three creep specimens with uncoated bars were tested at the 30,000 psi stress level and average loaded-end slip was .00164 inch and the average free-end slip was .00077 inch, at 45 days. The agreement between the three loaded-end slip values and also the three free-end values was excellent for this type of experiment. The range and percent coefficient of variation for the loaded-end data was 0.00022 inch and 4.0 percent, and .00013 inch and 7.1 percent for the free-end slip data. Because the variation in concrete strength was minor, no compensating adjustments were made in the slip values.

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FIGURE 22. Free-end and loaded-end slips in creep specimens versus time. Roman numbers indicate concrete batch number, while Arabic numbers identify the coating materials (table 1).

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#### Table 15. Creep of Coated and Uncoated Bars Embedded in Concrete Prisms at 45 Days

Coating No.	Compressive Strength of Concrete	Steel Stress (psi)	Slip at Loaded End (inch)	Slip at Free End (inch)	$\frac{\text{Slip}}{\text{Ratio}} = \frac{\text{Coa}}{\text{Und}}$	<u>ated Bar 1</u> / coated Bar
					Loaded End	Free End
1	5665	15,000	.00144	.00079	2.1	2.2
1	5665	30,000	.00260	.00150	1.6	2.0
18	5494	15,000	<u>2</u> /	.00063		1.8
18	5494	30,000	.00149	.00118	0.90	1.5
19	5665	15,000	.00053	.00008	0.77	0.22
19	5665	30,000	.00114	.00063	0.70	0.82
25	5494	15,000	.00080	.00031	1.2	0.86
25	5494	30,000	.00150	.00085	0.91	1.1
29	5494	15,000	.00079	.00007	1.1	0.19
29	5494	30,000	<u>2</u> /	.00103		1.3
30	5494	15,000	.00434	.00384	6.3	10.7
30	5494	30,000	.01215	.01229	7.4	16.0
31	5665	15,000	.00059	.00008	0.86	0.22
31	<b>5</b> 665	30,000	.00134	.00026	0.82	0.34
38	5665	15,000	.00069	.00017	1.0	0.47
38	5665	30,000	.00168	.00080	1.0	1.0
39	5665	15,000	.00080	.00017	1.2	0.47
39	5665	30,000	.00158	.00097	0.96	0.99
41	5494	15,000	.00110	.00008	1.6	0.22
41	5494	30,000	.00212	.00100	1.3	1.3
U.C.	5494	15,000	.00069	.00036		
U.C.	5494	30,000	.00176	.00071		
U.C1	5665	30,000	.00163	.00084		
U.C2	5665	30,000	.00154	.00076		

1/ Slip for uncoated bars at tensile stress of 30,000 psi is average of 3 specimens, i.e. slip of .00164 inch at loaded-end and .00077 inch at free-end.

2/ Malfunction of dial gage.

The slip at the 30,000 psi stress level for the nine specimens with epoxy coated bars ranged from .00114 (No. 19) to .00260 (No. 1) for the loaded-end and from .00026 (No. 31) to .00079 inch (No. 1) for the free-end. Excessive slips of .0122 inch for the loaded-end and .0123 for the free-end developed for the specimen with the polyvinyl chloride coated bar (material No. 30). The slip of the coated bars are compared with the average slip of the uncoated bars by computing the following slip ratio

(7)

# slip ratio = $\frac{\text{slip of coated bar}}{\text{average slip of uncoated bar}}$

for both loaded and free-end slip. These respective ratios are listed under columns 6 and 7 in table 15. At the 30,000 psi stress level, the ratios for the loaded-end slip ranged from 0.70 to 2.1 for epoxy coated bars and was 7.4 for the polyvinyl chloride coated bar. The similar ratios for the free-end slip of epoxy coated bars varied from 0.34 to 2.2, and was 16.0 for the polyvinyl chloride coated bar.

Unequivocal interpretation of the significance of the ratios of slip of coated bar to average slip of uncoated bar is difficult, because criteria for allowable creep of uncoated reinforcing bars, subjected to tensile stresses, have not been established. Obviously, the high ratios of 7.4 and 16.0 for the polyvinyl chloride coated bar (material No. 30), should preclude its use as protective coating for concrete reinforcement. Probably, all the bars coated with epoxy materials, with the possible exceptions of Nos. 1 and 18, had acceptable slip ratios. Values of the slip ratios for the bar coated with No. 1 was about 2.0 for both the free and loaded-end slip. The slip ratio of 2.0 for the loaded-end is about twofold greater than the ratio with most other epoxy coated bars, and the slip ratio for the free-end is about 2 to 6 times greater than the similar ratio for the epoxy coated bars (except for No. 18). Therefore, it is felt that the bar coated with material No. 1 had undesirable creep characteristics. Similarly, analysis of the slip ratio for the bar with coating No. 18 indicates it had acceptable loaded-end creep but possibly unacceptable free-end creep (considering slip ratios at both 15,000 and 30,000 psi). It is felt that a reasonable criterion would require that bars coated with an approved coating material should have both acceptable loaded-end and free-end creep characteristics, when subjected to tensile stresses near the level it would actually experience if used as the reinforcement in concrete.

Further studies are necessary to determine the values of acceptable slip ratios expressed by equation 7. Based on the results of the current creep study, the following values are proposed as being reasonable: maximum slip ratio of 1.6 for the loaded-end; and maximum slip ratio of 1.3 for the free-end.

#### 6. DISCUSSION

Altogether, 47 coating materials (table 1) were evaluated and four coatings have been judged, on the basis of results in the testing program, to have overall acceptable properties as potential coatings for the reinforcing bars of concrete of bridge decks. These four materials are Nos. 25, 31, 39, and 41, all powder epoxy coatings. In the following section, the pertinent experimental results which lead to the selection of the four coating materials, will be briefly discussed.

#### 6.1 Evaluation of Coating Materials

The evaluation of coating materials as protective coating for steel reinforcing bars embedded in concrete of bridge decks was based on the following four general test categories:

- 1. Chemical resistance of cured coatings.
- 2. Physical durabilities of coatings on reinforcing bars.
- 3. Corrosion protection of reinforcing bars by coatings.
- 4. Structural characteristics of coated reinforcing bars in concrete.

Established tests were selected and when necessary new tests developed so that the probable performance of coatings on bars embedded in concrete of bridge decks could be evaluated.

The implications of the results of these tests will be discussed in this section. The major emphasis in the present study has been given to epoxy coatings because of anticipated unacceptable structural characteristics of reinforcing bars coated with thermoplastics, which has been subsequently experimentally confirmed (Section 6.1.4).

#### 6.1.1 Chemical Resistance of Coating Materials

Chemical resistance studies were implemented to make projected evaluations of the long-term durability of coatings when in concrete. The resistance of coatings to aggressive aqueous solutions similar to those in portland cement concrete was assessed by immersion studies with specimens of both pure coatings (table 4) and coating on bars (table 5). The weight changes of cured specimens of liquid epoxies (table 4) are in the range reported by other investigators [40]. It is felt that with the exception of the three solvent-containing epoxy systems, Nos. 7, 8, and 9, the liquid epoxy systems performed satisfactorily and probably will not be degraded by long-term embedment in concrete.

The immersion of coated bars in aqueous solutions of 3.5 percent (0.5M) NaCl was an excellent discriminatory test. Specimens in this test included bars coated with both the powder epoxies and most of the liquid epoxies, and with polyvinyl chloride materials. Seven coatings consisting of 4 powder epoxy and three polyvinyl chloride materials, had ratings of 1 and 2 indicating they had sufficient chemical resistances to adequately protect the reinforcing bars from corrosion. The long-term durability of polyvinyl chloride in concrete, however, is still regarded by the authors to be of major concern, for if hydrolysis should take place sufficient amounts of chloride ions could be liberated to cause corrosion of the bars.

The performance of a coating on bars in the immersion tests is not entirely governed by the chemical resistance of the coating materials but it is also dependent on the film integrity of the coating. Some coatings had poor ratings (below 2) because of poor application techniques by applicators, inadequate film thicknesses (below 5 mils) and uneven film coverage which left the top of the deformation either thinly coated or bare.

Phosphatizing the surface of the metal substrates has been considered advantageous to inhibiting corrosion [36]. In the present study, however, the coated bars with phosphatized steel surfaces, Nos. 38-Phosp, 39-Phosp and 40-Phosp, were rusted when immersed in saturated  $Ca(OH)_2$  and 3.5 percent NaCl, to a greater extent than the companion coated bars with blasted surfaces. Furthermore, the epoxy coatings over the phosphatized surfaces softened while the coatings over the blasted surfaces were still hard after a year of immersion.

#### 6.1.2 Physical Durabilities of Coatings

Reinforcing bars are normally subjected to harsh physical treatment while being shipped to the site of bridge construction and during the placement process. Furthermore, steel reinforcing bars are still being bent to form hooks, in accordance with the specifications of some state highway departments. The ability of coatings on bars to withstand a reasonable amount of rough treatment with minimum damage, therefore, it is a necessary prerequisite.

The relative physical durabilities of coatings were assessed by measuring the impact and abrasion resistances of coatings on steel plates (table 7), bending coated reinforcing bars (table 9), impact tests on coated bars (table 10), and hardness measurements of coatings (table 11), with the bending test probably being the most important physical test.

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The performance of a coated bar in the bend test gives significant information concerning the flexibility of a coating; proper cure of the coating; surface preparation of the steel; and film thickness. Coatings with little flexibility will crack when subjected to tensile forces caused by bending. Polyvinyl chlorides are inherently flexible materials and performed well, even with film thicknesses up to 35 mils. Although epoxies are intrinsically more brittle than polyvinyl chlorides, the relative performance of epoxy films ranged from complete failure to excellent. Interestingly, the flexibilities do not appear to be directly related to the type of epoxy system, i.e. powder or liquid. Flexibilities of epoxy coatings will often be decreased by improper cure caused by such factors as mixing incorrect ratios of resin to hardener or by curing powder epoxies at improper temperatures. The flexibilities of epoxy coatings decrease inversely with their film thickness. Based on the present study, it is recommended that the maximum allowable film thickness should be determined for each epoxy coating and consistent with good flexibilities and structural properties (Section 6.1.4) should not exceed an average thickness of 10 mils.

#### 6.1.3 Corrosion Protective Qualities of Coatings

The relative effectiveness of barrier-type organic coatings in protecting steel reinforcing bars from accelerated corrosion attributed to chloride ions can be associated with the following: physical and chemical durabilities of the coatings (discussed in the previous sections); intrinsic chloride ion permeabilities; film integrity and film thickness; formulation of the coating, including corrosion inhibitors [41].

The present study confirms the results of others [40] that epoxies absorb measurable amounts of water and, therefore, thin epoxy films, about 2-10 mils, are not entirely impervious to moisture. Chloride ion permeability rates, however, may be much lower than those of pure water. Little if any data on the rates of migration of chloride ions through epoxy films have been previously reported. The results of the present study do indicate that many thin epoxy films are essentially impervious to chloride ions (at least during the test time of this study).

The film integrity of coatings on reinforcing bars is an important consideration because holidays are potential sites of corrosion. In general, the coatings on bars with few or no holidays (table 8) had acceptable protective ratings of 1 or 2 (table 12). Holidays can be produced by solvent evaporation, poor flow characteristics of coatings, mechanical damage, and inadequate film coverage. Note in table 8 that films of all the solventcontaining systems had significant amounts of holidays (over 10 per 4 foot bar), regardless of the application method. Liquid epoxies have the tendency to flow-off the higher portions of the deformations, before hardening, thereby accumulating in the lower lying regions and resulting in an inadequate thin film over the deformations. In almost every corrosion study, coating failures were first observed to occur on the deformations. The large number of holidays in some powder epoxy films can possibly be attributed to either poor coating practices or to low film thicknesses. Holiday-free films can be obtained by thick film buildups, however, the maximum permissible film thickness must be consistent with good structural and flexibility requirements.

The powder epoxy coatings, when properly applied to a film thickness of greater than 4 mils, usually adequately protected reinforcing bars from corrosion caused by chloride ions.

#### 6.1.4 Bond Strengths and Creep Characteristics of Coated Bars in Concrete

An important aspect of this study was the determination that reinforcing bars coated with certain epoxy materials had both adequate bond strengths and satisfactory creep rates when embedded in concrete.

The bond strengths of coated bars embedded in concrete were measured by pullout tests and compared to the values obtained with uncoated bars. The applied load corresponding to the critical bond strength of pullout specimens with bars having epoxy coatings 1 to 11 mils thick ranged from 17,000 to 21,500 lb with an average value of 19,000 lb, equivalent to an average bond stress of 677 psi. Those average values are about six percent less than the respective averages of 20,300 lb and 720 psi for pullout specimens with uncoated bars, and are believed to be in the acceptable range. In contrast, bars coated with polyvinyl chloride materials and epoxy coating with a film thickness above 15 mils, were judged to have developed unacceptable bond strengths.

The creep characteristics of coated bars were evaluated by comparing their slip-time relationships (figure 22) and free-end and loaded-end slip values at 45 days (table 15) with those of uncoated bars. In general, the epoxy coated bars which had adequate bond strength, also had acceptable creep properties (Section 5.2). However, bars coated with epoxy materials Nos. 1 and 18 had unacceptable slip ratios, although these bars performed well in the pullout test. Apparently, the creep test is more discriminating than the pullout test. The poor creep characteristics of the bar coated with material No. 18 is easily rationalized: No. 18 is an epoxy-coal tar mixture and coal tar materials are susceptible to high creep rates, therefore, the epoxy-coal tar mixture should have larger creep rates than the more pure epoxy coatings. The high creep of the bars coated with material No. 1 is not easily understood.

Based on the results of both the pullout test and creep test, it is felt that the rebars coated with epoxy materials Nos. 19, 25, 29, 31, 38, 39 and 41 can be incorporated into existing bridge designs without any compromise in the structural integrity of the bridge.

The polyvinyl chloride materials which were part of this study should not be used to protect reinforcing bars embedded in concrete because of unacceptable bond strengths and creep characteristics. Of particular interest is the coal tar epoxy which has the highest pullout strength recorded, but with very poor creep results. Based on this phenomenon, it is recommended that epoxy coatings in which are incorporrated modifiers (such as coal tar, polysulfides, etc) should be tested in creep in the prequalification test as described in the last section of table 16.

#### 6.2 Proposed Qualification Criteria for Coating Materials

Probably, the determination that four epoxy materials had sufficient attributes to merit their selection as coatings for bars to be used in experimental construction was fortuitous, because none of the 47 coating materials evaluated in this study were purposely formulated to serve as protective coatings for steel reinforcing bars. It is anticipated, however, that if the experimental bridge decks constructed with epoxy-coated reinforcement perform well, uniquely-formulated coatings will become available. Based on the results of the evaluation program, proposed minimum performance levels are listed in table 16 which can serve as a basis for the development of prequalification specifications for organic coatings.

#### 6.3 Implementation of Epoxy-Coated Reinforcing Bars

Powder epoxy coatings have performed sufficiently well in this relatively short-term study, to warrant their implementation in experimental bridge construction. The success of epoxy coatings in protecting the bars from corrosion will be governed by the application, fabrication, and installation processes. Crucial aspects of the application process include: proper substrate preparation prior to coating; correct powder application, resulting in a cured film about 7  $\pm$  2 mils thick and essentially free from holidays; and proper thermal treatment leading to well-cured, flexible epoxy films. In their fabrication, reinforcing bars are bent to specific shapes and cut to prescribed lengths. The present fabrication techniques for uncoated bars will certainly cause some damage to the epoxy coatings. The extent of such damage can probably be reduced by using bearing rollers, and bending wheels and anvils covered with pliable materials such as nylon. An alternate, and preferred method, might be to coat prefabricated reinforcing bars. Presently, reinforcings are subjected to harsh treatment in their shipping and installation. Although epoxy coatings on bars can withstand a moderate level of abuse, present handling methods should be modified, such as bundling coated bars together with nylon rope and protecting them from rough treatment at the construction site. Extensively damaged areas should be repaired with an approved material (such as a liquid epoxy) after being placed in the forms just prior to casting the concrete.

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#### TABLE 16. PROPOSED CRITERIA FOR QUALIFYING COATING MATERIALS

Test Method	Test Specimen	Section Describing Test	Test Period <sup>1/</sup> Test Condition	Acceptable Performance Level
Chemical Resistance	Coated Bar	4.1.2	45 days	The coating must not blister, soften, disbond nor develop holidays.
Chloride Permeability	3 mil thick film	4.1.3	45 days	Accumulative concentration of chloride ion permeating through film shall be less than 1 x $10^{-4}$ .
Abrasion Resistance	Coated Steel Plate	4.2	CS-10 wheels and 1000g load per wheel	Weight loss shall not exceed 100 mg per 1000 cycles.
Bend Test	Coated No. 6 Bar	4.4.1	Bars bent 120° over 3 inch mandrel	No visible crack in coating.
Impact Test (Falling Weight Method)	Coated No. 6 Bar	4.4.2.2	Impact of 80 in-1b	Area of damage should not exceed 0.15 in. <sup>2</sup> .
Hardness Determin- ation	Coated Bar	4.4.3.2	10 gram load	Hardness shall equal or be greater than 16 KHN.
Applied Voltage Test	Coated Bar	4.5.1	Maximum of 31 days	No evolution of H <sub>2</sub> at c <b>a</b> thode or rusting at anode within one hour. No undercutting during test.
Pullout Test	Coated and Uncoated No. 6 Bar	5.1	Concrete age of 27-29 days	Mean critical bond strengths for coated bars should be no less than 80 percent of the strengths for uncoated bars.
Creep Test	Coated and Uncoated No. 6 Bar	5.2	45 days at tensile stress of 30,000 psi	Average slip ratios for free-end creep should not exceed 1.3 and the ratio for loaded-end creep should not exceed 1.6 (Ratio of slip of coated versus uncoated bar).

 $\underline{1}/$  Exact test conditions are given in the section describing the test methods.

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The corrosion state of uncoated reinforcing bars in concrete of existing bridge decks is currently determined by taking electrical potential measurements [34]. As previously discussed in section 4.5.2, electrical potential measurements were not found to be reliable indicators of the corrosion state of coated bars.

Wolstenholme [42] has discussed the difficulties of interpreting electrical potential measurements and concluded that in general electrochemical tests have not been informative. Therefore, the use of epoxy-coated reinforcing bars will probably necessitate the development of other electrochemical tests to monitor the corrosion condition of the reinforcing steel. Suggested methods are electrical resistance measurements [43] and electrical polarization measurements [43-45].

#### 7. CONCLUSIONS AND RECOMMENDATIONS

#### 7.1 Conclusions and Recommendations

The following conclusions and recommendations are based upon the results of the experimental investigation described in this report.

- 1. The powder epoxies, in general, have better overall properties as barrier coatings (considering chemical resistance, chloride permeability and corrosion protective qualities) than the liquid epoxies; within the liquid epoxy series the solvent-free materials performed better than the solvent-containing systems.
- Epoxy films on reinforcing bars can withstand a moderate amount of abuse. Some modifications appear to be necessary in the current fabrication, shipping, and installation practices to prevent damage to the coating.
- 3. For any epoxy coating to perform well, good application techniques are important. Epoxy coatings should be applied to blasted steel surfaces as both phosphatized and mill scale surfaces are brittle and modest mechanical forces can cause disbondment. The electrostatic spray gun method is the most effective application method in producing thin films free of defects. Proper curing of the epoxy film is important as undercured materials are very brittle and susceptible to mechanical damage.
- 4. Some epoxy coating materials have sufficient flexibilities in cured film thicknesses below about 10 mils that coated bars can be bent to the normal shapes prescribed in most existing bridge designs, with minimal damage to the coating.
- 5. All of the epoxy coated bars, with film thicknesses not greater than 11 mils, tested in the pullout studies had acceptable bond to concrete. Nine epoxy coatings on bars were tested in the creep studies and seven were judged to have acceptable creep characteristics. Therefore, it is felt that selected epoxy coated bars can be used in existing bridge designs without compromising the structural integrity of the bridge. The polyvinyl chloride coated bars tested in this study have unacceptable bond and creep characteristics and, therefore, should not be used in reinforced concrete. Although the coal tar epoxies had excellent pullout properties their poor creep characteristic makes them unacceptable.
- 6. Considering flexibility, bond strength and creep characteristics, and minimum corrosion protective requirements, the optimum film thickness of epoxy films on steel reinforcing bars is about 7  $\pm$  2 mils.

7. Four powder epoxies, Nos. 25, 31, 39 and 40 are judged to have the best overall properties as potential coating materials for steel reinforcing bars. It is recommended that these coatings be further evaluated in experimental bridge decks constructed using bars coated with these materials.

#### 7.2 Recommended Further Studies

The following studies are recommended to complete certain aspects of this study:

- 1. Although the information on relative bond strengths of coated and uncoated reinforcing bars determined by means of pullout tests are believed valid by the investigators, it is recommended that tests of flexural members (slabs) be carried out to confirm these results.
- 2. Further creep studies of flexural members (slabs) should be performed with coated and uncoated bars to determine acceptable slip ratio for both loaded-end and free-end creep. Different size of bars and concrete test specimens should be included in further studies.
- 3. Electrical resistance and linear polarization measurements should be investigated as methods to monitor the condition of epoxy-coated reinforcing bars in service in bridge decks. Electrical potential measurements were not found to be reliable indicators of the corrosion state of coated bars in the present study.

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#### APPENDIX A

### FIRMS SUBMITTING COATING MATERIALS FOR EVALUATION $\frac{1}{2}$

Adhesive Engineering Company

Carboline

Celanese Coatings Company

CIBA-GEIGY Corporation Resins Department

E. I. du Pont de Nemours Company, Inc.

H. B. Fuller Company

General Mills Chemicals, Inc.

Hercules Incorporated

Michigan Chrome & Chemical Company

Minnesota Mining and Manufacturing Company

Mobil Chemical Company

NORDSON Corporation

Polymer Corporation

H. C. Price

Products Research and Chemical Corporation

Republic Steel Corporation

Robroy Industries

Rowe Products, Inc.

Royston Laboratories

SCM Corporation Gates Engineering Division

Shell Chemical Company

SIKA Corporation

United States Steel Corporation

Whittaker Corporation Narmco Materials Division

Wailes Bitumastic Ltd.

Witco Chemical Corporation

 $\underline{1}$ / These firms submitted coatings materials which they handled for evaluation. They are not necessarily the manufactureres of the coating materials.

#### APPENDIX B

#### CONVERSION FACTORS OF U. S. UNITS TO SI UNITS

In view of the accepted practice in the United States at present, the units in this report are those commonly used in the technological field for which the report is intended. In recognition of the position of the U.S.A. as a signatory to the General Conference on Weights and Measures and the action of the U. S. Congress, readers interested in using the metric (SI) units may use the conversions below, excerpted from Standard Metric Practice Guide, E380-72 (a guide to the use of SI - the international system of units), published by the ASTM, 1916 Race Street, Philadelphia, Pa.

To Convert From

degree Fahrenheit (°F)	To degree celsius (°C)	$\frac{\text{Multiply by}}{t_{c}} = \frac{t_{F} - 32}{1.8}$
<pre>inch (in.) gallon (gal.) inch<sup>2</sup> (in.<sup>2</sup>) kip (1000 lbf) kip/in.<sup>2</sup> (ksi)</pre>	metre (m) metre <sup>3</sup> (m <sup>3</sup> ) metre <sup>2</sup> (m <sup>2</sup> ) newton (N) pascal (Pa)	$2.540,000 \times 10^{-2}$ $3.785,412 \times 10_{-4}$ $6.451,600 \times 10_{-3}$ $4.448,222 \times 10_{-3}$ $6.894,757 \times 10^{-6}$
pound-mass (lb.m. avordipois) pound-force/inch <sup>2</sup> (psi)	kilogram (Kg) pascal (Pa)	$4.535,924 \times 10^{-1}$ $6.894,757 \times 10^{3}$

\* Exact Conversion Factor

GPO 881-087

