A novel interpenetrating polymer network coating for the protection of steel reinforcement in concrete

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Abstract

A cost-effective better performing epoxy/phenolic interpenetrating polymer network (IPN) coating for the protection of steel reinforcement in concrete exposed to aggressive environment (marine and industrial) has been developed. Physicomechanical properties along with chemical resistance against some acids, alkalies, fertilizers, and water have been determined. To assess the corrosion protection efficacy from end use application, point of view chemical resistance, adhesion by bend test, bond strength by pull-out test method, and accelerated corrosion cycling test by weight change method have been performed. On the basis of the data, it may be concluded that the IPN-coated reinforcing rebars have acceptable bond strength with concrete, and have better corrosion resistance than other commercially available treatment used for similar applications. The economics of the treatment is quite attractive since treatment costs about 15 to 20% of the cost of steel. Hence it may be said that IPN-coated reinforcing steel bars fulfill the minimum requirements laid down in various standard specifications. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Interpenetrating; Reinforcement; Corrosion; Polymer; Accelerated

1. Introduction

From the beginning of twentieth century, reinforced concrete has become one of the most widely used material of construction due to its inherent properties, especially its strength to sustain imposed loading conditions. It is believed that reinforced concrete structures are durable and maintenance-free for the whole of its design life, approximately more than 60 years [1–3]. However, the corrosion of reinforcing steel in concrete exposed to aggressive environment affects the life of the concrete and thus has rapidly become a serious problem throughout the world. Parking structures, bridges, buildings, and other reinforced concrete structures exposed to marine and industrial environments are being severely damaged due to corrosion of reinforcing steel within periods as short as 10 to 20 years [4,5]. From these instances, it can be realised that the free life of concrete structures is far less in comparison to the design life unless some effective protective measures are taken at the initial stages. Protection of steel reinforcement in marine and industrial environments is essential, since lack of protection may cause direct loss due to loss of production in

case of unserviceable factory buildings and hazards to human lives as a result of collapse of residential and public buildings.

Concrete normally provides a high degree of protection to embedded steel against corrosion. This is due to highly alkaline environment provided by concrete at the concrete-
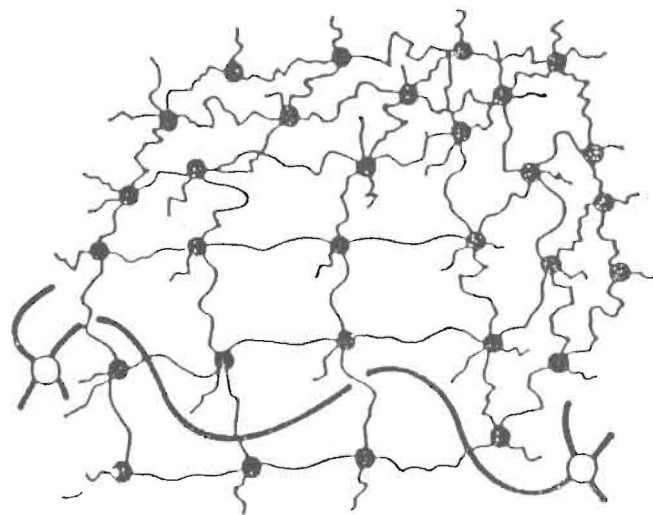

Fig. 1. Schematic view of a part of IPN showing entanglement of polymer A (thin lines) with polymer B (thick lines).

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Table 1
Physicomechanical properties of the coating

Test	Epoxy system	Epoxy/phenolic (IPN)	Standards followed
Tensile strength (N/mm²)	22.4	24.4	ASTM D-2370-7
Elongation (%)	8.0	21.0	ASTM D-2370-7
Modulus of elasticity (N/mm²)	1244.4	1031.0	ASTM D-2370-7
Specific permeability (mg/cm²/mm/24 hr)	0.2043	0.1354	ASTM D-1653-74
Glass transition temperature (°C)	107.0	94.0	DSC Method
Coefficient of linear expansion, °C × 10^{-5} (30–80)°C	1.714	0.885	ASTM D-696-7
Shear strength (Kg/cm²)	58.00	77.00	ASTM D 1002-64
Bond strength (Kg/cm²), steel substrate	25.00	30.00	BS 3900 -E-10-79
Impact resistance (falling weight method)	No failure	No failure	BS 3900-E-7-74
Scrub resistance (10,000 cycle)	No failure	No failure	ASTM D-2468-79
Salt fog (720 h)	No failure	No failure	ASTM B-117-73
Scratch hardness (1500 g load)	No failure	No failure	BS3900-E-2-70

steel interface resulting in the formation of a protective film on the steel [6]. This protective film is stable when pH is more than 12.0 and becomes unstable when pH is less than 11.0 [7]. The pH may become lowered if the concrete contains chlorides, sulphates, and other deleterious chemicals.

These chemicals diffuse through the concrete and lower the pH value of the water in the pores of concrete. As a result, protective oxide film is pierced by these chemicals, which will then attack the reinforcement [2,8].

Normal precautions such as ensuring proper mix proportion, compaction of concrete, adequate thickness of the concrete cover, and curing may not be sufficient to give proper protection to concrete and adequate reinforcement when used in an aggressive environment. Such structures need some protective measures like addition of inhibitor or admixture in
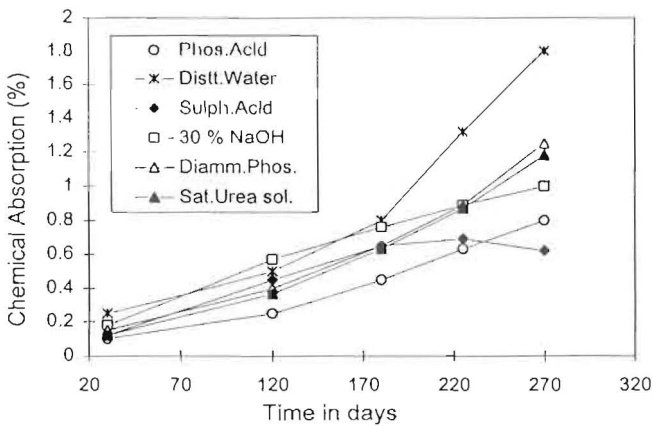

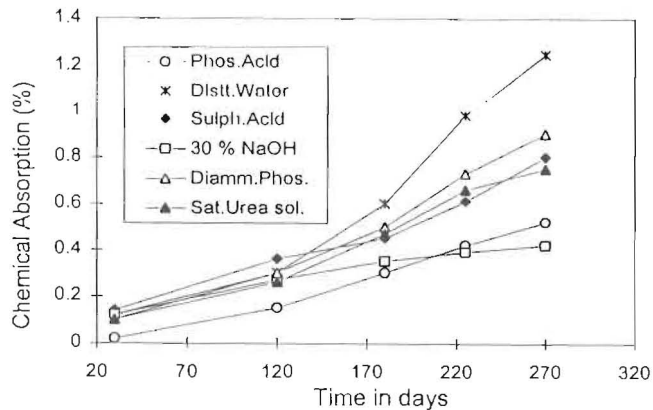Fig. 2. Percent absorption of chemicals in epoxy system.

Fig. 3. Percent absorption of chemicals in epoxy phenolic system.
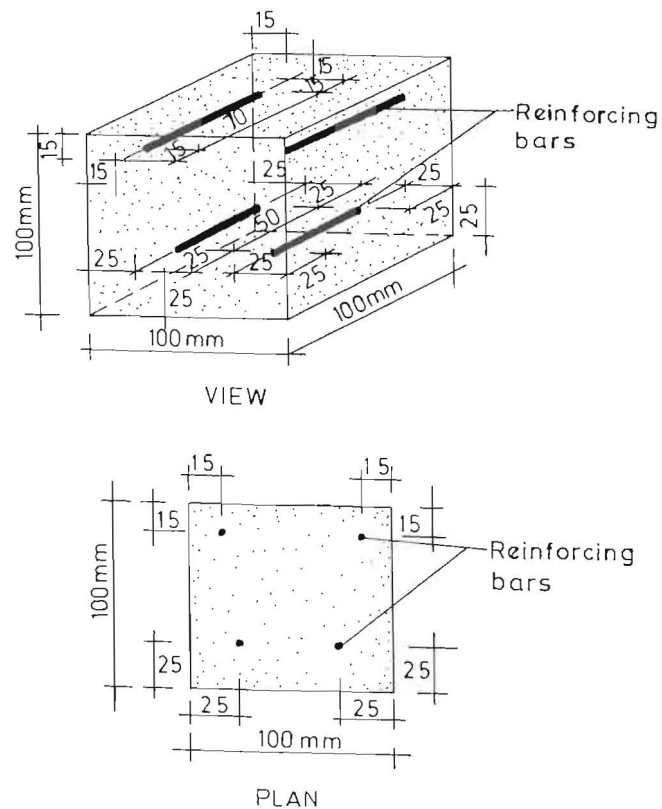

Fig. 4. Details of placement of reinforcing bars in concrete cubes.

Table 2
Chemical resistance test as per IS: 13620

Coatings	Chemicals			
	3 M CaCl$_2$	3 M NaOH	Saturated Ca(OH)$_2$	Distilled water
IPN	No failure up to 6 months	No failure up to 6 months	No failure up to 6 months	One blister after 6 months
Epoxy	No failure up to 6 months	No failure up to 6 months	No failure up to 6 months	Some blisters & Discolouration after 4 months
PU	No failure	Coating failed within 7 days	No failure up to 6 months	Some blisters debonding with substrate after 5 months
Zinc-rich epoxy coating	Discolouration after 2 months and blisters after 5 months	Some blisters after 15 days	No failure up to 6 months	Some blisters after 5 months

concrete, surface coating of concrete, or coating of the reinforcement steel [9–15]. Surface coating of the concrete reduces the ingress of oxygen, water, gases, and chemicals present in the atmospheric and industrial environments and coating of reinforcement is an effective method of increasing the life of reinforcement steel. But the corrosion protection efficiency of the coating system depends upon a number of factors, such as choice of polymer, composition of the coating, method of application, and, moreover, the environment in which it is going to be used. Therefore, a detailed study was needed for the development of a suitable coating system for the protection of reinforcing bars.

Central Building Research Institute in Roorkee has developed an "interpenetrating polymer network system (IPN polymer)"-based coating for the protection of concrete structures that are exposed to an aggressive environment. The basic coating system has been modified to make it suitable for the protection of reinforcing steel in concrete. The salient features of the developed two-component coating designated as "IPN-R," along with its corrosion protection efficacy for the protection of reinforcing steel, are highlighted in this paper.

2. Development of coating system

A cost-effective better performing epoxy/phenolic IPN coating for the protection of steel reinforcement in concrete exposed to aggressive environment (marine and industrial) has been developed. In the present development a prepolymer of epoxy resin (based on diglycidyl ether of bisphenol A) and

phenolic resin (based on cashew nut shell liquid, a by-product of the cashew industry and a renewable resource) are made to polymerize with their respective hardeners in such a manner that the prepolymers cross-links simultaneously with their respective hardeners by a separate noninterfering mechanism (Fig. 1). The IPN system thus developed has been evaluated for its physicomechanical properties and chemical resistance (against saturated urea solution, diammonium phosphate, 30% sulphuric acid, 30% phosphoric acid, 30% sodium hydroxide solution, and distilled water) as per the relevant standards and has been compared with epoxy coating under a similar set of conditions (Table 1, Figs. 2 and 3). A coating for the protection of steel reinforcement was developed by incorporating certain additives, such as reactive diluents, pigments, fillers, flow controlling agents, wetting agents, and thickening agents, among others.

3. Experimental

3.1. Corrosion protection efficacy of developed coating system

To assess the efficacy of the developed IPN-R coating for corrosion protection of steel reinforcement in concrete, following tests were conducted.

Table 3
Bond strength of coated and uncoated rebars with concrete

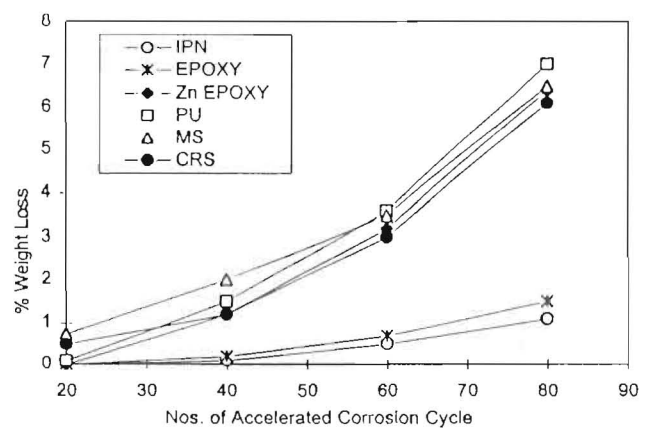| Diameter of the rebars (mm)	Condition of rebars	Bond stress at rupture (kg/cm^2)
12	Chemical resistance steel	114
12	Mild steel	113
12	IPN-coated MS rebars	110
12	Fusion-bonded epoxy-coated rebars	109
12	Epoxy-coated MS rebars	109
12	Polyurethane-coated MS rebars	106
12	Zinc-rich epoxy-coated rebars	110

Fig. 5. Weight loss (%) vs. number of acceleration corrosion cycle when rebars are embedded in M-25 concrete at 15-mm cover.
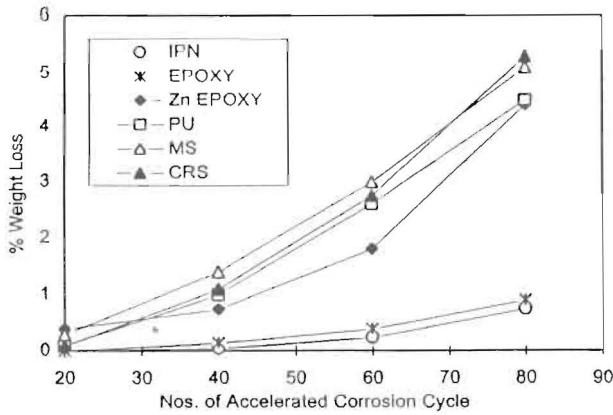
Fig. 6. Weight loss (%) vs. number of acceleration corrosion cycle when rebars are embedded in M-25 concrete at 25-mm cover.
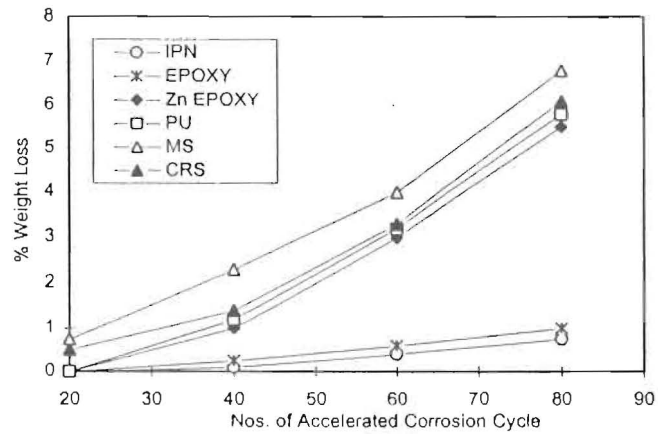

Fig. 8. Weight loss (%) vs. number of acceleration corrosion cycle when rebars are embedded in M-20 concrete at 25-mm cover.

1. chemical resistance
2. adhesion by bend test
3. bond strength with concrete by pull out method
4. accelerated corrosion test by weight loss measurements

3.2. Chemical resistance test

Samples for the chemical resistance test were prepared by applying two coats of the coatings selected for study on thoroughly cleaned reinforcing bars. After curing the coating for 3 days at room temperature, these samples were subjected for chemical resistance test as per IS 13620-1993 [16]. In this test, coated reinforcing bars were immersed in distilled water, saturated solution of calcium hydroxide, 3-M solution of sodium hydroxide, and 3-M solution of calcium chloride for a period of 180 days. Visual observations were recorded after different intervals and are reported in Table 2.

3.3. Adhesion by bend test

The adhesion test was carried out by bend test as described in IS 13620-1993 [16]. The reinforcing bars were

thoroughly cleaned and two coats of IPN coating were applied. The coated bars were cured at room temperature for 3 days and then tested for adhesion by bending the bars 120° around the 100-mm diameter mandrel. Then the bars were inspected for any signs of failure, such as cracking or debonding of the coating.

3.4. Bond strength by pull-out test method

A pull-out test as described in IS 2770 Part 1—1976 [17] was used for determining the bond strength of reinforcing bars with concrete. Specimens for the pull-out test were prepared in such a way that coated and uncoated deformed bars were placed centrally in concrete cubes. A concrete mix of
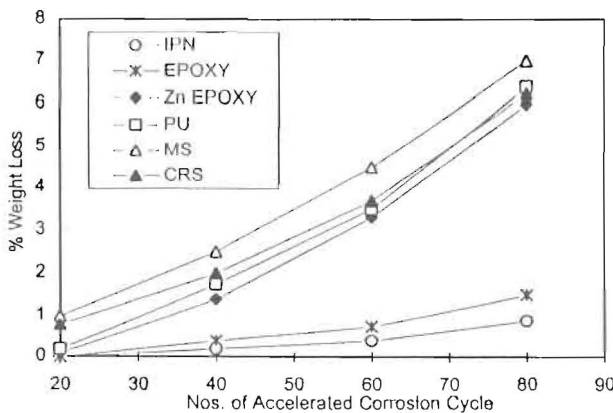

Fig. 7. Weight loss (%) vs. number of acceleration corrosion cycle when rebars are embedded in M-20 concrete at 15-mm cover.

Fig. 9. Concrete cube having uncoated MS rebars (45 cycles).

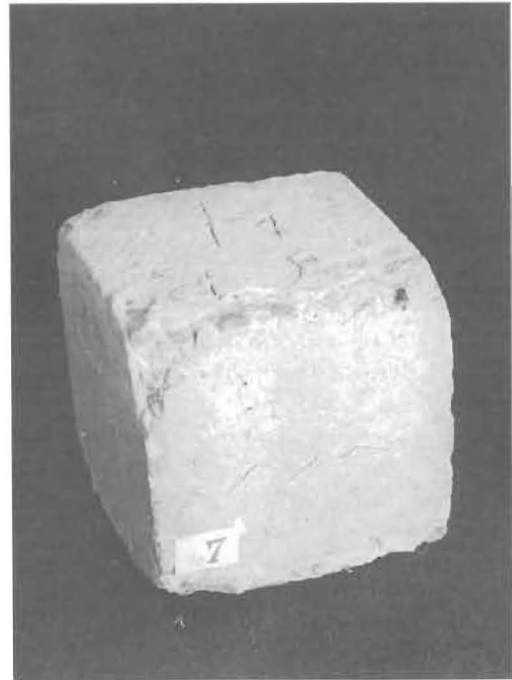Fig. 10. Concrete cube having uncoated CRS rebars (50 cycles).

Fig. 12. Concrete cube having uncoated CRS rebars (80 cycles).

M-25 grade (compressive strength, 270 Kg/cm^2) was used to prepare the specimens. After casting, the cubes were de-moulded after 24 h and then cured in water for 28 days and dried. Bond strength after 28 days was determined using a universal testing machine. Load at break was determined and results are reported in Table 3. The bond strength at rupture was calculated by dividing the load at break with the surface area of the embedded length of the bar.

3.5. Accelerated corrosion test by weight loss measurements

Loss of weight of coated and uncoated steel reinforce-ment bars embedded in concrete specimens was determined

Fig. 11. Concrete cube having uncoated MS rebars (80 cycles).

Fig. 13. Concrete cube having zinc-rich epoxy-coated MS rebars (80 cycles).

Fig. 14. Concrete cube having polyurethane-coated MS rebars (80 cycles).

Fig. 16. Concrete cube having epoxy-coated MS rebars (80 cycles).

before and after exposing the concrete specimens to acceler-
ated corrosion test. The following method for the prepara-
tion of specimens, their exposure to accelerated corrosion
test [i.e., 1 day of immersion in 3% sodium chloride solu-
tion followed by 3 day at room temperature (27 ± 2°C) and

3 days at 60°C in an air-circulating oven] was used. Fusion-
bonded epoxy-coated rebars were also used in our studies.
Only visual observations were recorded because the initial
weight of the reinforcing bars could not be determined since
these are factory made.

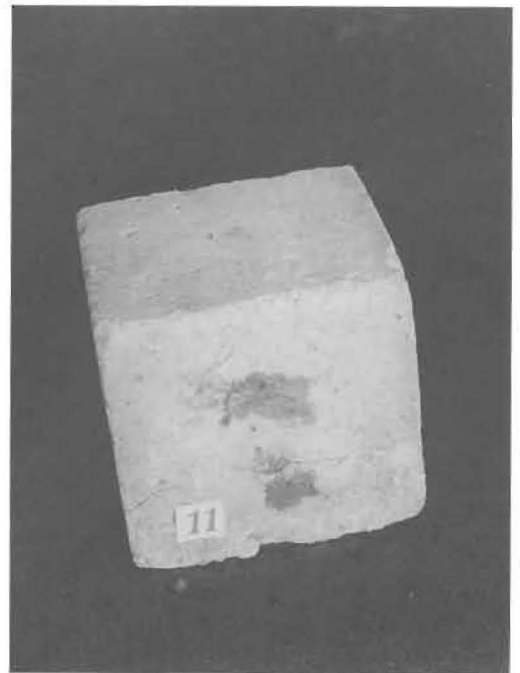Fig. 15. Concrete cube having IPN-coated MS rebars (80 cycles).

Fig. 17. Concrete cube having fusion-bonded epoxy-coated rebars (80
cycle).

Fig. 18. Condition of MS uncoated rebars after 60 cycles.

3.6. Preparation of test specimens

For weight loss measurements, reinforcement bars of required sizes were thoroughly cleaned and the initial weight of each bar was recorded. Two coats of the coatings under study were applied on the cleaned bars. In this study two types of steel bars, corrosion resistant steel (CRS) and ordinary mild steel (MS) bars, along with fusion-bonded epoxy-coated bars were used. After curing the coated bars for 7 days at 27 ± 2°C, the bars were embedded in concrete cubes in such a way that concrete covers of 15 and 25 mm were obtained. Two type of concrete mixes, M-25 (compressive strength of the concrete cube is 270 Kg/cm^2) and M-20 (compressive strength is 230 Kg/cm^2), were used. Concrete specimens for carrying out the accelerated corrosion tests were cast in a 100-mm steel mould and placed on the vibrating table, using each type of above-mentioned concrete mix. Details of the placement of the reinforcement bars are shown in Fig. 4. All the sets of concrete specimens for accelerated corrosion test were cast and cured in the same way. After drying, the specimens were subjected for accelerated corrosion cycling test. On completion of respective cycles, rebars were removed from the concrete. After the removal of adhered concrete, the samples were derusted and finally washed with distilled water. Change in weight along with visual observation after 20, 40, 60, and 80 cycles were recorded and percent change in weight after different intervals is shown in Figs. 5, 6, 7, and 8. Conditions of the cubes after different cycles are shown in Figs. 9, 10, 11, 12, 13, 14, 15, 16, and 17 and rebars after 60 cycles are shown in Figs. 18, 19, and 20.

4. Results and discussion

Table 1 shows that the properties of the epoxy/phenolic IPN system is better than the epoxy resin alone. Permeability of IPN is reduced, which shows the reduction in ingress of detrimental ions, and percent elongation is also better than the epoxy system. This shows the retention of adhesion even at higher fatigue limits is better. Bond strength of the IPN system, both in shear as well as perpendicular to surface, is better than epoxy. From Figs. 2 and 3 it is observed that the chemical resistance against acids, alkalies, and fertilizers is better than epoxy alone. The above-mentioned properties combined with excellent chemical resistance make the IPN system ideal for the protection of steel reinforcement in concrete.

The chemical resistance of the developed coating was determined by exposing the coated panels to various chemicals as described in IS 13620-1993 [16]. IPN and epoxy coating passes the chemical resistance test. Polyurethane and zinc-rich epoxy-coated samples failed in 3 M NaOH solution, but passed in three other chemicals, 3 M calcium chloride solution, saturated calcium hydroxide solution, and distilled water. Thus, IPN and epoxy-coated samples satisfy the requirements laid down in IS 13620-1993.

Bond strength of the coated and uncoated bars were determined as per IS 2770 Part I—1976. Bond strength data given in Table 3 show that there is slight reduction in the case of coated specimens. Reduction in bond strength in the case of coated rebars varies from 90–95% in comparison to uncoated bars. As per IS 13620—1993 the critical bond strength of the coated rebars should be 80% of the mean bond strength for
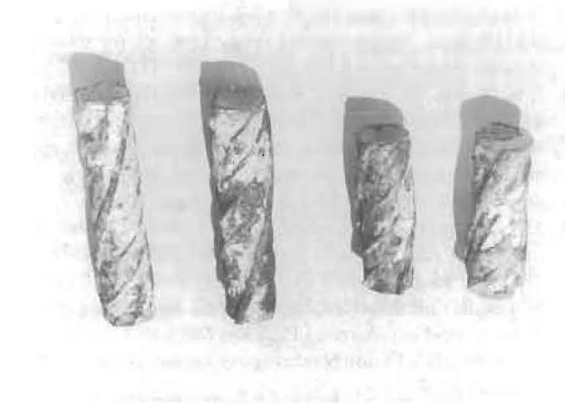

Fig. 19. Condition of IPN-coated rebars after 60 cycles.

Fig. 20. Condition of fusion-bonded epoxy-coated rebars after 60 cycles.

uncoated bars. Therefore, the reduction in bond strength in this case is within permissible limits. From this data it may be concluded that IPN-coated reinforcing bars have acceptable bond strength with concrete and hence the coating can be used for the protection of steel reinforcement in concrete to be used in a highly aggressive environment.

Results of accelerated corrosion test by weight change method has been shown in Figs. 5, 6, 7, and 8. From the curves shown, it is observed that the difference in loss of weight of coated and uncoated bars is very small after 20 cycles. However, it continues to increase with an increase in the number of cycles. Loss of weight of uncoated bars becomes excessively high with the increase in number of accelerated cycles for both the cover thickness of 15 mm and 25 mm, indicating localized corrosion. In contrast, in the case of IPN- and epoxy-coated rebar samples, the weight losses are small for both the cover thicknesses, in comparison to uncoated rebars. However, in the case of zinc-rich epoxy and polyurethane coatings, weight loss after 40 cycles is quite high and match uncoated rebars, and hence are unsafe as protective coating for steel reinforcement in an aggressive environment, while IPN and epoxy coating are found to be safe as steel reinforcement in concrete. The trend shown in Figs. 5, 6, 7, and 8 indicates that the IPN coating is better than the epoxy coating. In comparison to mild steel-reinforcing bars, CRS rebars are found to be superior up to 60 cycles, but after 80 cycles both are found to be almost same. General condition of the concrete cubes and reinforcement after 20, 40, 60, and 80 cycles of accelerated corrosion test was also recorded. All the cubes were intact up to 40 cycles but in case of cubes of M-20 concrete having MS steel reinforcement, a hair crack was seen after 45 cycles (Fig. 9), while with CRS a very small hair crack was seen after 50 cycles (Fig. 10). In the case of M-25 concrete cube that had MS reinforcement, a hair crack was seen after 50 cycles, while in the case of CRS a very small hair crack was seen after 55 cycles. All these cracks were further widened after 80 cycles (Figs. 11 and 12). A similar cracking pattern was also seen in the case of cubes that had polyurethane-coated rebars and zinc-rich epoxy-coated rebars (Figs. 13 and 14), and hence are not suitable for the protection of steel reinforcement in a highly aggressive condition. All the cubes having IPN-coated (Fig. 15) and epoxy-coated (Fig. 16) rebars were intact and hence can be used as a protective coating. In the concrete cubes that had fusion-bonded epoxy-coated bars, a small hair crack was seen in the concrete surface after 80 cycles of accelerated corrosion cycles (Fig. 17). The condition of coated and uncoated rebars after 60 cycles is shown in Figs. 18, 19, and 20.

5. Field applications

The laboratory studies carried out have generated considerable interest and therefore the work was carried out in the field, mainly for marine structures in the west coast. Some of the work included use of IPN coatings in bridges, cul-

verts, and marine structures in Maharashtra and Andhra Pradesh pile cage of transmission towers located in marshy creeks in Bombay. A recent development that points to the promising prospects for this technology is that Rolls-Royce Industrial Power (India) Ltd., a multinational company, has selected this material for use in the prestigious Godavari Gas Power Project near Kaki Nada.

6. Conclusions

It can be concluded from the above studies that the IPN-coated steel reinforcement rebars would have a more extended life in comparison to uncoated reinforcement. In IPN coating, part of the epoxy resin has been replaced by low-cost resin, and hence is less costly than epoxy. Moreover, the economics of the treatment is quite attractive since treatment costs about 15 to 20% of the cost of steel.

Acknowledgment

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