CHAPTER 5

EXPERIMENTAL INVESTIGATION

5.1 INTRODUCTION

Experiments to certain extent do reflect the true behaviour of a structure or a member. In corrosion studies experiments are the only means by which the performance of any coating system can be evaluated to a near accurate level. The following criteria are taken into account while evaluating the performance of protective coatings on steel rebars.

- The coating should have excellent corrosion resistance properties and shall not affect the bond strength of steel to concrete.
- The coating should withstand the handling stresses at the site and the aggressiveness of the test should meet the relevant ASTM / IS standards.

The details of tests conducted to evaluate the performance of protective coating systems are discussed in this chapter.

- Chemical resistance test
- Applied voltage test
- Impressed voltage test
- Open circuit potential test
- Macrocell corrosion test
In the construction site, the coated bars can be subjected to damage caused by improper handling and storage methods. It is necessary to study the performance of damaged coated bars. The performance of uncoated and coated bars were studied under the following conditions in the impressed voltage test, open circuit potential test, macrocell corrosion test and impedance technique.

- Uncoated bar in control concrete
- Uncoated bar in inhibitor admixed concrete
- Coated bar in control concrete
- Coated bar in inhibitor admixed concrete
- Coated bar (1% damage) in control concrete
- Coated bar (1% damage) in inhibitor admixed concrete

Control concrete specimens were made with a mix ratio 1: 2.18 : 2.84 and with a water-cement ratio 0.52. In the case of inhibitor admixed concrete, the inhibitor was added at 4% by weight of cement.

### 5.2 CHEMICAL RESISTANCE TEST

#### 5.2.1 Introduction

Chemical resistance tests were conducted for evaluating the resistance of coating materials when exposed to various concentration of reagents and was done as per ASTM A775 / A775M - 2001, IS 13620 - 1993.
5.2.2 Test Programme

Totally 192 coated Cold Twisted Deformed (CTD) rebars and Thermomechanically Treated (TMT) rebars with different type of coatings were tested. Figure 5.1 shows the overall chemical resistance test programme and Figure 5.2 shows the details of chemical resistance test in each system.

Total number of specimens tested = 192

- Undrilled specimens
- Drilled specimens

Figure 5.1 Overall chemical resistance test programme
Note: In each medium three specimens were tested

**Figure 5.2 Details of chemical resistance test in each system**

### 5.2.3 Test Procedure

Deformed coated rebars of 12 mm diameter and 200 mm length were used for the test and mediums such as distilled water, 3M aqueous solution of CaCl$_2$, 3M aqueous solution of NaOH and saturated Ca(OH)$_2$ solution were used. A single coated rebar was positioned vertically in a test container and the reagents were filled in such a way that the liquid level covers one half of the coated bar up to the point midway between the two intentionally made holes of 6mm diameter drilled through the coating. A rubber cork is provided at the bottleneck to prevent evaporation of reagents and contamination. The bottom surface of the rebar was sealed with epoxy material. Figure 5.3 shows the schematic diagram of chemical resistance test and Figure 5.4 shows the chemical resistance test in progress.
Figure 5.3 Schematic diagram of chemical resistance test

Figure 5.4 Chemical resistance test in progress
Specimens without holidays and specimens with 6 mm diameter intentional holes drilled through the coating were tested. The test was continued for 45 days at room temperature. After the specified period, the performance of coating was evaluated based on its blistering, softening and bond loss characteristics including development of holidays. Holidays are discontinuity in a coating that is not discernible to a person with normal or corrected vision. In the case of drilled specimens undercutting behaviour of the coating around the drilled area was observed. Uncoated CTD and TMT rebars were also subjected to chemical resistance test. At the end of test period, the corrosion rate exhibited by various test mediums were calculated using gravimetric weight loss method. The severity of corrosion offered by various test mediums were analysed and reported.

5.3 APPLIED VOLTAGE TEST

5.3.1 Introduction

The effects of electrical and electrochemical stresses on the bond of coating to steel and the film integrity of the coating was assessed by conducting applied voltage test. This is an accelerated type of corrosion test carried out as per IS 13620 - 1993.

5.3.2 Test Programme

Six tests were conducted in each coating system which comprises of 3 CTD and 3 TMT coated rebars each of 16 mm diameter and 600 mm length. Totally twenty four tests were conducted.
5.3.3 Test Procedure

Two coated bars were used in this test, one acts as an anode and the other acts as a cathode. Copper wire of 14 gauge was soldered to the exposed end of each bar. The other end of the bar was sealed with an epoxy material up to a length of 10 mm. The exposed coating surface area on which the test is to be conducted shall not be less than 232 cm². The remaining exposed area has to be applied with an epoxy based insulating material.

A non-conductive plastics container of size 150 × 150 × 620 mm was used for the test. The container was filled with 7% sodium chloride solution up to a height of 580 mm. The coated rebars were suspended vertically so as to have a clearance of 25 mm from the bottom, 40 mm from the sides and 40 mm between the rebar. A potential of 2V was impressed for a period of 60 min between the coated rebars using a suitable DC power supply. The coating failure as evidenced by the evolution of hydrogen gas at the cathode or the appearance of corrosion products of iron at the anode was monitored during the test period. A Saturated Calomel Electrode (SCE) with saturated potassium chloride as electrolyte and salt bridge made of agar-agar was used for measuring reference electrode potential. Figure 5.5 shows the schematic diagram of applied voltage test. The potential measurements were observed at every 5 min interval during the test period using high impedance multimeter. The current development was also monitored at regular intervals. Figure 5.6 shows the applied voltage test in progress and the close-up of applied voltage test is shown in Figure 5.7.
Figure 5.5 Schematic diagram of applied voltage test

Figure 5.6 Applied voltage test in progress
5.4 IMPRESSED VOLTAGE TEST

5.4.1 Introduction

This is an accelerated corrosion test carried out to assess the performance of coated rebars under accelerated electrochemical corrosion conditions. The test was carried out as per the procedure prescribed by the Structural Engineering Research Centre (SERC), Chennai, India and Central Electrochemical Research Institute (CECRI), Karaikudi, India.

5.4.2 Test Programme

In each coating system 24 tests were conducted on both CTD rebars and TMT rebars which comprise of coated bars in control concrete, coated bars in inhibitor admixed concrete, coated bars with 1% damage in control concrete and coated bars with 1% damage in inhibitor admixed concrete. In the case of uncoated control rebars, 12 tests were conducted in CTD rebars and TMT rebars with control and inhibitor admixed concrete. Totally 108 tests were conducted.
5.4.3 Test Procedure

The test specimen comprises of 70mm diameter and 115 mm height concrete cylinder. 12 mm diameter and 75 mm length coated rebars were embedded centrally such that it gives a clear cover of 25 mm from all the sides. The coated rebar acts as a working electrode and the top surface was soldered with 14 gauge wire. Both top and bottom surfaces were sealed with epoxy material. The top and bottom surfaces of the concrete specimens were also sealed with epoxy coating so as to allow chloride ingress from the sides to replicate site conditions.

A non conductive plastics container of size 150 × 150 × 150 mm was used for the test. The concrete cylinder was placed centrally in the container surrounded by a stainless steel mesh which acts as a cathode / counter electrode and filled with 3% NaCl electrolyte. A constant potential of 12 V was applied to the system using a DC power supply regulator. Figure 5.8 shows the schematic diagram of impressed voltage test. The variation in the development of corrosion current was monitored at regular intervals using a high impedance multimeter. The time required for the visible observation of cracking of specimens were monitored carefully. The time required for the initiation of first crack was considered as a measure of the relative resistance of coating material against chloride ingress and subsequent corrosion. Figure 5.9 shows the close-up of impressed voltage test in progress.
Figure 5.8 Schematic diagram of impressed voltage test

Figure 5.9 Close-up of impressed voltage test
5.5 OPEN CIRCUIT POTENTIAL TEST

5.5.1 Introduction

Open circuit potential test was conducted to evaluate the performance of coated rebars in nullifying and interfering the ingress of chloride ions under alternate wetting and drying conditions. This is a qualitative method of assessing the corrosion condition of the rebar inside concrete. The half cell potential measurements were carried out as per ASTM C876-1999.

5.5.2 Test Programme

In each coating system 24 tests were conducted on both CTD rebars and TMT rebars which comprise of coated bars in control concrete, coated bars in inhibitor admixed concrete, coated bars with 1% damage in control concrete and coated bars with 1% damage in inhibitor admixed concrete. In the case of uncoated control rebars, 12 tests were conducted in CTD rebars and TMT rebars with control and inhibitor admixed concrete. Totally 108 tests were conducted.

5.5.3 Test Procedure

The test specimen comprises of 70mm diameter and 115 mm height concrete cylinder. 12 mm diameter and 75 mm length coated rebars were embedded centrally such that it gives a clear cover of 25 mm from all the sides. The coated rebar acts as a working electrode and the top surface was soldered with a 14 gauge wire. Both top and bottom surfaces were sealed with epoxy material. The top and bottom surfaces of the concrete specimen were also sealed with epoxy coating so as to allow chloride ingress only from the sides. After casting, the specimens were cured in water for 28 days. Then the specimens were subjected to alternate wetting and drying cycle for a period of
330 days. Wetting cycle comprises of immersion in 3% NaCl for 10 days followed by 10 days drying at room temperature which forms the drying cycle. The potential of the rebar was measured against a Saturated Calomel Electrode (SCE) at the end of every wetting and drying cycle using high impedance multimeter. Figure 5.10 shows the schematic diagram of open circuit potential test and Figure 5.11 shows the open circuit potential test in progress. The observed potential readings were compared with ASTM criteria for corrosion of steel in concrete in case of uncoated bars, inhibited cement slurry coated bars and cement polymer anticorrosive coated bars in control and inhibitor admixed concrete. For galvanized bars and cement polymer composite coated bars, potential variation in the long-term were monitored and analysed accordingly. At the end of test period, the specimens were broken and the condition of rebar inside was examined to find the extent of corrosion.

Figure 5.10 Schematic diagram of open circuit potential test
5.6 MACROCELL CORROSION TEST

5.6.1 Introduction

Macrocell corrosion test is a long-term field performance test which is performed to assess the efficiency of the protective coating system under naturally occurring corrosion conditions. These tests are useful to assess the realistic durability factor under most aggressive corrosion conditions. The test was carried out as per ASTM G109-92.

5.6.2 Test Programme

Twenty four tests were conducted on both CTD rebars and TMT rebars in each coating system which comprises of coated bars in control concrete, coated bars in inhibitor admixed concrete, coated bars with 1% damage in control concrete and coated bars with 1% damage in inhibitor admixed concrete. In the case of uncoated control rebar, 16 tests were conducted on CTD rebars and TMT rebars with control and inhibitor admixed concrete. Totally 112 specimens were tested.

Figure 5.11 Open circuit potential test in progress
5.6.3 Preparation of Test Specimen

The size of the concrete prism was 280 × 115 × 150 mm, in which two numbers of 12 mm diameter bars were placed at 25 mm from the bottom (cathode) and one number of 12 mm diameter bar was placed at 20 mm from the top (anode). In the case of uncoated control specimens, rusted bars were pickled in 10% sulphuric acid solution for 10 min, dried and cleaned by wire brush before concreting. Rebar has to be placed in the moulds with longitudinal rib horizontal such that 40 mm length of the bar is protected within each exit end from the concrete by tightly wrapping with an electroplaters tape. This will expose 200 mm length of steel rebar in concrete and the protruding portion of the rebars are protected by electroplaters tape to prevent rusting during curing.

Concrete was placed in the moulds in three layers with proper compaction and consolidation. Wood float finish was given to the top concrete surface. After removing the forms, the specimens were cured in water for 28 days. After curing, the concrete surface to be exposed was roughened by steel wire brush cleaning. Four vertical sides of the concrete prism was sealed with epoxy based coating. A plastics dam of size 150 × 75 × 75 mm was then fixed centrally over the exposed concrete surface using an epoxy compound. The top concrete surface, outside the dam was also sealed with epoxy coating. One end of the rebar was soldered with a 14 gauge copper wire and a 100 ohm resistor was placed between the top and bottom bars. Figure 5.12 shows the macrocell corrosion test specimens.
5.6.4 Test Procedure

The specimens were placed over 15 mm thick wooden strips to allow airflow under most of the specimen. The plastics dam was then ponded with 3% sodium chloride solution to a height of 40 mm. The volume of sodium chloride solution is approximately 400 ml. After 10 days, the solution was vacuumed off and allowed to dry for another 10 days. This comprises of one cycle. Voltage across the resistance was measured at the beginning of every cycle using a high impedance multimeter. From the measured voltage, $V_j$ across the 100 ohm resistor, current flowing, $I_j$ is calculated.

$$I_j = \frac{V_j}{100}$$

Macrocell current density and total integrated current values were calculated as per procedure outlined in ASTM G 109-92. Corrosion potential of anode rebars against SCE were observed at the end of wetting cycle to study the status of rebar inside concrete and establishment of active macrocell
corrosion conditions. At the end of test period, the concrete prisms were broken to assess the condition of anode rebar for the extent of corrosion damage. The rebar surfaces were slightly cleaned with steel wire brush and the corroded area was traced followed by scanning. From the scanned images, the corroded area was accurately found out using Geographical Information System (GIS) and expressed as corrosion rate. Free chloride analysis of concrete samples near anode and cathode rebar in control and inhibitor admixed concrete was done by conducting standard titrimetric analysis involving silver nitrate. Figure 5.13 shows the schematic diagram of macrocell corrosion test and Figure 5.14 shows the macrocell corrosion current and potential measurements.

![Figure 5.13 Schematic diagram of macrocell corrosion test](image)

Free chloride analysis of concrete samples near anode and cathode rebar in control and inhibitor admixed concrete was done by conducting standard titrimetric analysis involving silver nitrate. Figure 5.13 shows the schematic diagram of macrocell corrosion test and Figure 5.14 shows the macrocell corrosion current and potential measurements.
5.7 IMPEDANCE TECHNIQUE

5.7.1 Introduction

AC impedance spectroscopy is a useful non-destructive technique for quantifying the corrosion of steel rebars inside concrete. In this technique an AC signal is applied to the embedded rebar and the response is monitored in terms of the phase shift of the current and voltage components and their amplitude. This is done in the time domain or frequency domain using a spectrum or frequency response analyser. Impedance is the ratio of AC voltage to AC current. An alternating voltage of about 10 to 20 mV is applied to the rebar and the resultant current and phase angle are measured for various frequencies.

5.7.2 Test Programme

Sixteen tests were conducted in each coating systems on CTD rebars and TMT rebars comprising of coated bar without damage and coated bar with 1% damage embedded in control and inhibitor admixed concrete. Eight tests were conducted on uncoated bar with control and inhibitor admixed concrete. Impedance tests were conducted on specimens at the age of 28 days (control) and at 360 days (after subjecting the specimens to 18 alternate...
wetting and drying cycle with immersion in 3% NaCl for 10 days followed by 10 days atmospheric drying). Totally 72 tests were conducted.

5.7.3 Test Procedure

The test specimen comprises of 70mm diameter and 115 mm height concrete cylinder. 12 mm diameter and 75 mm length coated rebars were embedded centrally such that it gives a clear cover of 25 mm. The coated rebar acts as a working electrode and the top surface was soldered with 14 gauge wire. Both top and bottom surface was sealed with epoxy material. The top and bottom surface of the concrete specimen was also sealed with epoxy coating so as to allow chloride ingress from the sides.

A non conductive plastics container of size 100 × 100 × 100 mm was used for the test. The concrete cylinder was placed centrally in the container surrounded by a stainless steel circular mesh which acts as counter electrode. The container was filled with distilled water for the impedance measurement of control specimens and filled with 3% NaCl for specimens subjected to alternate wetting and drying cycle for 360 days. Salt bridge made of agar-agar jell was made to be in contact with concrete surface at one end and the other end was placed in a beaker containing saturated calomel electrode with potassium chloride solution as electrolyte. The working electrode, counter electrode and Saturated Calomel Electrode (SCE) were connected to the AUTOLAB type frequency response analyser appropriately. Figure 5.15 shows the schematic diagram of impedance technique test. A frequency range of 10 mHz to 50 kHz was used. An AC signal of 10 mV amplitude was applied to the system and the resultant current and phase angle were measured at various frequencies. The impedance test results were obtained in the form of Bode plots. Figure 5.16 shows the impedance test in progress and Figure 5.17 shows the close-up of impedance cell set-up. From the Bode plot, the total coating resistance values offered by the uncoated and coated rebars in control and inhibitor admixed concrete were calculated and analysed (Saravanan et al 2007).
Figure 5.15  Schematic diagram of impedance technique test

Figure 5.16  Impedance test in progress
5.8 ATMOSPHERIC EXPOSURE TEST

5.8.1 Introduction

This test is used to evaluate the performance of protective coating material when they are exposed to varied exposure conditions. In the construction sites, there is a time lag which exists between the application of protective coating and the actual embedment in concrete. Atmospheric exposure test gives an indication of the maximum period the coated rebars can be stored in the site without affecting its original performance. In the case of uncoated control rebars, the atmospheric exposure test gives the corrosion rate of steel pertaining to that area. Accordingly type of rebar and coating system can be selected considering the vulnerability to corrosion. Exposure conditions are complex and are varied which depend on climate (i.e.) temperature and humidity and the presence of pollutants in the atmosphere. Since it is difficult to define or measure precisely all the factors that cause degradation due to weathering, results of outdoor exposure test must be taken as indicative only.
5.8.2 Test Programme

Three numbers of coated rebars each in CTD and TMT were exposed to atmospheric conditions in all the coating systems. Forty numbers of uncoated control specimens which include 20 CTD bars and 20 TMT bars were also exposed. All the test specimens are of 12 mm diameter and 200 mm in length. Totally sixty four specimens were subjected to atmospheric exposure test.

5.8.3 Test Procedure

The exposure panel consists of a stainless steel frame of size 680 × 680 mm in which waterproof coated wooden exposure racks were fabricated with rebar holding arrangement. The racks were positioned such that the exposed surfaces of sample are at an angle of 5° to the horizontal. The panel was located at a station 8 km away from the Bay of Bengal, the nearest Indian Ocean. The uncoated control specimens were pickled in 10% H₂SO₄ for 10 min followed by wire brush cleaning, dried, weighed and kept in the exposure racks. Figure 5.18 shows the atmospheric exposure test in progress.

Figure 5.18 Atmospheric exposure test in progress
The rebars were subjected to atmospheric exposure from 1st October, 2006 to 30th September, 2007 for a period of 365 days. During the exposure period, the meteorological data such as rainfall, humidity, minimum and maximum temperatures of the area were observed and recorded. At the end of exposure period, the coated rebars were investigated for loss of gloss, colour, blistering and disbondment of coating including presence of rust spots. In the case of uncoated control rebars, three numbers of CTD and TMT rebars were removed every month and the corrosion rate was studied by conducting gravimetric weight loss method.

5.9 BOND STRENGTH TO CONCRETE TEST

5.9.1 Introduction

The objective of this test is to find the effectiveness of protective coating on the bond strength of steel reinforcement with concrete. Pull-out tests were carried out on bars with different protective coating systems and also on uncoated bars as per IS 2770 - Part I - 1967 considering the modifications outlined in IS 1786-1985. The test results were compared with IS 13620 - 1993.

5.9.2 Test Programme

CTD rebars and TMT rebars three in each were tested in each coating system. Three control uncoated rebars of CTD and TMT were also tested. 20 mm diameter CTD rebars and 16 mm diameter TMT bars were used and totally thirty pull-out tests were conducted.

5.9.3 Description of Test Set up

Universal Testing Machine model 400 kN Losenhausenwerk was used to carry out the tests. Specimens for pull out test were prepared as per IS 2770 - Part I -1967. Concrete cubes of size 150 mm were cast with centrally embedded rebar provided upto 20 mm from the bottom face of the
cube. The rebar extended over the top face for a sufficient length to facilitate gripping the rod on the machine. Helical reinforcement of 6 mm diameter is wound with a pitch of 25 mm for a diameter of 130 mm. The rebar bonded length in concrete was restricted to 5 times the diameter of rebar. To avoid the bond near loaded end, plastics sleeves were provided in the remaining length. Figure 5.19 shows the arrangement inside the mould for bond strength test. Along with each set of test specimens, three 150 mm cubes were also cast in order to determine the compressive strength of the concrete of the test specimens. Test specimens and cubes were water cured for 28 days.

5.9.4 Testing Procedure

The projecting rebar was gripped firmly by the bottom wedge grips and the cube was pushed up by the platen of the machine. The test was carried out as per IS 2770 - Part I - 1967. The load verses slip behaviour for different coating systems were studied. Figure 5.20 shows the schematic diagram of pull-out test set-up and Figure 5.21 shows the pull-out test in progress. The free end slip readings were considered for calculating bond strength. From the load-slip behaviour of the free end, the mean load corresponding to a free end slip of 0.025 mm and 0.25mm were calculated, which is the usable bond strength.

Figure 5.19 Arrangement inside the mould for bond strength test
Figure 5.20 Schematic diagram of pull-out test set-up
5.10 IMPACT TEST

5.10.1 Introduction

The protective coating must resist mechanical damage during transportation, handling and fabrication and this depends on the impact resistance of the coating system. This test provides a systematic approach to test the coating materials. The resistance of the protective coating of the reinforcing bar towards mechanical damage is determined by the falling weight test. This test was carried out as per ASTM G-14 in accordance with IS 13620-1993 and ASTM A775/A775-2001.

5.10.2 Test Programme

CTD rebars and TMT rebars, three in each were tested in each coating system. Twenty four tests were conducted. The diameter of the bars chosen was 16 mm and the length of the bars were restricted to 150 mm.
5.10.3 Test Procedure

Impact test apparatus consists of a tup, a drop tube and a specimen holder. Tup having a weight of 18 kN was used over a drop range of 0.5 to 0.7 m. The head of the tup is made hemispherical with a 16mm diameter nose. Drop tube consists of a mild steel tube of 1.5 m length and 25 mm outer diameter used to contain the tup and guide it during freefall offering minimum friction to the falling tup. A scale was attached to the drop tube to measure the height of fall. The base of the apparatus was provided with an arrangement for holding the specimen in line with the axis of the vertical drop tube during testing. The specimen was kept in such a way that impact shall occur on the areas between deformations and ridges.

All the coating systems were subjected to an impact of 9 Nm at room temperature. Shattering, cracking or bond loss of coating material were observed around the impact area. Figure 5.22 shows the impact test set-up and Figure 5.23 shows the impact test in progress.

Figure 5.22 Impact test set-up
5.11 ADHESION TEST

5.11.1 Introduction

Reinforcement bars were subject to cutting, bending and shaping operation before they are laid in concrete. To resist these mechanical stresses, the coating should exhibit good adhesion and flexibility characteristics. Coating flexibility test was carried out as per IS 13620-1993 and ASTM A775/A775M-2001.

5.11.2 Test Programme

Three CTD bars and three TMT bars were subjected to flexibility test in each coating system. A total of twenty four tests were conducted. CTD and TMT coated bars of 12 mm diameter and length 1m was used for testing.

5.11.3 Test Procedure

Coated rebars were positioned over the bar bending table such that the two longitudinal protrusions are placed in a plane perpendicular to the mandrel radius and the rebar is kept at a thermal equilibrium of 24 ± 2°C. The
rebars were bent around 150mm diameter mandrel to 180° after rebound with suitable levers. The bending was done at a uniform rate of 12° per second. Figure 5.24 shows the adhesion test in progress and Figure 5.25 shows the inhibited cement slurry coated bars after adhesion test.

Figure 5.24 Adhesion test in progress

Figure 5.25 Inhibited cement slurry coated bars after adhesion test
5.12 SHORT-TERM AND LONG-TERM TESTS

Majority of short-term tests are of accelerated type and they are electrochemical in nature. Long-term tests are normally conducted on rebars embedded in concrete. The duration of the test varies from few months to several years. Long-term tests are performed to assess the efficiency of the protective coating system under naturally occurring corrosion conditions. These test methods are useful to study the realistic durability factor in most aggressive corrosion conditions.

Each and every test method has its own limitations and it is necessary to perform both short-term as well as long-term tests to analyse the realistic durability of a protective system. It is a fact that different coating systems have different durability factors under different test methods. Durability factor can be defined as the ratio of corrosion rate of uncoated control rebars and the coated rebars. In prescribing minimum codal requirements to be fulfilled by various coating systems, it is necessary to arrive at an optimum durability factor after carefully considering the following aspects.

- The nature of protective system
- Accelerating factor for particular test
- Minimum durability factor to ensure a design life
- Representative environmental condition
- Rapid evaluation coupled with realistic evaluation
- Reliability