# CHAPTER 1
# INTRODUCTION

## 1.1    Background of Study

Buried pipelines are subjected to corrosion that happens due to surrounding environment. Acidic soil, soil movement and microbial attack are only a few factors that can lead to corrosion [1]. Thus, good protection method to avoid corrosion must be used.

Normal corrosion controls are using coating as the primer protection and Cathodic Protection (CP) as the second protection. Coating will prevent corrosion on pipelines by isolate the pipelines from environment. If there is any defect on the coating, the exposed area supposes to corrode. However, if CP is installed on the pipelines, it will protect the pipeline when there is any defect on the coating. Coating used to protect the pipelines should has good characteristic to prevent corrosion even if CP is applied.

The main problem when we applied both types of protection is cathodic disbonding (CD) effect. Cathodic disbonding is phenomena when the coating starts to peel off from the surface of piping because of the conditions applied do not meet the requirements. It could be due to over potential of CP, coating is too thick or any other parameters that do not suit the conditions. Instead of protecting the bare steel area, CP has cause the coating near the defected area to dissolute [2]. Regard to the coating, different type of coatings gives different type of performance against cathodic disbonding. The factors that affect cathodic disbonding phenomena are always differing from one type of coating to the others.

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## 1.2    Problem Statement

### 1.2.1    Problem Identification

CP over potential can cause cathodic disbondment of coating used. Besides that, coating that are too thick (Dry Film Thickness) or too thin will also affect cathodic disbonding phenomena. There is a need to ensure that optimum potential of CP and suitable dry film thickness (DFT) of coating is being applied.

### 1.2.2    Significant of the Project

This project will determine suitable applied potential of CP that can give optimum protection to the piping. It will also determine suitable thickness of coating that can work well with CP. The result from this project can be guidelines for the industry in applying coating for buried pipelines.

## 1.3    Objective and Scope of Study

The objectives of the project are:
    a) To determine suitable applied potential of CP to prevent CD.
    b) To determine DFT of coating that can avoid CD due to CP over potential.

In this project, two different experiments were conducted to achieve both objectives. In these experiments, size of the coating defect, appearances of the coating surface and weights of the samples were monitored as indications whether corrosion and CD were happening on the samples or not.

# CHAPTER 2
# LITERATURE REVIEW

## 2.1    Buried Pipelines Corrosion

The corrosion of underground structures is a very widespread problem. Structures such as natural gas and crude oil pipelines and water mains are only some of the many structures reported to have been affected by soil corrosion all around the world [1]. When a gas or crude oil pipelines fails, there is a high degree of environmental, human and economic consequences. For example, in August 1983, a major pipeline (Ogoda-Brass 24") failed at Oshika village in Ahoada Local Government Area of Rivers State and an estimated 5,000 barrels was spilled. The cost of the incident was conservatively put at $1.5 million [2].

Buried pipelines are located within ever changing environmental conditions that may lead to a corrosive environment. Factors that may prevent or contribute to the initiation and attack on buried pipelines include the following [3]:

a)  **Pipe Coatings**

Buried pipe is coated to offer protection from the surrounding environment. A breakdown in the coating will result in pipeline metal being exposed. The material used for coating pipes varied over the years as technology evolved. For example; in the 1940's and 50's coal tar, wax, and vinyl tape were used; in the 1960's asphalts were used; and in the 1970's to present day fusion bond

epoxy was and is being used. Polyethylene tape and extruded polyethylene jacket material was also used from the early 1950's to the present day.

**b) Cathodic Protection**

The introduction of an electrical current on a buried pipe such that the electrode potential of the buried pipe is lowered creates an environment where metal loss is reduced. Soil conditions, such as moisture content and mineralogy influence the effectiveness of the cathodic protection, as does the type of coating on the pipe. For example, pipe coated with polyethylene material is shielded from cathodic protection more than pipes coated with asphalts.

**c) Soil conditions**

Soil structure and conditions will not only impact the effectiveness of the cathodic protection but also may contribute to the creation of a corrosive environment. Factors such as soil type, drainage, temperature, $CO_2$ concentration, and electrical conductivity all contribute to the environment surrounding the pipe.

**d) Temperature**

The temperature of the soil as well as the temperature of the pipe may create favorable conditions for attack on pipeline materials. Liquid and gas lines have slightly different operating temperature characteristics but both are still susceptible. For example, with gas pipelines both the pipe and surrounding ground can vary from a high of $40^{o}C$ upon leaving the compressor station down to $5^{o}C$ at distances from the station.

**e) Stresses (Residual and Others)**

Stresses in the pipe may lead to premature degradation of the pipeline strength. Stresses acting on the pipe include:

- residual stress from the manufacturing process,

- external stress such as those incurred due to bending, welding, mechanical gouges, and corrosion, and

- Secondary stresses due to soil settlement or movement.

f) **Pipe pressure**

Corrosion, in particular cracking, is related to the pressures exerted on the pipe. As the pressures within the pipe are increased, the growth rates for cracks also increase. The circumferential stress (hoop stress) generated by the pipeline operating pressure is usually the highest stress component that exists.

g) **Cyclic loading effects**

Conditions where the pipe is under cyclic loads may result in increased crack growth rates. Operating pressures for large diameter pipe can measure up to 8700kPa (1250psi). The pipeline pressure continually fluctuates due to loading and unloading of product and is influenced by pump activity. This applies to both gas and liquid lines but has greater influence in liquid systems.

## 2.2    Coating for Buried Pipelines

Corrosion protection is required to maintain the integrity of a buried pipeline system and coatings are the primary protection for a pipeline. As a buried pipeline is subject to corrosive attack if it is in contact with a wet environment, coating the pipeline to isolate it from this corrosive environment is an obvious approach to corrosion control. Since no coating system is defect free, cathodic protection is used to provide supplementary protection. Most countries have regulations that require pipelines to be coated and in general stipulate that a coating possess the following properties [13]:

a)    Electrically isolates the external surfaces of the pipeline from its environment.

b)      Have sufficient adhesion to resist underfilm migration of electrolyte.

c)      Be sufficiently ductile to resist cracking.

d)      Resist damage due to soil stress and normal handling.

e)      Be compatible with cathodic protection.

f)      Resist deterioration due to the environment and service temperature.

## 2.3      Polyurea Coating

In the past 10 years, the use of polyurea-based thick film coating materials for substrate protection and corrosion prevention has taken an exponential rise in the marketplace. When properly installed, polyurea coatings offer a variety of performance characteristics. Further, these coatings cure rapidly, are compliant with volatile organic compound (VOC) regulations and are high build, so they can provide multiple coat performance in a single coat application, making them attractive to facility owners representing many different industries [4]. Polyurea cannot solve your protective coating or joint fill needs by itself. It must be specified and installed in combination with proper surface preparation and primers, correct manufacturer's material formulation, proper equipment, quality control inspection and trained applicators [5].

New technology polyurea coatings and their hybrids offer the industry an environmentally compliant, high performance option (with very attractive film forming properties) for corrosion prevention and asset protection. However, like all industrial protective coatings they have performance limitations and minimum surface preparation requirements. Use of these materials outside of the recommended service environments or over marginally prepared surfaces can result in catastrophic failure and costly rework.

### 2.3.1   Definition of Polyurea Coating

The urethane coatings chemistry can be divided into three sub segments [6]:
a)      Polyurethane coatings,
b)      Polyurea coatings, and

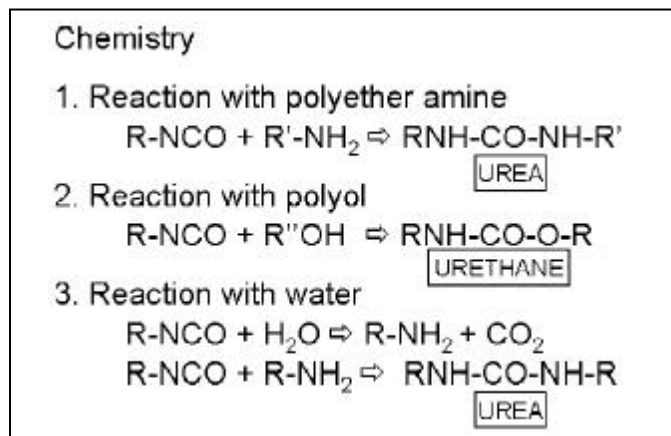c)     Hybrid polyurethane/polyurea coatings, all linked to different isocyanate reactions (refer Figure 1).

Chemistry

1. Reaction with polyether amine
   $R\text{-}NCO + R'\text{-}NH_2 \Rightarrow RNH\text{-}CO\text{-}NH\text{-}R'$
   UREA
2. Reaction with polyol
   $R\text{-}NCO + R''OH \Rightarrow RNH\text{-}CO\text{-}O\text{-}R$
   URETHANE
3. Reaction with water
   $R\text{-}NCO + H_2O \Rightarrow R\text{-}NH_2 + CO_2$
   $R\text{-}NCO + R\text{-}NH_2 \Rightarrow RNH\text{-}CO\text{-}NH\text{-}R$
   UREA

Figure 1: Isocyanate reactions [6]

Each of these segments deals with systems, which can be aromatic, aliphatic, or a blend of both aromatic and aliphatic. Pigments, fillers, solvents and/or additives can be introduced to all of them.

A purely **polyurethane** coating is the result of a reaction between an isocyanate component and a resin blend made with only hydroxyl-containing resins. The final coating film will contain no intentional urea groups. A polyurethane system will most probably contain one or more catalysts.

**A polyurea** coating is the result of a one-step reaction between an isocyanate component and a resin blend component. The isocyanate can be monomer based, a pre-polymer, a polymer or a blend. For the pre-polymer, amine- and/or hydroxyl-terminated resins can be used. On the other hand, the resin blend should only contain amine-terminated resins and/or chain extenders and not any hydroxyl reactive polymer components. All the polyurea coatings mentioned in the paper comply with this requirement.

A **polyurethane / polyurea hybrid** coating has a composition which is a combination of the above-mentioned two coating systems. The isocyanate component can be the same as for the "pure" polyurea systems. The resin blend is a blend of amine-terminated and hydroxyl-terminated polymer resins and/or chain extenders. The resin blend may also contain additives, or non-primary components. To bring the reactivity of the hydroxyl-containing resins to the same level of reactivity as the amine-terminated resins, the addition of one or more catalysts is necessary.

The **water**/isocyanate reaction also produces urea-groups at the end of the process. However, this reaction should not be considered a polyurea reaction since the mechanism is a two-step process, which is controlled by the much slower isocyanate/water reaction, and produces carbon dioxide.

### 2.3.2   Polyurea Coating Advantages and Benefits [5]

A few polyurea coating advantages and benefits that can be listed here are:

a)  No volatile organic compounds (voc) and little to no odor

b)  Weather tolerant: cures at -25ºF to >300ºF even in high humidity

c)  Excellent resistance to thermal shock

d)  Flexible: bridges cracks

e)  Waterproof, seamless and resilient

f)  Unlimited mil thickness in one application

g)  Spray, hand mix and caulk grade materials

h)  Excellent bond strengths to properly prepared substrates

i)  Resistant to various solvents, caustics and mild acids

j)  Low permeability, excellent sustainability

## 2.4    Cathodic Protection

Cathodic protection is fundamental to preserving a pipeline's integrity. Cathodic protection is a method of corrosion control that is achieved by supplying an external direct current that neutralizes the natural corrosion current arising on the pipeline at coating defects. Current required to protect a pipeline is dependent on the environment and the number and size of the coating defects. Clearly, in a particular environment, the greater the number and size of coating defects, the greater the amount of current required for protection. Coating plays an integral part in the functioning of a pipeline's cathodic protection system. Where a coating system has badly deteriorated, cathodic protection requirements and costs can increase exponentially.

### 2.4.1    Principles of Cathodic Protection

The principles of cathodic protection may be explained by considering the corrosion of metal M in acid environment. Electrochemical reaction occurring are the dissolution of metal and the dissolution of hydrogen gas; for example, metallic corrosion by interfering with both anodic and cathodic reactions, thus causing anodic and cathodic polarization.

$$M \longrightarrow M^{n+} + ne \qquad \text{(Equ. 1.0)}$$

$$2H^+ + 2e \longrightarrow H_2 \qquad \text{(Equ 2.0)}$$
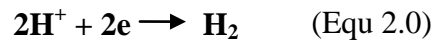
Equations (1.0) and (2.0) indicates that the addition of electron to the structure will tend to suppress metal dissolution and increase the rate of hydrogen evolution. Consider the current flow from (+) to (-), the metal structure is protected if current enters it from the electrolyte. Conversely, accelerated corrosion occurs if current passes from the metal to the electrolyte [7].

### 2.4.2 Types of Cathodic Protection

There are two main types of CP systems which are sacrificial anodes and impressed current. Impressed-current CP systems represent the vast majority of CP systems for transmission pipelines. Impressed-current systems (Figure 1) can be readily adjusted to compensate for changes in the amount of current required to adequately protect the structure; however, they may also contribute to the interference of other structures in the vicinity. Depending   upon soil, pipe coating properties, and pipe size, impressed-current CP systems can be used to protect long lengths of pipe. However, impressed-current CP systems require more expensive installation and equipment, increased monthly monitoring, and greater power consumption charges than that of sacrificial anode systems [7].
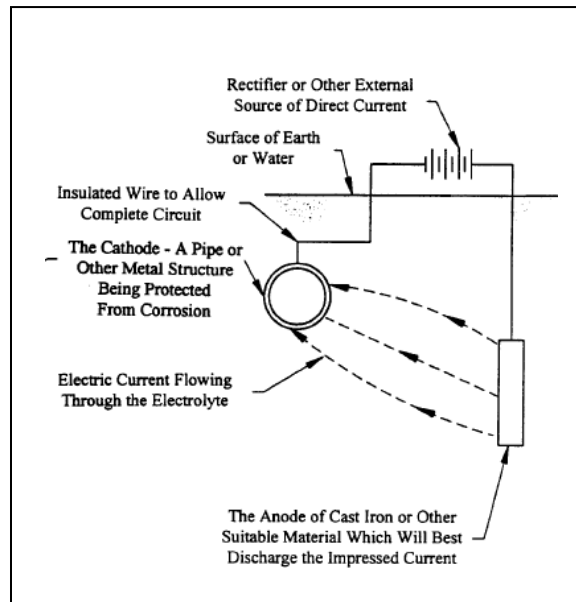


Figure 2: Impressed Current System [7]

Sacrificial anode CP systems are used extensively to protect gas distribution pipelines, but are applied more as a remedial measure for problem areas on transmission pipelines. Sacrificial anodes (Figure 2) are relatively inexpensive, do not require an external power supply, and require no regular monitoring of the anode (rectifiers for impressed-current systems require bimonthly monitoring to ensure proper operation). Due to their low driving voltages, however, sacrificial anodes are not applicable in all environments and do not have the power to protect long lengths of pipeline [7].
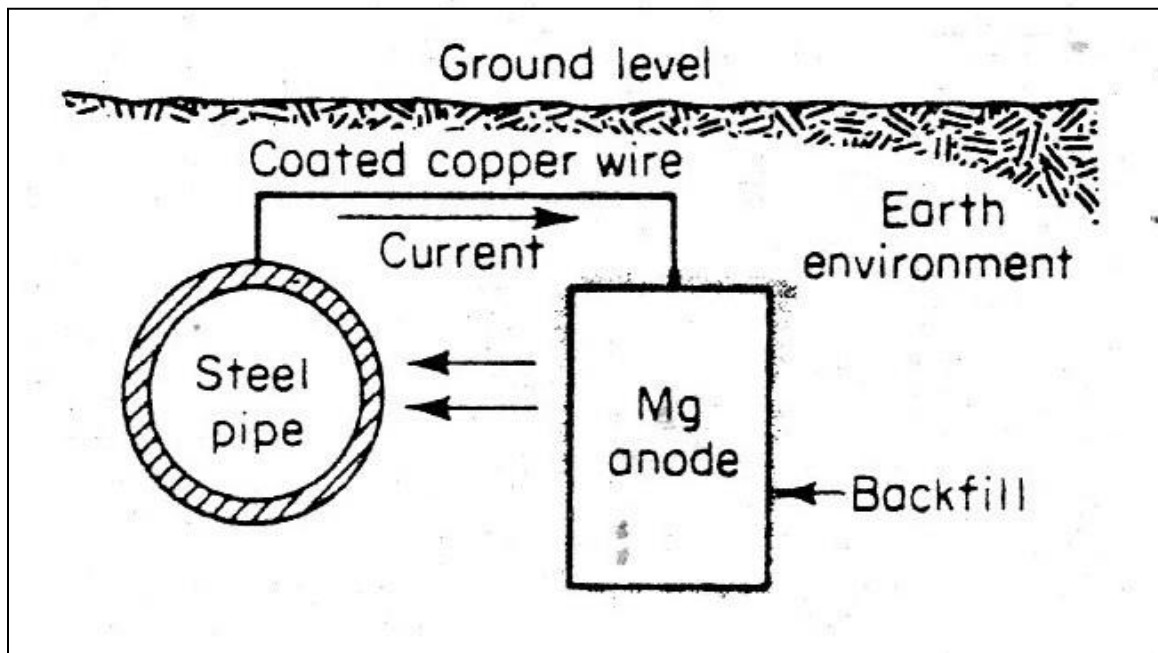


Figure 3: Underground protection of magnesium sacrificial anode, (Fontana, 1986) [7]

## 2.5     Cathodic Disbondment

Cathodic protection current passing onto the metal causes the release of hydrogen which disbonds the coating [8]. In reality this is rarely a problem, and a careful study reveals why.

The current will only pass onto the metal at a coating fault, and the density of the current will depend on the size of the coating fault and the current locally available. As the current blows the coating from the metal, the volts drop at the interface will decrease, and equilibrium will be reached with a very small increase in additional disbondment.

If there is no coating fault, then no cathodic disbondment will occur as recognized in the British Standard Code of Practice for testing the coating manufacturer's specification. This requires a specific size of coating fault on a steel coupon, to be subjected to an increasing voltage over a specified period. The test cannot be carried out on a coupon with perfect coating as the disbondment is observed under the coating at the edge of the fault.

It is logical to deduce that if cathodic disbondment is caused by current and that if all current is prevented by a perfect coating, then no disbondment will take place. This is not common sense, however as many excavations have been dug in areas where high 'pipe-to-soil potentials' have caused concern about cathodic disbondment. In the event, it has proved the logic (above) and no disbondment has been found.

### 2.5.1 Cathodic Disbondment Chemical Reaction

Chang and Asein (1989) in their work reported that at coating holidays the following chemical reactions occur due to a negative polarization of more than    -1.05V Vs $Cu/CuSO_4$ reference electrode [9].

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \qquad \text{below -1.1V (Equ. 3.0)}$$

$$1/2\ O_2 + H_2O + 2e^- \rightarrow 2OH^- \qquad \text{above -1.1 V (Equ. 4.0)}$$

These reactions which take place at the coating holidays are detrimental to the coating. Various roles have been suggested for hydrogen gas and hydroxyl ion in the disbondment process. Coulson and Temple (1983) in their extensive work reported that the hydrogen produced by the electrochemical reaction enters the holiday and builds up pressure between the coating and the pipe surface, resulting in the delamination of coatings.

Leidheiser (1981) in his work pointed out that it is the accumulation of OH ions in the holidays that caused the delamination of coatings from the pipe surface.

### 2.5.2    Cathodic Blistering

One of CD sign is blistering effect. Blistering was taking place with dissolution of coating material. The development of cathodic activity and formation of blisters in regions alongside the disbonds spreads progressively outwards over an extended time period [14].

Cathodic blistering is caused by this electrochemical reduction of oxygen beneath intact and sometimes defective coatings [15]. Water, oxygen, and alkali metal cations (e.g., sodium cations) diffuse through the coating to cathodic sites to produce strongly alkaline solutions. Anodic sites may be located at nearby corroding sites as shown in figure 4. As the reaction is localized beneath the coating film, the hydroxyl ions are trapped by the semi-permeable film at the site of adhesion loss (the incipient blister). There, ionic accumulation equates with increasing pH. The mechanism of cathodic blistering and delamination is presented in Figure 4. Figure 5 and 6 show images of blister defect seen under SEM.
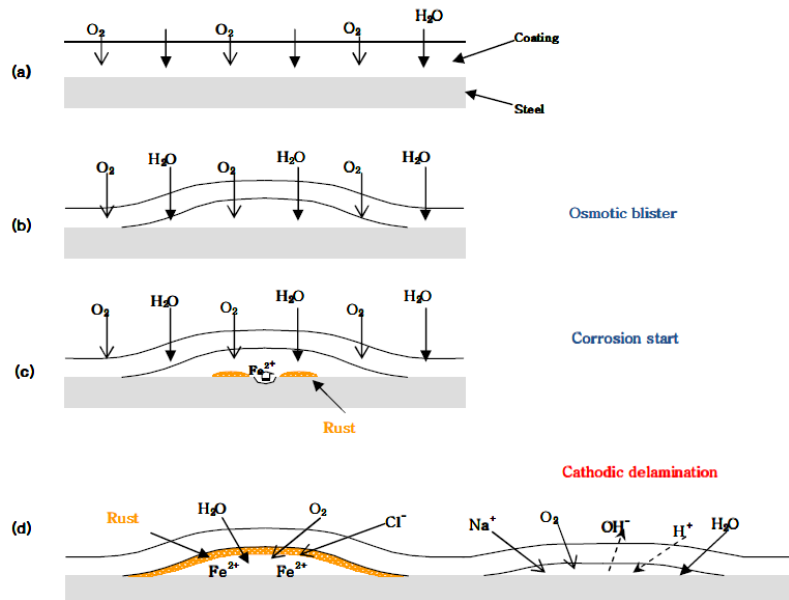


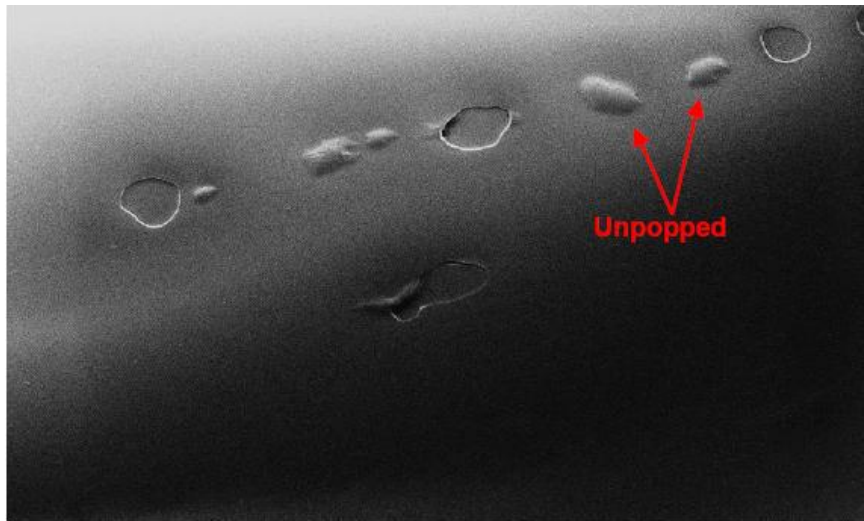Figure 4: Mechanism of Blistering and Delamination [15]

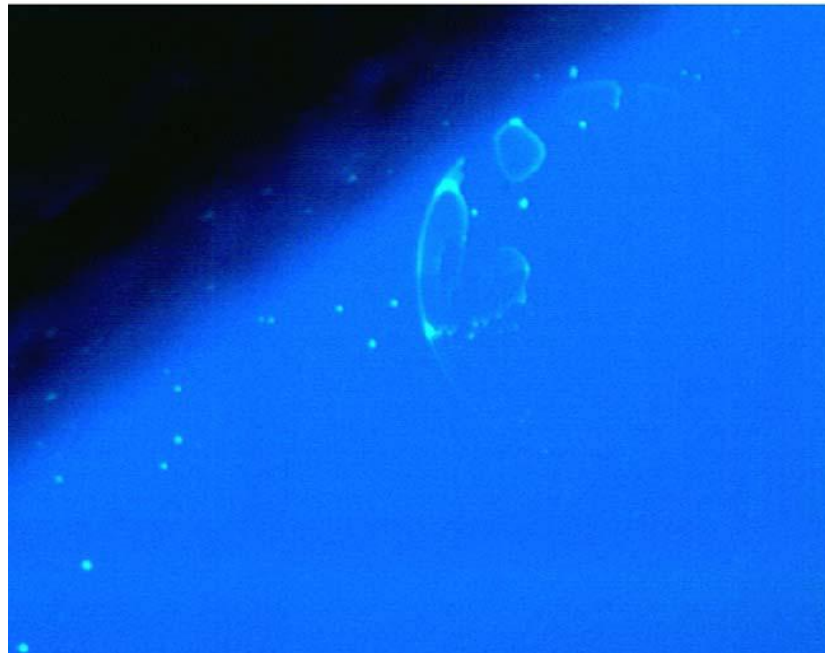Figure 5: Blisters Defect Captured by SEM at 30X Magnification [16]



Figure 6: Blister Defect at 20X Magnification [16]

Blistering can be caused by a number of different conditions [16]:

a) Soluble salts contaminating the substrate or contaminating the surface between coats. No coatings are 100% water proof. The moisture vapour passing through the coating can dissolve salt into a concentrated solution. Pressure in the high concentration liquid will cause blisters. This phenomen is called osmosis.

b) Contamination of the surface (e.g. oils, waxes, dust, etc.) will not allow proper adhesion of the coating. The moisture vapour tends to be concentrated in these areas of low adhesion. In this case, the blisters are so-called "dry" blisters.

c) Poor or inadequate solvent release from the coating. Entrapped solvents can increase the water absorption and moisture vapour transmission of the coating and lead to blistering. Solvent odour is usually connected with retained solvents. If the blistering is widespread on a construction: reblast and wash before a new system is applied. For local areas: blast or carry out other mechanical cleaning before recoating.

In an experiment conducted by J.D. Crossen, he used Epoxy-polyamide as the coating to observe cathodic disbonding effect [14]. Figure 7 below shown result of his experiment. In the pictures, we can see how disbondment spreads away from the defect.
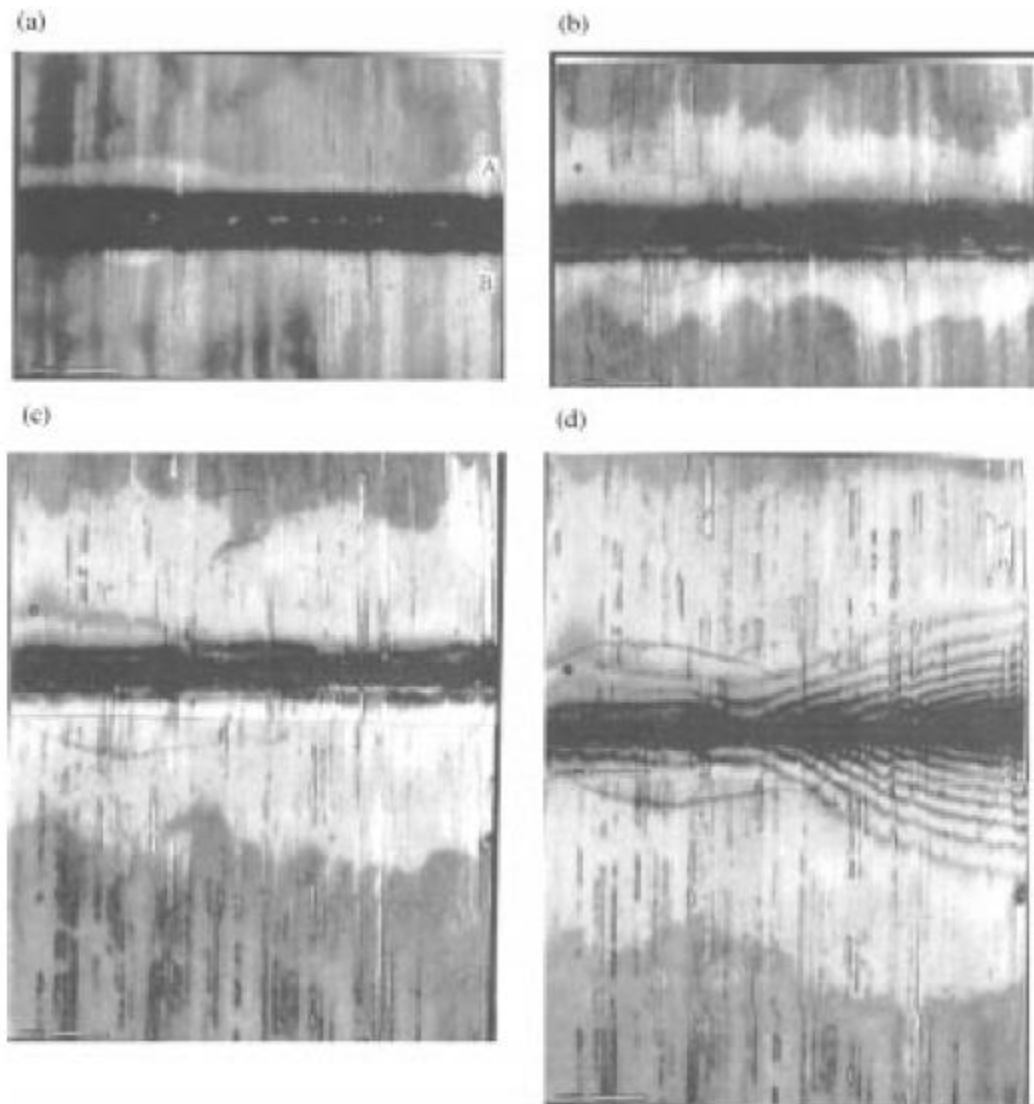


Figure 7: SAM images showing early disbondment of an epoxy-polyamide coating from mild steel at a scribed defect after (a) 10, (b) 32, (c) 42, (d) 54 min of immersion in 0.05 M NaCl solution. Scale bars are 100 lm. [14]

## 2.6　Related Works

Research made by Ole Oystein Knudsen and Jan Ivar Skar which title " Cathodic Disbonding of Epoxy Coating – Effect of Test Parameters" did mention about effects of applied potential and coating thickness in CD effect.

### 2.6.1　Effect of applied potential

Using Epoxy 0 in his experiment, he found out a linear relationship between applied potential and disbonding rate. As we can see in figure 8, decreasing in potential will cause decreasing in disbonding rate. However, another relationship cannot be ruled out, since the variation between parallels was rather high [2]. The correlation coefficient for the straight line is 0.72. A linear relationship between applied potential and disbonding rate indicates that the process is controlled by an Ohmic resistance.
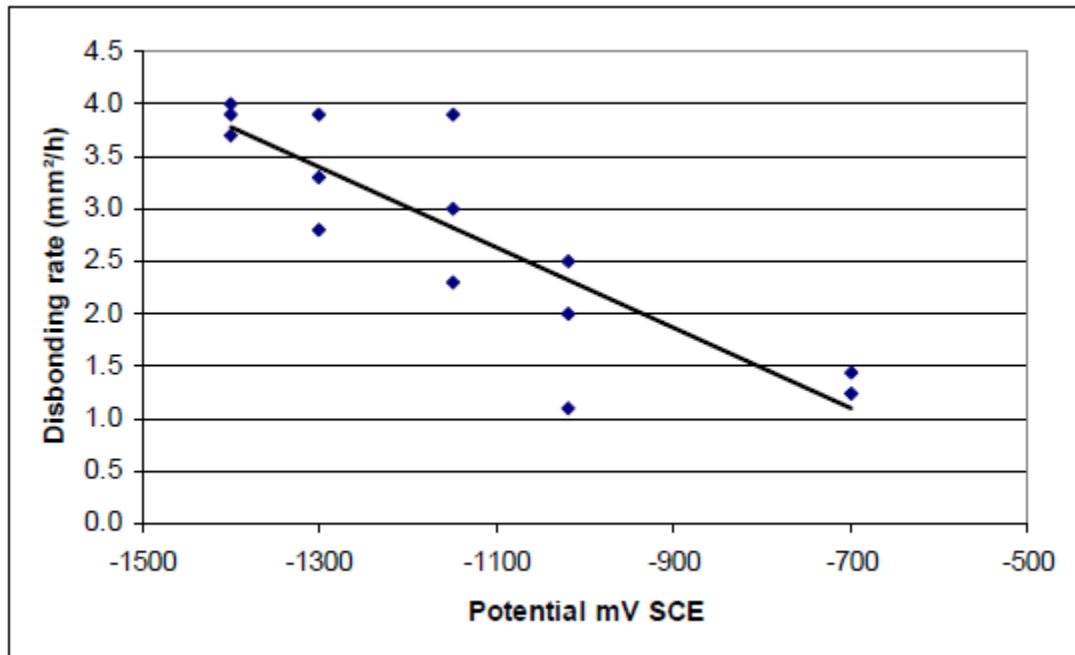


Figure 8: Disbonding rate for Epoxy 0 as function of applied potential. Test conditions: 0.5%. [2]

### 2.6.2    Effect of Film Thickness on Cathodic Disbonding

Knudsen and Skar used Epoxy 0 and Epoxy 5 to study the effect of film thickness on cathodic disbonding. The coatings were applied in thickness between 100 and 500 μm. The test conditions were not the same for the two coatings. Less aggressive conditions were used for the Epoxy 0 samples in order to avoid blistering of the coating, since the film thickness was very low for some of the samples.

As we can see in Figure 9, Epoxy 0 showed a linear decrease in cathodic disbonding with increasing film thickness. Increasing the film thickness usually improves the barrier properties of the film, which may explain the effect. The film thickness did not seem to have any effect on the disbonding rate for Epoxy 5. Epoxy 5 was pigmented with aluminum flakes, which has been shown to have a significant effect on cathodic disbonding. The ruling factor is probably a reaction of aluminum with hydroxyl ions [2].Hence, the effect of film thickness on disbonding rate will vary between coating products.
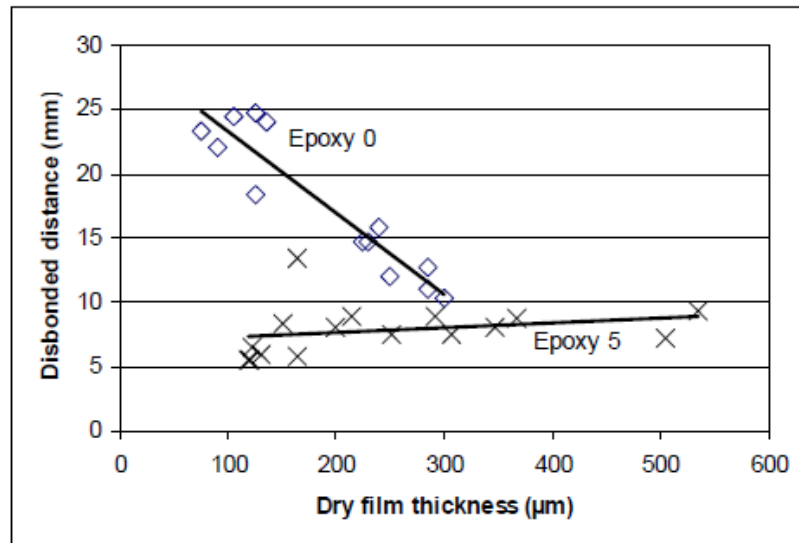


Figure 9: Effect of dry film thickness on cathodic disbonding. Experimental conditions for Epoxy 5: substitute seawater, 25°C, -1050 mV SCE, blast cleaned steel substrate. Experimental conditions for Epoxy 0: 1.5% NaCl, 20°C, -700 mV SCE, blast cleaned steel substrate.

# CHAPTER 3

# METHODOLOGY

## 3.1    Project Methodology

Figure 10 shows the overall project flow. This project started with problem identification. From the problem identified objective and scope of study been set. All these have been done in the early stage of this project.

Later on, some study had been done in the related topic of this project. Theory and procedure from other journals and articles were used to get better understanding about this project. It was actually a continuous process throughout the project. When method of the project was decided, we continued the work with sample preparation for the experiments. During this time, we need to do proper surface preparation and coating process to the samples. When the samples were ready, experiments started and all the data important data were recorded. Further analysis of the samples were done by using Scanning Electron Microscopy (SEM) machine.

After all the important data and results were recorded, those data were analyzed and discussed. Base from the analysis, conclusion was made and a few recommendations were suggested.
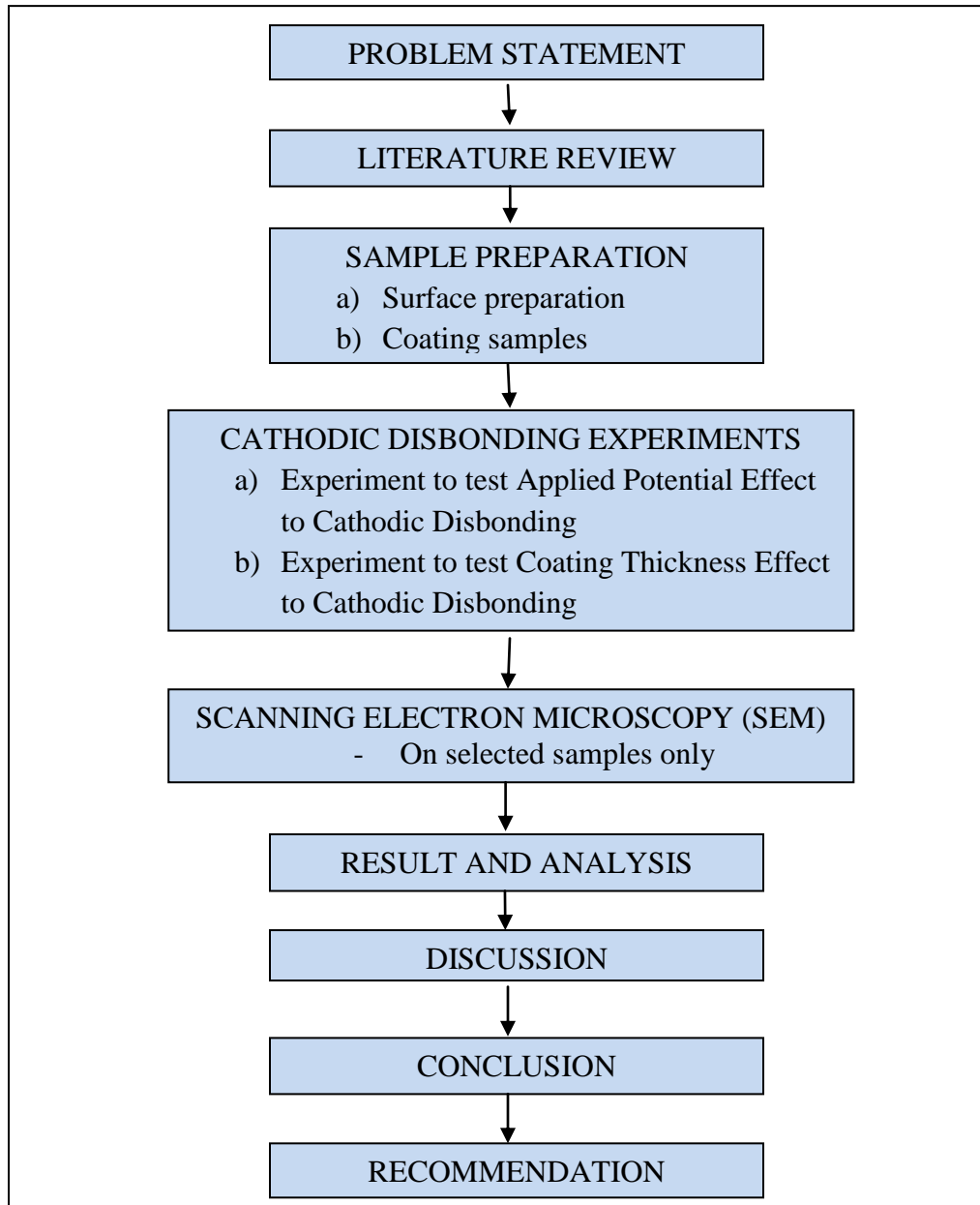
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- SAMPLE PREPARATION
  - a) Surface preparation
  - b) Coating samples

CATHODIC DISBONDING EXPERIMENTS
- a) Experiment to test Applied Potential Effect to Cathodic Disbonding
- b) Experiment to test Coating Thickness Effect to Cathodic Disbonding

SCANNING ELECTRON MICROSCOPY (SEM)
- On selected samples only

RESULT AND ANALYSIS

DISCUSSION

CONCLUSION

RECOMMENDATION

Figure 10: Overall Project Flow

## 3.2 Sample Preparation

Samples for the experiment was taken from cold rolled steel plate. The raw material was 1m x 1m plate with 1.4mm thickness. It was cut into small pieces which are 15cm x 3 cm dimension. Coating thickness and applied potential for every sample was shown in Table 1 below.

Table 1: Sample Matrix

| Experiment Group | Samples Code | Coating Thickness (mm) | Applied Potential (mV) | Quantity |
|---|---|---|---|---|
| Applied Potential Testing | A | 1.5 | -600 | 1 |
| | B | 1.5 | -850 | 1 |
| | C | 1.5 | -1000 | 1 |
| | D | 1.5 | -1500 | 1 |
| Coating Thickness Testing | E | 1.0 | -850 | 1 |
| | F | 1.5 | -850 | 1 |
| | G | 2.0 | -850 | 1 |
| | H | 2.5 | -850 | 1 |

### 3.2.1 Surface Preparation

Coating performance depends on adequate surface preparation and proper application. About 60% of all coating failures are due to improper surface treatment [11]. The surface samples need to achieve at least Sa 2.5 in order to adequate polyurea coating.

Method for steel surface preparation used:

a) **Scraping & Wire brushing (Figure 11 and 12):** loose rust, mill scale & old paint can be removed by hand or power tools such as power wire brush

& grinders are more effective than hand tools. It can give surface preparation up to St 3 which is equivalent to Sa 2.5. This method was used during first trial of sample preparation.



Figure 11: Wire brush machine



Figure 12: During wire brush process

b)   **Sand Blasting:** This method was used during second trial of sample preparation. It is the most effective method of cleaning steel. It is recommended for removal of mill scale, heavy rust, rust – scale & previous coating from extensive prior to sand blasting it is essential to first

degrease the steel. Generally four grades can be achieved by abrasive blasting to the Swedish standard or British Standard, that is, as below:

- Light blast cleaning GR SA – 1
- Through blast cleaning GR SA–2 / BS 4232 3rd quality
- Very thorough blast cleaning GR SA-2.5 BS 4232 2nd quality
- Blast cleaning to white metal GR SA-3 /BS 4232 1ST quality

### 3.2.2   Coating Process

Polyurea coating was sprayed on the samples using low pressure equipments. It requires minimal application pressure and do not require added heat which allow for the use of less complex and less expensive equipment. A few equipments are shown in Figure 13.

|  |  |
|---|---|
| (a)  Air Compressor | (b) Static-mixer |
|  |  |
| (c)  Pour Gun | (d) Low Pressure Equipments |
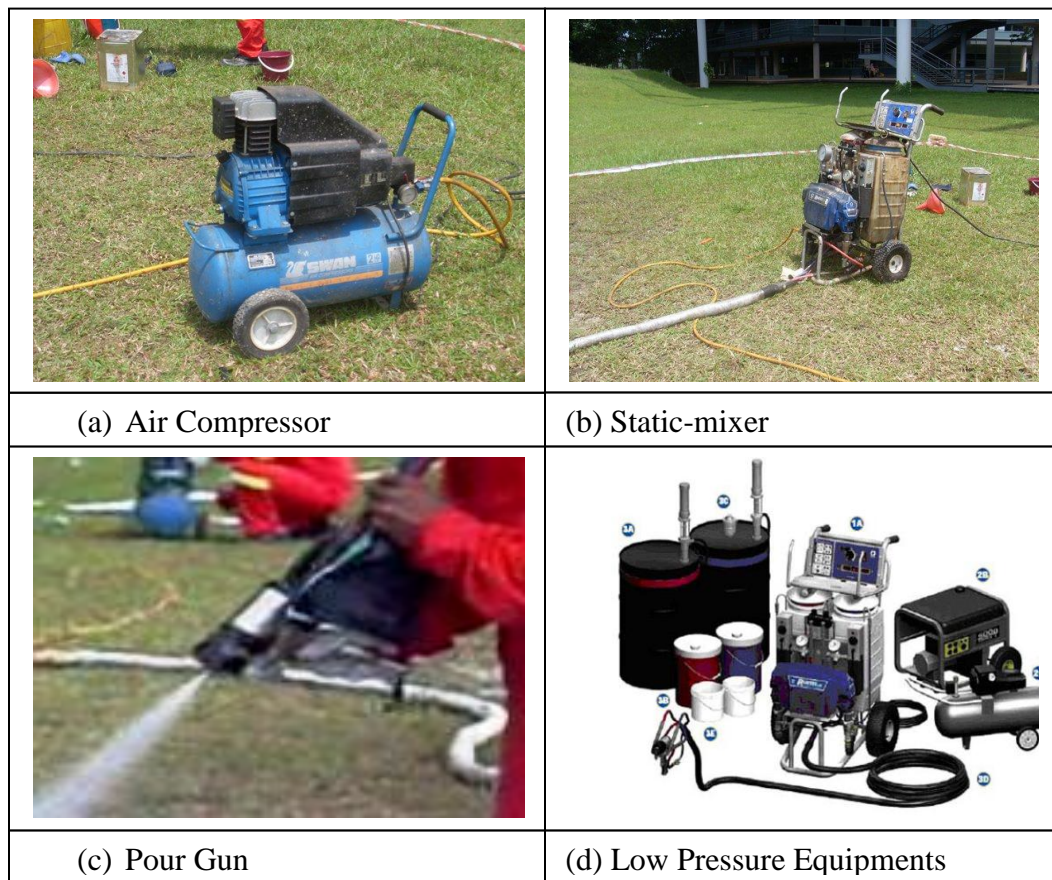
Figure 13: Low Pressure Equipments to Coat Polyurea

## 3.3     Cathodic Disbonding Experiment

### 3.3.1    Apparatus and Materials

a) *Test Vessel*—a nonconducting material shall be used for the vessel or as a lining in a metallic vessel. Dimensions of the vessel shall permit the following requirements:
   - Test specimens shall be suspended vertically in the vessel with some  clearance from the bottom.
   - Each test specimen shall be separated from the other specimens, from the anodes and from the walls of the test.

b) *Samples* – All samples having size of 15 cm (length) x 3 cm (width) x 1.4 cm (thickness) before coated (Figure 17). Coating thickness and applied potential can be refer on Table 1.

c) *Anode* –using uncoated cold rolled steel. 3 cm x 15 cm size.

d) *Connectors*—wiring from anode to positive terminal of power supply and from negative terminal to test specimen.

e) *High-Resistance Voltmeter*, for direct current, having an internal resistance of not less than 10 MV and having a range from 0.01 to 5 V for measuring potential to the electrodes (Figure 14).

f) *Power Supply, DC Converter* – act as rectifier (Figure 15).

g) *Electrolyte*- consist of potable tap water with the addition of 1 mass % of sodium chloride. Freshly prepared solution was used for each test.

h) *Stirrer* – used to stir solution of electrolyte (Figure 16).

i) *Mounting Board* introduced through holes in the top ends of test specimens have been found suitable for suspending test specimens from the vessel cover.

Figure 14: Power supply



Figure 15: Voltmeter
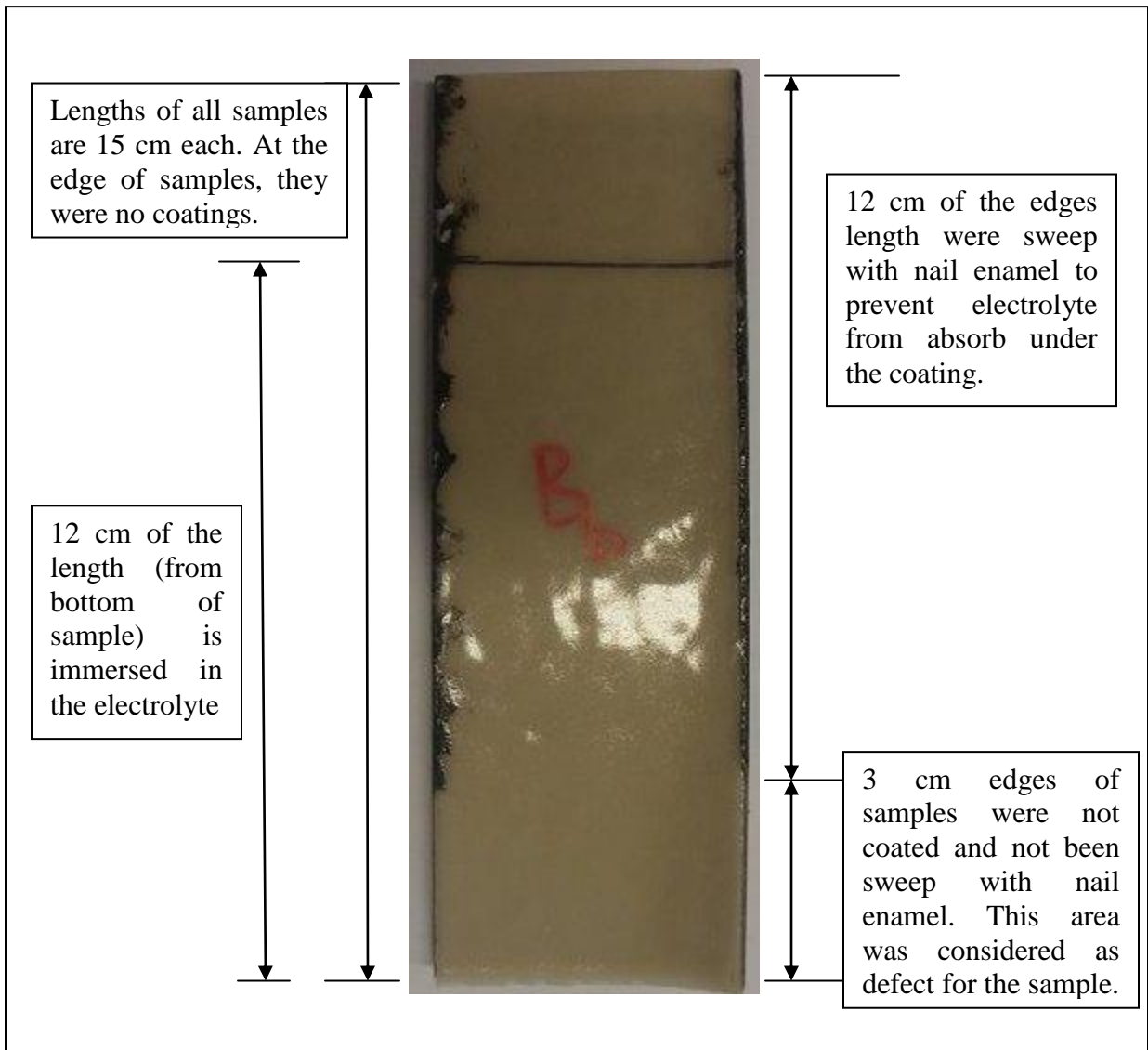


Figure 16: Stirrer

Figure 17: Typical Test Samples Used in Experiment

### 3.3.2   Experiment Assembly and Procedure

1. Experiments were assembled as shown in figure 18. Samples were connected to negative terminal of power supply and anodes (bare steel plate) were connected to positive terminal.
2. Before experiment start, picture of the samples were taken and weight were measured. (Initial condition of the samples).
3. Experiment conditions were setup based on table 1. For samples A until D, potential applied were varied from -600 mV to -1500 mV. For samples E until H, potential applied were fixed to -850 mV but the coating thickness of the samples were varied from 1.0 mm to 2.5 mm.
4. Experiments were run for 7 days. Potential applied were monitored and adjusted to the required potential.
5. On $4^{th}$ day of the experiments, weights of the samples were taken.
6. On $7^{th}$ day of the experiments, power supplies were turn off. Picture of the samples were taken (after cleaning) and weights of the samples were recorded.
7. On $10^{th}$ day of the experiments, weights of the samples were taken again and surface of the samples under coating were observed and pictures were taken.
8. After identified 2 samples from each group of experiments (which consider the least and the most corroded samples) from visual inspection and weight data, those coating surface samples were observed using Scanning Electron Microscopy (SEM) machine.
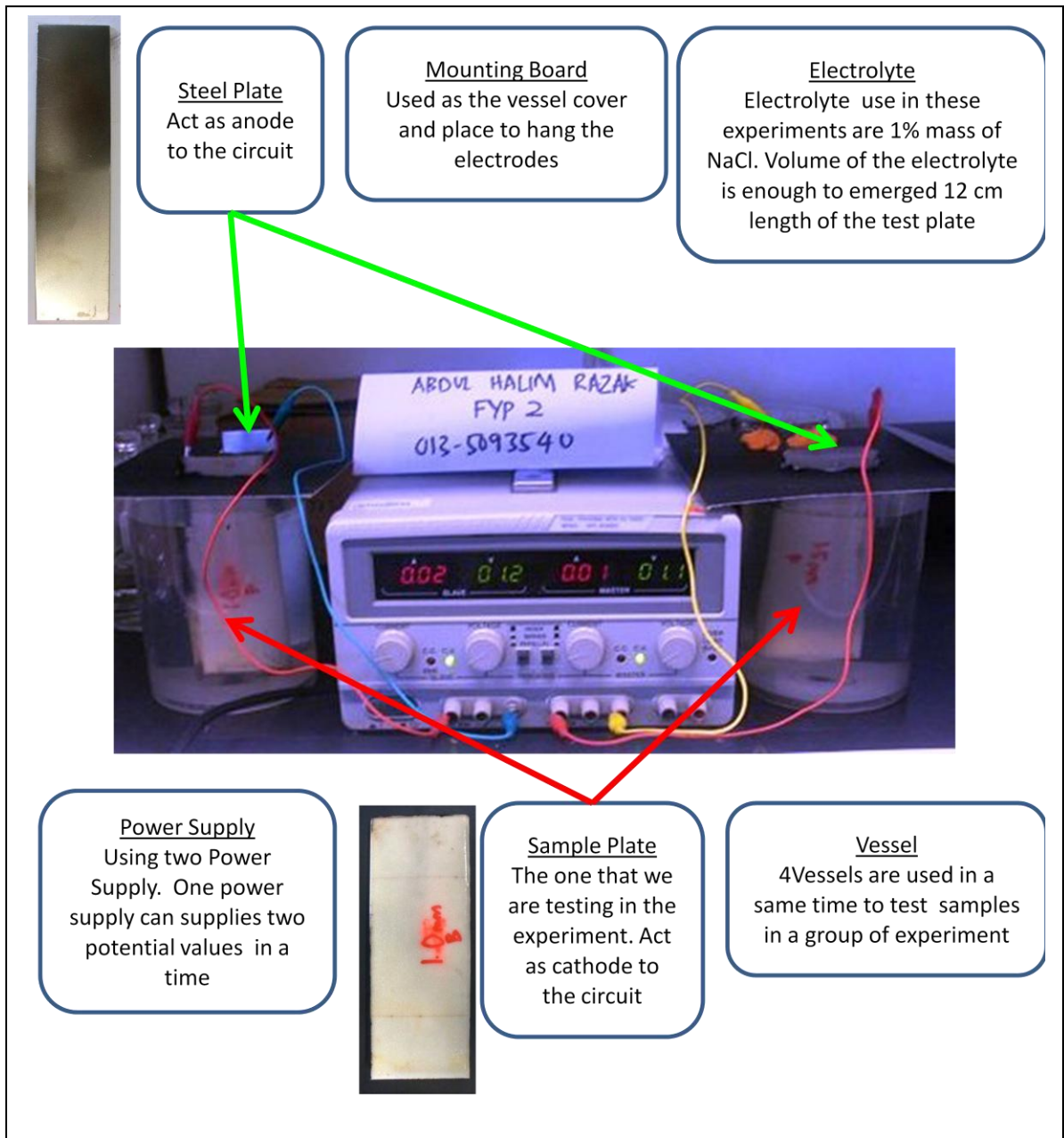
**Steel Plate**
Act as anode to the circuit

**Mounting Board**
Used as the vessel cover and place to hang the electrodes

**Electrolyte**
Electrolyte use in these experiments are 1% mass of NaCl. Volume of the electrolyte is enough to emerged 12 cm length of the test plate

**Power Supply**
Using two Power Supply. One power supply can supplies two potential values in a time

**Sample Plate**
The one that we are testing in the experiment. Act as cathode to the circuit

**Vessel**
4Vessels are used in a same time to test samples in a group of experiment

Figure 18: Experimental Test Setup

# CHAPTER 4
# RESULTS AND DISCUSSIONS

## 4.1     Result of Cathodic Disbonding Experiment

### 4.1.1   Potential Varied

For applied potential testing, there are no obvious signs of cathodic disbonding happened on any of the samples. Visual inspection that being done immediately after experiment shows no corrosion happened at the defected area. This means CP were protecting the defected area. Difference between the coatings surface could not been identified using visual inspection.

However, observation of surface under coating showed that water was coming under the coating from the defected area (edge of sample). It happened on every sample. However, water cannot penetrate directly to the surface through the coating. Even though, polyurea coating are absorbing electrolyte, the electrolyte were still contain inside the polymer and did not reach until steel surface. Appendix 1 shows pictures of the samples at the initial of the experiment, after experiment and pictures of samples without coating.

### 4.1.2   Thickness Varied

For coating thickness testing, there were also no obvious signs of cathodic disbonding happened on any of the samples. Visual inspection gives same result as the first experiment. No difference can be detected between the samples in term of coating surface condition. Observation of the surface under coating also showed that water was coming

under the coating from the defected area (edge of sample). It happened on every sample. Water cannot penetrate directly to the surface through the coating. Even though, polyurea coating are absorbing electrolyte, the electrolyte were still contain inside the polymer and did not reach the steel surface. Appendix 2 shows pictures of the samples at the initial of the experiment, after experiment and pictures of samples without coating.

## 4.2    SEM Result

### 4.2.1   Potential Varied

Figure 19 shows SEM image of samples exposed to -600 mV. The surface of the samples is still smooth and there are no signs of blistering. This shows that lower potentials which are -600 mV and -850 mV are able to reduce the effect of CD. Figure 20 shows the same surface but with bigger magnification to verify the conditions of the coating surface.
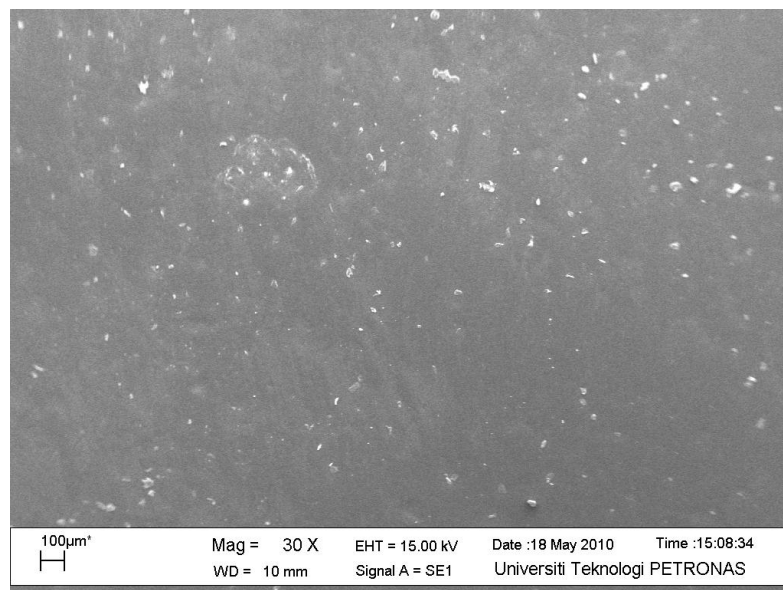


Figure 19: Coating Surface for -600 mV potential Applied (Mag = 30X)

| 100μm* | Mag = 100 X | EHT = 15.00 kV | Date :18 May 2010 | Time :15:09:17 |
| | WD = 10 mm | Signal A = SE1 | Universiti Teknologi PETRONAS | |
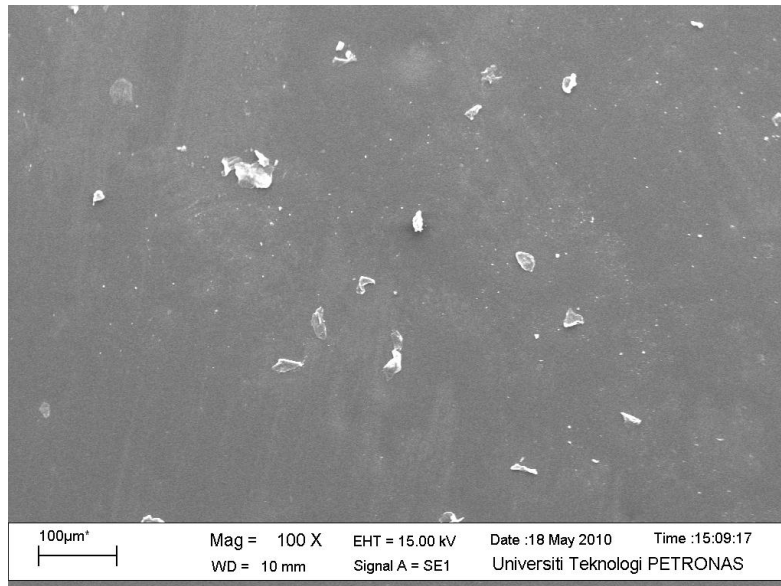
Figure 20: Coating Surface of -600 mV potential testing (Mag = 100 X)

Figure 21 shows SEM image of samples exposed to -1500 mV. The image shows that blistering has occurred rapidly when exposed to the potential. The blistering has grown big and even holes are already developed at this stage. This image shows that blistering and dissolution of coating will start occurred at potential lower than -1500 mV. Figure 22 shows the same surface but with bigger magnification.



| 100μm* | Mag = 30 X | EHT = 15.00 kV | Date :18 May 2010 | Time :15:16:48 |
| | WD = 10 mm | Signal A = SE1 | Universiti Teknologi PETRONAS | |
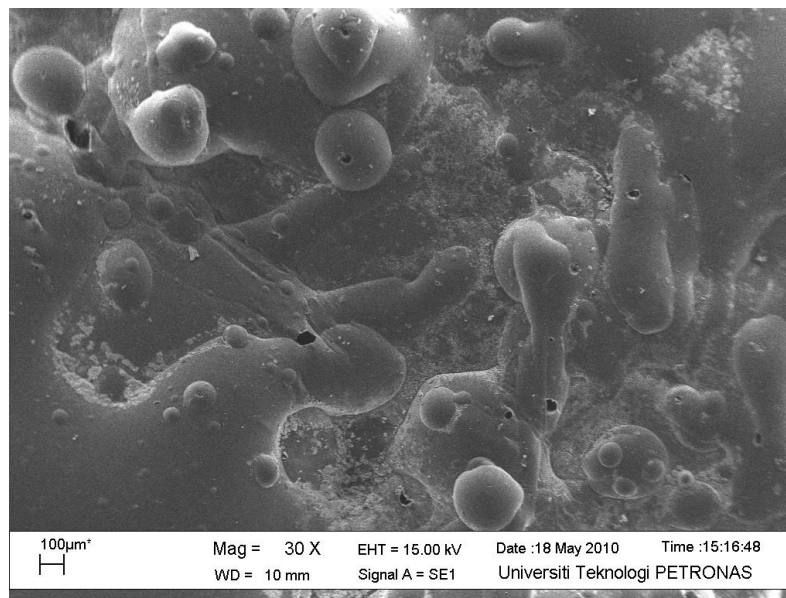
Figure 21: Coating Surface for -1500 mV potential testing (Mag = 30X)

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| 100μm* | Mag = 100 X | EHT = 15.00 kV | Date :18 May 2010 | Time :15:20:21 |
| | WD = 10 mm | Signal A = SE1 | Universiti Teknologi PETRONAS | |
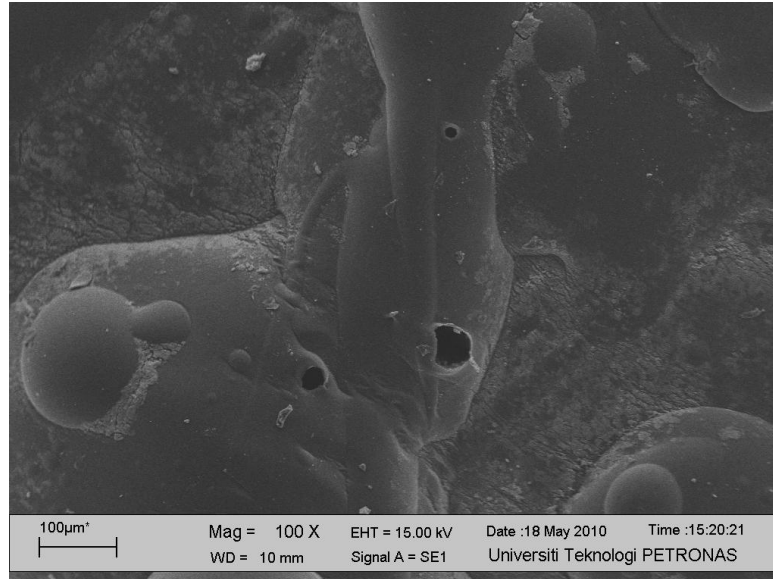
Figure 22: Coating Surface for -1500 mV potential testing (Mag = 100X)

### 4.2.2 Thickness Varied

From Figure 23, it could be seen that a few image of blistering start to appeared on coating surface of 1.5 mm thickness. The blisters are still small and not concentrated on close area. Only one or two of the blister are already popped. In Figure 24, it shows the same image of blister but in a bigger magnification.



| 100μm* | Mag = 30 X | EHT = 15.00 kV | Date :18 May 2010 | Time :15:11:37 |
| | WD = 10 mm | Signal A = SE1 | Universiti Teknologi PETRONAS | |
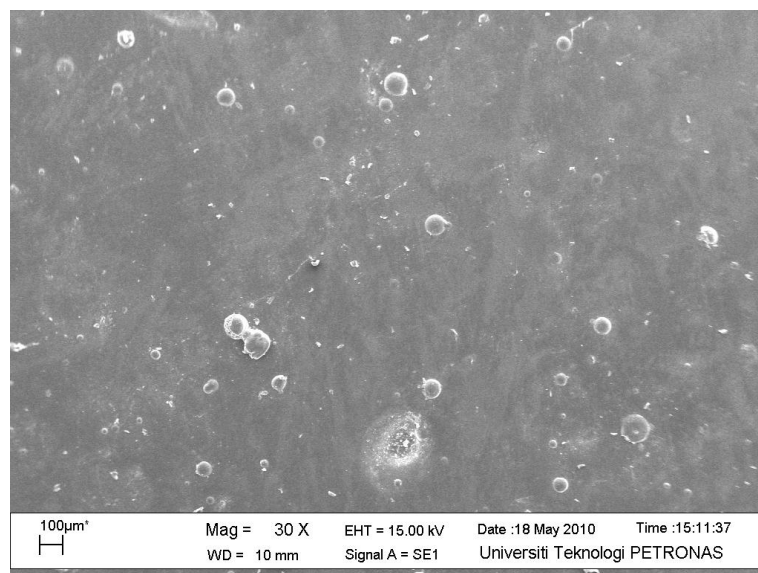
Figure 23: Coating Surface for 1.5 mm coating thickness testing (Mag = 30X)
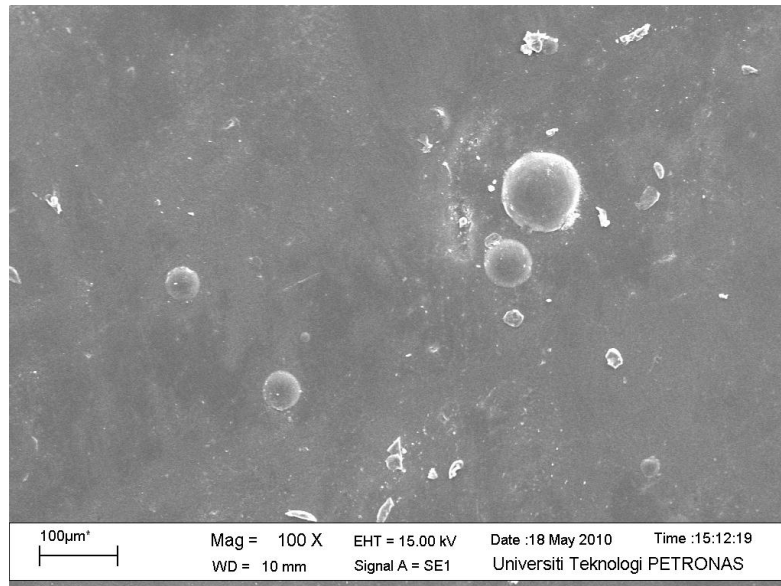
Figure 24: Coating Surface for 1.5 mm coating thickness testing (Mag = 100X)

From Figure 25, it could be seen that blisters on coating surface of 1.5 mm thickness have grown bigger. The blisters are located close to each other which mean blistering are occurring rapidly on this coating compare to 1.5 mm coating thickness. They are also already popped blisters. In Figure 26, it shows the same coating surface image but in a bigger magnification.
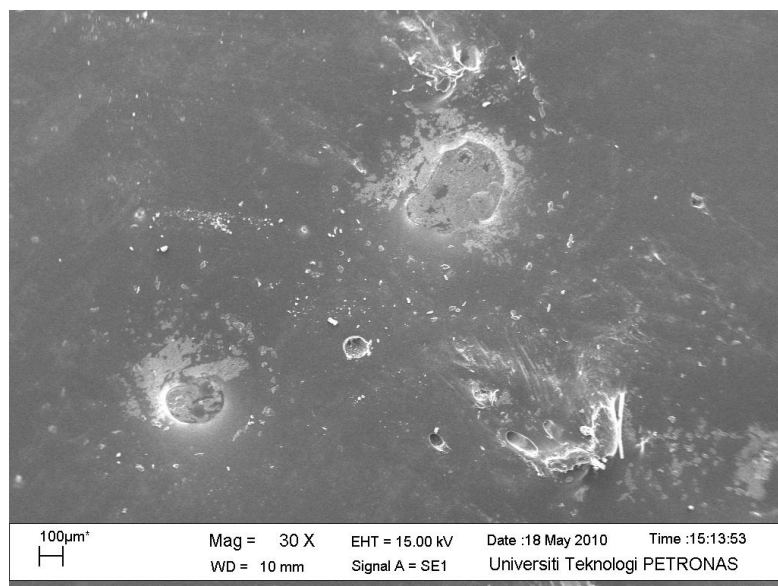


Figure 25: Coating Surface of 2.5 mm coating thickness testing (Mag = 30X)
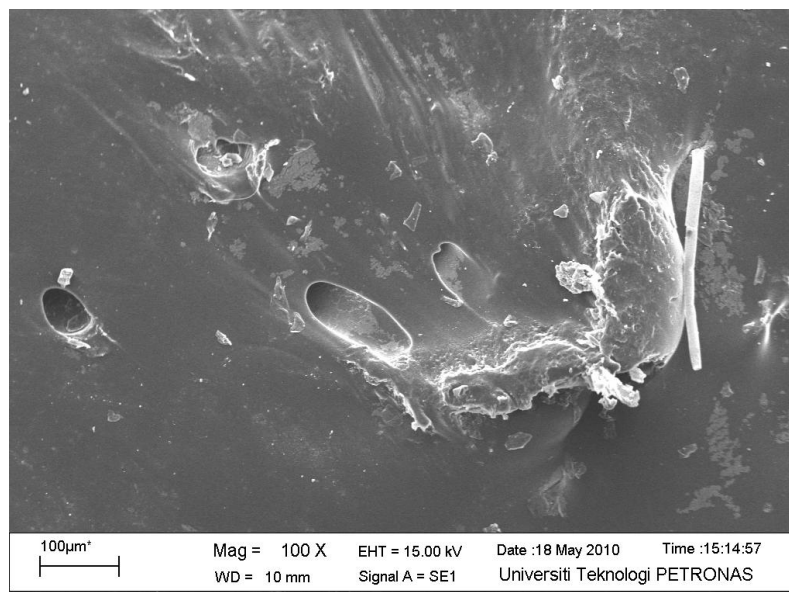
Figure 26: Coating Surface for 2.5 mm coating thickness testing (Mag = 100X)

## 4.3 Water Absorption

From weight measured on fourth, seventh and tenth day, it could be seen that there is weight increment on the samples (refer Table 2 and 3). It was expected because most polymer having water absorption characteristic. After samples were taken out from electrolyte on day seven, the samples were let dry and there were weight decrement because electrolytes inside polyurea were vaporized. Electrolyte absorbed by the coating caused high concentration liquid inside it. Pressure in the high concentration liquid will cause blisters and will add to the effects of blistering due to CP. It also could be concluded that as the coating thickness increase, water absorption are also increase.

Table 2: Percentage of Weight Increment or Reduction for Applied Potential Testing

| | Percentage of Weight Increment/Reduction (%) | | |
|---|---|---|---|
| Samples / Days | Fourth | Seventh | Tenth |
| A | 0.2620 | 0.2480 | -0.0004 |
| B | 0.2150 | 0.1994 | -0.0400 |
| C | 0.2211 | 0.2249 | -0.0416 |
| D | 0.1704 | 0.1959 | -0.0872 |

Table 3: Percentage of Weight Increment or Reduction for Coating Thickness Testing

| Samples / Days | Percentage of Weight Increment/ Reduction (%) | | |
|---|---|---|---|
| | Fourth | Seventh | Tenth |
| E | 0.0962 | 0.0979 | -0.3979 |
| F | 0.2378 | 0.2512 | -0.2890 |
| G | 0.2271 | 0.2595 | -0.3075 |
| H | 0.2245 | 0.2330 | -0.4055 |

# CHAPTER 5

# CONCLUSION AND RECOMMENDATION

## 5.1    Conclusions

From experiments of applied potential and coating thickness testing, a few conclusions can be made:

a) At coating thickness of 1.5mm, -850 mV is the best applied potential of CP that can avoid CD.

b) At coating thickness of 1.5mm, -1500 mV applied potential cause a lot of CD effects.

c) At -850 mV applied potential, 1.5 mm is the optimum coating thickness that can reduce CD effects.

d) At -850 mV applied potential, CD effects increase as the coating thickness increases more than 1.5 mm.

e) Absorption of water increase as the coating thickness increase.

## 5.2    Recommendations

If this project is going to be developed further in the future, there are a few recommendations to improve the project. Suggested future works are:

a) Samples must be cut first before being coated to avoid damage to the coating.

b) Experiments must be done in a month time to have better result of CD.

c) At least 3 samples must be tested in one condition to get average of the data.

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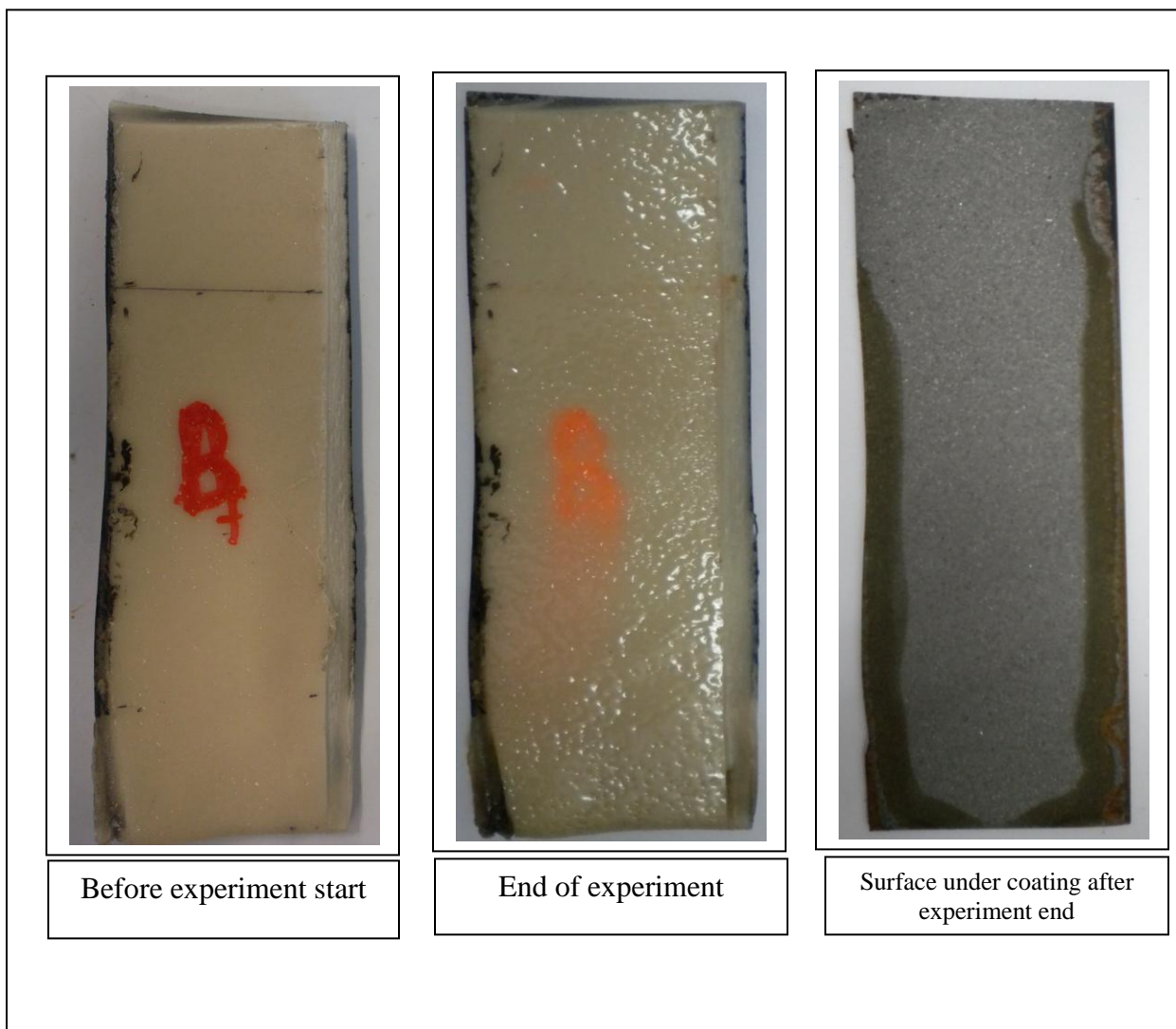20020115.nsf&v=1102&e=uk&m=922&c=52CB8C0DAD610F78C1256C40006
C2D04>

# APPENDIX

**APPENDIX 1(a)**: APPEARANCE OF THE SAMPLES FOR APPLIED POTENTIALTESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATING - Samples for Applied Potential Testing – 600 mV



| Before experiment start | End of experiment | Surface under coating after experiment end |
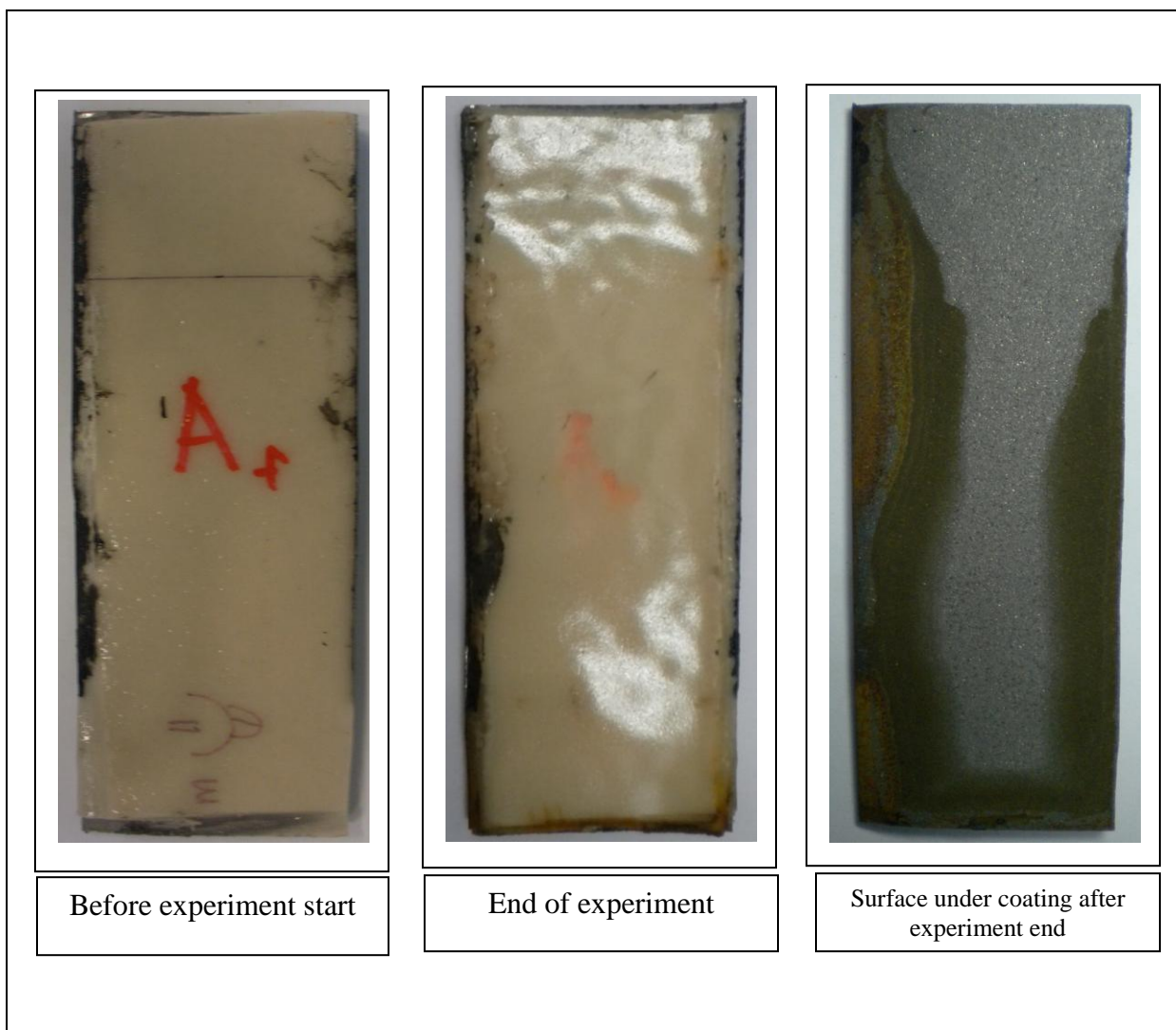
**APPENDIX 1(b)**:    APPEARANCE OF THE SAMPLES FOR APPLIED POTENTIALTESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATING - Samples for Applied Potential Testing – 850 mV



| Before experiment start | End of experiment | Surface under coating after experiment end |

**APPENDIX 1(c)**: APPEARANCE OF THE SAMPLES FOR APPLIED POTENTIALTESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATING - Samples for Applied Potential Testing – 1000 mV



| Before experiment start | End of experiment | Surface under coating after experiment end |

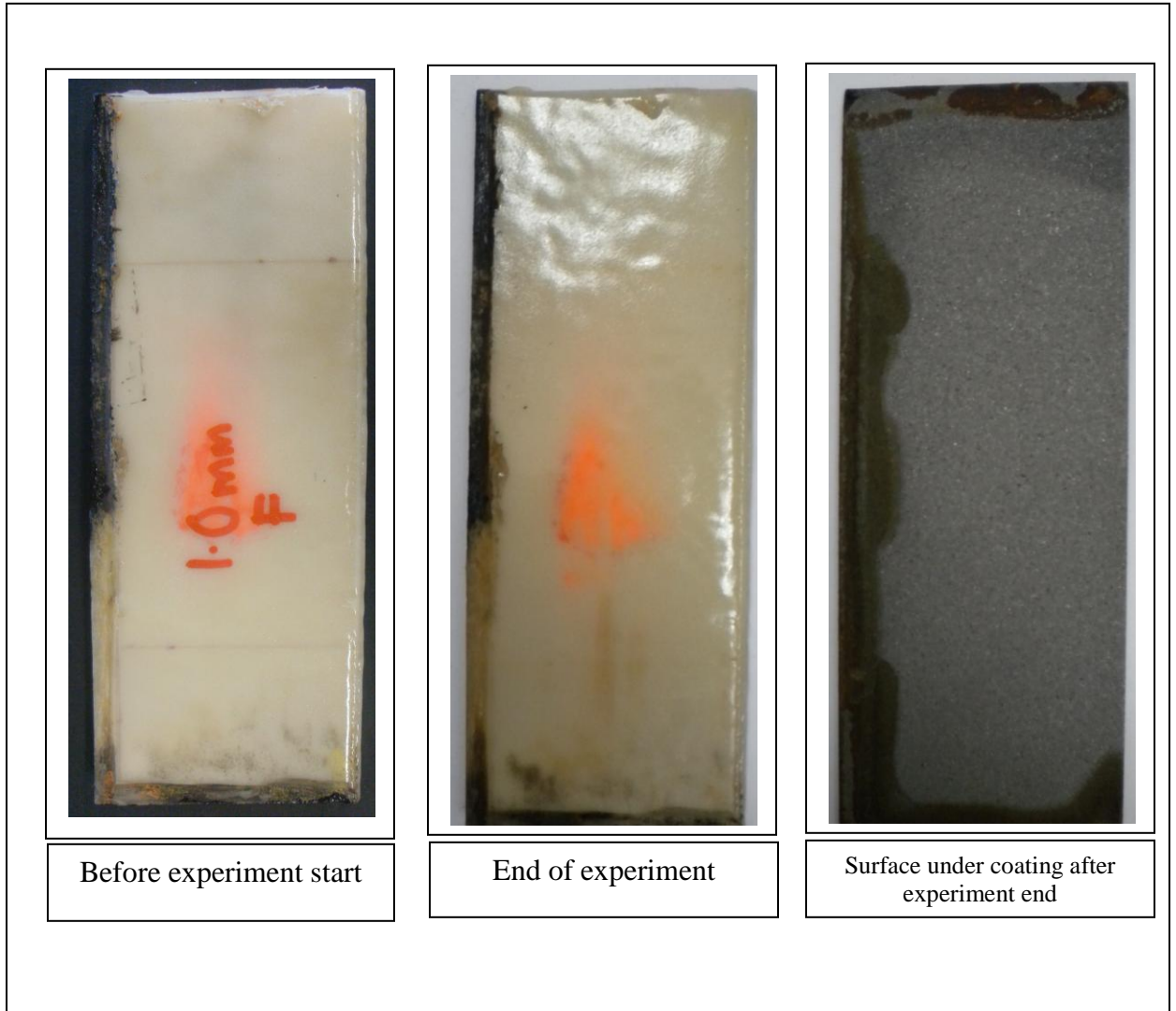**APPENDIX 1(d)**: APPEARANCE OF THE SAMPLES FOR APPLIED POTENTIALTESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATING - Samples for Applied Potential Testing – 1500 mV
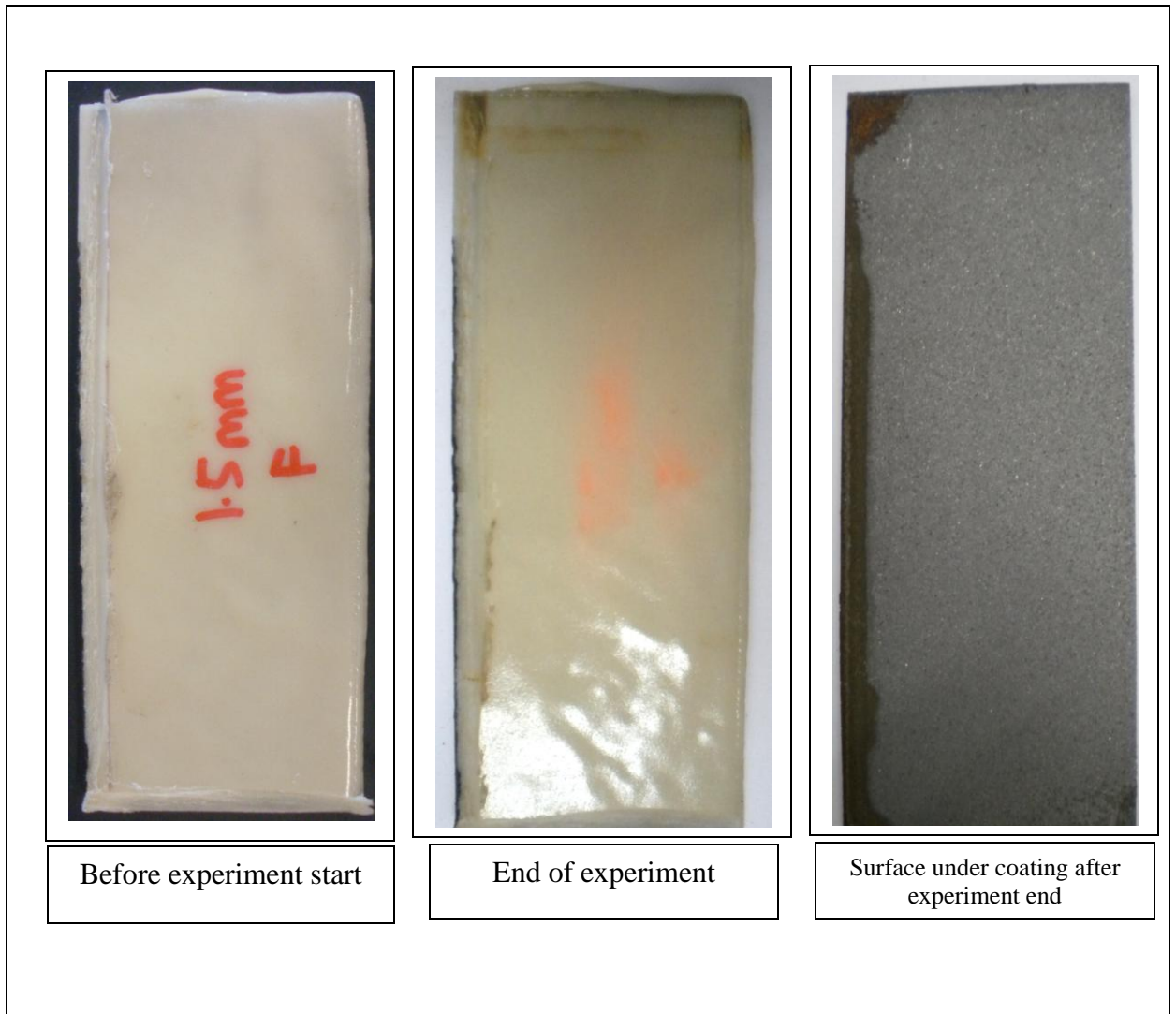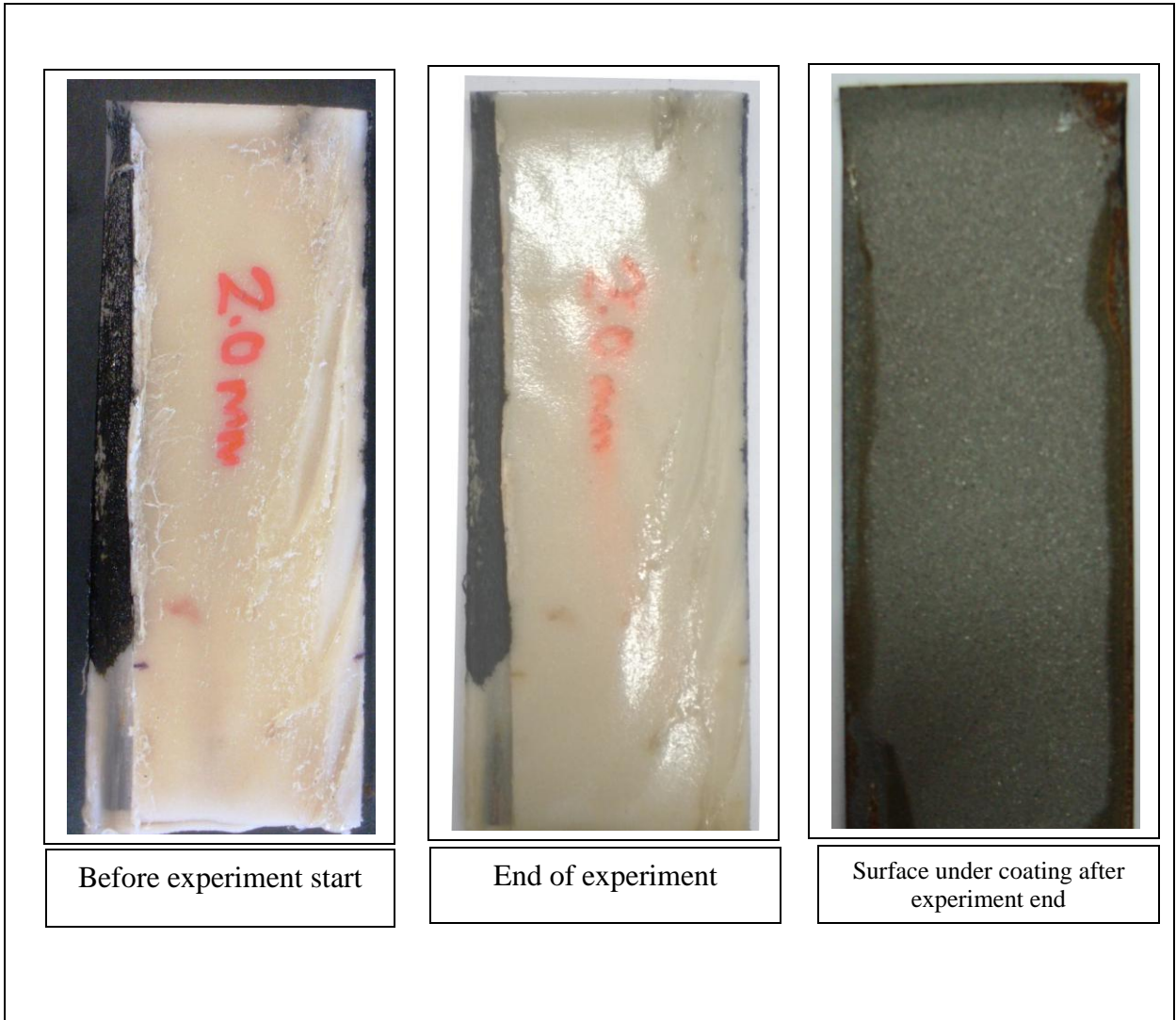


| Before experiment start | End of experiment | Surface under coating after experiment end |
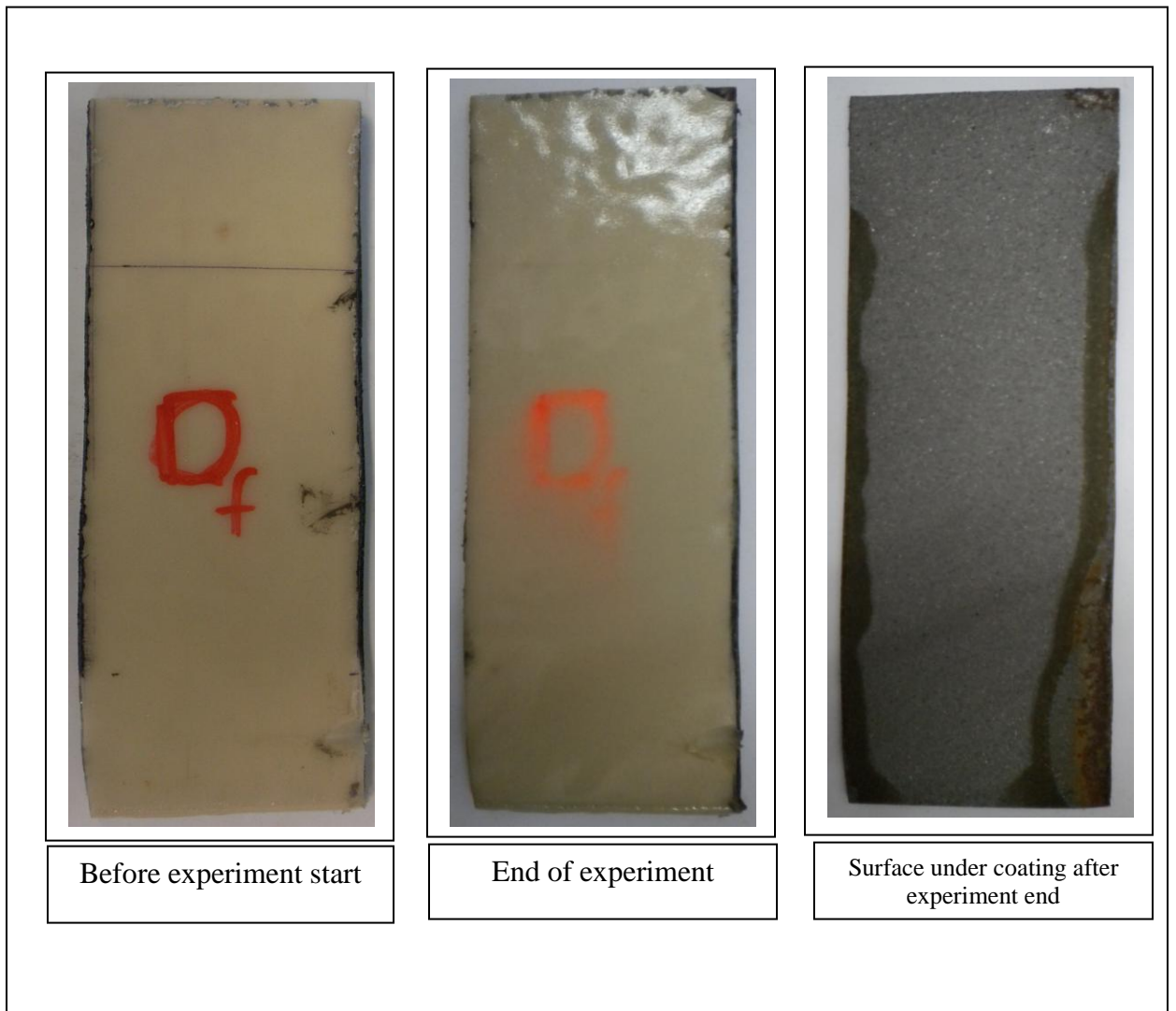
**APPENDIX 2(a):** APPEARANCE OF THE SAMPLES FOR COATING THICKNESS TESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATIN - Sample for Coating Thickness Testing – 1.0 mm



| Before experiment start | End of experiment | Surface under coating after experiment end |

**APPENDIX 2(b)**: APPEARANCE OF THE SAMPLES FOR COATING THICKNESS TESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATIN - Sample for Coating Thickness Testing – 1.5 mm



| Before experiment start | End of experiment | Surface under coating after experiment end |

**APPENDIX 2(c)**: APPEARANCE OF THE SAMPLES FOR COATING THICKNESS TESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATIN - Sample for Coating Thickness Testing – 2.0 mm



| Before experiment start | End of experiment | Surface under coating after experiment end |
|---|---|---|

**APPENDIX 2(d)**: APPEARANCE OF THE SAMPLES FOR COATING THICKNESS TESTING DURING INITIAL OF EXPERIMENT, END OF EXPERIMENT WITH COATING AND END OF EXPERIMENT WITHOUT COATIN - Sample for Coating Thickness Testing – 2.5 mm



| Before experiment start | End of experiment | Surface under coating after experiment end |
|---|---|---|