

# **Effect of Surface Roughness on Adhesion and Corrosion Properties for Metal Surface**

by

**Ahmad Shahizan Bin Shamshuddin**

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the requirement for the  
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**Bandar Seri Iskandar**

**31750**

**Perak Darul Ridzuan**

**CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the

Mechanical Engineering Programme

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Approved by,



**(MR KAMAL ARIFF BIN ZAINAL ABIDIN)**

**UNIVERSITI TEKNOLOGI PETRONAS**

**TRONOH, PERAK**

**August 2011**

## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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AHMAD SHAHIZAN BIN SHAMSHUDDIN

## **ABSTRACT**

This report highlights the research work done by the author for the final year project entitled "Effect of Surface Roughness on Adhesion and Corrosion Properties for Metal Surface". The aspects that are covered in this report include the introduction of the project, literature review, the methodology used in achieving the objective of the project as well as the project planning and milestone, results and discussion, conclusion and recommendation. The objective of this project is to attain the clear correlation between the effect of various surface roughness towards the adhesion and corrosion properties of the coating process applied on the metal surface. The methodology part contains project research process flow and project planning with Gantt chart as the attachment. The discussion part will explain on the discovery from research and the way forward of the project. The study recover that there is a need to improve the coating system for the manufacturing purpose in the future. The conclusion consists of the overall conclusions and recommendations regarding the project.

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

An organic coating composition is described, which can be used to enrich the surface region of a metal-based substrate. Many paints, coatings and high performance organic coatings have been developed as a need to protect equipment from environmental damage. Of prime importance in the development of protective coatings was the industry that produces most of the basic ingredients from which most synthetic resins were developed.

Surface roughness is the measure of the finer surface irregularities in the surface texture. These are the result of the manufacturing process employed to create the surface. Surface roughness, Ra is rated as the arithmetic average deviation of the surface valleys and peaks expressed in micro inches or micro meters. ISO standards use the term CLA (Centre Line Average). Both are interpreted identical.

The ability of a manufacturing operation to produce a specific surface roughness depends on many factors. For example, in end mill cutting the final surface depends on the rotational speed of the end mill cutter, the velocity of transverse, the rate of feed, the amount and type of lubrication at the point of cutting, and the mechanical properties of pieces being machined. A small change in any of the above factors can have a significant effect on the surface produce. <sup>[1]</sup>

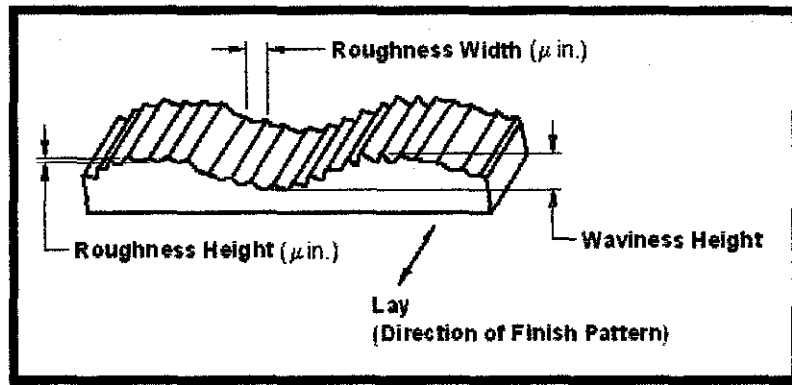


Figure 1: Surface Roughness

## 1.2 Problem Statement

Inappropriate surface preparation which is the substrate surface is not sufficiently prepared for the coating process. This may include cleaning, chemical pretreatment or surface roughening. [2]

Roughness is usually not well known to be taken care of; but it is difficult and expensive to control in manufacturing. Decreasing the roughness of a surface will usually increase exponentially its manufacturing costs. This often results in a trade-off between the manufacturing cost of a component and its performance in application.

## 1.3 Objective and Scope of Study

The main objectives of this study are:

1. Attain the clear correlation between the effect of various surface roughness towards the adhesion and corrosion properties of the coating process applied on the metal surface.
2. Record and analyze the physical condition of the coating after undergone the accelerated corrosive environment.

Scope of work of this research is to experiment and compare the effect of surface roughness measured on different metal surface to the adhesion and corrosion properties of the applied oil modified alkyl-based enamel coating on the surface. The metal plate used is aluminium sized 20 x 70 x 4.5 mm. All the experiment will be carried out in the laboratory. The real-environment conditions will be replaced by the using of the Salt-Spray Corrosion Chamber for an accelerated corrosive environment.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2. INTRODUCTION**

This study is regarding some major field that very important to be understood in order to complete the research. Some of the main areas in this study are Surface Roughness, Coating Material, Profilometer, Salt Fog Test and Scratch Test.

##### **2.1 Surface Roughness**

Roughness is a measure of the texture of a surface. It is quantified by the vertical deviations of a real surface from its ideal form. If these deviations are large, the surface is rough; if they are small the surface is smooth. Roughness is typically considered to be high frequency, short wavelength component of a measured surface.

Roughness plays an important role in determining how real object will interact with environment. Rough surface usually wear more quickly and have higher friction coefficients than smooth surfaces. Roughness is often a good predictor of the performance of a mechanical component, since irregularities in the surface may form nucleation sites for cracks or corrosion. Roughness may be measured using contact or non-contact methods. Contact methods involve dragging a measurement stylus across the surface. <sup>[4]</sup>

## **2.2 Coating Material**

Corrosion resistant coatings protect metal components against degradation due to moisture, salt spray, oxidation or exposure to a variety of environmental or industrial chemicals. Coating materials are applied in a thin film to provide protection or decoration to a surface. Most of films are thin in comparison to the work piece. In order to achieve the desired characteristics from the thin film, the coating material formulation must be carefully considered in relation to the part characteristics, surface preparation, application technique and curing method. The correct combination of components and process steps can lead to a film that provides long-lasting beauty and defense against the elements. <sup>[15]</sup>

## **2.3 Profilometer**

Profilometer is a measuring instrument used to measure a surface's profile, in order to quantify its roughness. Vertical resolution is usually in the nanometer level, though lateral resolution is usually poorer.

Contact profilometer is a diamond stylus is moved vertically in contact with a sample and then moved laterally across the sample for a specified distance and specified contact force. A profilometer can measure small surface variations in vertical stylus displacement as a function of position. A typical profilometer can measure small vertical features ranging in height from 10 nanometers to 1 millimeter. The height position of the diamond stylus generates an analog signal which is converted into a digital signal stored, analyzed and displayed. The radius of diamond stylus ranges from 20 nanometers to 25  $\mu\text{m}$ , and the horizontal resolution is controlled by the scan speed and data signal sampling rate. The stylus tracking force can range from less than 1 to 50 milligrams. <sup>[6]</sup>

#### Advantages of contact profilometers:

- **Acceptance:** Most of the world's surface finish standards are written for contact profilometers. To follow the prescribed methodology, this type of Profilometer is often required.
- **Surface Independence:** Contacting the surface is often an advantage in dirty environments where non-contact methods can end up measuring surface contaminants instead of the surface itself. However, because the stylus is in contact with the surface, this method is not sensitive to surface reflectance or color.
- **Resolution:** The stylus tip radius can be as small as 20 nanometers, significantly better than white-light optical profiling.
- **Direct Technique:** No modeling required. <sup>[6]</sup>

#### **2.4 Salt Fog Test**

Salt Fog Testing is typically performed on coated or painted samples for marine, automotive, and military equipment. Salt Fog Testing is also an excellent way to test the permeability of coating and seals. Salt spray test is an accelerated corrosion test that produces a corrosive attack to the coated samples in order to predict its suitability in use as a protective finish. The apparatus for testing consists of a closed testing chamber, where a salted solution is sprayed by means of a nozzle. This produces a corroding environment in a chamber and thus, parts in it are attacked under this severe corroding atmosphere. Chamber construction, testing procedure and testing parameters are standardized under national and international standards, such as ASTM B 117 and ISO 9227. These standards describe the necessary information to carry out this test; testing parameters such as temperature, air pressure of the sprayed solution, preparation of the spraying solution, concentration, pH, etc. the method of coating application on the surface will vary.

The apparatus for testing consists of a closed testing chamber, where a salted solution is sprayed by means of a nozzle. This produces a corroding environment in the chamber and thus, parts in it are attacked under this severe corroding atmosphere. Typical volumes of these chambers are of 15 cubic feet because for historical reasons that was the smallest volume accepted by ASTM B 117, since the 90's there is no request about volume in ASTM, ISO recommends that the chamber should not be smaller than 200 liters in order to receive an acceptable amount of test samples, chambers are available from sizes as small as 9.3 cu ft (260 L) up to 2058 cubic feet (58300 L), most common machines range from 15 to 160 cubic feet (420 - 4500 L). Tests performed with a solution of NaCl. [7]

## **2.5 Scratch Test**

Scratch Test is a new method in determining the adhesion strength of a coating. During the scratch, the stage moves in the X-direction and probe remains stationary while applying a controlled load on the specimen. The load is applied by cantilever system. The three load modes include constant, incremental, and progressive loads. In a progressive load mode, the load of failure or adhesion strength is at the load where the probe first eliminates the coating. The specimen must be flat with a length and width preferably between 0.5 inch and 1.25 inches. On the other hand the coating must have a roughness less than 5 $\mu$ m. Difference preparation in the surface of the substrate and application procedure of the coating will alter results.

However, there are some challenges for using scratch test for testing such as there are no ASTM standards on how fast, load rate and length to scratch the surface of the coated specimen. Then, the determination of adhesion strength of the coatings is difficult for a scratch test because the failure point of each coating has different characteristics of failure during a progressive load scratch. [8]

Scratch tests were performed using a CSM instruments with a spherical micro-indentor used in progressive mode. For spherical contact geometry, the imposed

effective strain depends on the depth of penetration and linearly proportional to the ratio of the radius of efficient contact,  $r$ , to the radius of the spherical geometry,  $R$ .<sup>[9]</sup>



# CHAPTER 3

## METHODOLOGY

### 3.1 Gantt Chart

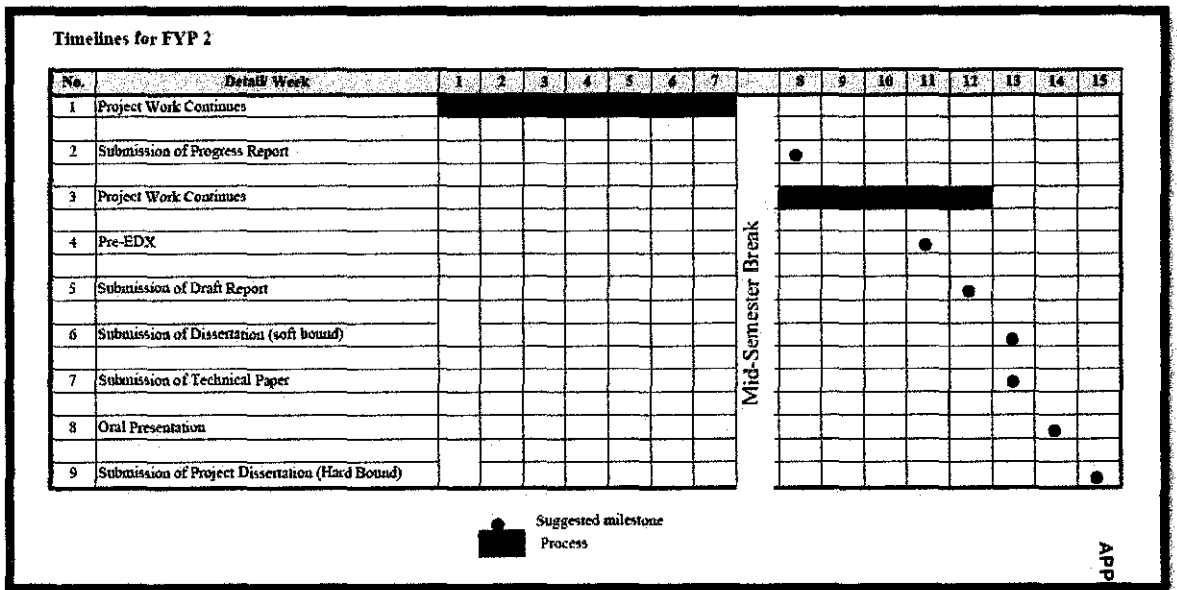
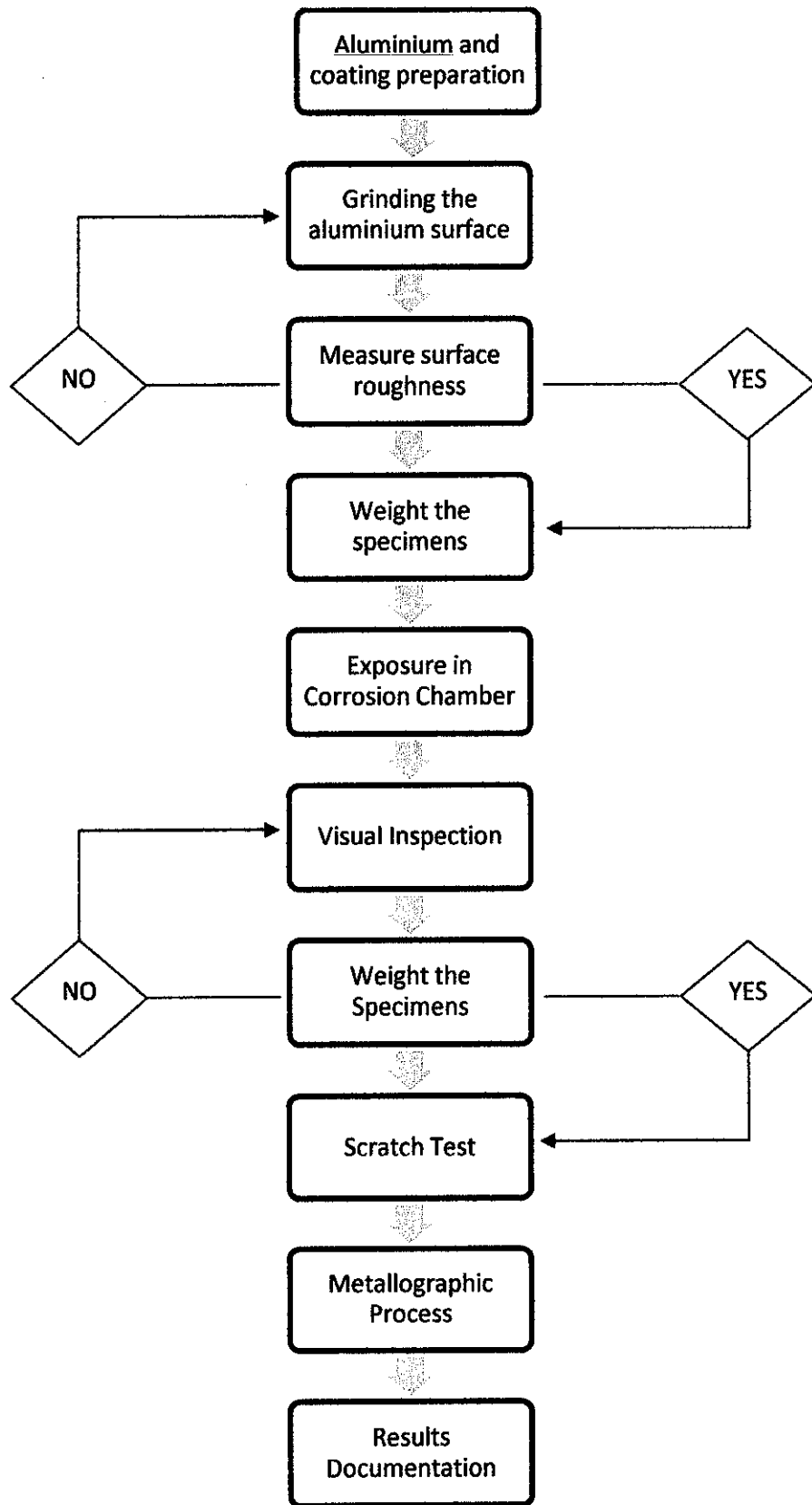


Chart 1: Project Gantt chart

### 3.2 Project Activities



### 3.3 Activities Descriptions

#### 3.3.1 Metal and Coating Preparation

This stage is to find the metal and paint as the coating system in this project. In this study, the type of metal used is Aluminium. The size is determined that way in order to make later stage of the research easier to handle without damaging the specimens. The metal is then cut into 70 x 20 x 4.5 mm size.

Paint that has been chosen for the research is International Interthane 990 formulated for interior and exterior metal and wood surface. This type of paint is chosen due to its availability and the quality that it offers.

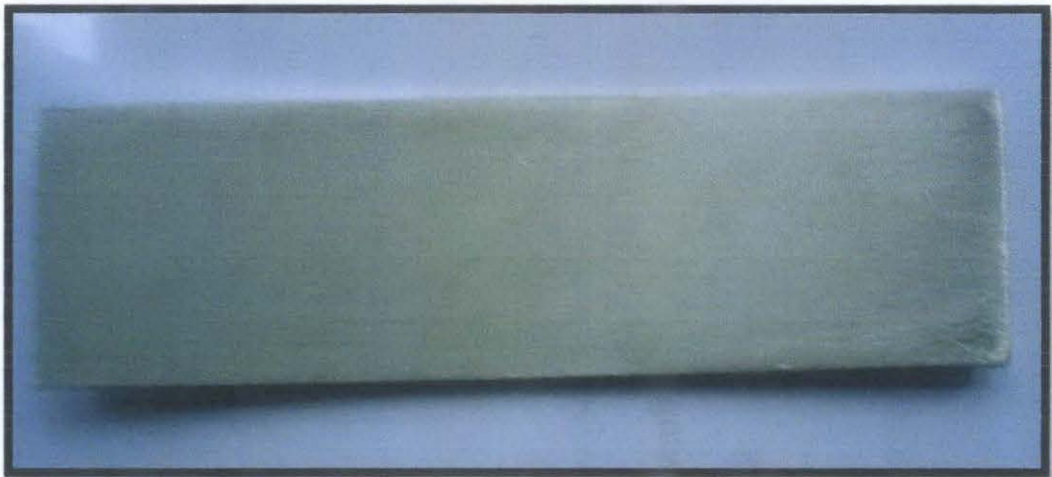


Figure 2: Prepared Samples

#### 3.3.2 Surface Grinding

Specimen that has been cut is then polished using Polish and Grinded Machine using different Grit Paper. Three samples have been prepared. These three samples were prepared to have different surface roughness from one another. These samples were marked as Smooth, Medium, and Rough. Grit paper that has been used was Grit 80 for rough surface, Grit 280 for medium and Grit 600 for smooth.



Figure 3: Example of Grit Paper

Rough surface sample is polished using only one grit paper which is Grit 80, but for medium surface the sample is first polished with Grit 80 and then Grit 280, and the same thing goes for smooth surface sample. Time taken for each polishing is roughly 15 minutes for Smooth, 10 minutes for Medium, and 5 minutes for Rough as each grit paper use 5 minutes with constant speed throughout all polishing processes.



Figure 4: Grinding and Polishing Machine

### 3.3.3 Measure Surface Roughness

The three samples are then measured to obtain the reading of surface roughness for each sample. The measurement is done using Profilometer to indicate the roughness different between each surface significantly with the name given to each sample. If there is any reading that unconvincing to the surfaces, polishing processes is going to be repeated again to obtain the desired reading. If the readings that had been taken are significant enough with the desired roughness, we may proceed to the next step.

### 3.3.4 Coating Specimen

Three specimens is then coat with International Interthane 990 according to the International product datasheet. Recommended paint system is according to the data sheet as well as recommended paint application method. The weight measurement of the samples is then taken before proceed to the next stage.



Figure 5: International Paint Three Coat System for Metal

### 3.3.5 Exposure to the Corrosive Environment

All samples are placed inside the corrosion chamber following the ASTM B 117 – 09 are followed throughout the process. Exposure period for the samples are eight days starting July 4<sup>th</sup> to 11<sup>th</sup>. [8]

Before the exposure, each of the samples was weight to obtain the original weight before undergone the exposure.

### 3.3.6 Weight the Samples

After taking off from the corrosion chamber, visual inspection going to be done on the sample to take note any visible changes occur towards the coated surface of the sample. Some blistering and peeling are expected to occur on the paint of the sample. Sample is not to be touched and none of the paint on the metal is removed.

The samples are then weight on the scientific balance to determine of weight loss of the metal. If the result of weight measurement is shown clear different between initial and final condition, project work proceed to the next stage. [8]

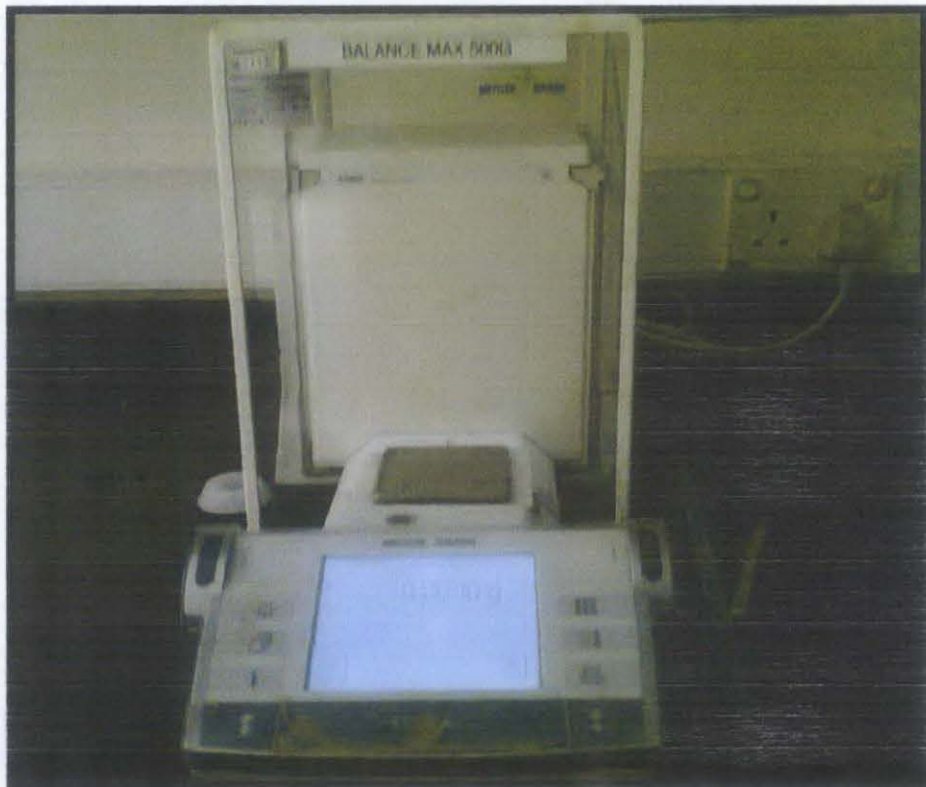


Figure 6: Scientific Balance to Weight the Samples

### 3.3.7 Scratch Test

Scratch test is one of the tests that can be performed in order to obtain the adhesion properties of the coating system. Some other test that can be done such as three bend test, tape test and bend test. Based on the availability of the test to be performed, scratch test is chosen.

The scratch tester moves a Rockwell diamond tip with a radius of 200 $\mu$ m across the coated surface of a substrate at a constant velocity while an increasing normal force is applied with constant loading rate. The scratch test introduces stresses to the interface between coating and substrate causing delamination or chipping of the coating. The critical normal force at which failure of the coating is detected is termed the critical load  $L_c$ .<sup>[9]</sup>

Scratch Test machine is dedicated instruments for characterizing the surface mechanical properties of thin films and coatings, e.g. adhesion, fracture and deformation. The tester has the ability to characterize the film-substrate system and to quantify parameters such as friction and adhesive strength, using a variety of complementary methods, makes it an invaluable tool for research, development and quality control.<sup>[10]</sup>

This test is done to get more knowledge of the adhesion properties of the coating system that has been applied with the effect of different surface roughness. Based on general understanding of adhesion properties of organic coating, the coating will fail as the load is over the adhesion properties of the coating.

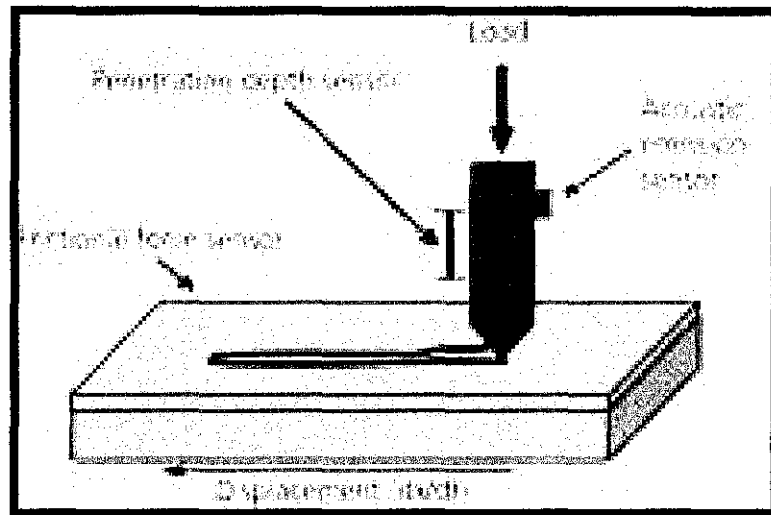


Figure 7: Diagram of Scratch Test

### 3.3.8 Metallographic Process

Metallographic is the study of a materials microstructure. Analysis of a materials microstructure aids in determining product reliability and for determining product reliability and for determining why a material failed. The basic steps for proper metallographic specimen preparation include Sectioning and Cutting, Planar Grinding, Rough Polishing, Final Polishing, Etching, Microscopic Analysis, and Hardness Analysis. <sup>[11]</sup>

### 3.3.9 Results Documentation

All the finding along the research phase is documented to generate a research paper for this research entitles Effect of Surface Roughness on Adhesion and Corrosion Properties for Metal Surface.



## CHAPTER 4

### RESULT

#### 4.1 Coating Specification

Coating system that going to be used in the research work is International tri-coat coating system International Interthane 990, International Interlac Undercoat and International Red Oxide Primer. These paints are oil modified alkyl-based enamel paint, specially formulated for interior and exterior metal and wood surfaces giving them a touch of brilliance. <sup>[12]</sup>

This gloss paint will provide a lasting beauty even in harsh weather and helps prevent fungus. It is recommended use for decoration and protection of internal and external wood and metal surfaces.

This organic paint is composing of three major parts which are:

Pigments	Mainly Titanium Dioxide, Iron Oxides, Carbon Black, Organic Pigments
Binders	Soya Bean Oil Modified Long Oil Alkyd
Thinner	White Spirit or Mineral Turpentine

Table 1: Paints Composition

Recommendation for the coating process:

Recommended Number of Coats	2-3 times
Drying time <ul style="list-style-type: none"> <li>• Touch dry</li> <li>• Hard Dry</li> </ul>	2 hours (depend on temperature and humidity) Maximum 8 hours
Recoating Intervals	16 hours

Table 2: Recommended Paint Applications

Recommended Paint System for Steel and Iron:

Sequence	Product Name	No of Coats
Primer	International Red Oxide Primer	1
Undercoat	International Interlac Undercoat/ Economy Undercoat	1
Finish	International Interthane 990	2-3

Table 3: Recommended Paint System

#### 4.2 Aluminium Selection

Aluminium is the most widely used non-ferrous metal. Pure aluminium has a low tensile strength, but when combined with thermo-mechanical processing, aluminium alloys display a marked improvement in mechanical properties, especially when tempered. Some of the many uses for aluminium metal include in transportation (automobiles, aircraft, trucks, railway cars, marine vessels, and bicycles) as sheet, tube, and castings. It also used in packaging (cans, and foil.), construction (windows, doors, siding, and building wire) and a wide range of household items, from cooking utensils to baseball bats and watches. [3]

Aluminium is chosen due to its availability and it is easy to process to produce the work piece with the size that suit the work later on. The plate cut into that size due to same size limitation to work with scratch test machine afterward.

If the metal is already cut into the size of allowable work piece to work of bench of scratch test machine, the sample will not be damage and test can be conducted as original state as possible as it is taken out from the corrosion chamber without has to be cut off.

### 4.3 Grinding Process

In order to differentiate the roughness of the surface of the samples, grinding process is done. Three samples prepared shown different surface roughness. The surface of each sample is observed under the Optical Microscope to get a clear view of the real surface.

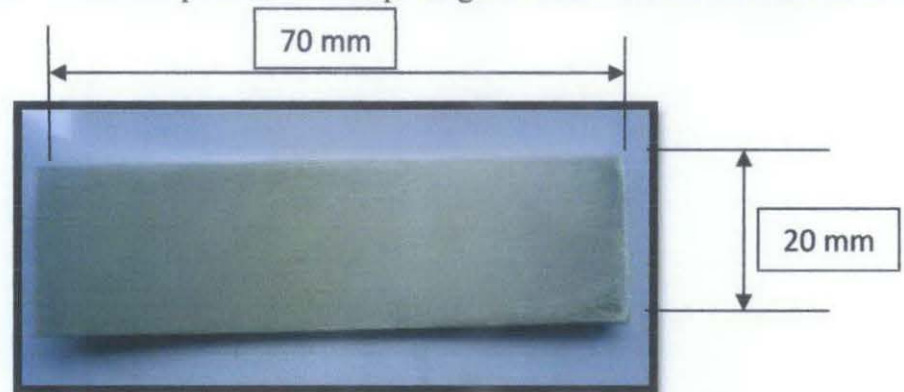


Figure 8: Metal Sample Size

Sample A is indicated as Rough surface sample, Sample B is Medium surface sample, and Sample C is Smooth surface sample.

Figures show the microscopic condition of each surface under Optical Microscope.

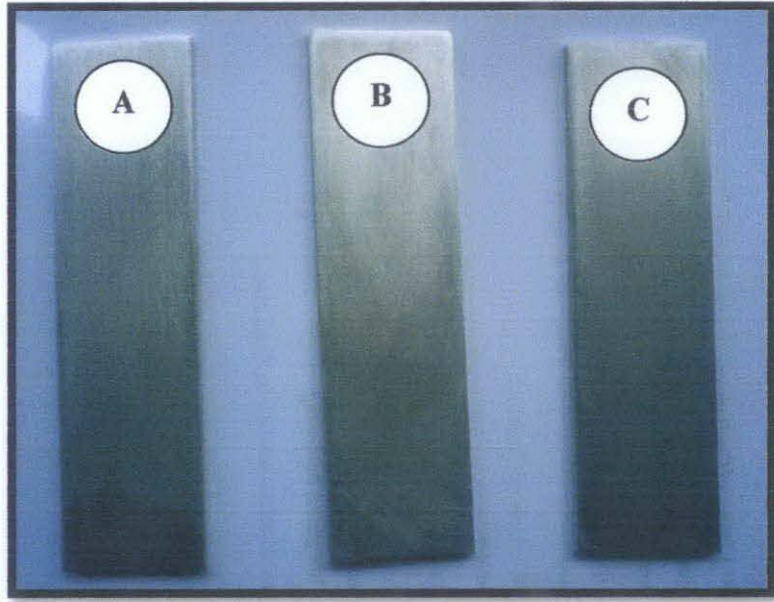


Figure 9: Three samples prepared with different surface roughness



Sample A: Rough



Sample B: Rough



Sample C: Smooth

Figure 10: Microscopic View of Each Surface at Mag 10x

#### 4.4 Profilometer

This test is to measure the exact surface roughness of each sample. After the measurement is complete, the sample will be coated according to the product data sheet from the paint manufacturer. If the measurement of the profile of the surface does not show significant difference of the each surface roughness, samples will be polished again accordingly. Mahr Perthometer basic specification and information:

- Tactile measuring system, profilometer
- Drive unit PGK-20, tracing length 20mm
- Pick-up, RFHTB-250, MFW-250
- Dynamic noise < 8nm, Static noise Rz < 2nm
- Maximum resolution vertical: 1nm, horizontal:100nm
- Static measuring force 0.6 mN, 1mN
- Evaluation software Mahr and PTB-Reference Software RPTB <sup>[11]</sup>



Figure 11: Mahr Perthometer

The reading was taken three times at different spots to detect the reading of surface roughness of the specimen, and then the average of the reading is determined and assumed as the overall surface roughness of the specimen. The reading measured shows that the surface roughnesses were different from one another as it is polished using different grit paper.

Sample	A	B	C
1 <sup>st</sup> reading	0.43	0.38	0.26
2 <sup>nd</sup> reading	0.84	0.64	0.16
3 <sup>rd</sup> reading	0.65	0.36	0.21
Average	0.64	0.46	0.21

Table 4: Surface Roughness Measurement

#### 4.5 Coat the Samples

The coating process will be done according to the method recommended in the product data sheet using paint brush. To make the coat almost identical to one another, 3 layer of third coat Gloss Finish will be apply to each sample after the application of first coat Red Oxide, and White Undercoat as second coat. The application of the coating will be in one single direction and path.

#### 4.6 Weight The Sample after Coating

All coated sample that has reached the drying time recommended is then weight for the documentation. The weight of each samples are taken into account to make sure that the deposited amount of thickness layer of coating is almost equal in every pass for all three samples. This practice is important to make sure that there is consistency of the coating applied on top of the metal surface every single time. Samples will be weighted using scientific balance with normal precautions such as take the average reading and etc.

	Raw	Primer Oxide	Undercoat	First Coat	Second Coat
Sample A	53.485	53.875	54.148	54.871	55.134
Sample B	52.748	53.154	53.454	53.478	53.84
Sample C	57.156	57.587	57.914	58.245	58.647

Table 5: Weight of Samples after Certain Process

#### 4.7 Corrosion Chamber Exposure

Corrosion chamber is acting like an accelerated environment for the corrosion process to occur. In this research Sodium Chloride (NaCl) is used for the salt solution. 5 +/- 1 part of NaCl will be dissolved in 95 part of water. The salt used shall be NaCl with not more than 0.3% by mass total impurities. [7]

A common formula to calculate the amount of salt required by mass to achieve a 5% salt solution of a known mass of water is:

$$0.053 \times \text{Mass of Water} = \text{Mass of NaCl Required}$$

Equation 1: Salt Solution Equation

The mass of water is 1 g per 1 mL. To calculate the mass of salt required in grams to mix 1L of a 5% salt solution, multiply 0.053 by 1000g. This formula yields a result of 53g of NaCl required for each liter of water to achieve a 5% salt solution by mass. [7]



Figure 12: Cyclic Corrosion Chamber

The pH of the salt solution shall be such that when atomized at 35°C, the collected solution will be in the pH range from 6.5 and 7.2. [7]

#### 4.8 Visual Inspection

As the samples are taken out from the corrosion chamber, the samples are inspected visually to identify any defects that has happen to all the samples after 200 hours of



exposure to the corrosive environment. Some of the observable changes that obviously can be seen are the yellow brownish sediment on the sample surface. As the coating system is coated in white color, it is much easier to have the contrast between the coating and the foreign elements. The coating failures that had occurred on the coating surface including cracks, peeling and pinhole.

Table below summarize all the failure that occurred for all the samples:

	A	B	C
Cracking	Yes	Yes	Yes
Peeling	Yes	No	No
Discoloration	Yes	Yes	Yes

Table 6: Samples Failure

Cracking is small breaks in coating to substrate of various geometries normally resulting from stresses due to continued polymerization or oxidation. Peeling in contrast is strips or sections of paint peel loose from the surface, usually due to moisture and/or inadequate surface preparation. In the meantime, discoloration is the looks of some yellowish, grayish, or darkening on the coated surface as a result of weathering or chemical reaction. <sup>[2]</sup>

#### 4.9 Weight the Samples

After 8 days in cyclic corrosion chamber, samples need to be weighted again to obtain the final weight of the samples. This measurement is taken to calculate the percentage of weight different for each step for the whole process.

The samples is weighted in three condition which are, at first it is weight directly after taking out from the cyclic corrosion chamber, secondly the cleaning using soft cloth and the thirdly using Ultrasonic Cleaner. These cleaning processes ensure that the foreign elements are taken out from the coated surface and the final weight of the samples and the reading during final weighting is valid enough to do more research on the founding.

Sample	After Exposure	First Cleaning	UT Cleaning
A	54.974	54.387	53.687
B	53.456	53.247	52.478
C	58.478	58.264	57.814

Table 7: Weight of the Sample after Cleaning Process

#### 4.10 Scratch Test

The typical scratch tester has three methods of detecting coating failure; a load cell to measure the change in friction, acoustic emission or observation of the scratch channel using an attached optical microscope. The best scratch adhesion testers use all three methods of coating failure detection. The intensity of the acoustic emission is dependent on the type of coating failure during the adhesion test e.g. cracking, chipping (cohesive failure) and delamination (adhesive failure). It is therefore important to observe the coating failure after the adhesion test using an optical microscope to confirm the critical load. <sup>[9]</sup>

##### 4.10.1 Features of the Scratch Test Machine

- Proven method to quantify adhesion of coatings
- Acoustic Emission, Frictional Force, Penetration Depth and Optical Observation
- Unique force feedback actuator
- Wide range of different indenters
- Very high throughput and reproducibility
- Handling of large samples (up to 300mm)
- Works for both hard and soft materials
- Wear testing in multi pass mode
- Automated optical microscope inspection
- Industrial platform for quality control <sup>[10]</sup>

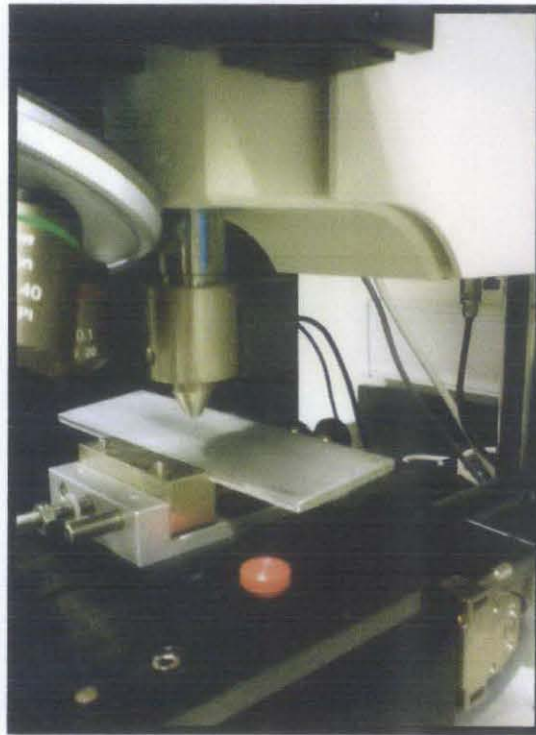
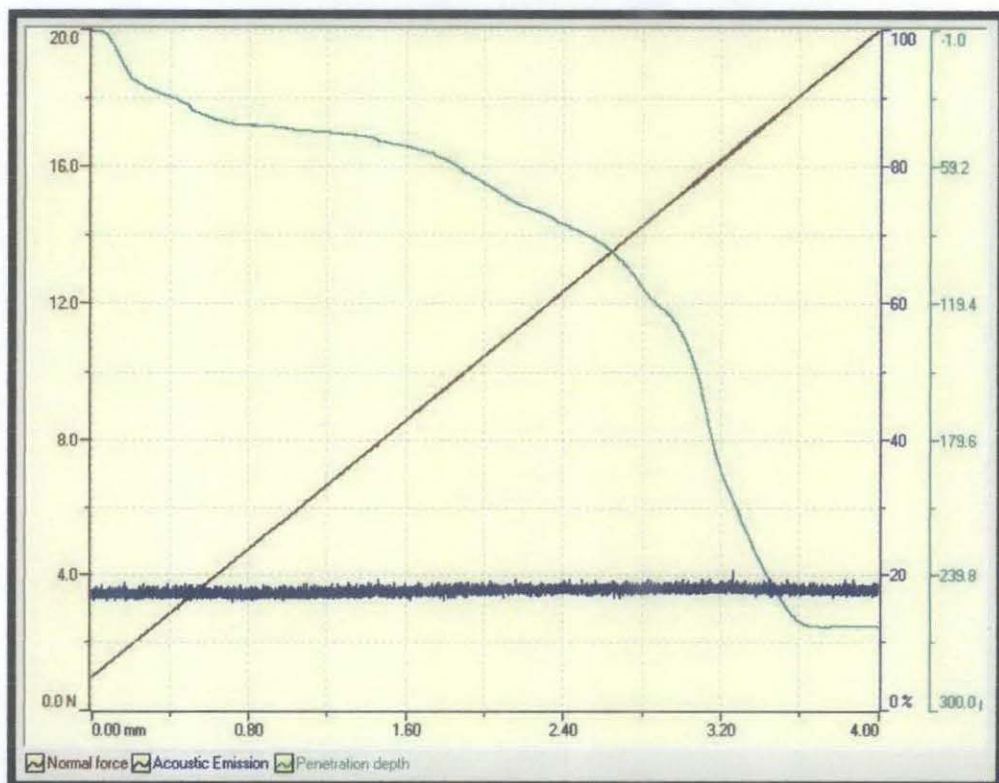
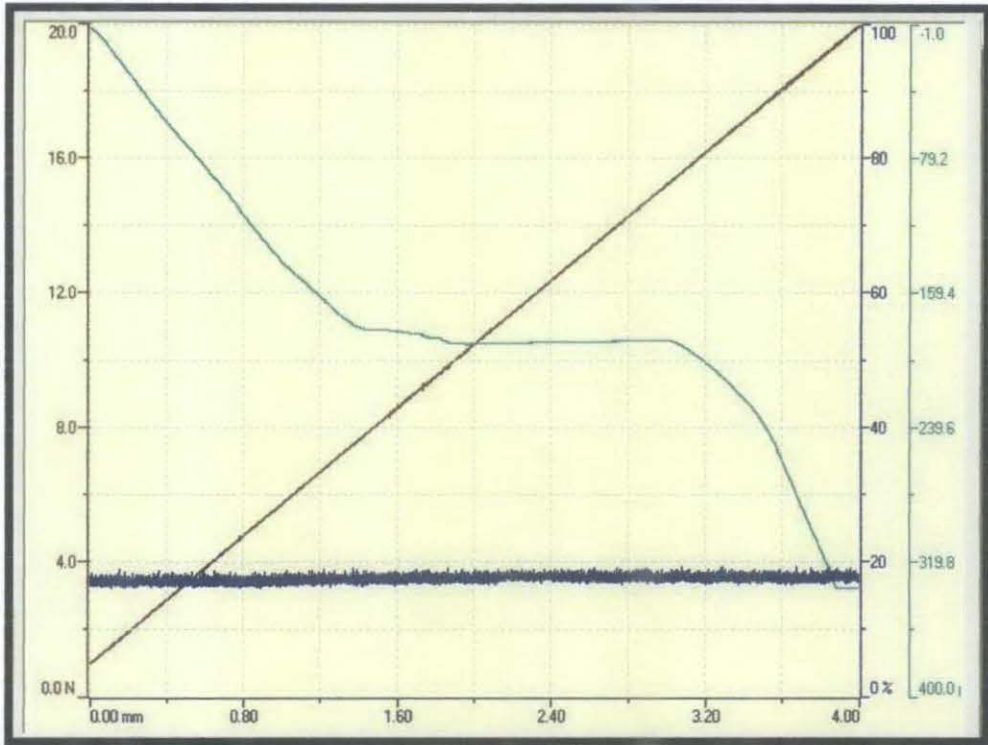


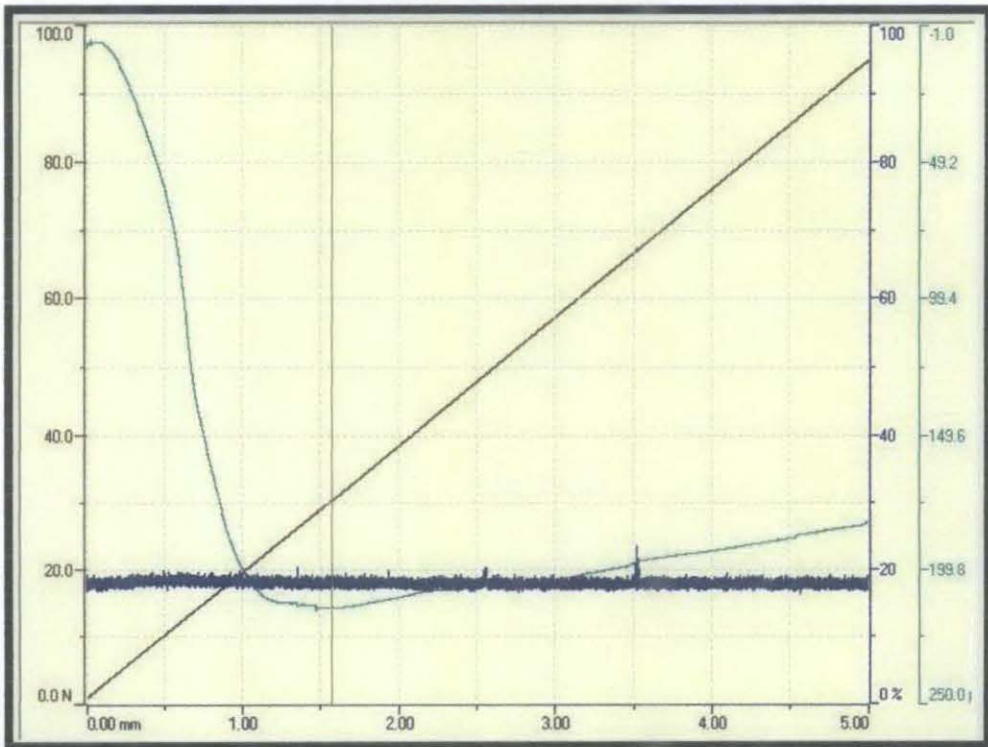
Figure 13: Sample Being Test by Scratch Test



Graph 1: Sample A



Graph 2: Sample B



Graph 3: Sample C

## **4.11 Metallographic Process**

Method that going to be practiced during metallographic are:

### **4.11.1 Sectioning and Cutting**

Proper sectioning is required to minimize damage, which may alter the microstructure and produce false metallographic characterization. Proper cutting requires the correct selection of abrasive type, bonding, and size; as well as proper cutting speed, load and coolant. <sup>[11]</sup>

Cutting and section has to be done properly to avoid any peeling and of the coating system which would affect the result and finding.

### **4.11.2 Mounting**

The mounting operation accomplishes three functions which is to protects the specimens edge and maintains the integrity of materials surface features, to fills voids in porous materials and improves handling of irregular shaped samples.

For metal, compression mounting is widely used. Phenolics are popular because they are low cost, whereas the diallyl phthalates and epoxy resins find applications where edge retention and harder mounts are required. The acrylic compression mounting compounds are used because they have excellent clarity. <sup>[11]</sup>



Figure 14: Automatic Mounting Machine

#### **4.11.3 Planar Grinding**

Grinding is required to planarize the specimen and reduce the damage created by sectioning. The planar grinding step is accomplished by decreasing the abrasive grit or particle size sequentially to obtain surface finishes that are ready for polishing. Care must be taken to avoid being too abrasive in this step, and creating greater specimen damage than produces during cutting. <sup>[11]</sup>

#### **4.11.4 Rough Polishing**

The purpose of the rough polishing step is to remove the damage produced during cutting and planar grinding. Proper rough polishing will maintain specimen flatness and retain all inclusions or secondary phases. By eliminating the previous damage and maintaining the micro structural integrity of the specimen at this step, a minimal amount of time should be required to remove the cosmetic damage at final polishing step.

Rough polishing is accomplished primarily with diamond abrasive ranging from 9 micron down to 1 micron diamond. Polycrystalline diamond because of its multiple and small cutting edges, produces high cut rates with minimal surface damage, therefore it is the recommended diamond abrasive for metallographic rough polishing on low napped polishing cloths. <sup>[11]</sup>

#### **4.11.5 Final Polishing**

The purpose of final polishing is to remove only surface damage. It should not be used to remove any damage remaining from cutting and planar grinding. If the damage from these steps is not complete, the rough polishing step should be repeated or continued. <sup>[11]</sup>

#### **4.11.6 Etching**

The purpose of etching is to optically enhance microstructural features such as grain size and phase features. Etching selectively alters these microstructural features based on composition, stress, or crystal structure. The most common technique for etching is selective chemical etching and numerous formulations have been used over the years. Other techniques such as molten salt, electrolytic, thermal and plasma etching have also found specialized applications,

Chemical etching selectively attacks specific microstructural features. It generally consists of a mixture of acids or bases with oxidizing or reducing agents. For more technical information on selective chemical etching consult corrosion books which discuss the relationship between pH and Eh (oxidation/reduction potentials), often known as Eh-pH diagrams or Pourbaix diagrams. <sup>[11]</sup>

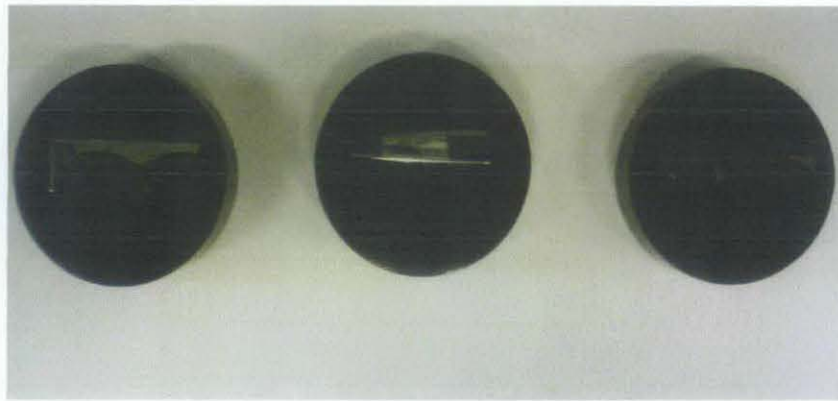


Figure 15: Samples after being mounted

#### 4.12 Microscopic Analysis

Optical Microscope is used to observe the condition of the microstructure and the coating layer on the metal.

Result obtain from the measurement shows that the thickness is almost precise from one to another. The differences were in acceptable range and the different was not too big.

	Sample A ( $\mu\text{m}$ )	Sample B ( $\mu\text{m}$ )	Sample C ( $\mu\text{m}$ )
Red Oxide Primer	45	58	45
Undercoat	84	94	81
First Coat	56	46	53
Second Coat	41	54	42

Table 8: Thickness of the Coating Measured Under Microscope



## **CHAPTER 5**

### **DISCUSSION**

#### **5.1 Aluminium**

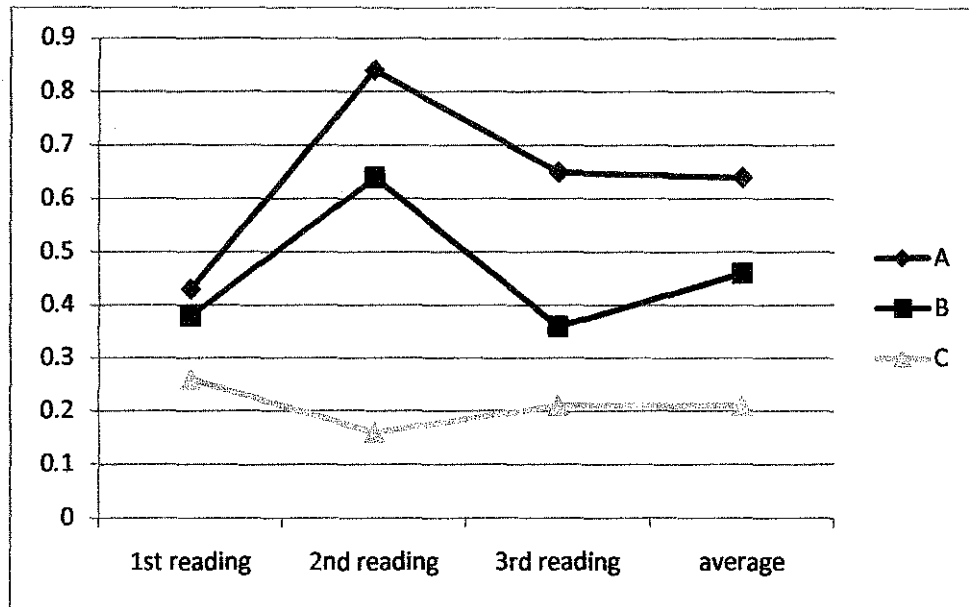
Aluminium plate is sized 70 x 20 x 4.5 mm. The metal is cut into that size to ensure that later stage of experiment will not be affected by its excessive size. Size of plate is about to fit the testing bed of Scratch Test Machine which the maximum width of allowable work piece on the testing bed is about 1.5 inch width.

So, the specimen is prepared earlier to fit the testing bed. Sizing the specimens that already fit to the testing bed will avoid cutting and sectioning the specimen after the exposure in the Corrosion Chamber. Cutting and sectioning the specimen after the exposure might affect the condition of the coating system of the specimen. Some defect that would occur due to cutting and sectioning are cracking and peeling which then lead to the early failure of the coating system before endure the scratch test.

#### **5.2 Grinding Process**

The grit paper is chosen with different number to demonstrate the significant different of the surface roughness after and grinding process done. The entire specimen is grinded using the same speed, 150 rpm for 10 minutes for each paper individually.

Sample A was grind with grit No 80, Sample B was grind with grit No. 80 and grit No. 280, meanwhile Sample C was grinded 3 times using No. 80, No. 280 and finally No.600.



Graph 4: Sample's Surface Roughness

From the chart, it is noticeable that each sample has its own surface roughness patterns that differ from one another. For each sample, the reading is taken at different spot which lead to the different of reading of the roughness. Based from these three reading for each samples, the average of the reading is taken as the sample's surface roughness. The roughness reading is 5 mm for each specimen.

This practice can avoid the same reading is taken on continuous pattern on the surface but the reading is taken adjacent to one another. The average of the readings will provide enough amount of information required to take into account as the surface roughness of the metal.

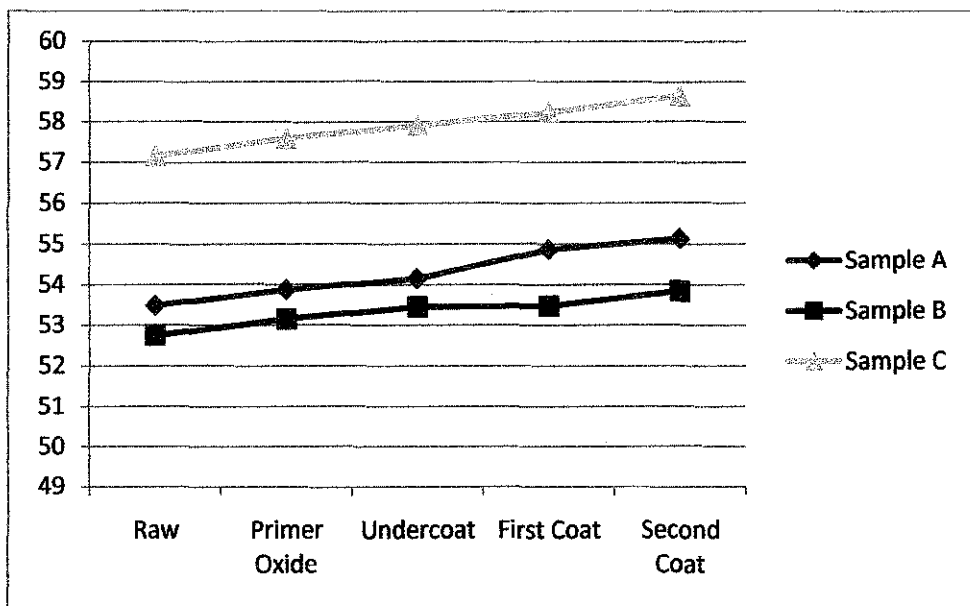
Notice that for Sample A, there is a highest measurement taken about more than  $0.8\mu\text{m}$  recorded. This may due to lack of surface contact towards the SiC Grit paper during grinding process. The condition is preferable for this study as it is concerning with the variation of the surface roughness. It shows that on that particular rough metal surface sample the variation of peak and valley present there.

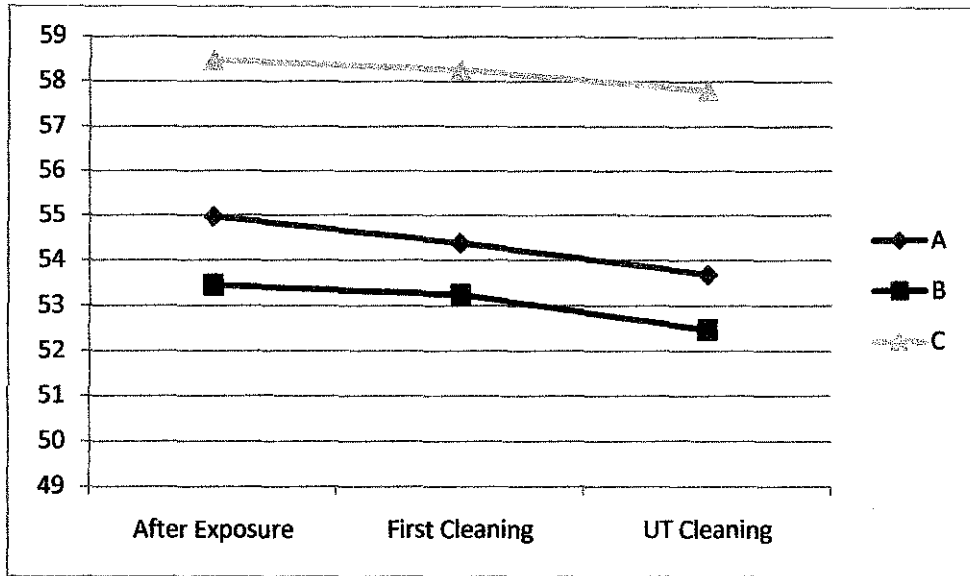
On the other hand, sample B and Sample C shows a very small different of variation from three reading which mean the measurement of the surface roughness is consistent

and precise with the average surface roughness taken. Although Sample A has a wide variation from first reading to the second reading, the first reading and third time reading is taken and recorded with small measurement variation from one to another.

### 5.3 Weight the Samples

For this research, the method is to determine the preciseness and consistency of each coating process is by the weight measuring method. Using this method, we can determine the amount of coating being applied and deposited on the metal surface every single time.





Graph 5: Samples Weight Curve

To observe the consistency of deposited layer coating the percentage different of single coating is calculated using the equation:

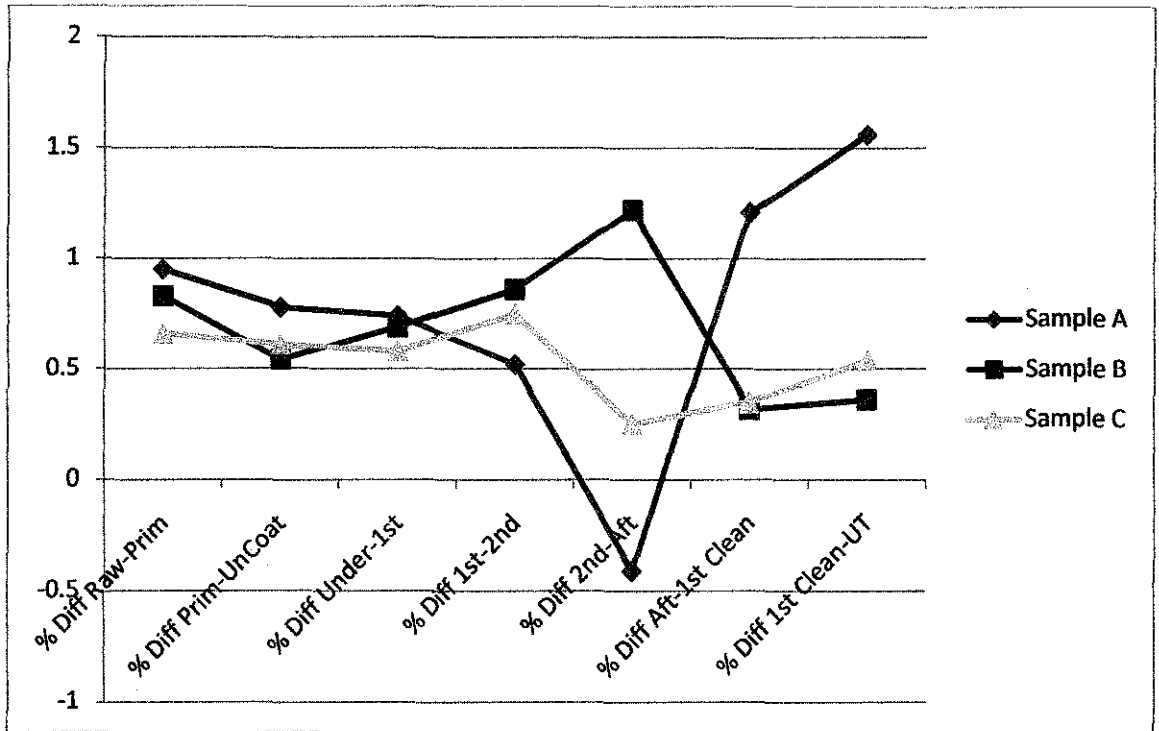
$$\% \text{ different} = \frac{|\text{Current Weight} - \text{Previous Weight}|}{|\text{Previous Weight}|}$$

Equation 2: Coating Percentage Different

	% Diff Raw-Prim	% Diff Prim-UnCoat	% Diff Under-1st
Sample A	0.95	0.78	0.74
Sample B	0.83	0.54	0.69
Sample C	0.66	0.61	0.58

% Diff 1st-2nd	% Diff 2 <sup>nd</sup> -Aft	% Diff Aft-1st Clean	% Diff 1 <sup>st</sup> Clean-UT
0.52	-0.41	1.21	1.56
0.86	1.22	0.32	0.36
0.75	0.25	0.35	0.54

Table 9: Percentage Weight Different of the Samples



Graph 6: Scatter Plot of Samples Percentage Different

As this study is using the sample's weight to measure the consistency of the applied coating, it is noticed that during the process of coating the samples, percentage different of the applied coating is almost uniform and consistency with one another. From the scatter plot above, we can say that the average percentage different is about 0.75%.

From the scatter plot, it is noticeable that Sample A and Sample C have almost identical physical plot meanwhile Sample B have slightly different from those two. Sample B has one inclined point taken for the percentage different for the second coat weight and the weight taken after one week exposure in corrosion chamber.

This is might be the result of the corrosion and other sediments forming on the bare surface of the metal such as water or air trapped inside the corrosion area as the samples is taken to the scientific balance straight away after being removed from the corrosion chamber. No cleaning is involved for this process therefore no foreign material or corrosion precipitate is removed from the surface.

For Sample A and C, there are significant different percentage of the sample's weight during the surface measurement of the sample after first cleaning and the freshly taken out sample from the corrosion chamber. This may due to the loss of the contaminants, sediment or any foreign material that attached to the sample during the exposure.

This first cleaning is the process of removing noticeable and physical contaminant that is precipitated on the surface. For this purpose, simple hand tool such as wire brush, and scrapper gently used to remove all the foreign material that precipitated on the surface. Then the samples are dried using hair drier to remove the entire water particle on the surface in order to keep the surface dry.

#### 5.4 Coating

Apart from surface preparation, the actual coating application is the most visible and important of the coating process itself.

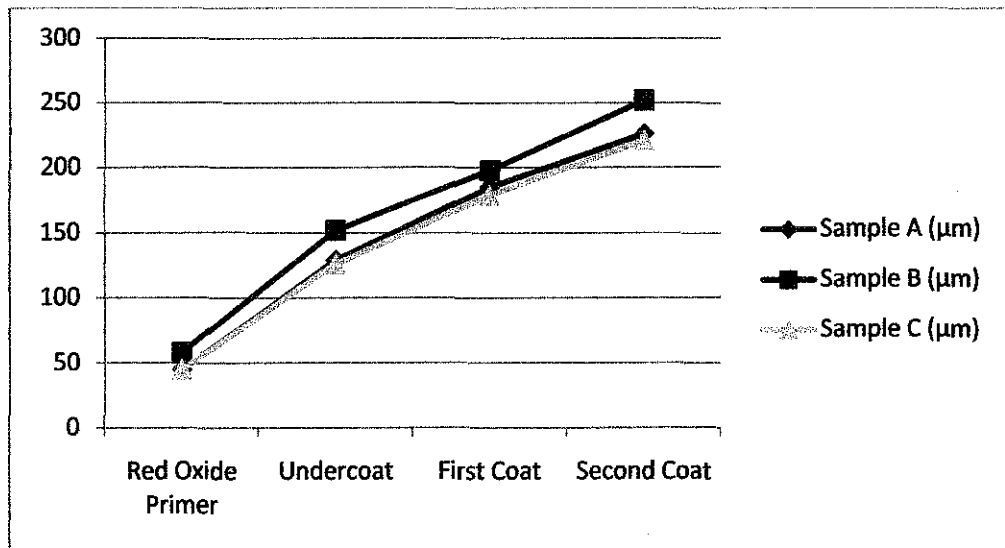
For brush purpose, the brush should be dipped about one and half of its bristle length into the paint. The bristle tips need to be brushed lightly against the side of the container to prevent dripping, and as fully a loaded a brush as possible should be maintained. This will result in a more even coating film and help ensure thorough wetting of the metal surface.

Brushing is more efficient than spraying for working paint into depressed irregularities, pits or crevices. Care should be taken to ensure that the coating is not brushed out too thin, particularly on projections and corners.

	Sample A ( $\mu\text{m}$ )	Sample B ( $\mu\text{m}$ )	Sample C ( $\mu\text{m}$ )
Red Oxide Primer	45	58	45
Undercoat	129	152	126
First Coat	185	198	179

Second Coat	226	252	221
-------------	-----	-----	-----

Table 10: Collective Thickness of Coated Sample



Graph 7: Collective Thickness of Coated Sample Curve

From the curve, it is noticeable that the thickest coating applied is on Sample B measured about 252  $\mu\text{m}$ , and the least thick is on Sample C measured about 221  $\mu\text{m}$ . The curve has shown no major different between those three samples.

- The percentage different of Sample A to Sample B = 7.6%.
- Percentage different between Sample B to Sample C = 18.2%.
- Percentage different between Sample A to Sample C = 11.5%.
- Average percentage different between these three samples = 12.4%.

### 5.5 Visual Inspection

Sample A with surface roughness 0.64  $\mu\text{m}$  shows some momentous defect such as discoloration, peeling and cracking on the metal surface after being exposed for 8 days in cyclic corrosion chamber. Meanwhile, Sample B and Sample C only show the sign of cracking and discoloration without any indication of peeling found.

This situation might be the result of roughest surface of Sample A compared to the other samples. Rough surface will affect the coating adhesion strength on the metal surface. The adhesion properties of the coating material rely on the roughness of the metal surface. This condition might also be one of the results if the surface preparation is not performed adequately to overcome this problem as one of the affected areas of surface preparation is surface roughness.

On Sample A, peeling only occurred at one side of the metal at two spot along the edge of the sample. First defect was measured and read the length at 25.2 mm and the other location measured to be 30.8 mm. This giving the coating adhered length to be around 14 mm only out of total length which is 70 mm. Only 20% of the length of the applied coating is adhered and the other 80% is completely peeled off from the metal surface.

Sample A has four different spots with cracks shows that the coating is very weak and easily influenced by the corrosive environment. All the cracks are originated at the corner or the edge of the samples with none of them originated from the center which indicates that the edge and corner area should not be taken for granted during surface preparation. The coating system provide greater adhesion towards the better surface roughness sample such as Sample B and C and corrosion can be reduce with only two cracking found each.

## **5.6 Scratch Test**

Scratch test is performed using CSM Instrument and it is conducted in the progressive loading test. Three reading was taken precaution measure. The orientation of the samples also varies from one run to the other. Meanwhile the location for the test is randomly chosen from the coated surface.

Scratch Test is conducted after the sample is dried overnight inside the moisture absorber to make sure the samples are completely dry. After underwent Scratch Test,



Sample A demonstrate further cracking and severe peeling of the coating system which initiated from the scratch test area. This also may due to the rough surface of the sample.

## **CHAPTER 6**

### **RECOMMENDATION**

Further study on this topic can be done in many ways possible:

#### **6.1 Focusing on One Major Factor**

Further study can be conducted if more concentrate on one major result either adhesion properties or corrosion properties. We may focus more on specific major and can allow more understanding and development of the process. For the adhesion properties, the research only involves scratch test alone to determine the adhesion properties of the samples although we got several type of adhesion testing that could be performed such as tape test and bend test.

Meanwhile, the corrosion properties for the organic coating, beside visual inspection, corrosion penetration rate could be obtain if allocation time for cyclic corrosion chamber exposure is long enough to have the failed the coating system. Further inspection also could be done using modern gadget such as Ultrasonic Test to obtain the coating thickness should it loss some.

#### **6.2 Overall Coating of Samples**

For this study, the sample is coated on one surface only and the rest were left bare and uncoated. Having full coated samples will reduce the possibility of edge peeling of the coating system towards the sample surface. However, having full coated sample has

another problem which is the entire surface of sample must have almost equal surface roughness from another to obtain the precise result. When the samples coated on every faces of the samples, the scratch also need to be done on every single faces, therefore using thicker samples is possible with dimension 70 mm x 20 mm x 20 mm. If the sample is cut in the size, scratch test can be conducted easily.

### **6.3 Longer Exposure Time**

Exposure time in the cyclic corrosion chamber for this study is 8 days continuously without stop. In order to obtain greater visible result, the samples shall be exposed for longer period such as one whole month and meet and follow the requirement from ASME B117. This practice will allow the failure, if there is any will appear more significantly on the sample's surface.

### **6.4 Coating Thickness Measuring**

Coating thickness measuring is the process of measuring the wet and dry coating thickness using specific film thickness gauge. However for this study, weight different is use instead. Using film thickness gauge is better than measure the weight of the coated sample because, each of the coating layers applied on metal surface can be determined its thickness during the coating is wet and dry. The information gathered from the measurement can be used to calculate overall coating thickness applied on the metal surface straight away.

## CHAPTER 7

### CONCLUSION

Based on the research work that has been completed, the flow of this project is constructed firmly and structurally. Coating is very ideal for decorative and corrosion protection purpose but it is really related with the surface preparation done before the process. By varies the method of surface preparation, the surface roughness of the metal is varied and produce different result from one another in term of adhesiveness to the organics coating applied.

Direct relationship is clearly obtained from the study that has been completed that the one of the concern of having surface perfection is roughen of the metal surface. Different level of surface roughness affected the adhesion properties of the coating system. For example surface roughness of Rough Sample,  $R_a = 0.64 \mu\text{m}$  has the Critical Load about 3 N. Meanwhile, Smooth Sample with surface roughness,  $R_a = 0.21 \mu\text{m}$  has Critical Load about 25 N which represent the adhesion strength of the organic coating applied.

Different level of surface roughness affected the adhesion properties of the coating system. The corrosion rate also depends on the adhesion properties of the coating material towards the surface. Rough surface provide less adhesion between coating material and metal surface. It was found out that the coating on Sample A has peel off about 56 mm at the edge of the sample which is about 80% from sample's total length after the exposure to corrosive environment. Evident from the scratch testing on the

other hand shows that Sample A had peel off in the brittle manner and continues to crack further more.

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Designation: B 117 – 03

## Standard Practice for Operating Salt Spray (Fog) Apparatus<sup>1</sup>

This standard is issued under the fixed designation B 117; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the Department of Defense.*

### 1. Scope

1.1 This practice covers the apparatus, procedure, and conditions required to create and maintain the salt spray (fog) test environment. Suitable apparatus which may be used is described in Appendix X1.

1.2 This practice does not prescribe the type of test specimen or exposure periods to be used for a specific product, nor the interpretation to be given to the results.

1.3 The values stated in SI units are to be regarded as standard. The inch-pound units in parentheses are provided for information and may be approximate.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

B 368 Method for Copper-Accelerated Acetic Acid-Salt Spray (Fog) Testing (CASS Test)<sup>2</sup>

D 609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products<sup>3</sup>

D 1193 Specification for Reagent Water<sup>4</sup>

D 1654 Test Method for Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments<sup>3</sup>

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode<sup>5</sup>

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method<sup>6</sup>

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee G01 on Corrosion of Metals and is the direct responsibility of Subcommittee G01.05 on Laboratory Corrosion Tests.

Current edition approved October 1, 2003. Published October 2003. Originally approved in 1939. Last previous edition approved in 2002 as B 117 – 02.

<sup>2</sup> Annual Book of ASTM Standards, Vol 02.05.

<sup>3</sup> Annual Book of ASTM Standards, Vol 06.01.

<sup>4</sup> Annual Book of ASTM Standards, Vol 11.01.

<sup>5</sup> Annual Book of ASTM Standards, Vol 15.05.

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.02.

### G 85 Practice for Modified Salt Spray (Fog) Testing<sup>7</sup>

### 3. Significance and Use

3.1 This practice provides a controlled corrosive environment which has been utilized to produce relative corrosion resistance information for specimens of metals and coated metals exposed in a given test chamber.

3.2 Prediction of performance in natural environments has seldom been correlated with salt spray results when used as stand alone data.

3.2.1 Correlation and extrapolation of corrosion performance based on exposure to the test environment provided by this practice are not always predictable.

3.2.2 Correlation and extrapolation should be considered only in cases where appropriate corroborating long-term atmospheric exposures have been conducted.

3.3 The reproducibility of results in the salt spray exposure is highly dependent on the type of specimens tested and the evaluation criteria selected, as well as the control of the operating variables. In any testing program, sufficient replicates should be included to establish the variability of the results. Variability has been observed when similar specimens are tested in different fog chambers even though the testing conditions are nominally similar and within the ranges specified in this practice.

### 4. Apparatus

4.1 The apparatus required for salt spray (fog) exposure consists of a fog chamber, a salt solution reservoir, a supply of suitably conditioned compressed air, one or more atomizing nozzles, specimen supports, provision for heating the chamber, and necessary means of control. The size and detailed construction of the apparatus are optional, provided the conditions obtained meet the requirements of this practice.

4.2 Drops of solution which accumulate on the ceiling or cover of the chamber shall not be permitted to fall on the specimens being exposed.

<sup>7</sup> Annual Book of ASTM Standards, Vol 03.02.



4.3 Drops of solution which fall from the specimens shall be returned to the solution reservoir for respraying.

4.4 Material of construction shall be such that it will not affect the corrosiveness of the fog.

4.5 All water used for this practice shall conform to Type IV water in Specification D 1193 (except that for this practice limits for chlorides and sodium may be ignored). This does not apply to running tap water. All other water will be referred to reagent grade.

**Test Specimens**

5.1 The type and number of test specimens to be used, as well as the criteria for the evaluation of the test results, shall be defined in the specifications covering the material or product being exposed or shall be mutually agreed upon between the purchaser and the seller.

**Preparation of Test Specimens**

5.1 Specimens shall be suitably cleaned. The cleaning method shall be optional depending on the nature of the surface and the contaminants. Care shall be taken that specimens are not recontaminated after cleaning by excessive or careless handling.

5.2 Specimens for the evaluation of paints and other organic coatings shall be prepared in accordance with applicable specification(s) for the material(s) being exposed, or as agreed upon between the purchaser and the supplier. Otherwise, the test specimens shall consist of steel meeting the requirements of Practice D 609 and shall be cleaned and prepared for coating in accordance with the applicable procedure of Practice D 609.

5.3 Specimens coated with paints or nonmetallic coatings shall not be cleaned or handled excessively prior to test.

5.4 Whenever it is desired to determine the development of corrosion from an abraded area in the paint or organic coating, a scratch or scribed line shall be made through the coating with a sharp instrument so as to expose the underlying metal before testing. The conditions of making the scratch shall be as defined in Test Method D 1654, unless otherwise agreed upon between the purchaser and the seller.

5.5 Unless otherwise specified, the cut edges of plated, coated, or duplex materials and areas containing identification marks or in contact with the racks or supports shall be protected with a suitable coating stable under the conditions of practice.

NOTE 1—Should it be desirable to cut test specimens from parts or from plated, painted, or otherwise coated steel sheet, the cut edges shall be protected by coating them with paint, wax, tape, or other effective media that the development of a galvanic effect between such edges and the uncoated plated or otherwise coated metal surfaces, is prevented.

**Position of Specimens During Exposure**

6.1 The position of the specimens in the salt spray chamber during the test shall be such that the following conditions are met:

6.1.1 Unless otherwise specified, the specimens shall be supported or suspended between 15 and 30° from the vertical, preferably parallel to the principal direction of flow of fog through the chamber, based upon the dominant surface being tested.

7.1.2 The specimens shall not contact each other or any metallic material or any material capable of acting as a wick.

7.1.3 Each specimen shall be placed to permit unencumbered exposure to the fog.

7.1.4 Salt solution from one specimen shall not drip on any other specimen.

NOTE 2—Suitable materials for the construction or coating of racks and supports are glass, rubber, plastic, or suitably coated wood. Bare metal shall not be used. Specimens shall preferably be supported from the bottom or the side. Slotted wooden strips are suitable for the support of flat panels. Suspension from glass hooks or waxed string may be used as long as the specified position of the specimens is obtained, if necessary by means of secondary support at the bottom of the specimens.

**8. Salt Solution**

8.1 The salt solution shall be prepared by dissolving 5 ± 1 parts by mass of sodium chloride in 95 parts of water conforming to Type IV water in Specification D 1193 (except that for this practice limits for chlorides and sodium may be ignored). Careful attention should be given to the chemical content of the salt. The salt used shall be sodium chloride with not more than 0.3 % by mass of total impurities. Halides (Bromide, Fluoride, and Iodide) other than Chloride shall constitute less than 0.1 % by mass of the salt content. Copper content shall be less than 0.3 ppm by mass. Sodium chloride containing anti-caking agents shall not be used because such agents may act as corrosion inhibitors. See Table 1 for a listing of these impurity restrictions. Upon agreement between the purchaser and the seller, analysis may be required and limits established for elements or compounds not specified in the chemical composition given above.

**TABLE 1 Maximum Allowable Limits for Impurity Levels in Sodium Chloride<sup>A,B</sup>**

Impurity Description	Allowable Amount
Total Impurities	≤ 0.3 %
Halides (Bromide, Fluoride and Iodide) excluding Chloride	≤ 0.1 %
Copper	< 0.3 ppm
Anti-caking Agents	0.0 %

<sup>A</sup> A common formula used to calculate the amount of salt required by mass to achieve a 5 % salt solution of a known mass of water is:

$$.053 \times \text{Mass of Water} = \text{Mass of NaCl required}$$

The mass of water is 1 g per 1 mL. To calculate the mass of salt required in grams to mix 1 L of a 5 % salt solution, multiply .053 by 1000 g (35.27 oz., the mass of 1 L of water). This formula yields a result of 53 g (1.87 oz.) of NaCl required for each liter of water to achieve a 5 % salt solution by mass.

The 0.053 multiplier for the sodium chloride used above is derived by the following:

1000 g (mass of a full L of water) divided by 0.95 (water is only 95 % of the total mixture by mass) yields 1053 g. This 1053 g is the total mass of the mixture of one L of water with a 5 % sodium chloride concentration. 1053 g minus the original weight of the L of water, 1000 g, yields 53 g for the weight of the sodium chloride. 53 g of total sodium chloride divided by the original 1000 g of water yields a 0.053 multiplier for the sodium chloride.

As an example, to mix the equivalent of 200 L (52.83 gal) of 5 % sodium chloride solution, mix 10.6 kg (23.37 lb) of sodium chloride into 200 L (52.83 gal) of water. 200 L of water weighs 200,000 g. 200,000 g of water x .053 (sodium chloride multiplier) = 10,600 g of sodium chloride, or 10.6 kg.

<sup>B</sup> In order to ensure that the proper salt concentration was achieved when mixing the solution, it is recommended that the solution be checked with either a salimeter hydrometer or specific gravity hydrometer. When using a salimeter hydrometer, the measurement should be between 4 and 6 % at 25°C (77°F). When using a specific gravity hydrometer, the measurement should be between 1.0255 and 1.0400 at 25°C (77°F).

8.2 The pH of the salt solution shall be such that when atomized at 35°C (95°F) the collected solution will be in the pH range from 6.5 to 7.2 (Note 3). Before the solution is atomized it shall be free of suspended solids (Note 4). The pH measurement shall be made at 25°C (77°F) using a suitable glass pH-sensing electrode, reference electrode, and pH meter system in accordance with Test Method E 70.

NOTE 3—Temperature affects the pH of a salt solution prepared from water saturated with carbon dioxide at room temperature and pH adjustment may be made by the following three methods:

(1) When the pH of a salt solution is adjusted at room temperature, and atomized at 35°C (95°F), the pH of the collected solution will be higher than the original solution due to the loss of carbon dioxide at the higher temperature. When the pH of the salt solution is adjusted at room temperature, it is therefore necessary to adjust it below 6.5 so the collected solution after atomizing at 35°C (95°F) will meet the pH limits of 6.5 to 7.2. Take about a 50-mL sample of the salt solution as prepared at room temperature, boil gently for 30 s, cool, and determine the pH. When the pH of the salt solution is adjusted to 6.5 to 7.2 by this procedure, the pH of the atomized and collected solution at 35°C (95°F) will come within this range.

(2) Heating the salt solution to boiling and cooling to 35°C (95°F) and maintaining it at 35°C (95°F) for approximately 48 h before adjusting the pH produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

(3) Heating the water from which the salt solution is prepared to 35°C (95°F) or above, to expel carbon dioxide, and adjusting the pH of the salt solution within the limits of 6.5 to 7.2 produces a solution the pH of which does not materially change when atomized at 35°C (95°F).

NOTE 4—The freshly prepared salt solution may be filtered or decanted before it is placed in the reservoir, or the end of the tube leading from the solution to the atomizer may be covered with a double layer of cheesecloth to prevent plugging of the nozzle.

NOTE 5—The pH can be adjusted by additions of dilute ACS reagent grade hydrochloric acid or sodium hydroxide solutions.

## 1. Air Supply

9.1 The compressed air supply to the Air Saturator Tower shall be free of grease, oil, and dirt before use by passing through well-maintained filters. (Note 6) This air should be maintained at a sufficient pressure at the base of the Air Saturator Tower to meet the suggested pressures of Table 2 at the top of the Air Saturator Tower.

NOTE 6—The air supply may be freed from oil and dirt by passing it through a suitable oil/water extractor (that is commercially available) to trap any oil from reaching the Air Saturator Tower. Many oil/water extractors have an expiration indicator, proper preventive maintenance intervals should take these into account.

9.2 The compressed air supply to the atomizer nozzle or nozzles shall be conditioned by introducing it into the bottom of a tower filled with water. A common method of introducing the air is through an air dispersion device (X1.4.1). The level of the water must be maintained automatically to ensure adequate humidification. It is common practice to maintain the temperature in this tower between 46 and 49°C (114–121°F) to offset the cooling effect of expansion to atmospheric pressure during the atomization process. Table 2 in 9.3 of this practice shows the temperature, at different pressures, that are commonly used to offset the cooling effect of expansion to atmospheric pressure.

9.3 Careful attention should be given to the relationship of tower temperature to pressure since this relationship can have

a direct impact to maintaining proper collection rates (Note 7). It is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog as listed in Table 2.

TABLE 2 Suggested Temperature and Pressure guideline for the top of the Air Saturator Tower for the operation of a test at 35°C (95°F)

Air Pressure, kPa	Temperature, °C	Air Pressure, PSI	Temperature, °F
83	46	12	114
96	47	14	117
110	48	16	119
124	49	18	121

NOTE 7—If the tower is run outside of these suggested temperature and pressure ranges to achieve proper collection rates as described in 10.2 of this practice, other means of verifying the proper corrosion rate in the chamber should be investigated, such as the use of control specimens (panels of known performance in the test conducted). It is preferred that control panels be provided that bracket the expected test specimen performance. The controls allow for the normalization of test conditions during repeated running of the test and will also allow comparisons of test results from different repeats of the same test. (Refer to Appendix X3, Evaluation of Corrosive Conditions, for mass loss procedures).

## 10. Conditions in the Salt Spray Chamber

10.1 *Temperature*—The exposure zone of the salt spray chamber shall be maintained at 35 + 1.1 – 1.7°C (95 + 2 – 3°F). Each set point and its tolerance represents an operational control point for equilibrium conditions at a single location in the cabinet which may not necessarily represent the uniformity of conditions throughout the cabinet. The temperature within the exposure zone of the closed cabinet shall be recorded (Note 8) at least twice a day at least 7 h apart (except on Saturdays, Sundays, and holidays when the salt spray test is not interrupted for exposing, rearranging, or removing test specimens or to check and replenish the solution in the reservoir).

NOTE 8—A suitable method to record the temperature is by a continuous recording device or by a thermometer which can be read from outside the closed cabinet. The recorded temperature must be obtained with the salt spray chamber closed to avoid a false low reading because of wet-bulb effect when the chamber is open.

10.2 *Atomization and Quantity of Fog*—Place at least two clean fog collectors per atomizer tower within the exposure zone so that no drops of solution will be collected from the test specimens or any other source. Position the collectors in the proximity of the test specimens, one nearest to any nozzle and the other farthest from all nozzles. A typical arrangement is shown in Fig. 1. The fog shall be such that for each 80 cm<sup>2</sup> (12.4 in.<sup>2</sup>) of horizontal collecting area, there will be collected from 1.0 to 2.0 mL of solution per hour based on an average run of at least 16 h (Note 9). The sodium chloride concentration of the collected solution shall be 5 ± 1 mass % (Notes 9–11). The pH of the collected solution shall be 6.5 to 7.2. The pH measurement shall be made as described in 8.2 (Note 3).

NOTE 9—Suitable collecting devices are glass or plastic funnels with

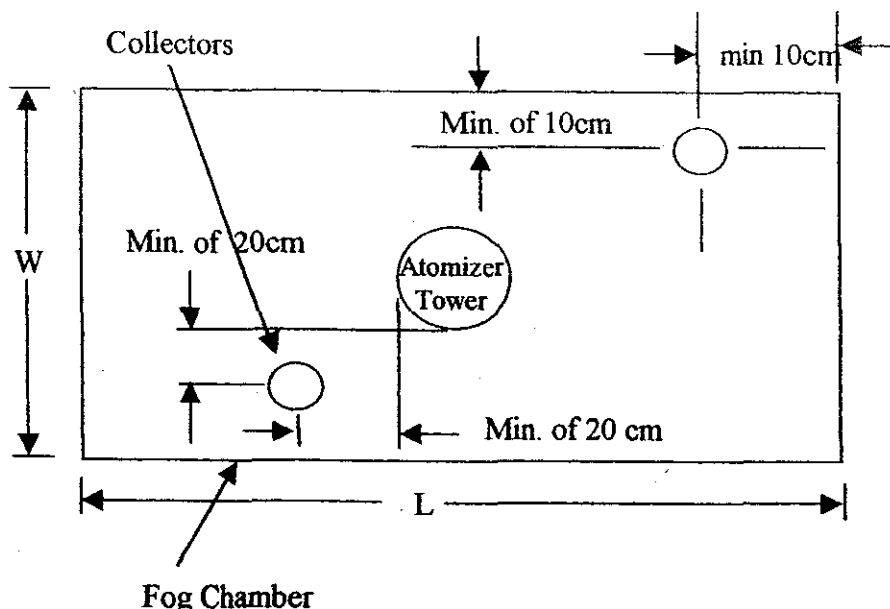


FIG. 1 Arrangement of Fog Collectors

Note—This figure shows a typical fog collector arrangement for a single atomizer tower cabinet. The same fog collector arrangement is also applicable to multiple atomizer tower and horizontal (“T” type) atomizer tower cabinet constructions as well.

stems inserted through stoppers into graduated cylinders, or crystallizing dishes. Funnels and dishes with a diameter of 10 cm (3.94 in.) have an area of about 80 cm<sup>2</sup> (12.4 in.<sup>2</sup>).

Note 10—A solution having a specific gravity of 1.0255 to 1.0400 at 20°C (77°F) will meet the concentration requirement. The sodium chloride concentration may also be determined using a suitable salinity meter (for example, utilizing a sodium ion-selective glass electrode) or volumetrically as follows. Dilute 5 mL of the collected solution to 100 mL with distilled water and mix thoroughly; pipet a 10-mL aliquot into an evaporating dish or casserole; add 40 mL of distilled water and 1 mL of 1% potassium chromate solution (chloride-free) and titrate with 0.1 N silver nitrate solution to the first appearance of a permanent red coloration. The solution that requires between 3.4 and 5.1 mL of 0.1 N silver nitrate solution will meet the concentration requirements.

Note 11—Salt solutions from 2 to 6% will give the same results, though for uniformity the limits are set at 4 to 6%.

10.3 The nozzle or nozzles shall be so directed or baffled that none of the spray can impinge directly on the test specimens.

#### 11. Continuity of Exposure

11.1 Unless otherwise specified in the specifications covering the material or product being tested, the test shall be continuous for the duration of the entire test period. Continuous operation implies that the chamber be closed and the spray generating continuously except for the short daily interruptions necessary to inspect, rearrange, or remove test specimens, to check and replenish the solution in the reservoir, and to make necessary recordings as described in Section 10. Operations shall all be so scheduled that these interruptions are held to a minimum.

#### 12. Period of Exposure

12.1 The period of exposure shall be as designated by the specifications covering the material or product being tested or mutually agreed upon between the purchaser and the seller.

Note 12—Recommended exposure periods are to be as agreed upon between the purchaser and the seller, but exposure periods of multiples of 24 h are suggested.

#### 13. Cleaning of Tested Specimens

13.1 Unless otherwise specified in the specifications covering the material or product being tested, specimens shall be treated as follows at the end of the test:

13.1.1 The specimens shall be carefully removed.

13.2 Specimens may be gently washed or dipped in clean running water not warmer than 38°C (100°F) to remove salt deposits from their surface, and then immediately dried.

#### 14. Evaluation of Results

14.1 A careful and immediate examination shall be made as required by the specifications covering the material or product being tested or by agreement between the purchaser and the seller.

#### 15. Records and Reports

15.1 The following information shall be recorded, unless otherwise prescribed in the specifications covering the material or product being tested:

15.1.1 Type of salt and water used in preparing the salt solution,

15.1.2 All readings of temperature within the exposure zone of the chamber,

15.1.3 Daily records of data obtained from each fog-collecting device including the following:

15.1.3.1 Volume of salt solution collected in millilitres per hour per 80 cm<sup>2</sup> (12.4 in.<sup>2</sup>),

15.1.3.2 Concentration or specific gravity at 35°C (95°F) of solution collected, and

15.1.3.3 pH of collected solution.

- 15.2 Type of specimen and its dimensions, or number or description of part,
- 15.3 Method of cleaning specimens before and after testing,
- 15.4 Method of supporting or suspending article in the salt spray chamber,
- 15.5 Description of protection used as required in 6.5,
- 15.6 Exposure period,
- 15.7 Interruptions in exposure, cause, and length of time, and

15.8 Results of all inspections.

NOTE 13—If any of the atomized salt solution which has not contacted the test specimens is returned to the reservoir, it is advisable to record the concentration or specific gravity of this solution also.

## 16. Keywords

16.1 controlled corrosive environment; corrosive conditions; determining mass loss; salt spray (fog) exposure

## APPENDIXES

### (Nonmandatory Information)

### XI. CONSTRUCTION OF APPARATUS

#### X1.1 Cabinets

X1.1.1 Standard salt spray cabinets are available from several suppliers, but certain pertinent accessories are required before they will function according to this practice and provide consistent control for duplication of results.

X1.1.2 The salt spray cabinet consists of the basic chamber, an air-saturator tower, a salt solution reservoir, atomizing nozzles, specimen supports, provisions for heating the chamber, and suitable controls for maintaining the desired temperature.

X1.1.3 Accessories such as a suitable adjustable baffle or central fog tower, automatic level control for the salt reservoir, and automatic level control for the air-saturator tower are pertinent parts of the apparatus.

X1.1.4 The size and shape of the cabinet shall be such that the atomization and quantity of collected solution is within the limits of this practice.

X1.1.5 The chamber shall be made of suitably inert materials such as plastic, glass, or stone, or constructed of metal and lined with impervious plastics, rubber, or epoxy-type materials or equivalent.

X1.1.6 All piping that contacts the salt solution or spray should be of inert materials such as plastic. Vent piping should be of sufficient size so that a minimum of back pressure exists and should be installed so that no solution is trapped. The exposed end of the vent pipe should be shielded from extreme air currents that may cause fluctuation of pressure or vacuum in the cabinet.

#### X1.2 Temperature Control

X1.2.1 The maintenance of temperature within the salt chamber can be accomplished by several methods. It is generally desirable to control the temperature of the surroundings of the salt spray chamber and to maintain it as stable as possible. This may be accomplished by placing the apparatus in a constant-temperature room, but may also be achieved by surrounding the basic chamber of a jacket containing water or air at a controlled temperature.

X1.2.2 The use of immersion heaters in an internal salt solution reservoir or within the chamber is detrimental where

heat losses are appreciable because of solution evaporation and radiant heat on the specimens.

#### X1.3 Spray Nozzles

X1.3.1 Satisfactory nozzles may be made of hard rubber, plastic, or other inert materials. The most commonly used type is made of plastic. Nozzles calibrated for air consumption and solution-atomized are available. The operating characteristics of a typical nozzle are given in Table X1.1.

X1.3.2 It can readily be seen that air consumption is relatively stable at the pressures normally used, but a marked reduction in solution sprayed occurs if the level of the solution is allowed to drop appreciably during the test. Thus, the level of the solution in the salt reservoir must be maintained automatically to ensure uniform fog delivery during the test.<sup>8</sup>

X1.3.3 If the nozzle selected does not atomize the salt solution into uniform droplets, it will be necessary to direct the spray at a baffle or wall to pick up the larger drops and prevent them from impinging on the test specimens. Pending a complete understanding of air-pressure effects, and so forth, it is important that the nozzle selected shall produce the desired

<sup>8</sup> A suitable device for maintaining the level of liquid in either the saturator tower or reservoir of test solution may be designed by a local engineering group, or may be purchased from manufacturers of test cabinets as an accessory.

**TABLE X1.1 Operating Characteristics of Typical Spray Nozzle**

Siphon Height, cm	Air Flow, dm <sup>3</sup> /min				Solution Consumption, cm <sup>3</sup> /h			
	Air Pressure, kPa				Air Pressure, kPa			
	34	69	103	138	34	69	103	138
10	19	26.5	31.5	36	2100	3840	4584	5256
20	19	26.5	31.5	36	636	2760	3720	4320
30	19	26.5	31.5	36	0	1380	3000	3710
40	19	26.6	31.5	36	0	780	2124	2904

Siphon Height, in.	Air Flow, L/min				Solution Consumption, mL/h			
	Air Pressure, psi				Air Pressure, psi			
	5	10	15	20	5	10	15	20
4	19	26.5	31.5	36	2100	3840	4584	5256
8	19	26.5	31.5	36	636	2760	3720	4320
12	19	26.5	31.5	36	0	1380	3000	3710
16	19	26.6	31.5	36	0	780	2124	2904

dition when operated at the air pressure selected. Nozzles not necessarily located at one end, but may be placed in the center and can also be directed vertically up through a suitable orifice.

**4 Air for Atomization**

X1.4.1 The air used for atomization must be free of grease, oil, and dirt before use by passing through well-maintained filters. Room air may be compressed, heated, humidified, and cooled in a water-sealed rotary pump if the temperature of the air is suitably controlled. Otherwise cleaned air may be introduced into the bottom of a tower filled with water through porous stone or multiple nozzles. The level of the water must be maintained automatically to ensure adequate humidification. A chamber operated in accordance with this method and Appendix X1 will have a relative humidity between 95 and 98%. Since salt solutions from 2 to 6% will give the same results (though for uniformity the limits are set at 4 to 6%), it is preferable to saturate the air at temperatures well above the chamber temperature as insurance of a wet fog. Table X1.2 shows the temperatures, at different pressures, that are required to offset the cooling effect of expansion to atmospheric pressure.

**TABLE X1.2 Temperature and Pressure Requirements for Operation of Test at 95°F**

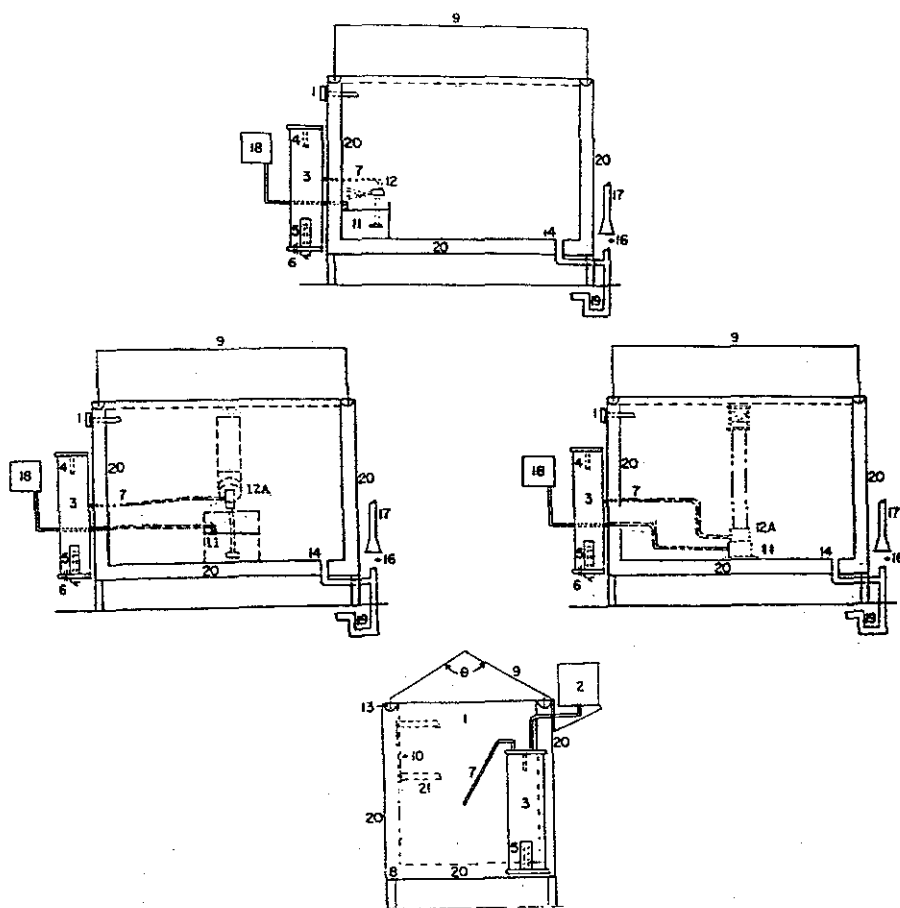
	Air Pressure, kPa			
	83	96	110	124
Temperature, °C	46	47	48	49
	Air Pressure, psi			
	12	14	16	18
Temperature, °F	114	117	119	121

X1.4.2 Experience has shown that most uniform spray chamber atmospheres are obtained by increasing the atomizing air temperature sufficiently to offset heat losses, except those that can be replaced otherwise at very low-temperature gradients.

**X1.5 Types of Construction**

X1.5.1 A modern laboratory cabinet is shown in Fig. X1.1. Walk-in chambers are usually constructed with a sloping ceiling. Suitably located and directed spray nozzles avoid ceiling accumulation and drip. Nozzles may be located at the ceiling, or 0.91 m (3 ft) from the floor directed upward at 30 to 60° over a passageway. The number of nozzles depends on type and capacity and is related to the area of the test space. An 11 to 19 L (3 to 5-gal) reservoir is required within the chamber, with the level controlled. The major features of a walk-in type cabinet, which differs significantly from the laboratory type, are illustrated in Fig. X1.2. Construction of a plastic nozzle, such as is furnished by several suppliers, is shown in Fig. X1.3.

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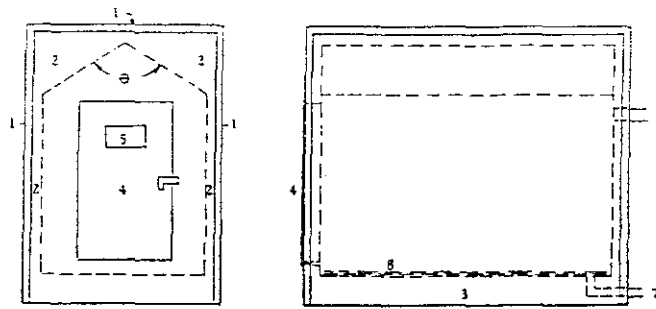


NOTE 1— $\theta$ —Angle of lid, 90 to 125°

- 1—Thermometer and thermostat for controlling heater (Item No. 8) in base
- 2—Automatic water leveling device
- 3—Humidifying tower
- 4—Automatic temperature regulator for controlling heater (Item No. 5)
- 5—Immersion heater, nonrusting
- 6—Air inlet, multiple openings
- 7—Air tube to spray nozzle
- 8—Heater in base
- 9—Hinged top, hydraulically operated, or counterbalanced
- 10—Brackets for rods supporting specimens, or test table
- 11—Internal reservoir
- 12—Spray nozzle above reservoir, suitably designed, located, and baffled
- 12A—Spray nozzle housed in dispersion tower located preferably in center of cabinet (typical examples)
- 13—Water seal
- 14—Combination drain and exhaust. Exhaust at opposite side of test space from spray nozzle (Item 12), but preferably in combination with drain, waste trap, and forced draft waste pipe (Items 16, 17, and 19).
- 16—Complete separation between forced draft waste pipe (Item 17) and combination drain and exhaust (Items 14 and 19) to avoid undesirable suction or back pressure.
- 17—Forced draft waste pipe
- 18—Automatic leveling device for reservoir
- 19—Waste trap
- 20—Air space or water jacket
- 21—Test table or rack, well below roof area

NOTE 2—This figure shows the various components including alternate arrangements of the spray nozzles and solution reservoir.

**FIG. X1.1 Typical Salt Spray Cabinet**



NOTE—The controls are the same, in general as for the smaller laboratory type cabinet (Fig. X1.1), but are sized to care for the larger cube. The chamber has the following features:

- Angle of ceiling, 90 to 125°
- Heavy insulated outer panels
- Air space
- Low-watt density heaters, or steam coils
- Single- or double-, full-opening door (refrigeration type), with inward sloping door sill
- Viewing window/s
- Inner chamber vent
- Inner chamber drain
- Duct boards on floor

FIG. X1.2 Walk-in Chamber, 1.5 by 2.4 m (5 by 8 ft) and Upward in Overall Size

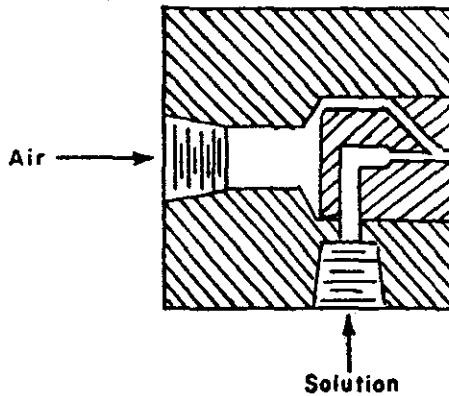


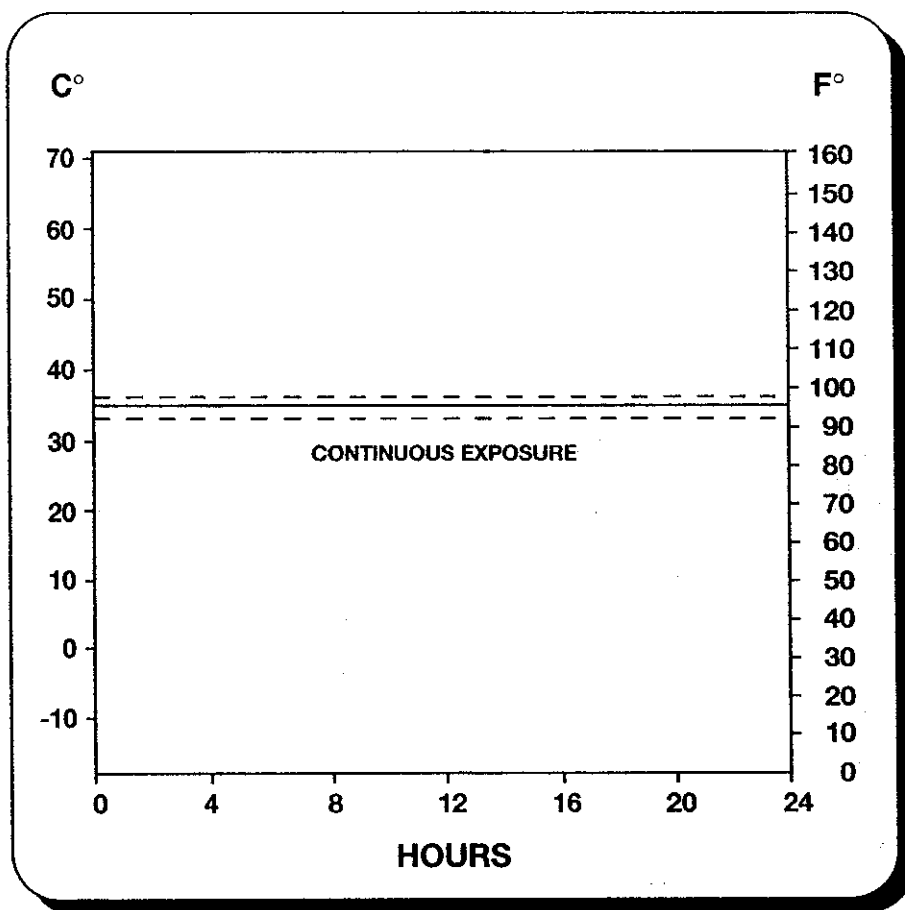
FIG. X1.3 Typical Spray Nozzle

## X2. USE OF THE SALT SPRAY (FOG) TEST IN RESEARCH

X2.1 This practice is primarily used for process qualification and quality acceptance. Regarding any new applications, it is essential to correlate the results of this practice with actual field exposure results. (See Fig. X2.1.)

X2.2 The salt spray has been used to a considerable extent for the purpose of comparing different materials or finishes. It should be noted there is usually not a direct relation between salt spray (fog) resistance and resistance to corrosion in other media, because the chemistry of the reactions, including the formation of films and their protective value, frequently varies greatly with the precise conditions encountered. Informed personnel are aware of the erratic composition of basic alloys, the possibility of wide variations in quality and thickness of plated items produced on the same racks at the same time, and the consequent need for a mathematical determination of the

number of specimens required to constitute an adequate sample for test purposes. In this connection it is well to point out that Practice B 117 is not applicable to the study or testing of decorative chromium plate (nickel-chromium) on steel or on zinc-base die castings or of cadmium plate on steel. For this purpose Method B 368 and Practice G 85 are available, which are also considered by some to be superior for comparison of chemically treated aluminum (chromated, phosphated, or anodized), although final conclusions regarding the validity of test results related to service experience have not been reached. Practice B 117 and Practice G 85 are considered to be most useful in estimating the relative behavior of closely related materials in marine atmospheres, since it simulates the basic conditions with some acceleration due to either wetness or temperature, or both.



(1) Salt Solution:  $5 \pm 1$  parts by mass of sodium chloride (NaCl) in 95 parts by mass of Specification D 1193 Type IV water.

(2) pH 6.5 to 7.2 of collected solution.

(3) The exposure zone of the salt spray chamber shall be maintained at  $35 + 1.1 - 1.7^\circ\text{C}$  ( $95 + 2 - 3^\circ\text{F}$ ). Each set point and its tolerance represents an operational control point for equilibrium conditions at a single location in the cabinet which may not necessarily represent the uniformity of conditions throughout the cabinet.

(4) Fog at a rate of 1.0 to 2.0 mL/hr per 80 cm<sup>2</sup> of horizontal collection area.

Note: Dashed chart lines indicate temperature tolerance limits.

Note: Reprinted with permission.

FIG. X2.1 Standard Practice for Operating Salt Spray (Fog) Apparatus

### X3. EVALUATION OF CORROSIVE CONDITIONS

**X3.1 General**—This appendix covers test panels and procedures for evaluating the corrosive conditions within a salt spray cabinet. The procedure involves the exposure of steel test panels and the determination of their mass losses in a specified period of time. This may be done monthly or more frequently to ensure consistent operation over time. It is also useful for correlating the corrosive conditions among different cabinets.

**X3.2 Test Panels**—The required test panels, 76 by 127 by 0.8 mm (3.0 by 5.0 by .0315 in.), are made from SAE 1008 commercial-grade cold-rolled carbon steel (UNS G10080).

**X3.3 Preparation of Panels Before Testing**—Clean panels before testing by degreasing only, so that the surfaces are free of dirt, oil, or other foreign matter that could influence the test results. After cleaning, weigh each panel on an analytical balance to the nearest 1.0 mg and record the mass.

**X3.4 Positioning of Test Panels**—Place a minimum of two weighed panels in the cabinet, with the 127-mm (5.0 in.) length supported 30° from vertical. Place the panels in the proximity of the condensate collectors. (See Section 6.)

**X3.5 Duration of Test**—Expose panels to the salt fog for 48 to 168 h.

**X3.6 Cleaning of Test Panels After Exposure**—After removal of the panels from the cabinet, rinse each panel immediately with running tap water to remove salt, and rinse in reagent grade water (see Specification D 1193, Type IV). Chemically clean each panel for 10 min at 20 to 25°C in a fresh solution prepared as follows:

Mix 1000 mL of hydrochloric acid (sp gr 1.19) with 1000 mL reagent grade water (D 1193, Type IV) and add 10 g of hexamethylene tetramine. After cleaning, rinse each panel with reagent grade water (Type IV) and dry (see 13.2).



X3.7 *Determining Mass Loss*—Immediately after drying, determine the mass loss by reweighing and subtracting panel mass after exposure from its original mass.

TABLE X3.1 Repeatability Statistics

NOTE—Based on two replicates in every test run. No. = number of different salt spray cabinets in test program;  $r$  = 95 % repeatability limits,  $C_v = S_r/\text{avg}$ , coefficient of variation, %; and  $S_r$  = repeatability standard deviations, g.

Materials	Test Duration, h	Average Mass Loss, g	$S_r$ , g	$C_v$ , %	$r$ , g	No.
QP1	48	0.8170	0.0588	7.20	0.1646	12
QP1	96	1.5347	0.1048	7.28	0.2934	12
QP1	168	2.5996	0.2498	9.61	0.6994	12
AP	48	0.7787	0.0403	5.17	0.1128	10
AP	96	1.4094	0.0923	6.55	0.2584	10
AP	168	2.4309	0.1594	6.56	0.4463	10
QP2	48	0.8566	0.0686	8.01	0.1921	5
QP2	96	1.5720	0.0976	6.21	0.2733	5
QP2	168	2.7600	0.2588	9.38	0.7246	5

TABLE X3.2 Reproducibility Statistics

NOTE—No. = number of different salt spray cabinets in test program;  $R$  = 95 % reproducibility limits, g;  $C_v = S_R/\text{avg}$ , coefficient of variation, %; and  $S_R$  = reproducibility standard deviation, g.

Materials	Test Duration, h	Average Mass Loss, g	$S_R$ , g	$C_v$ , %	$R$ , g	No.
QP1	48	0.8170	0.0947	11.58	0.2652	12
QP1	96	1.5347	0.2019	14.02	0.5653	12
QP1	168	2.5996	0.3255	12.52	0.9114	12
AP	48	0.7787	0.0805	10.33	0.2254	10
AP	96	1.4094	0.1626	11.54	0.4553	10
AP	168	2.4309	0.3402	14.00	0.9526	10
QP2	48	0.8566	0.1529	17.85	0.4281	5
QP2	96	1.5720	0.1319	8.39	0.3693	5
QP2	168	2.7600	0.3873	14.03	1.0844	5

X3.7.1 Data generated in the interlaboratory study using this method are available from ASTM as a Research Report.<sup>9</sup>

<sup>9</sup> Available from ASTM Headquarters. Request RR No. G1-1003.

X3.8 *Precision and Bias—Steel Panel Test:*

X3.8.1 An interlaboratory test program using three different sets of UNS G10080 steel panels, 76 by 127 by 0.8 mm (3.0 by 5.0 by .0315 in.) has shown that the repeatability of the mass loss of the steel panels, that is, the consistency in mass loss results that may be expected when replicate panels are run simultaneously in a salt spray cabinet, is dependent upon exposure time and the panel lot or source. The interlaboratory program yielded repeatability standard deviations,  $S_r$ , from which 95 % repeatability limits,  $r$ , were calculated as follows (see Practice E 691):

$$r = 2.8 S_r \tag{X3.1}$$


The values of  $S_r$  and  $r$  are reported in Table X3.1. Note that the corrosion rate of steel in this environment is approximately constant over the exposure interval and that the ratio of the standard deviation to the average mass loss, the coefficient of variation,  $C_v$ , varies between 5 and 10 % with a weighted average of 7.4 % and an  $r$  of  $\pm 21$  % of the average mass loss.

X3.8.2 This interlaboratory program also produced results on the reproducibility of results, that is, the consistency of mass loss results in tests in different laboratories or in different cabinets in the same facility. This program yielded reproducibility standard deviations,  $S_R$ , from which 95 % reproducibility limits,  $R$ , were calculated as follows (See Practice E 691):

$$R = 2.8 S_R \tag{X3.2}$$

The values of  $S_R$  and  $R$  are reported in Table X3.2. Note that the ratio of standard deviation to the average mass loss, the coefficient of variation,  $C_v$ , varies between 8 to 18 % with a weighted average of 12.7 % and an  $R$  of  $\pm 36$  % of the average mass loss.

X3.8.3 The mass loss of steel in this salt spray practice is dependent upon the area of steel exposed, the temperature, time of exposure, salt solution make up and purity, pH, spray conditions, and the metallurgy of the steel. The procedure in Appendix X3 for measuring the corrosivity of neutral salt spray cabinets with steel panels has no bias because the value of corrosivity of the salt spray is defined only in terms of this practice.

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