CHAPTER 1
INTRODUCTION

1.1 Background Study

The structure material such as steel is commonly used in the building structures such as stadiums, theme parks, petrochemical plants, offshore drilling platforms and also high rise buildings. Steel exhibits a good ductility but it begins to lose its structural properties between 470ºC to 500ºC [2]. Therefore, the protection for this material against fire has become an important issue in construction industry.

The fire protection materials issues become vital when the great fire in London 1666, where it spread throughout the city. Then, in 1871 there was a fire in Chicago and at the San Francisco earthquake in 1906 led to fire. These fires showed how a small fire could grow to encompass a building, then a group of building, and finally a portion or a complete section of a city [18]. Beside that, in 2002, investigations into the collapse of the World Trade Center have raised concern over the reliability of fire protection materials.

In fact, prevention for structural collapse is vital as to ensure the safety of the people from the building. Intumescent coatings are designed to prevent the heat, flames or fire from spreading. This coating will expand for many times of its original thickness to produce insulative layer of char and char form when it is being exposed to heat. It is one of the easiest and economical coatings where it can be used to metallic materials, polymers, textiles and wood. The advantages of intumescent coating are it does not modify the mechanical properties of the material and it can prevent the heat from spreading to the coated structure.

However, in term of performance of the coating, the ability of the coating to adhere properly to the material is most crucial element. The coating might flake away or detach from the surface due to the poor adhesion bonding thus allow moisture or corrosion products degrade the coating film at the damage areas. Moreover, if the coating is not adheres sufficiently to the steel structure the coating will not perform their function to protect steel structure from the heat efficiently.

In this paper, the types of testing to study on the interface layer between intumescent coating and steel substrate are presented.

1.2 Problem Description

Currently there is still no official study on the adhesive bonding between the intumescent coating film and steel substrate. The coating cannot play its important role to prevent the steel from the heat if the adhesive bonding is poor. Hence, the coating will peel off from the steel substrate and this brings a problem to the steel structure of the building where it can become ductile then collapse if there is fire occurred. Furthermore this will allow the corrosion products to degrade the steel at the exposed and damage area. In addition, the quality or integrity between intumescent coating and steel structure is an issue to look further since poor adhesion also cause by the incomparability of these two elements.

1.3 Objectives

Upon completing the project, a few objectives need to be achieved. The objectives of the study are as follows:

i. To investigate the adhesive bonding between intumescent coating and steel structures with respect to its physical and mechanical properties before fire exposure.

ii. To determine the quality or integrity between the intumescent coating and steel substrate.

1.2 Scope of Work

The scope of work for the project will be:

i. Investigation on the adhesive bonding between intumescent coating and steel structure with respect to physical and mechanical properties.

ii. Testing will be conducted to see the capability, sustainability and integrity of the coating with the steel substrate.

CHAPTER 2
LITERATURE REVIEW AND THEORY

There were many literatures found about the interface study between coating and steel substrate and the mechanism of the intumescent coating, as well as the durability of the intumescent coating to steel structure. However, no literatures could be found about previous studies on interface between intumescent coating and steel substrate, therefore these literatures would be able to help in explaining the results that are will be obtained from the testing for the intumescent coating.

2.1 Intumescent Coating

Intumescent coating is a mineral based or organic resin based product functioning as fire retardant coating where it can be applied to metallic materials, polymers, textiles, wood as well as structural steel in buildings, storage tank in order to protect them from weakening when encounter elevated temperature in a fire. This coating will expand for many times of its original thickness to produce insulative layer of char and char form when it is being exposed to heat to protect the substrate from the rapid increase of temperature.

Randoux et. al, 2002 said there were two main classes of flame retardant coatings that can be distinguished. The first class referred as flame retardant coating, concerns coatings that delay ignition and hinder flame spread. The other one which is second class concerns coatings that combine the properties of the first class with the protection of the substrate by formation of an insulation layer during its combustion also called as intumescent coating [12].

The intumescent coating composed of three components which are: an acid source, a carbon source and a blowing agent [2]. These three ingredients must have a suitable matching thermal behaviour in order to have an effectively protective coating. The acid source breaks down to yield a mineral acid, then it takes part in the dehydration of the carbonization agent to yield the carbon char and finally blowing agent decomposes to yield gaseous products [2].

According to Sawko et. al (1977), the general composition of intumescent coating composed of diammonium phosphate, ammonium phosphate or monoammonium phosphate as catalysts which function by acting as dehydrating acid which react with a carbon containing material such as starch. The resin component such as melamine formaldehyde acts as a binder for the various ingredients. The special blowing agent is functioning to release gases for the swelling or foaming action of the coating at the proper decomposition temperature. The insulating char stops fire and remains on the substrate, it offers better fire and thermal protection under severe fire conditions than non-flammable type coatings.

Hanafin et al. (2000), describes that typical intumescent coating contain at least one type of epoxy resin which functions as a binder, at least one char forming agent, at least one spumific and at least one epoxy curing agent. Char forming agent cause the formation of the char when the intumescent coating is exposed by fire. Char forming agent controls the rate and mechanism of thermal decomposition of the epoxy, resulting in the formation of carbonaceous char instead of formation of carbon dioxide, carbon monoxide and water. The common composition in fire retardant for char forming agent is phosphorous.

A spumific compound function as to release an expansion gas such as nitrogen, carbon dioxide or water vapour hence, expanding the char to increase the char thickness. The spumific agent will off gas below the temperature of formation carbonaceous char. So, the gas is retained in the char thus better insulation for the steel substrate. The other component is filler which is low in density to reduce the intumescent coating density and insulate the coated substrate by reducing the thermal conductivity of the coating [19].

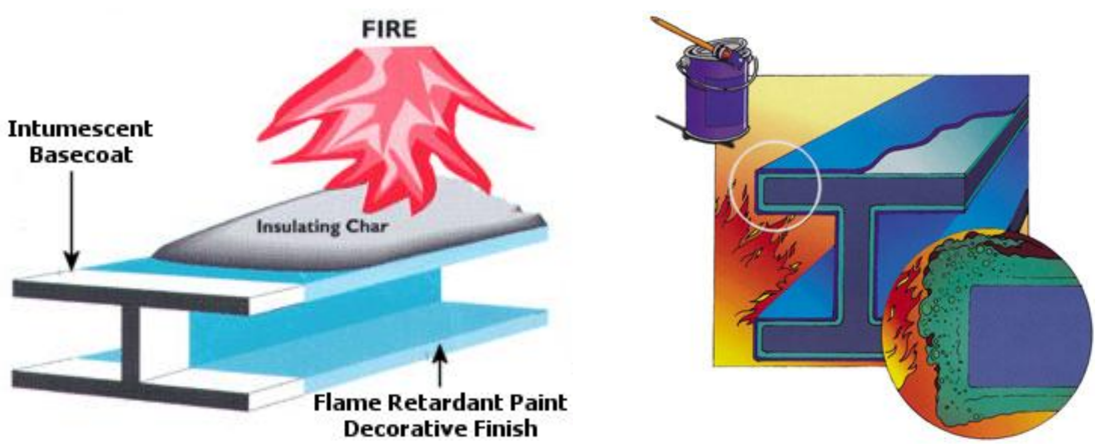
In “Low Density, Light Weight Intumescent Coating” journal by Hanafin et al. (2000) also explained about the blowing agent which is the same as Sawko et al. (1977). In the journal, blowing agent is said to enhance foaming within the char by release a large amount of gas during char formation. The example of blowing agent is carbonate salts which is calcium carbonate such as limestone that will degrade to release carbon dioxide.

Additional components is added to the intumescent coating are wetting agents and anti-forming agents. These two components are to ease the formulation of the fire retardants [19]. A common example of this agent is titanium oxide.

Since the intumescent coating contains a variety of compositions, the sequence of reactions which takes place is dependent on the decomposition temperature of each ingredient which must possess environmental stability in order to function at the proper temperature. For example, the blowing agent must decompose at a temperature above at which the charring of the mixture begins, but before the solidification of the liquid charring melt occurs.

There is another class of intumescent compositions which is formulated upon the bisulfate salts of nitroaniline isomers as the main ingredients. Another type of intumescent coating contains bisulfate salt of nitroaniline with a nitrile phenolic binder. All of these types of intumescent compositions degrade due to the environment [13].

Pollak (2006) have stated that there are two types of intumescent available either water-based or solvent-based coatings. Basically there are two added material to support the intumescent coating which are the primer and topcoat. The purpose of the primer is to enhance adhesion of the intumescent coating to the substrate while the topcoat is to provide a decorative appearance (e.g., color, gloss) or to enhance the durability of the coating. However, the primer and the topcoat must chemically compatible to the intumescent coating. According to Pollak the water-based products might not be able to accept an epoxy, hence will not have proper adhesion.



(Source from www.firepaint.com.au/FIRE1)

Figure 1: Intumescent Coating

2.2 Steel

Steel is an alloy consist mostly iron, with a carbon content (0.2 to 2.04%) depending on grade. Steel is widely used in construction industry because it played critical role especially in construction for bridges, building, cars, roads, infrastructure and also offshore platform. It is a non-combustible material where when expose to high temperature above 500ºC the strength and the stiffness of the steel decreased. If it has been exposed to a long time, the crystalline/ metallurgical structure of the steel will undergoes a transformation. Unprotected steel will visibly deform, twist and buckle. The significant heating effects of the fire could permanently alter the nature of the steel material. In order to prevent this from occur, a fire protection coating is been applied to the steel structure such as spray on materials, intumescent paints or membrane/gypsum boards [3].

Steel that has not been exposed to very high temperature, for a prolonged time will not be significantly deformed, its metallurgy will not be affected after cooling, and hence it will regain its original thermomechanical properties.

2.3 Physical Properties

The table below shows the physical properties of steel which depend on the temperature

Table 1: The Variation in Selected Physical Properties with Temperature. (Source from Carbon Steel Handbook, Gandy, D., 2007)

Temperature °F	**Thermal Conductivity British thermal units (Btu)/hr.ft°F**	**Thermal Diffusivity ft2/hr**	**Mean Coefficient of Thermal Expansion (70°F to Temp) 10-6 in./in./°F**	**Modulus of Elasticity Pounds per square inch (psi) x 10^6**	**Electrical Resistivity μΩ-m**
-200	-	-	5.8	30.6	-
-100	-	-	6.0	30.1	-
0	-	-	6.3	29.6	-
70	27.3	0.53	6.4	29.2	0.200
100	27.6	0.52	6.5	29.1	0.219
200	27.8	0.487	6.7	28.6	0.292
300	27.3	0.455	6.9	28.1	0.292
400	26.5	0.426	7.1	27.7	0.487
500	25.7	0.399	7.3	27.1	0.623
600	24.9	0.373	7.4	26.4	0.758
700	24.1	0.346	7.6	25.3	0.925
800	23.2	0.319	7.8	24.0	1.094
900	22.3	0.291	7.9	22.3	1.136
1000	21.1	0.263	8.1	20.2	1.167
1100	19.8	0.234	8.2	17.9	1.194
1200	18.3	0.204	8.3	15.4	1.219

2.4 Adhesion

Generally, the coating system is widely used to coat various types of surfaces such as wood, metal and plastic, the diverse structures likes porous, homogeneous and flat, and the many possible stresses to which it may exposed such as temperature, extensibility. Because of that, it is important to have adhesion promoter to ensure the strength or a good adhesive bonding between coating material and the coated material. Adhesion is about the physical and chemically intermolecular interactions in the interface or interface layer [4]. The adhesion promoter can be in a range of chemicals available includes silicones, amides, phosphates or modified polymers. Beside that, there are binders and additives. This adhesion promoter is to ensure the adhesive bonding between the coating and coated material is sufficient hence the coating functioning effectively to protect the coated surfaces.

Regardless of what excellent properties a coating may possess, it is useless unless it also has good adhesion. The coating's resistant to weather, chemicals, scratches or impact in only value while the coating remains on the substrate [10].

2.5 Corrosion

Corrosion is the deterioration of the properties, mechanical, aspect of a material due to the surrounding environment. It means a loss of an electron of metals reacting with water and oxygen. That is why coating is important to solve for corrosion problem. Coatings are used to prevent or control corrosion so know the basic concept of corrosion will help in coatings technology [7]. When coatings break down, then the steel will corrode where the corrosion of steel arises from its environment of exposure. The chemical reaction between the exposed steel with moisture and oxygen as below:

$$Fe + O_2 + H_2O \rightarrow Fe_2O_3.H_2O$$

(iron) (rust)

2.6 Coating Failures

In addition, there are some coating failures which can lead to the loss of adhesion. From these factors, the effect from these failures can be minimized when applying the coating to have a better result in searching for integrity of intumescent coating and steel substrate. These are the factors:

- Improper surface preparation – the substrate surface is not adequately prepared for the coating that is to be applied. This may include cleaning, chemical pretreatment or surface roughening.

- Improper coating selection – either the paint or coating selected is not suitable for the intended service environment, or it is not compatible with the substrate surface. In this case, the compatibility between the intumescent coating and steel substrate is the main subject.

- Improper application – this can be a problem with either shop-applied or field applied coatings, and occurs when the required specifications or parameters for the application are not met.

- Improper drying, curing and over coating times – again, this problem relates to a lack of conformance to the required specifications or parameters.

- Lack of protection against water and aqueous systems – this is a particularly serious problem with aqueous systems containing corrosive compounds such as chlorides.

- Mechanical damage – which results from improper handling of the painted or coated substrate, resulting in a breach in the paint or coating.

CHAPTER 3
METHODOLOGY

The steel substrate and the intumescent coating as the two vital elements were used in this project. Furthermore, the project involved the experimental works; prepare the specimen, conduct the metallography investigation in order to study on the physical properties of the steel. The mechanical properties on the specimen were analysed using hardness tester. The morphological structures of intumescent and steel substrate were studied using Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS).

The chart below shows the methodology for the overall project:

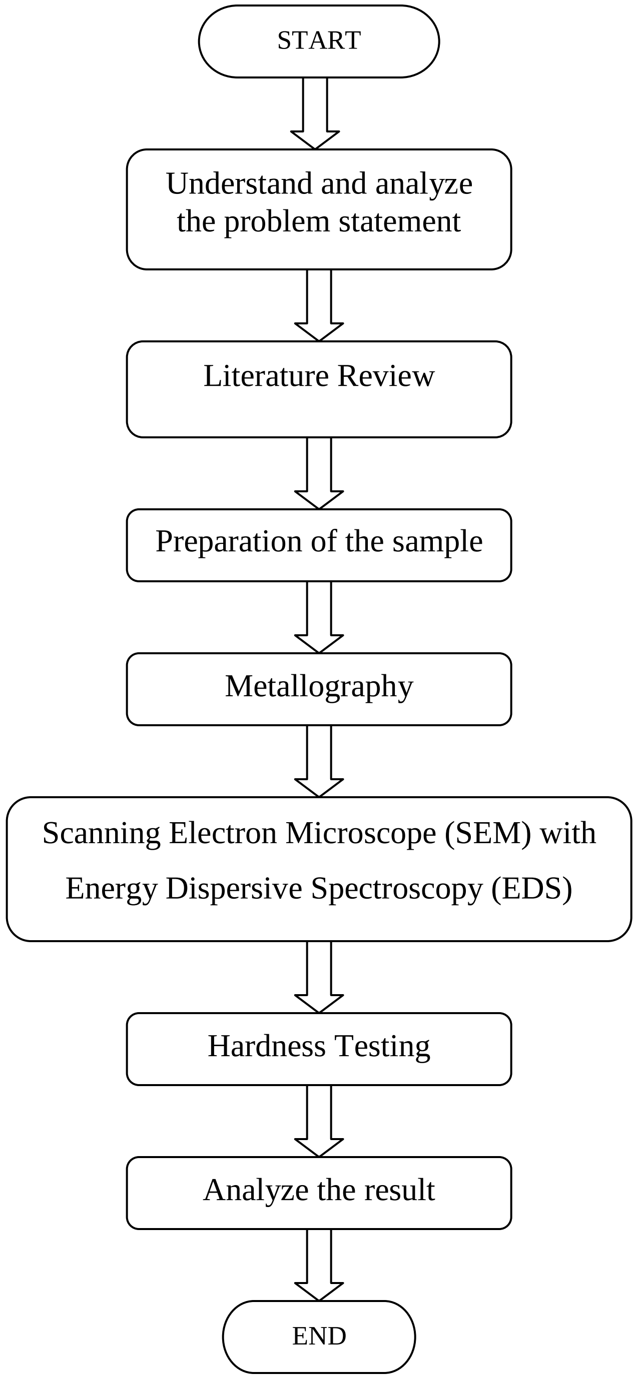


Figure 2: Flow Chart for project methodology

3.1 Preparation of coated steel substrate.

3.1.1 Coated the steel

The specimens were obtained from Norimax (M) Sdn. Bhd. which is the expert in the intumescent coating. The specimen was prepared by sand blasting of Sa2 ½ and through hot rolled process to apply the primer. After that the intumescent coating was sprayed layer by layer until 6mm followed by carbon fibre mesh. The intumescent coating was sprayed again. The last layer on top of the intumescent coating is the top coat.

The preparation for the intumescent coating to the steel is as below:

i. 1 spray coat Epigrip L425HS Zinc Phosphate Primer – Light Grey

ii. 1 spray coat Firetex M90 Epoxy PFP

iii. Firetex J120 Scrimcloth

iv. 1 spray coat Firetex M90 Epoxy PFP

v. 1 spray coat Resistex C137V2 Special Firish - Holly 14C39

3.1.2 Abrasive Cutting

The intumescent coated steel was cut to 1cm x 1cm using abrasive cutter and is shown in the figure below:

Figure 3: Abrasive Cutter Machine

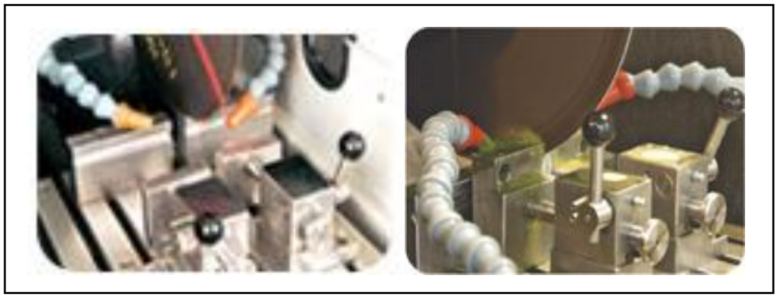


Figure 4: Abrasive Cutter

3.1.3 Mounting

After cutting the material the next step is mounting. There are two types of mounting which are hot mounting and cold mounting. In this project cold mounting was used because the coating is an epoxy-based intumescent where it can be affected by heat and pressure if the hot mounting is used. The materials that used to do the cold mounting were Epo-Kwick Fast-cure epoxy resin and Epo-Kwick hardener in ratio of five to one. Then the mixture was poured into a mould where the specimen was placed first and allowed to set for one day.

Figure 5: The mounting process

3.1.4 Grinding

Grinding is a finishing process used to improve surface finish, abrade hard materials, and tighten the tolerance on flat and cylindrical surfaces by removing a small amount of material.

During grinding, water must be used to flush the abrasive disk and keep the specimen cool. To obtain a flat surface with minimal deformation, the specimen should be held with the fingertips as close as possible to the grit paper. The specimen should be rotated 90^o between steps. The specimen surface should be examined periodically to determine whether the scratches from the previous grinding have been removed or not.

After final grinding, the specimen surface should be cleaned with water, flushed with alcohol and then dried.

Grit papers of 120, 240, 320, 400, 600 and 1200 were used to improve the surface finished of the samples after they have been mounted. After grinding has been completed, I've cleaned the specimen surface with ethanol and dried it.

3.1.5 Polishing

Polishing is the next step after grinding and its purpose is to remove the scratches and the surface deformation created by grinding. For this project where the material used is low carbon steel, the specimen had been polished using 6 μm diamond on a canvas for 1 min with heavy pressure. Then followed by 3 μm alumina for 2 min with firm pressure on a medium nap cloth.

Figure below shows the grinding and polishing machine.

Figure 6: Grinding and Polishing Machine

3.1.6 Etching

The specimen should be etched immediately after the final polishing. Etching process is to reveal the microstructure of low carbon and coated steel. In this project, nital is used to etch the low carbon steel as stated in the table below:

Table 2: Etchant for low carbon steel

Etchant	Ingredients	Application
2% nital	1-5 mL nitric acid (conc) 99 to 95 mL ethyl alcohol	Used to reveal ferrite grain boundaries and lath martensite (low carbon).

Etching times generally range from 5- 8s with the polished face held upward. After etching, the specimen was held under water to remove excess etchant, flushed with ethyl alcohol and dried.

3.2 Analysis and characterization

For coatings to perform satisfactorily, they must adhere to the substrates on which they are applied. A variety of recognized methods can be used to determine how well a coating is bonded to the substrate. In order to achieve the objectives of this project, the following testing will be conducted:

1. Metallography
2. Hardness Test
3. Scanning Electron Microscope (SEM) with Energy Dispersive X-ray Spectroscopy (EDS)

The testing is divided into physical and mechanical testing. Physical testing provides important characteristics of a paint or coating specimen which may reveal primary causes for the failure. Important physical and mechanical tests are metallography and hardness testing, respectively. The toughness of the steel substrate was investigated after the coating had been applied.

The morphological structure of intumescent and steel substrate was studied by Scanning Electron Microscope (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS).

3.2.1 Metallography

The purpose of this testing is to reveal microstructual constituents of the specimen. It is significant to look at the structure of the specimen since it shows the roughness and composition of the specimen. In this testing, ASM A36 was used for low carbon steel for the metallography process.

3.2.2 Hardness Testing

The hardness test was performed on the intumescent coating using Vickers Hardness Test. This hardness test was conducted in this project to observe the integrity of the intumescent coating to the steel substrate after the coating was applied. Then the result was compared with the normal steel to determine whether the process applied to the intumescent coating affected the hardness of the steel substrate or not.

3.2.3 Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS)

The SEM function is to observe the materials in micro and submicron ranges. The charring layer and the morphological structure was observed by using SEM in this project. This equipment is capable to analyze the topography features [14]. Moreover, when used with EDS the analyst can perform an elemental analysis on microstructure of the material and the contaminants that may present.

The SEM is a microscope that uses electron rather than light which provide the images of resolution up to 1 000 000 magnification. The preparation of the sample for this testing is easy because most of SEM only required the sample to be electrically conductive. The process is when SEM generates the high energy electrons and focused on the specimen. Whereas for the EDS use x-rays that are emitted from the specimen when bombarded by the electron beam to identify the elemental composition of the specimen.

These two techniques combined, when the sample is bombarded by the electron beam of the SEM, electrons are ejected from the atoms on the specimens' surface. A resulting electron vacancy is filled by an electron from a higher shell, and an x-ray is emitted to balance the energy difference between the two electrons. The EDS x-ray detector measures the number of emitted x-rays versus their energy. The energy of the x-ray is characteristic of the element from which the x-ray was emitted. This is because different elements have different energy level of electrons. A spectrum of the energy versus relative counts of the detected x-rays is obtained and evaluated for qualitative and quantitative determinations of the elements present [14]

CHAPTER 4

RESULT AND DISCUSSION

4.1 The coated steel

The material that has been coated is shown in the figure 7:

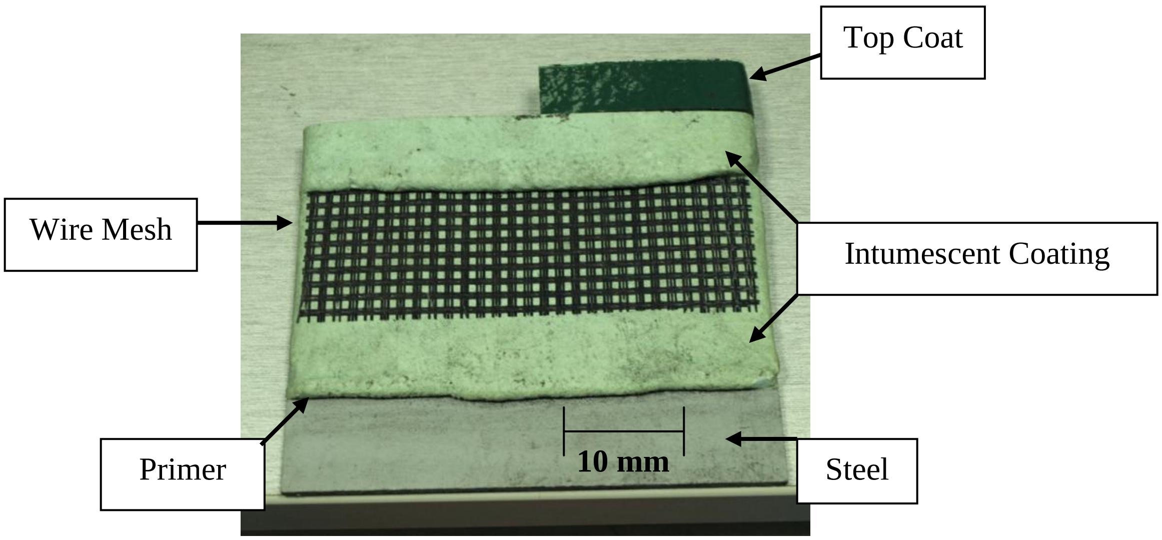


Figure 7: Layers of the coating

Figure 8: The coated steel

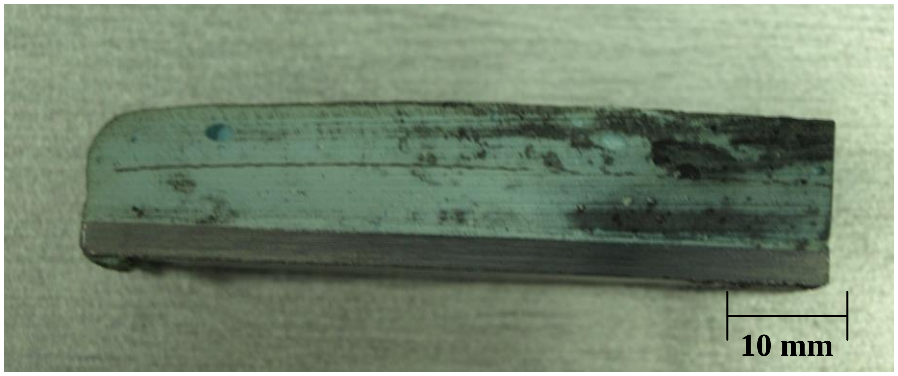


Figure 9: The coated steel (from side view)

4.2 Mounting

The figure below shows the sample after being mounted.

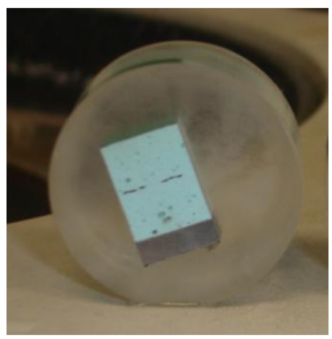


Figure 10: The sample after being mounted

4.3 Metallography

The figures 11 and 12 below shows the microstructure of pearlite and ferrite

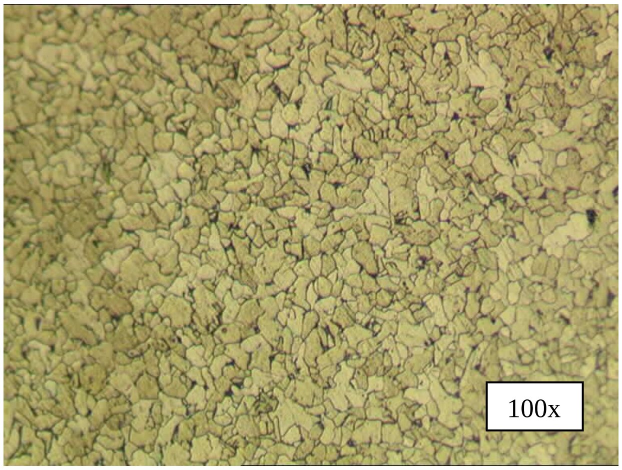


Figure 11: Microstructure of a low carbon steel which are pearlite and ferrite after etched with 2% nital.

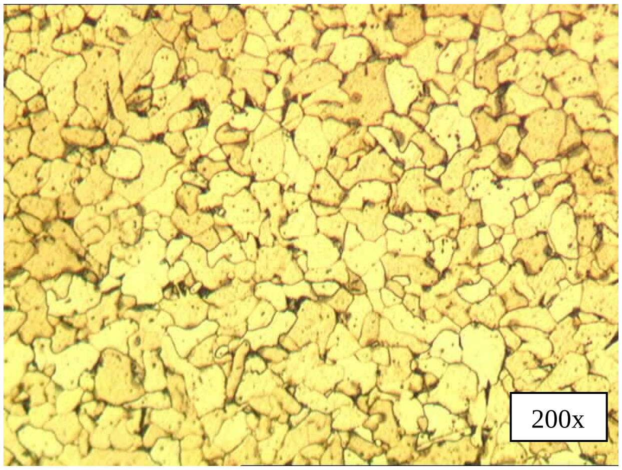


Figure 12: Microstructure of a low carbon steel after etched with 2% nital.

4.4 Hardness Testing

The data from the hardness testing which was taken by Vickers Hardness Tester are shown in Table 3:

Table 3: Vickers Hardness of steel substrate after applied coating

Sample before burned	
105.9	105.3
99.1	106.4
101.6	93.5
105.7	101.6
104.5	106.7
98.9	94.6
100.6	108.4
106.4	93.4
101.6	104.5
94.6	99.5

By comparing this result with the normal steel, this is in the range of 90 to 411 HV. It shows that the steel is not affected by the coating process.

4.5 Scanning Electron Microscope (SEM) with Energy Dispersive X-ray Spectroscopy (EDS).

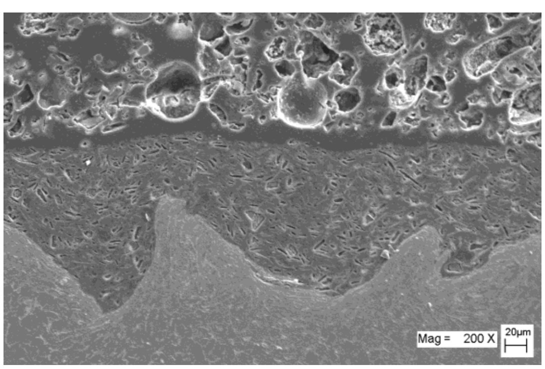


Figure 13: SEM images of three elements; steel, primer and intumescent coating

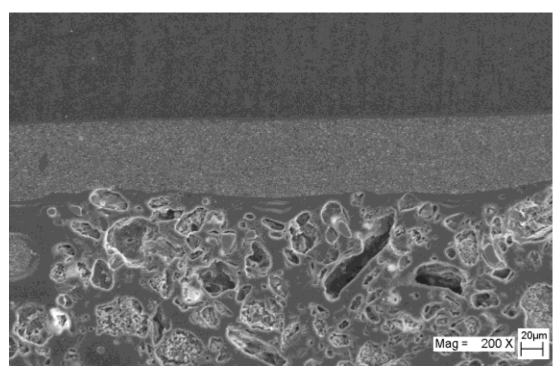


Figure 14: SEM of intumescent and top coat

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From Figure 13, there are different microstructures of each element. The bottom element is steel, the top is intumescent coating and in between it, is the primer. According to Pollack (2006), the purpose of the primer is to enhance adhesion of the intumescent coating to the substrate while the topcoat is to provide a decorative appearance (e.g., color, gloss) or to enhance the durability of the coating. However, the primer and the topcoat must chemically compatible to the intumescent coating.

In this project, the primer is zinc phosphate where it had been proved by the chemical analysis using Energy Dispersive X-ray Spectroscopy (EDS) and is shown in the Figure 15 below:

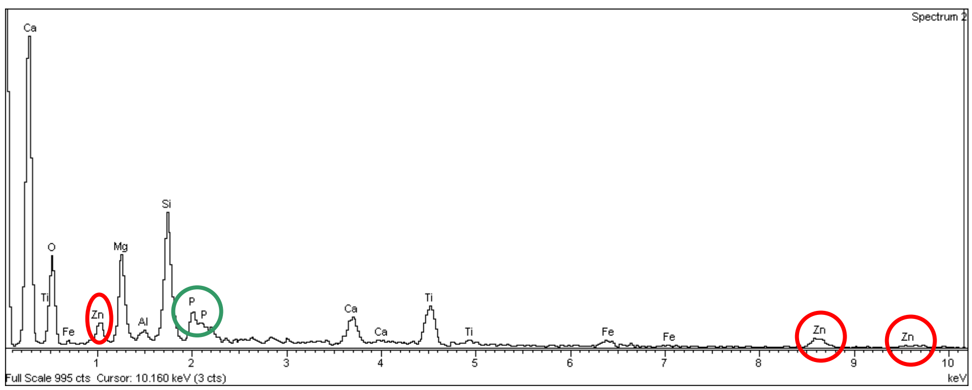

Figure 15: The elements in primer which are Zinc, Zn and Phosphate, P.

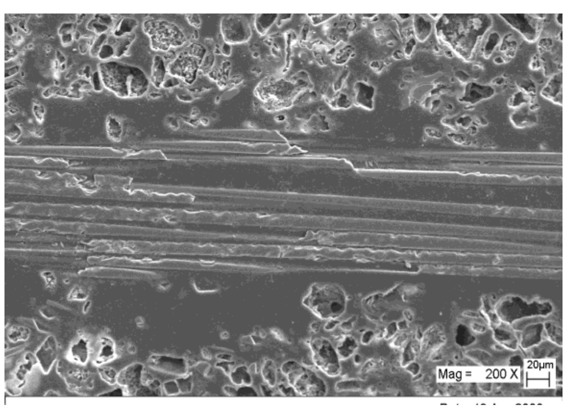

Figure 16: SEM of carbon fibre mesh

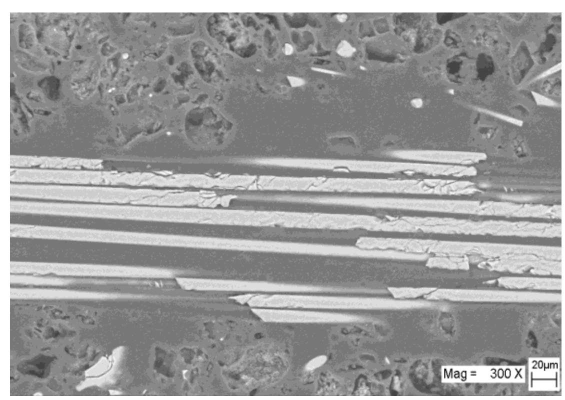

Figure 17: Quadrant Back Scatter Detector QBSD of carbon fibre mesh

The function of carbon fibre mesh between the intumescent coating as shown in the figures above is to reinforce the intumescent coating. Thus, the coating is not easily flaked off.

Intumescent elements

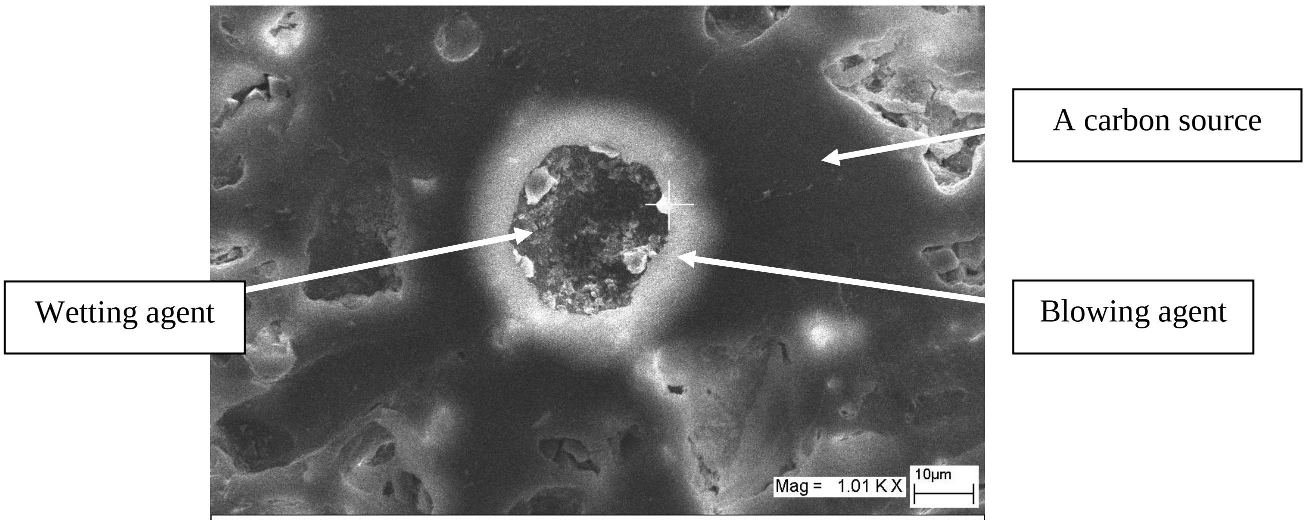

Figure 18: SEM of Intumescent coating

Based on literature review, there are three main elements in intumescent coating. There are a carbon source, blowing agent and acid source. Figure 17 above shows the three elements in the intumescent coating. Each of them has different element characterization and function.

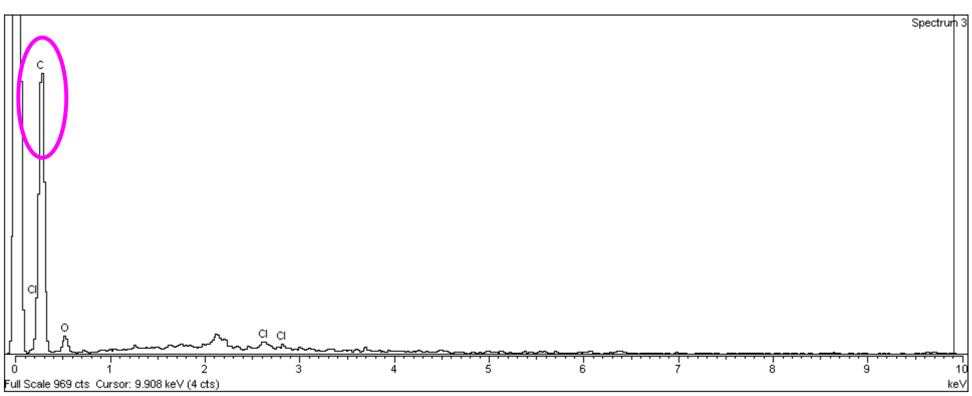

Figure 19: A carbon source in Intumescent coating.

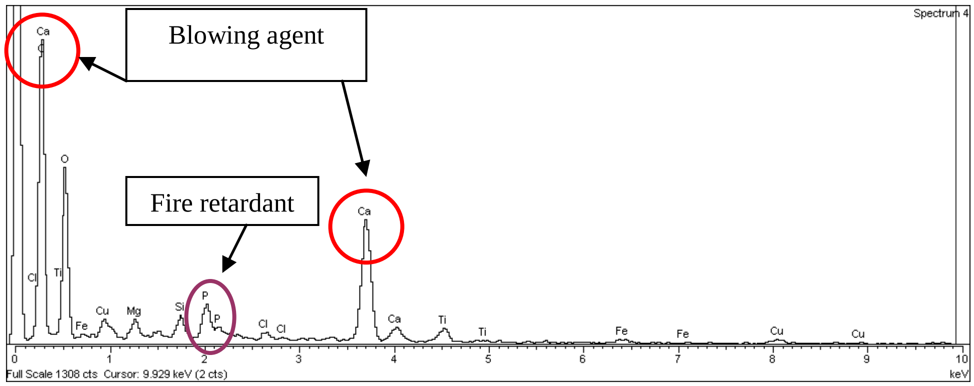

Figure 20: Blowing agent's element in Intumescent

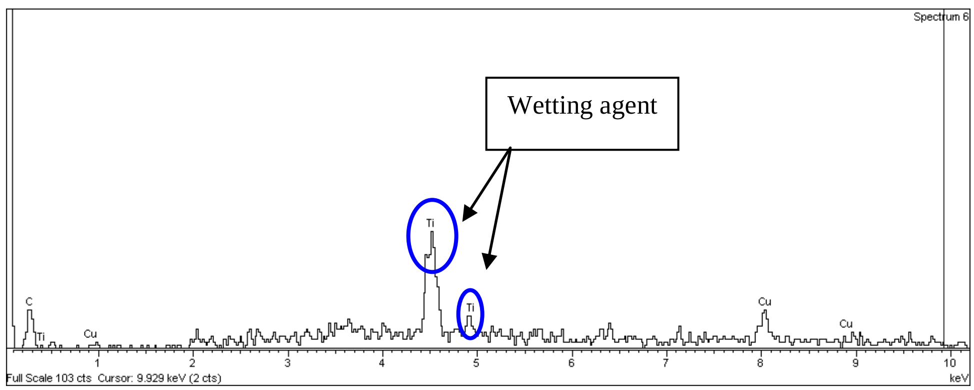

Figure 21: Filler's element in Intumescent coating

From Figure 19, the carbon element in the intumescent coating acts as a carbonization agent to yield carbon char. The other char-forming agent as fire retardant in intumescent is contains phosphorus which is shown in Figure 20. Blowing agent is to further enhance foaming within the char where from Figure 20, the blowing agent is carbon carbonate where it will degrade to release carbon dioxide. The gas released during char formation. Beside that, wetting agent and anti-foaming agent is additional components in the intumescent coating to ease formulation of the fire retardant. This is shown in the Figure 21 where the wetting agent is Titanium Dioxide, TiO_2.

Integrity between Intumescent and steel substrate through primer

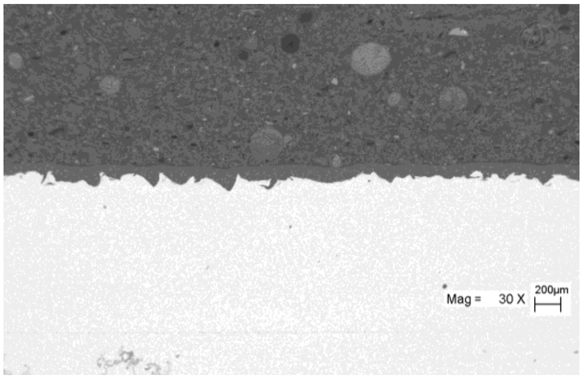

Figure 22: Steel profile using microscope

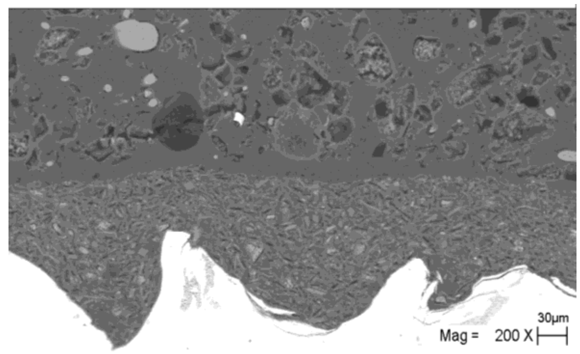

Figure 23: Closer look at steel profile using Quadrant Back Scatter Detector (QBSD)

From Figure 22 and 23, we can see the mechanical interlocking between steel substrate and primer. The roughness of the steel is 50 – 70 μm. Then the adhesive bonding between the primer and steel substrate was investigated by the SEM. The result is shown in Figure 24 and Figure 25 below:

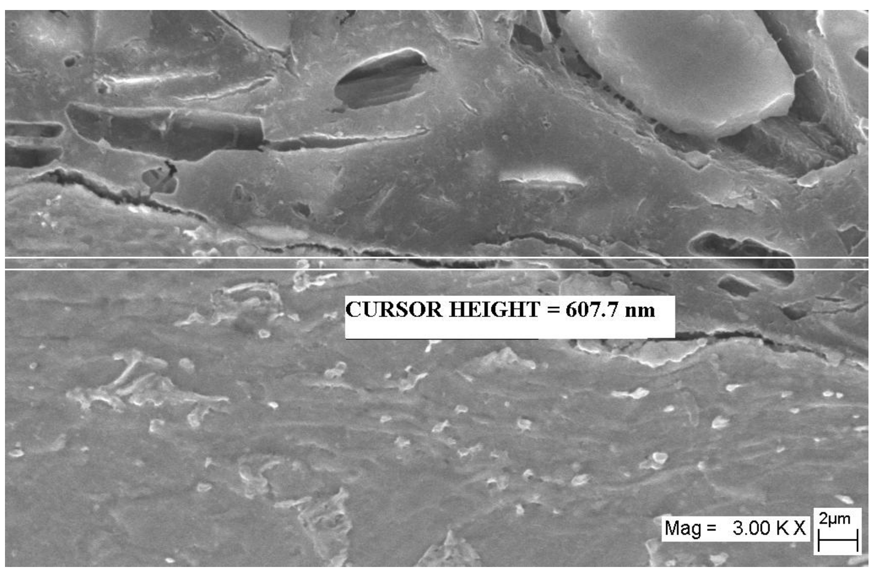

Figure 24: The gap between intumescent coating and primer before burned

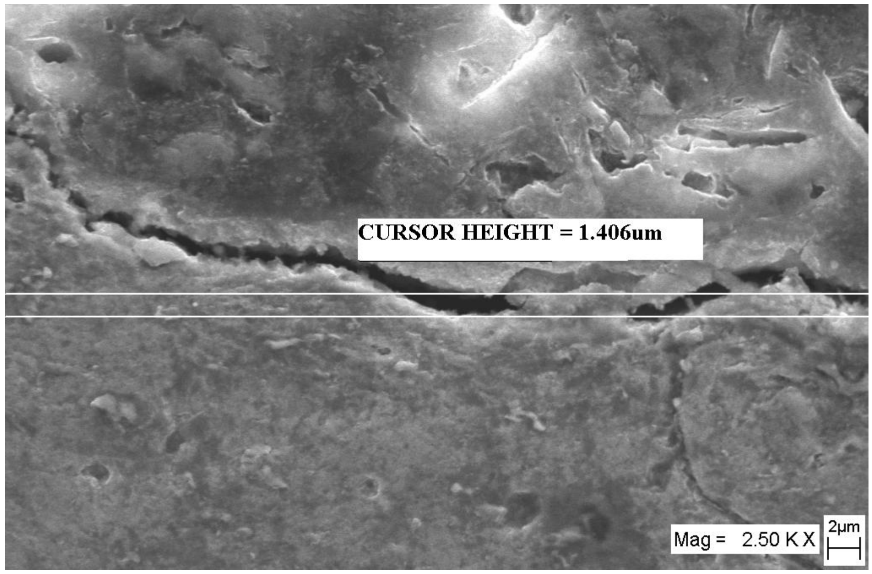

Figure 25: The gap between intumescent coating and primer after burned

From figure 24 and 25, it shows that there is a small effect from the burning process on intumescent coating where it can be negligible. Thus, the integrity between the intumescent coating and steel substrate is good when the primer is chemically compatible with intumescent and steel substrate.

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In the studied of intumescent, there are three main components in the intumescent coating; a carbon source, acid source and blowing agent. The aim of this study is to investigate the integrity of the intumescent coating with steel substrate. It was proven that the integrity between these two elements is good because the primer is chemically compatible to the steel and intumescent coating.

5.2 Recommendations

5.2.1 Primer Materials

Other material that can attach to the steel better should be examined. Therefore there is no possibility of the gap.

5.2.1 Testing

For better result in study on the adhesive bonding, the other testing such as scratch test should be performed. However there is no equipment available in UTP in order to prove the integrity between intumescent coating and steel substrate.