CHAPTER 1

INTRODUCTION

1.1 Project Background

Intumescent coatings represent an important class of passive-fire proofing materials, which concern insulating systems designed to decrease heat transfer from a fire to the substrate being protected. It has been used in the fire protection of steel structure for more than 20 years [1]. Using expandable graphite as an intumescent fire retardant has attracted more and more interests in both research and industry circles in recent years.

Some intumescent, however, are susceptible to environmental influences such as Ultraviolet (UV), heat and humidity which can reduce or negate their ability to function. Intumescent fire retardant coating was found to lose the majority of their capability due to leaching out of fire active ingredients in reaction to weathering [2].

1.2 Problem Statement

The issue of poor weathering properties of intumescent fire retardant coating is reported recently. Previous research indicates intumescent coating can lose fire endurance by leaching on long term weathering [2]. Some intumescent have very limited application because their beneficial properties can disappear within days of installation [3]. In reality, as an example, the structural of building is exposed to challenging environments. Hence, the coating should withstand rain, wind, humidity, UV exposure and other elements in order to give best protection to the substrate.

1.3 Objectives

The objective of this project is to study the effect of weathering on the expansion performance of intumescent fire retardant coating. Besides that, the difference of fire performance of intumescent fire retardant coating will be compared between the use of zirconium and without zirconium.

1.4 Scope of Study

There are some parameters and limitations in this project. The main material used is graphite which is in range of 212- 300 micron meter in particle size. Zirconium used as filler is in powder form. Structural steel sized 5cm x 5cm has been chosen as a substrate since it is widely used in the industry. Weathering chamber that has been set up to 37˚C and 70% relative humidity will be used for the test purpose. Fire test is conducted by using furnace only where the temperature is about 450˚C. For analysis purpose, Scanning Electron Microscope (SEM) and will be used.

CHAPTER 2

THEORY AND LITERATURE REVIEW

From the literature survey, there are some points that have been analyzed. Below are the details:

2.1 Intumescent coating

An intumescent coating is a substance that chemically reacts in a fire. The coating swells in size to form a char, which protects the steelwork for a specified period [4]. As the intumescent does not actively tackle a blaze it is known as passive fire protection.

2.2 Component of Intumescent Coating

Basically, this coating consists of four basic components which are carbon source, acid source, blowing agent and binder. A carbon source can be a carbon-rich polyhydric compound such as starch, glucose and pentaerythritol. Meanwhile, the acid source should be a dehydrating agent capable of promoting the formation of a carbonaceous chars from the carbon source. This is usually a source of phosphoric acid such as ammonium phosphate, diammonium phosphate and the other phosphate. For blowing agent, it is an agent that would expand the film of coating upon heating. This is usually a nitrogen or halogen releasing compound such as urea, melamine and melamine phosphate or chlorine paraffin. Tetra athleyne tetra amine is one of the binder source.

Ideal compatibility between these basic components is essential to produce an excellent fire retardant, that is dehydration to char and release of gas in a transitional semi-liquid state sufficient to enable foaming and expansion to occur followed by full carbonization without char collapse.

2.3 Principle of operation

Intumescent coatings are normally applied by airless spray to provide a smooth decorative finish, which remains stable at ambient temperatures. These coating compositions are based on organic resin binders, which are typically acrylated rubber or epoxy [5].

The resins are filled with active ingredients, which react in a fire at temperatures around 250°C to form a thermally insulating carbonaceous char or foam. The char reduces the rate of heating of the steel and hence prolongs its load bearing capacity.

As described above the basic formulation of an intumescent comprises of an organic binder, a carbonific, usually a penta or dipentaerythritol, a spumific or blowing agent which could be melamine or a melamine formaldehyde derivative, a source of an acid catalyst such as ammonium polyphosphate and additionally a char reinforcing pigment.

As the temperature rises the binder begins to melt and the blowing agent liberates gases causing a controlled expansion. At the same time there is degradation of the carbon backbone and fusion of the inorganic reinforcing materials, resulting in char solidification.

2.4 Expandable Graphite

Natural graphite is a special form of pure carbon [6]. The basic structure of graphite consists of hexagonal groups of carbon atoms, which form stable planar grids with only weak inter layer bonding.

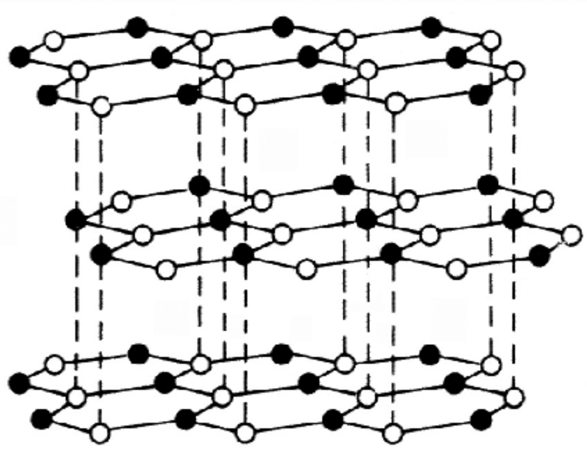


Figure 2.1: Crystal structure of graphite

Due to the layered structure of graphite, atoms or small molecules can be introduced between the carbon layers (intercalation). During this process a so-called expandable graphite salt or GIC (Graphite Intercalation Compound) is produced. High-grade expandable graphite has a greater proportion of intercalated layers. A wide variety of chemical species have been used to intercalate graphite materials. These include halogens, alkali metals, sulfate, nitrate, various organic acids, aluminum chloride, ferric chloride, other metal halides, arsenic sulfide, thallium sulfide, etc [7].

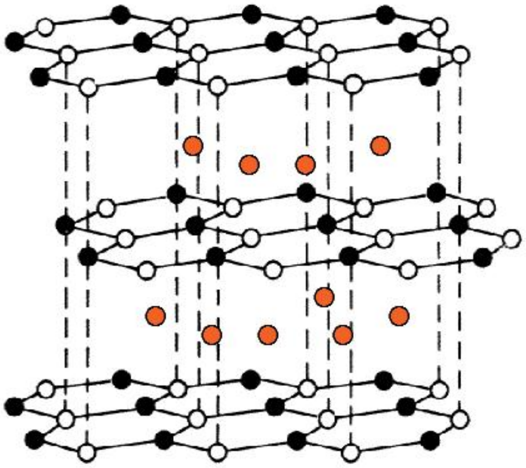


Figure 2.2: Graphite layers with intercalated molecules

Under the influence of heat, the layers separate like an accordion, and the graphite flakes expand [6]. Depending upon the grade of material, expansion can commence at as low as 150°C and can occur suddenly and rapidly. In the case of free expansion the final volume can be several hundred times greater than the initial volume. The properties of expandable graphite, i.e. initial expansion temperature and degree of expansion, are primarily defined by the quality of intercalation (proportion of intercalated layers) and by the intercalation agent.

2.5 Zirconium

Zirconium is a chemical element with the symbol Zr. Zirconium is used as filler in intumescent coating. The use of zirconia or zirconia-borate combination as additives in organic polymers reduces polymer flammability [8]. When heated to temperature at which fire retardant activity is needed and exhibited, zirconia will modify to the form in which the fire retardant effect is provided. It is preferred to use a powder form of zirconia such that it can be readily dispersed in the material in which it is incorporated.

2.6 Standard for Weathering Test

2.6.1 Underwriters Laboratories 1709 (UL1709) test standard

The standard that adequately addresses serious environmental exposures and the longevity of intumescents is UL1709 (Standard for Safety for Rapid Rise Fire Tests of Protection Materials for Structural Steel) [3]. UL1709 contains a very tough set of tests, as it should, considering the rigorous applications for exterior hydrocarbon fire protection. The test method also covers a small-scale fire exposure, intended to evaluate the ability of protective materials to withstand a variety of environmental conditions anticipated.

Preceding the fire exposure test is a whole battery of environmental exposures, some of which are mandatory. The “Simulated Environmental Exposures” provide that simulated exposure conditions may include but are not limited to the following:

- **Simulated aging**

  The sample is placed in a circulating air oven at a temperature of 153–163 °F for 270 days.

- **High humidity**

  The sample is placed in an environment with the following conditions: 92–98 °F and 97–100% relative humidity for 180 days.

- **Industrial atmosphere**

  The sample is placed in a chamber in which the gas contains 1% SO_2 and 1% CO_2. In addition, a small amount of water must be present at the bottom of the chamber, which must be maintained at 92–98 °F. The exposure duration is 30 days.

- **Salt spray**

  The sample is exposed to salt spray (fog) testing in accordance with the methods described in ASTM B 117-97.

- **Combination moisture, freeze, and dry heat cycling**

  The sample is exposed to a simulated rainfall of 0.005 mm/s for 72 h, followed by a temperature of –45 to –35 °F for 24 h, followed by a dry atmosphere at 135–145 °F for 72 h. This cycle is repeated 12 times, so the entire test takes 84 days.

- **Acid spray**

  The sample is exposed to a fog spray consisting of 2 vol % hydrochloric acid. This fog spray must provide 1–2 mL of solution per hour for each 80 cm^2 of horizontal sample surface area. The exposure duration is 5 days.

- **Solvent spray**

  Samples are sprayed with reagent-grade solvents at 65–75 °F. Typical solvents for this test are acetone and toluene. Using a standard spray gun, the solvent spray is applied until the entire surface area of the sample is covered with solvent and excess solvent is observed to run off the sample. One exposure cycle consists of applying the solvent, followed by drying the sample for 6 h, followed by another solvent application, then drying the sample for 18 h. This amounts to 1 day per cycle. The exposure cycle must be repeated 5 times, for a total test time of 5 days.

2.7 Performance of Intumescent

M. Jimenez et al in the journal entitled Characterization of The Performance of An Intumescent Fire Protective Coating evaluate the behavior and efficiency of different intumescent formulation designed to protect steel in case of hydrocarbon fire. The coating is based on a thermoset epoxy-amine resin system into which fire retardant agents, boric acid and ammonium polyphosphate derivative have been incorporated. They study the performance of the thermoset resin containing alone and in combination with additives [9].

Four different formulations were chosen and compared to the virgin steel plate which is marked as A. The four formulations are the thermoset resin alone which is marked as B, the thermoset resin mixed with the ammonium polyphosphate (APP) derivative which is marked as C, the thermoset resin mixed with boric acid which is marked as D and the thermoset resin mixed with both which is marked as E.

The time of failure of the steel plate covered with the thermoset resin is closed to the time of failure of the steel plate alone. It is implies that the thermoset resin does not provide any protective effect. When APP derivative is added to the thermoset resin, an improvement in performance is observed. Intumescent and charring take place, but the char falls off the plate before the end of the experiment. Addition of boric acid to the resin also leads to improved performance where the time of failure is increased. Development of intumescence is also observed however the char falls off the plate. The best result is obtained when both the APP derivative and boric acid are added to the resin. The time of failure increases and the char adheres to the plates.

The result show that the use of only one fire retardant additive leads to a significant increase in the time to failure. However, the combination of the two fire retardants additives leads to a higher time of failure.

As a conclusion, the combination of phosphate and boric acid is necessary to allow the formation of species which promote both intumescence and adhesion of intumesced coating to the steel plate.

2.8 Zirconium and Boron Containing Compound

J.W Gilman in the journal entitled New Non-Halogenated Fire Retardant for Commodity and Engineering Polymers discovers that the use of zirconia-borate combinations reduces flammability. Zirconium is used together with ammonium pentaborate, $NH_4B_5O_8$ where the result shows the effectiveness of intumescent is maintained at a very high heat fluxes [8]. Although zirconium may be used alone, it is preferred that the zirconium be used together with a boron-containing compound to exhibit the desired fire retardant activity. Preferred boron-containing compounds are boric acid or borate compound, particularly borate salt. Therefore, the author choose to use boric acid instead of ammonium pentaborate.

2.9 Fire Resistant of Passive Fire Protection (PFP) Coatings After Long Term Weathering

Shell UK in cooperation with Health and Safety Executive began a long term PFP weathering program to assess the fire performance of the coatings [10]. The program is conducted between years 1991 to 2002. They develop a program of real time weathering under known exposure conditions. A specimen of steel plate is designed to 300mm x 300mm x 5mm in size. The coating thickness and reinforcement was to be that which they deemed necessary to achieve at least 120minutes for a temperature rise of 139K above ambient as determined in a furnace-based fire test. Shell then applied coal tar epoxy to the back face of the steel and extended over the edges to give further protection and to restrict the effect of weathering to the one, major face. The basic requirements for real time weathering are direct and continuous exposure to the natural environment. Shell North Sea platform site was used for a real timing weathering.

Details of the weathering period (some products were added after the start of the program), application thickness and the top coat for the specimens used are summarized in table 2.

Table 2.1: Application detail of the product

Product	Weathering Years	Thickness (mm)	Topcoat
Chartek III	10.5	22	No topcoat applied
Chartek IV	8.3	16	Epoxy
Firetex M90	7.3	19	Acrylic urethane gloss finish
Mandolite 550	12.3	45	Yes
Nullifire System E	8.3	19	Acrylic urethane gloss finish
Pitt-Char	10.5	29	Acrylic urethane enamel
Thermo-lag 440	10.5	20	No specification provided

Below is the summary of the result for each product after the end of every weathering year exposure.

Table 2.2: Summary of weathering effect

Product	**Effect on bulk material**	**Effect on top coat**
Chartek III	Extensive surface cracking.	-
Chartek IV	Panel edge start to rust.	Initiation of cracking and loss of top coat from high spots
Firetex M90	Corrosion of panel edge initiated on one panel. Bulk intumescent obscured by algal growth at damage areas.	Continued erosion, cracking and loss of topcoat from high spot
Mandolite 550	Extensive loss of material.	-
Nullifire System E	Progressive erosion where exposed at deliberate damage to give open porous structure. Algal growth at intersection of cut slot	Cracking and loss of topcoat from high spots.
Pitt-Char	No cracking but fibres revealed at the surface.	Erosion, cracking and loss of topcoat from high spot. Surface cracking.
Thermo-lag 440	Surface of deliberately damaged areas obscured by algal growth at end of exposure. Delamination at weathered edges.	Crack at discontinuities and irregularities.

From this literature, it can be said that the differences between this project and the author project is the temperature and the coating used is produced by the author herself. Their specimen is only exposed to low temperature as the test is conducted at

North Sea platform. Meanwhile, fire test will be conducted right after the sample is exposed to the simultaneous environment in the weathering chamber.

CHAPTER 3

METHODOLOGY

3.1 Project Methodology

The figure below shows the main procedure in completing this project in order to achieve its objectives.

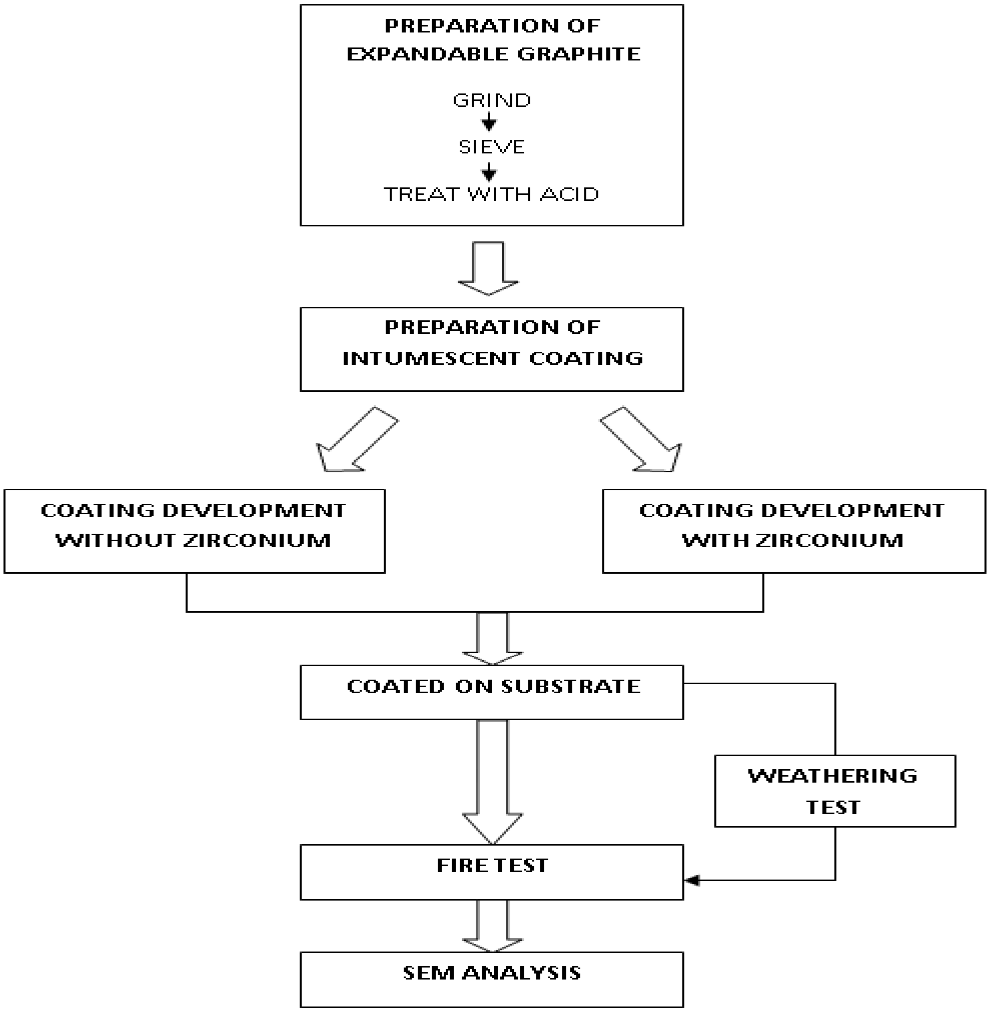


Figure 3.1: Methodology of project

3.2 Gantt Chart

Table 3.1: Gantt Chart for FYP I

No.	Action/Plan	WEEK														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	▨	▨ △						M I D S E M B R E A K							
2	Preliminary Research Work		🟥	🟥	🟥											
3	Submission of Preliminary Report				▨ △											
4	Research/ Literature Review					🟥	🟥	🟥		🟥	🟥	🟥	🟥	🟥		
5	Grind The Graphite					🟥										
6	Sieve The Graphite						🟥									
7	Treat The Graphite						🟥	🟥								
8	Submission of Progress Report									▨ △						
9	Seminar									▨ △						
10	Analyze The Graphite										🟥	🟥				
11	Interim Report Preparation												🟥	🟥		
12	Submission of Interim Report Final Draft														▨ △	
13	Oral Presentation															▨ △

LEGEND	
△	Milestone
🟥	Project Schedule
▨	FYP Schedule

Table 3.2: Gantt Chart for FYP II

No.	Action/Plan	WEEK														
		1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Prepare Intumescent Coating without using zirconium	🟥	🟥						M I D S E M B R E A K							
	Fire test			🟥	🟥											
	Choose the best samples				🟥											
2	Submission of Progress Report I				▨ △											
3	Prepare Intumescent Coating by using zirconium					🟥	🟥									
	Fire test						🟥	🟥								
	Choose the best samples							🟥								
4	Submission of Progress Report II									▨ △						
5	Weathering test									🟥						
6	Fire test										🟥	🟥	🟥			
7	Seminar										▨ △					
8	SEM Analysis												🟥	🟥		
9	Poster Exhibition												▨ △			
10	Analysed The Result														🟥	
11	Submission of Dissertation Final Draft															▨ △
12	Oral Presentation									During Study Week						
13	Submission of Dissertation									7 Days After Oral Presentation						

LEGEND	
△	Milestone
🟥	Project Schedule
▨	FYP Schedule

3.3 Preparation of Expandable Graphite

3.3.1 Grinded graphite

The container of grinder is cleaned by using brush to make sure there is no dirt. The graphite is put into container. The container in the grinder is tightened. The time is set to approximately 15 second. “START” button is pushed.

3.3.2 Sieved graphite

Each container of siever is cleaned by using brush to make sure there is no dirt and impurities. The graphite is poured into the largest size of siever. All the siever is arranged from the largest size to the smaller one on the machine. The time is set to 10minutes. “START” button is pushed.

3.3.3 Treated graphite

Graphite is weighed. Potassium permanganate is weighed according to the formula. Both materials are mixed well by using spatula. The three necks round bottom flask are set up in a bowl of water on the hot plate. The mixing ingredients and a magnet are put into the three necks round bottom flask. Acetic acid is measured and then poured into the the three necks round bottom flask. The speed of hot plate is set to 2.5rpm Sulphuric acid is measured and then dropped into the three necks round bottom flask using a dropper. The mixture is left for one hour on the functioning hot plate. The mixing then is put into a beaker. Distilled water is added into the beaker. The liquid then is poured until colourless. The mixing is put in the oven for two to three hours in 60˚C temperature.

3.4 Preparation of Intumescent Coating

APP, BA and melamine are weighed according to the formulation. All these materials are grinded to make sure they are homogenously mixed. EG is weighed then combined with the grinded material. This is called as ingredient A. TETA and BPA are weighed based on the formulation. By using mixer, the binder is mixed for about 5 minutes. Ingredient A is poured into the binder then they are mixed again homogenously. The coating is applied on the structural steel until it covered all the surface of the substrate. The coating is cured in the room temperature for two weeks. The thickness of each sample is measured and the data is recorded. For coating using zirconium, step number 1-9 is repeated but the zirconium flour is added in the first step.

3.5 Weathering Test

Weathering chamber is set to 37˚C temperature and 70% relative humidity. The sample is weighed and the data is recorded. The sample is put in the weathering chamber for one week. After one week, the sample is taken out. The weight of the sample is measured again.

3.6 Fire Test

The sample is put in the furnace that has been set based on the parameters below:

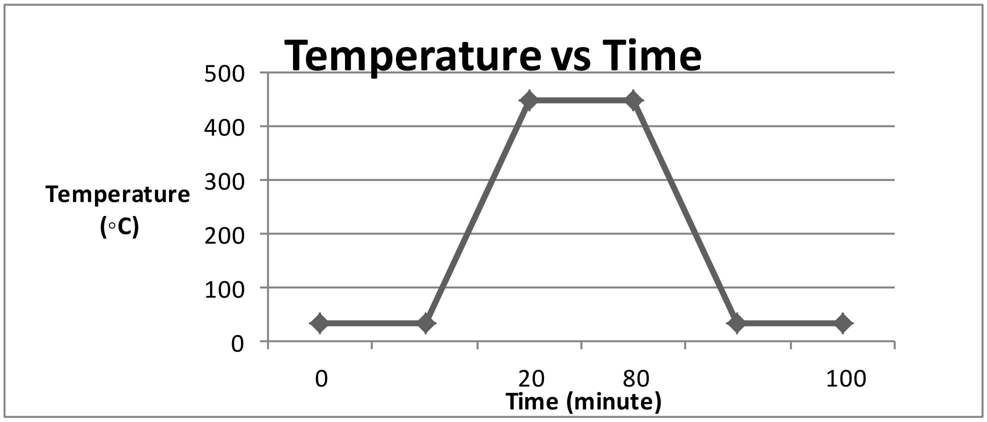


Figure3.2:Temperature vs Time

After about two hours, the sample is taken out from the furnace. The thickness is measured and recorded. Physical features of the coating are observed.

3.7 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity [11].

CHAPTER 4

RESULT AND DISCUSSION

4.1 Work Completed

Basically, all the process involved in finishing this project has been completed by the author. The preparation of expandable graphite has been successfully done by grinding, sieving and treating the graphite. Coating is developed based on the formulation. After the substrate is coated, weathering chamber test and fire test has been conducted. The result then is analysed by using Scanning Electron Microscope (SEM).

4.2 Data Presentation and Analysis

4.2.1 Intumescent Coating Using Boric Acid As Filler

Thirteen formulations have been developed using Taguchi Method. In the first stage, boric acid is used as filler. The table below shows the details of the formulation.

Table 4.1: Formulation using Boric Acid as filler

SAMPLE	WEIGHT PERCENTAGE (%)					
	EG	APP	MEL	BA	BPA	TETA
BI	11.11	11.11	5.56	5.56	44.44	22.22
B2	8.33	8.33	11.11	5.56	44.44	22.22
B3	11.11	5.56	11.11	5.56	44.44	22.22
B4	8.33	11.11	8.33	5.56	44.44	22.22
B5	5.56	11.11	11.11	5.56	44.44	22.22
B6	11.11	8.33	8.33	5.56	44.44	22.22
B7	5.56	8.33	11.11	8.33	44.44	22.22
B8	5.56	11.11	8.33	8.33	44.44	22.22
B9	8.33	8.33	8.33	8.33	44.44	22.22
B10	8.33	11.11	5.56	8.33	44.44	22.22
B11	5.56	5.56	11.11	11.11	44.44	22.22
B12	5.56	11.11	5.56	11.11	44.44	22.22
B13	5.56	8.33	8.33	11.11	44.44	22.22

4.2.2 Fire Test Using Furnace

Below is the thickness of the coating before the fire test:

Table 4.2: Thickness of coating before fire test

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
B1	3.98	4.32	4.20	3.80	4.36	4.20	4.14
B2	3.60	4.20	3.54	3.86	4.12	4.02	3.89
B3	3.96	4.12	3.90	3.76	4.18	4.02	3.99
B4	4.28	4.72	4.44	4.10	4.70	4.22	4.41
B5	4.60	4.88	4.14	4.30	4.12	4.60	4.44
B6	3.74	4.24	3.98	4.16	4.18	3.74	4.01
B7	4.72	4.64	4.58	4.40	4.50	4.12	4.49
B8	3.90	4.32	4.32	4.20	4.62	4.22	4.26
B9	3.74	3.92	3.84	3.96	4.16	3.66	3.88
B10	3.88	4.42	3.98	3.90	4.42	3.89	4.08
B11	3.94	4.52	4.34	4.38	4.66	4.24	4.35
B12	4.12	4.28	4.00	3.94	4.24	4.20	4.13
B13	4.12	4.72	4.14	4.72	3.96	4.26	4.32

After the test, the thickness of the coating is measured as in the table below:

Table 1.3: Thickness of coating after fire test

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
B1	8.68	8.32	8.38	8.64	8.54	8.68	8.54
B2	20.12	21.20	21.32	21.68	20.08	20.04	20.74
B3	10.00	10.10	10.06	10.98	10.14	10.02	10.22
B4	20.14	19.86	19.94	20.08	19.94	19.94	19.98
B5	11.02	11.06	11.02	11.14	11.08	11.04	11.06
B6	4.18	4.24	4.32	4.28	4.22	4.28	4.25
B7	4.12	4.28	4.40	4.40	4.50	4.22	4.32
B8	4.26	4.32	4.56	4.20	4.28	4.16	4.30
B9	8.32	8.40	8.38	8.32	8.38	8.38	8.36
B10	9.96	9.86	9.84	9.94	10.12	9.96	9.95
B11	13.08	12.86	13.24	13.18	13.16	13.10	13.10
B12	13.02	13.10	13.14	13.14	12.94	13.16	13.08
B13	9.08	8.86	8.82	8.94	8.98	8.92	8.93

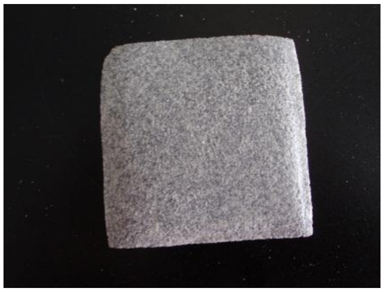
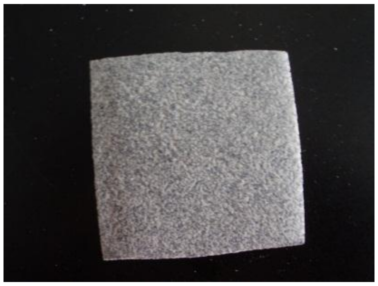
From the result obtained the analysis has been done to show how much differences occurred and percentage of the expansion in the data below:

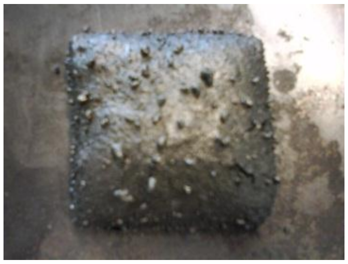
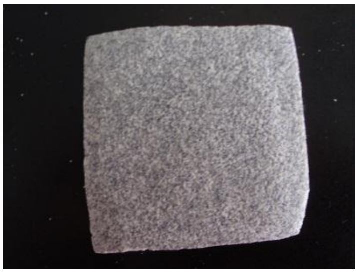
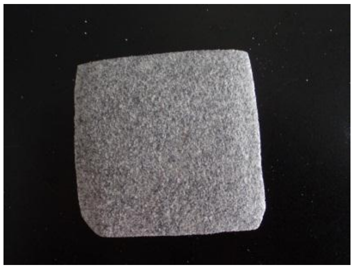
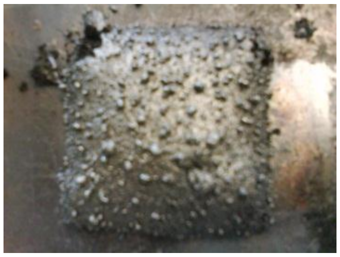
Table 4.4: Percentage of coating expansion

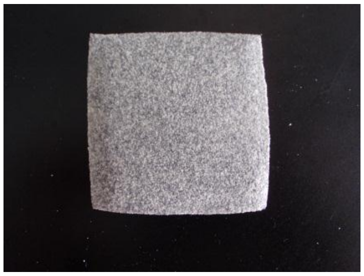
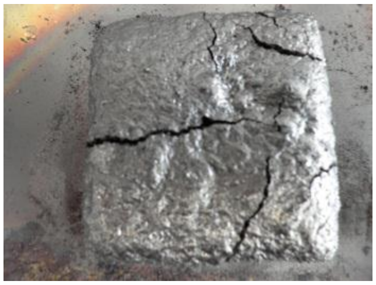
SAMPLE	**THICKNESS (mm)**		**PERCENTAGE OF EXPANSION (%)**
	Before Fire Test	**After Fire Test**	
B1	4.14	8.54	106.28
B2	3.89	20.74	433.16
B3	3.99	10.22	156.06
B4	4.41	19.98	353.14
B5	4.44	11.06	149.10
B6	4.01	4.25	6.07
B7	4.49	4.32	-3.79
B8	4.26	4.30	0.86
B9	3.88	8.36	115.55
B10	4.08	9.95	143.79
B11	4.35	13.10	201.23
B12	4.13	13.08	216.79
B13	4.32	8.93	106.79

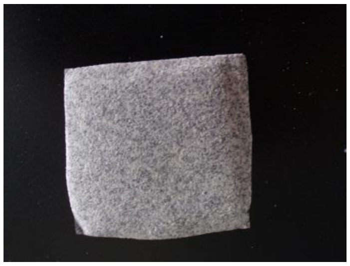
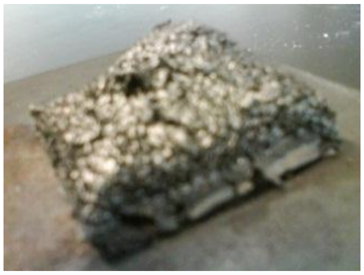
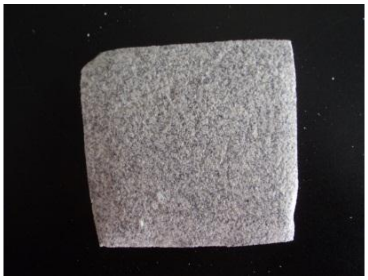
From the table, most of the samples expand more than 100%. However only three samples are not expanding which are sample B6, B7 and B8. The negative sign for B7 indicates that the coating is ruined and shattered after fire test.

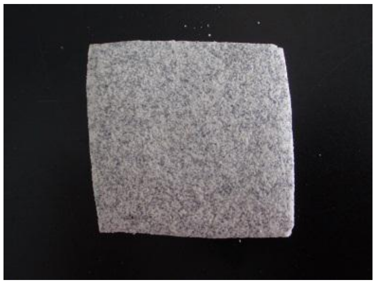
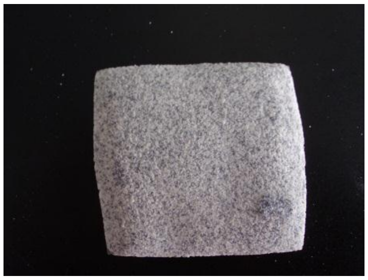
Observation to the coating surface has been done after the fire test. The differences before and after the fire test is shown as below:

SAMPLE	BEFORE FIRE TEST	AFTER FIRE TEST
B1	 Thickness of coating = 4.14mm	 Thickness of coating = 8.54mm Percentage of expansion = 106% (2 times of expansion) The coating is detached from the steel. The surface is cracked.
B2	 Thickness of coating = 3.89mm	 Thickness of coating = 20.74mm Percentage of expansion = 433% (5 times of expansion but expand unevenly) The coating is detached from the steel. The surface is rough.

B3	 Thickness of coating = 3.99mm	 Thickness of coating = 10.22mm Percentage of expansion = 156% (2.5 times of expansion) The coating is detached from the steel. The surface is rough.
B4	 Thickness of coating = 4.41mm	 Thickness of coating =19.98mm Percentage of expansion = 353% (4.5 times of expansion) The coating is detached from the steel at certain edge only. The surface is not too smooth.
B5	 Thickness of coating = 4.44mm	 Thickness of coating = 11.06mm Percentage of expansion = 149% (2.5 times of expansion) The coating is detached from the steel The surface is not too smooth.

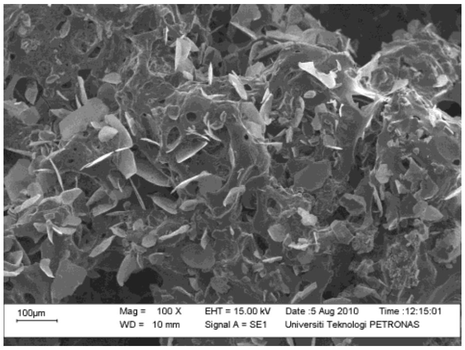
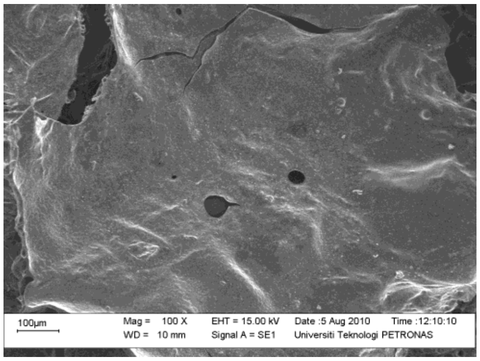
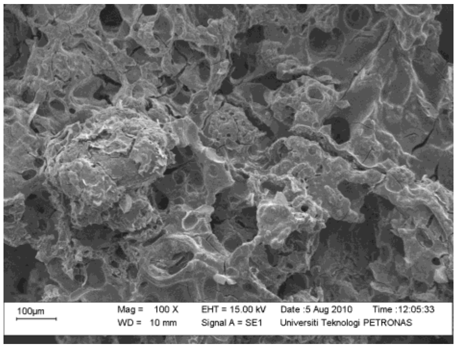
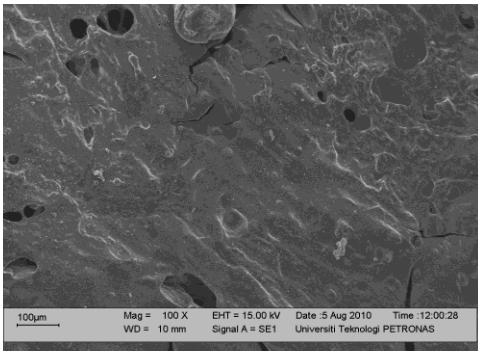
B6	 Thickness of coating = 4.01mm	 Thickness of coating = 4.25mm Percentage of expansion = 6% (no expansion) The coating is detached from the steel. The surface is rough.
B7	 Thickness of coating = 4.49mm	 Thickness of coating = 4.32 Percentage of expansion = -3.79 (no expansion) The coating is not detached from the steel. The surface is cracked.
B8	4  Thickness of coating = 4.26mm	 Thickness of coating = 4.30mm Percentage of expansion = 0.86% (no expansion) The coating is detached from the steel. The surface is cracked.

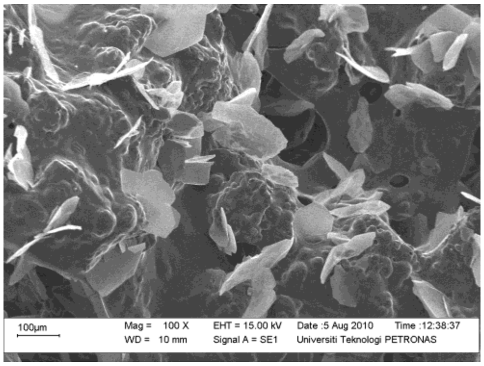
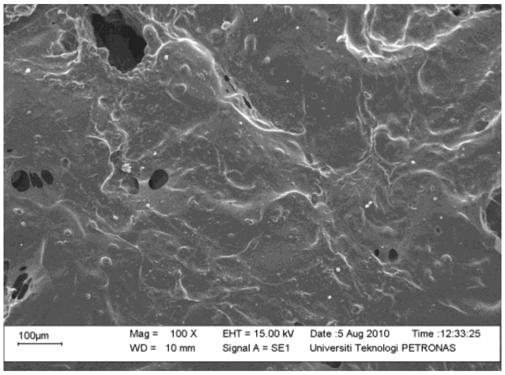
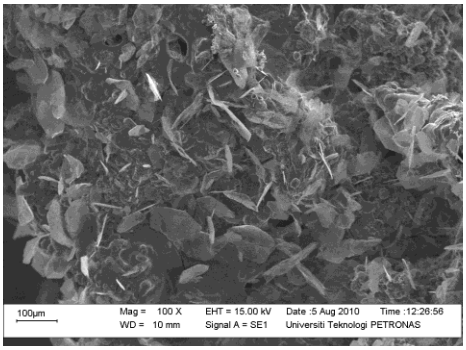
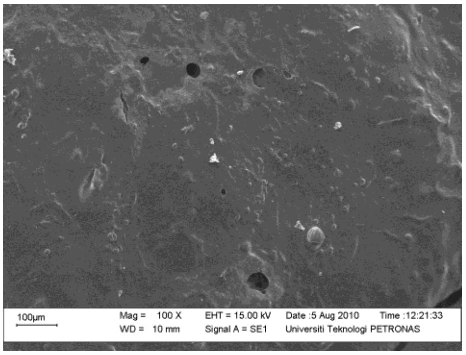
B9	 Thickness of coating = 3.88mm	 Thickness of coating = 8.36mm Percentage of expansion =115.55% (2 times of expansion) The coating is detached from the steel The surface is rough.
B10	 Thickness of coating = 4.08mm	 Thickness of coating = 9.95mm Percentage of expansion = 144% (2.4 times of expansion) The coating is not detached from the steel. The surface is rough.
B11	 Thickness of coating = 4.35mm	 Thickness of coating = 13.10mm Percentage of expansion = 201% (3 times of expansion) The coating is detached from the steel The surface is smooth.

B12	 Thickness of coating = 4.13mm	 Thickness of coating = 13.08mm Percentage of expansion = 217% (3 times of expansion) The coating is not detached from the steel The surface is smooth.
B13	 Thickness of coating = 4.32mm	 Thickness of coating = 8.93mm Percentage of expansion = 107% (2 times of expansion) The coating is not detached from the steel. The surface is rough.

4.2.3 Scanning Electron-Microscope (SEM) Analysis

From the result obtain, the best four (4) samples are analysed by using Scanning Electro-Magnetic (SEM) Machine. The analysis is described below:

B2  **Magnification: 100x (Inner layer)** The picture shows the coating has a lot of flakes. However, some holes and cracks can be seen.	**B2**  **Magnification: 100x (Outer layer)** The picture shows it has a lot of bubbles. Crack can also be seen and a little bit of holes is observed.
B4  **Magnification: 100x (Inner layer)** The picture shows a lot of holes and cracks. No flake can be seen.	**B4**  **Magnification: 100x (Outer layer)** The picture shows a lot of holes and cracks. There is also some bubbles.

B11  **Magnification: 100x (Inner layer)** This picture shows that this coating also have a lot of flakes crack is seen in this coating.	**B11**  **Magnification: 100x (Inner layer)** This picture shows the surface is rough and uneven. It has holes and some bubbles but no crack.
B12  **Magnification: 100x (Inner layer)** The picture shows that the coating has a lot of flakes This sample has no holes and bubbles inside.	**B12**  **Magnification: 100x (Outer layer)** The picture shows that this surface has only a few numbers of holes. Uneven surface is observed.

The formation of bubbles is due to the immersion of nitrogen, ammonia and carbon dioxide gas. Less bubble is better however if the bubble is too many and uniform on the surface, it is good as the gaseous will be released to make the expansion of the coating become higher.

Dehydration of water contributes to the formation of holes. Fewer holes are better because the hole can penetrate the heat to the substrate. Therefore, the time before failure will be shorter.

Cracks should be less too as it can allow the heat to go direct to the substrate where the coating cannot prevent any more to the substrate from failure in a short time.

4.2.4 Intumescent Coating Using Boric Acid And Zirconium As Filler

From the previous test result, the best result is set as reference to develop another coating by adding zirconium flour. Specific criteria must be fulfilled in order to choose the best result which is the most expanding coating, has the best char layer and adhesion. Based on these, formulation of B4, B11 and B12 are chosen to be the reference formulation to the next stage of coating development. The outcome is tabled below:

Table 4.5: Formulation using boric acid and zirconium as filler

SAMPLE	WEIGHT PERCENTAGE (%)						
	EG	**APP**	**MEL**	**BA**	**Zr**	**BPA**	**TETA**
Z-4-A	7.89	10.53	7.89	5.26	5.26	42.12	21.05
Z-11-A	5.26	5.26	10.53	10.53	5.26	42.11	21.05
Z-12-A	5.26	10.53	5.26	10.53	5.26	42.11	21.05
Z-4-B	7.50	10.00	7.50	5.00	10.00	40.00	20.00
Z-11-B	5.00	5.00	10.00	10.00	10.00	40.00	20.00
Z-12-B	5.00	10.00	5.00	10.00	10.00	40.00	20.00
Z-4-C	7.14	9.52	7.14	4.76	14.29	38.10	19.05
Z-11-C	4.76	4.76	9.52	9.52	14.29	38.10	19.05
Z-12-C	4.76	9.52	4.76	9.52	14.29	38.10	19.05

4.2.5 Fire Test Using Furnace

Below is the thickness of the coating before the fire test:

Table 4.6: Thickness of coating before fire test (with zirconium)

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
Z-4-A	4.32	5.74	5.08	5.40	5.36	4.98	5.15
Z-11-A	4.90	5.32	4.82	5.68	5.28	6.80	5.47
Z-12-A	4.80	5.64	5.00	5.22	5.00	5.20	5.14
Z-4-B	4.34	4.90	3.46	4.28	4.86	4.62	4.41
Z-11-B	4.98	5.48	4.34	4.98	5.02	5.58	5.06
Z-12-B	4.38	5.24	4.82	5.22	3.44	4.52	4.60
Z-4-C	5.40	5.66	5.76	5.70	5.70	5.90	5.69
Z-11-C	4.72	5.30	4.72	4.94	4.70	5.44	4.97
Z-12-C	5.08	6.60	6.08	6.54	6.18	6.26	6.12

After the test, the thickness of the coating is measured as in the table below:

Table 4.7: Thickness of coating after fire test (with zirconium)

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
Z-4-A	23.18	23.22	23.14	23.14	23.14	23.06	23.15
Z-11-A	21.68	21.96	21.88	21.84	21.96	21.92	21.87
Z-12-A	15.34	15.36	15.86	15.88	15.48	15.64	15.59
Z-4-B	17.64	17.72	17.68	17.58	17.72	17.74	17.68
Z-11-B	20.18	20.26	20.18	20.14	20.18	20.18	20.19
Z-12-B	15.88	15.86	15.64	15.72	15.74	15.66	15.75
Z-4-C	27.68	28.24	28.18	28.4	27.88	28.28	28.11
Z-11-C	21.38	21.46	21.36	21.28	21.36	21.32	21.36
Z-12-C	21.4	21.44	21.46	21.42	22.02	21.58	21.55

From the result obtained the analysis has been done to show how much differences occurred and percentage of the expansion in the data below:

Table 4.8: Percentage of coating expansion (with zirconium)

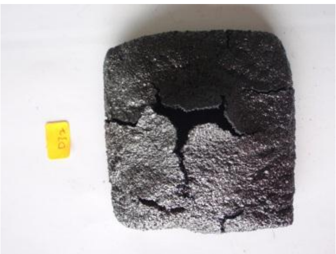
FORMULA NO.	THICKNESS (mm)		PERCENTAGE OF EXPANSION (%)
	Before Fire Test	After Fire Test	
Z-4-A	5.15	23.15	349.51
Z-11-A	5.47	21.87	299.82
Z-12-A	5.14	15.59	203.31
Z-4-B	4.41	17.68	300.91
Z-11-B	5.06	20.19	299.01
Z-12-B	4.6	15.75	242.39
Z-4-C	5.69	28.11	394.02
Z-11-C	4.97	21.36	329.78
Z-12-C	6.12	21.55	252.12

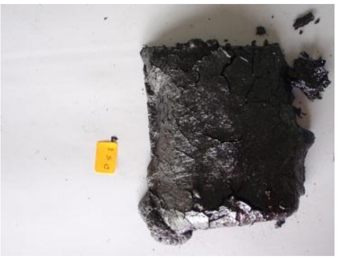
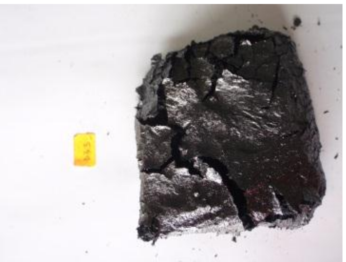
All of the samples expand more than 100%. It shows that most of the samples expand more than 2 times from the initial thickness.The result after fire test using furnace is shown as below:

SAMPLE	BEFORE FIRE TEST	AFTER FIRE TEST
Z-4-A	 Thickness of coating = 5.15mm	 Thickness of coating = 23.15mm Percentage of expansion = 350% (4.5 times of expansion) The coating is not detached from the steel. The surface is smooth but has a little crack at the edge.

Z-11-A	 Thickness of coating = 5.47mm	 Thickness of coating = 21.87mm Percentage of expansion = 300% (4 times of expansion) The coating is not detached from the steel The surface is smooth.
Z-12-A	 Thickness of coating = 5.14mm	 Thickness of coating = 15.49mm Percentage of expansion = 203% (3 times of expansion) The coating is not detached from the steel The surface is smooth but has a few cracks.

Z-4-B	 Thickness of coating = 4.41mm	 Thickness of coating = 17.68mm Percentage of expansion = 301% (4 times of expansion) The coating is not detached from the steel The surface is smooth.
Z-11-B	 Thickness of coating = 5.06mm	 Thickness of coating = 20.19mm Percentage of expansion = 299% (4 times of expansion) The coating is not detached from the steel The surface is smooth

Z-12-B	 Thickness of coating = 4.60mm	 Thickness of coating = 15.75mm Percentage of expansion = 242% (3.4 times of expansion) The coating is not detached from the steel The surface is smooth but has a big crack.
Z-4-C	 Thickness of coating = 5.69mm	 Thickness of coating = 28.11mm Percentage of expansion = 394% (5 times of expansion) The coating is not detached from the steel The surface is rough.

Z-11-C	 Thickness of coating = 4.97mm	 Thickness of coating = 21.36mm Percentage of expansion = 330% (4.3 times of expansion) The coating is not detached from the steel The surface is smooth
Z-12-C	 Thickness of coating = 6.12mm	 Thickness of coating = 21.55mm Percentage of expansion = 252% (3.5 times of expansion) The coating is detached from the steel The surface is cracked.

4.2.6 Weathering Chamber Test

After all the samples have been taking out from the weathering chamber, some observation has been conducted. It shows that the surface colour of the coating is changed to yellowish. This is due to the explosion to the Ultra-Violet (UV) light. The weight is also measured. Below is the weight of the sample before and after the weathering chamber test:

Table 4.9: Weight of samples before and after weathering test

SAMPLE	WEIGHT (g)		PERCENTAGE DIFFERENCES (%)
	Before Test	After Test	
B4	39.003	38.952	0.13
B11	40.398	40.353	0.11
B12	37.791	37.738	0.14
Z-4-A	43.847	43.825	0.05
Z-11-A	45.11	45.066	0.10
Z-12-A	42.448	42.397	0.12
Z-4-B	44.927	44.902	0.06
Z-11-B	43.466	43.447	0.04
Z-12-B	47.136	47.076	0.13
Z-4-C	48.154	48.104	0.10
Z-11-C	44.454	44.314	0.31
Z-12-C	49.915	49.746	0.34

Al the samples have lost the weight not more than 1%. Not too many weight is lost due to the duration of exposure to the simulated weathering which is only one week.

As usual, the thickness of the coating before and after fire test is measured. The data is presented in the tables below:

Table 4.10: Thickness of coating before fire test (after weathering test)

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
B4	4.24	4.68	4.62	4.20	4.42	4.32	4.41
B11	4.52	4.36	4.28	4.38	4.46	4.10	4.35
B12	4.34	4.10	4.02	4.12	4.16	4.04	4.13
Z-4-A	5.36	4.76	5.28	4.68	5.46	5.38	5.15
Z-11-A	5.52	5.72	5.52	5.38	5.16	5.54	5.47
Z-12-A	5.04	5.14	5.24	5.22	5.04	5.14	5.14
Z-4-B	5.12	4.92	4.78	5.42	4.98	5.04	5.04
Z-11-B	4.96	4.92	4.94	5.14	5.44	4.98	5.06
Z-12-B	4.88	4.72	4.78	4.88	4.40	4.26	4.65
Z-4-C	5.48	5.58	5.14	5.22	5.12	5.04	5.26
Z-11-C	5.34	5.18	5.08	5.12	5.16	5.18	5.18
Z-12-C	5.22	4.98	4.98	4.86	5.08	5.22	5.06

Table 4.11 Thickness of coating after fire test (after weathering test)

SAMPLE	READING 1(mm)	READING 2(mm)	READING 3(mm)	READING 4(mm)	READING 5(mm)	READING 6(mm)	AVERAGE THICKNESS (mm)
B4	17.96	17.56	17.42	17.66	17.26	17.48	17.56
B11	4.38	4.40	4.62	5.02	4.90	4.76	4.68
B12	14.46	14.16	14.28	14.42	14.62	13.88	14.30
Z-4-A	11.04	11.10	10.88	10.82	10.50	9.88	10.70
Z-11-A	5.32	5.48	.5.62	5.64	5.64	5.16	4.54
Z-12-A	13.16	13.18	13.32	13.40	13.64	13.58	13.38
Z-4-B	11.18	11.16	11.38	11.24	11.26	11.38	11.27
Z-11-B	4.78	5.44	5.32	5.42	5.18	4.92	5.18
Z-12-B	15.52	15.44	15.14	14.98	15.32	15.52	15.32
Z-4-C	17.66	17.82	17.52	17.42	17.16	17.18	17.46
Z-11-C	10.78	10.68	11.04	11.08	10.98	10.38	10.82
Z-12-C	7.78	7.72	8.14	8.22	7.88	8.04	7.96

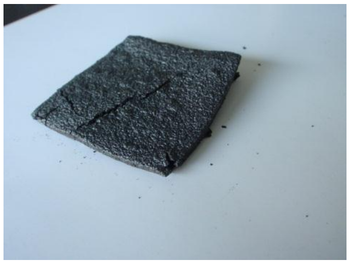
Therefore, the percentage of expansion can be summarised as follow:

Table 4.12: Percentage of coating expansion (after weathering test)

SAMPLE	THICKNESS (mm)		PERCENTAGE OF EXPANSION (%)
	Before Fire Test	**After Fire Test**	
B4	4.41	17.56	297.81
B11	4.35	4.68	7.59
B12	4.13	14.30	246.33
Z-4-A	5.15	10.70	107.70
Z-11-A	5.47	4.54	-17.05
Z-12-A	5.14	13.38	160.48
Z-4-B	5.04	11.27	123.40
Z-11-B	5.06	5.18	2.24
Z-12-B	4.65	15.32	229.23
Z-4-C	5.26	17.46	231.73
Z-11-C	5.18	10.82	109.08
Z-12-C	5.06	7.96	57.48

Most of the samples expand more than 100% but there are still one sample which is not expanding. This Z-11-A sample is scattered and ruined after the fire test is conducted. Which is indicates by the negative sign.

The fire test using furnace is conducted to see the performances after being exposed in the weathering chamber. The following analysis is the result obtains from the observation:

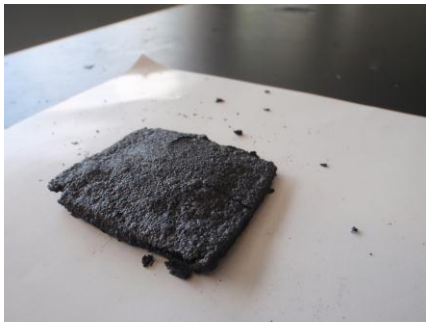
SAMPLE	BEFORE FIRE TEST	AFTER FIRE TEST
B4	 Thickness of coating =4.41mm	 Thickness of coating = 17.56mm Percentage of expansion = 298% (4 times of expansion) The coating is not detached from the steel. The surface is smooth.
B11	 Thickness of coating = 4.35mm	 Thickness of coating = 4.68mm Percentage of expansion = 8% (no expansion) The coating is detached from the steel. The surface is smooth but cracked.

B12	 Thickness of coating = 4.13mm	 Thickness of coating = 14.30mm Percentage of expansion = 246% (3.5 times of expansion) The coating is not detached from the steel. The surface is smooth.
Z-4-A	 Thickness of coating = 5.15mm	 Thickness of coating = 10.70mm Percentage of expansion = 108% (2 times of expansion) The coating is not detached from the steel. The surface has a few cracks.

Z-11-A	 Thickness of coating = 5.47mm	 Thickness of coating = 5.54mm Percentage of expansion = 1% (no expansion) The coating is detached from the steel The surface is smooth.
Z-12-A	 Thickness of coating = 5.14mm	 Thickness of coating = 13.38mm Percentage of expansion = 160% (2.6 times of expansion) The coating is detached from the steel The surface is smooth

Z-4-B	 Thickness of coating = 5.04mm	 Thickness of coating = 11.27mm Percentage of expansion = 124% (2.2 times of expansion) The coating is detached from the steel The surface is smooth but has crack
Z-11-B	 Thickness of coating = 5.06mm	 Thickness of coating = 5.18mm Percentage of expansion = 2% (no expansion) The coating is detached from the steel. The surface has cracks.

Z-12-B	 Thickness of coating = 4.60mm	 Thickness of coating = 15.32mm Percentage of expansion = 233% (3.3times of expansion) The coating is detached from the steel The surface is smooth.
Z-4-C	 Thickness of coating = 5.26mm	 Thickness of coating = 17.46mm Percentage of expansion = 232% (3.3 times of expansion) The coating is not detached from the steel The surface is smooth.

Z-11-C	 Thickness of coating = 5.18mm	 Thickness of coating = 10.82mm Percentage of expansion = 108% (2.1 times of expansion) The coating is detached from the steel The surface is smooth.
Z-12-C	 Thickness of coating = 5.06	 Thickness of coating = 7.96mm Percentage of expansion = 57% (1.6 times of expansion) The coating is detached from the steel The surface is smooth but has crack at the edge.

4.3 Discussion

After gathering all the result, it can be summarized that expansion of coating with zirconium flour as additive is better than using boric acid alone. The range of expansion for coating without zirconium is between 0 to 5 times of expansion from the original meanwhile for coating with zirconium is between 3 to 5 times of expansion from the original as shown in the figure 4.1 and figure 4.2. This is due to the function of zirconium that can reduce the flammability hence can sustain heat for a longer time.

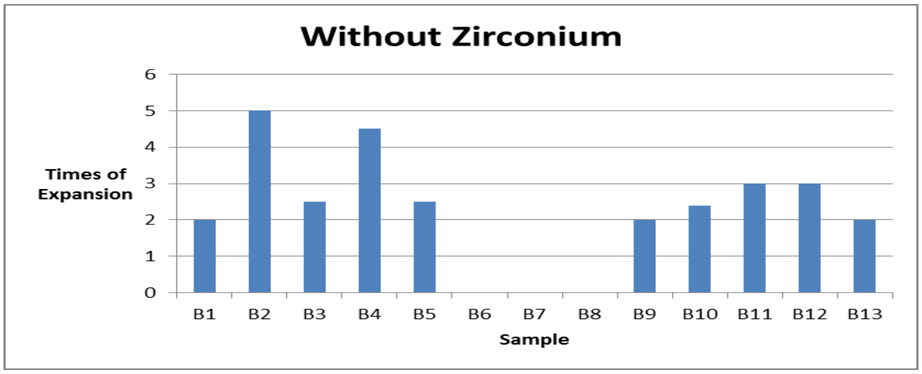


Figure 4.1: Expansion of coating without zirconium

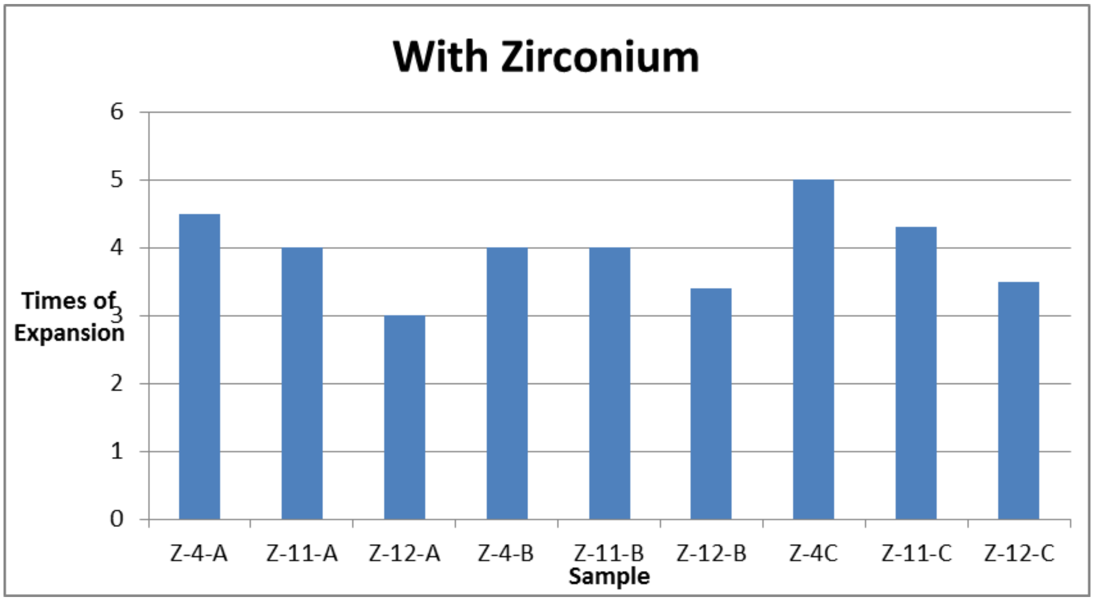


Figure 4.2: Expansion of coating with zirconium

Difference performance of expansion can be seen after the samples are exposed to the simulated weather. Figure 4.3below shows that the expansion of coating that has never been exposed to weather are having greater performance where the highest expansion is up to 5 times from the initial thickness. Meanwhile, for the coating that has been put in the weathering chamber, the highest expansion is only 4 times from the initial thickness. The below graph is also shows that none of the samples after weathering test are having higher expansion compared to the non-weathered sample.

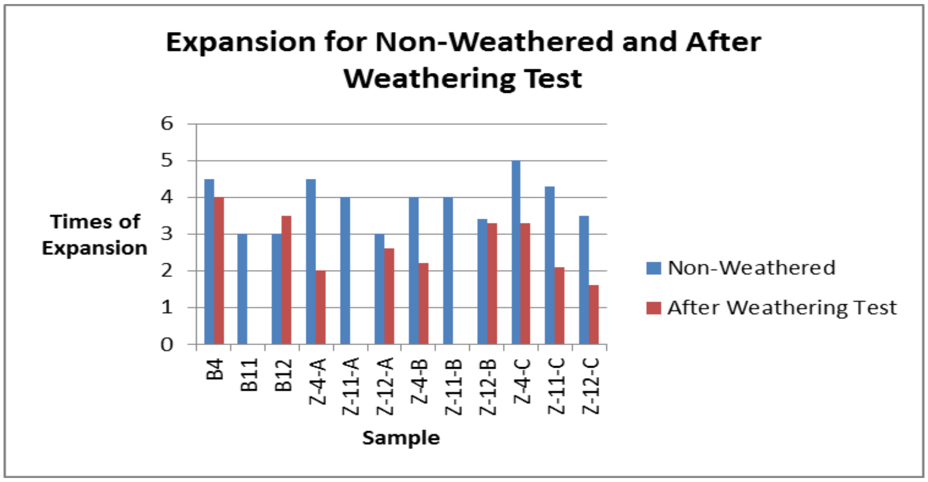


Figure 4.3: Expansion of coating for non-weathered and after weathering test

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This project has revealed that weather affects the performance of intumescent fire retardant coating in term of its expansion. From the result obtained, it shows that the weathering effect degrade the coating's expansion. This is due to the leaching out of its active ingredients. The coating expands more when they do not expose to the simulated weather. The usage of boric acid alone and the combination with zirconium flour as filler to the coating also have an implication. Coating with zirconium flour has greater expansion compared to the coating without zirconium flour. Therefore, there is no doubt that the weathering has reduces the expansion of the fire retardant coating and the usage of zirconium has improved the performance.

3.6 Recommendation

There is still room for development in working on future project. The size of graphite can be variety either become bigger or smaller. Other than that, another parameter of weathering properties are recommended to be studied such as humidity, oxidization and industrial atmosphere. Besides, the duration of time exposed to weather can be studied too for knowing the exact limit of the coating's endurance before it fails to protect the substrate. For fire test, the usage of Bunsen burner can be done to study how the performance differs when using a direct fire.

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