CHAPTER 1

INTRODUCTION

1.1 Background

Coating is a covering that is applied to the surface of an object, usually referred to as the substrate. In many cases coatings are applied to improve surface properties of the substrate, such as appearance, adhesion, wetability, corrosion resistance, wear resistance, and scratch resistance. Coatings may be applied as liquids, gases or solids [1]. Intumescent coating represents an increasingly used way to provide fire protection to the structural steel in the construction industry [2 ,3]. Upon heating, intumescent coating could swell and formed intumescent char layer, thus protecting the structural steel. Expandable graphite is the new intumescent additives and could provide good fire protection properties.

1.2 Problem Statement

Due to the poor thermal properties of Intumescent Fire Retardant Coating, the coating oxidizes and detached from the substrate material at high temperature and looses their fire protection properties. Besides, intumescent coating needs to protect the steel structure within 1 to 3 hours. Thereby, in order to maintain fire protection of the coating, it is necessary to improve anti-oxidation of the char layer with the improvements in char expansion in order to reduce the heat transfer to the substrate.

1.3 Objective

The objective of this project is to study the fire protection performance by using Expandable Graphite and Zinc Borate as additives. In doing so, the char expansion of intumescent coating composition will be measured and the char structure after fire test will be studied.

1.4 Scope of study

There are some limitations in this project. Expandable Graphite with particle size of 300 micron meter is used as the carbon source and Zinc Borate as the additives in intumescent coating. As for the substrate, mild steel is chosen. Furnace and Bunsen burner will be done as the fire test. Standard procedure of fire test is described in ISO/TR 834-3: 1994 standard [8]. The temperature of fire should be above 220^{o} for expandable graphite to start react. In order to measure the thickness of the char, Vanier caliper will be used. As for the analyzing part, Scanning Electron Microscopy (SEM) is chose to check the structure of the char after fire test.

CHAPTER 2

LITERATURE REVIEW

2.1 What is Traditional Coating?

Traditional coatings consist of three active ingredients: an acid source (ammonium polyphosphate, APP), a carbon source (pentaerythritol, PER) and a blowing agent (melamine, MEL) bound together by a binder. Although APP-PER-MEL coating has good intumescent and char formation, hydrophilic flame-retardant activities in the coating are very sensitive to water and char structure formed from the coating is easily oxidized at high temperature [2 ,3].

2.2 What is Intumescent Coating?

An intumescent is a substance which swells as a result of heat exposure, thus increasing in volume, and decreasing in density. Intumescents are typically used in passive fire protection [8] . Intumescent coatings are relatively thin-film products that expand rapidly in a fire to insulate steel. They come in various propriety formulas that include a mixture of binders and acids that react under temperature to expand up to many times the original thickness of the film, creating a char that insulates steel [7]. Expandable Graphite (EG) is a new generation of intumescent additives that can provide the good fire retardancy to various materials .EG is a kind of exfoliated expanding particles that have been treated with an agent that intercalates into the crystal structure of the graphite. The intercalated particles under heat source can expand in the direction perpendicular to the carbon layers in the crystal structure [4, 5]. Whereas, zinc borate is an inorganic compound. Zinc borate is primarily used as a flame retardant in plastics and cellulose fibers, paper, rubbers and textiles pigments. As a flame retardant, it can replace antimony trioxide as a synergist in both halogen-based and halogen-free systems. It is an anti-dripping and char-promoting agent, and suppresses the afterglow. Zinc borate also can be used not only as fire retardants, but also to reduce smoke release and after-glowing of the coating [6].

2.3 Types of Intumescent Char

There two types of Intumescent Char which are soft char producers and hard char producers. ;

2.3.1 Soft char producers

These intumescents produce a light char, which is a poor conductor of heat, thus retarding heat transfer. Typically, these materials also contain a significant amount of hydrates. As the hydrates are spent, water vapor is released, which has a cooling effect. Once the water is spent, it is only the insulation characteristics of the char that was produced, which can slow down heat transfer from the exposed side to the unexposed side of an assembly. Soft char producers are typically used in thin film intumescents for fireproofing of structural steel as well as firestop pillows. Typically, the expansion pressure that is created for these products is very low, because the soft carbonaceous char has little substance, which is beneficial if the aim is to produce a layer of insulation [8].

2.3.2 Hard expanding char producers

Harder chars are produced with sodium silicates and graphite. These products are suitable for use in plastic pipe firestops as well as exterior steel fireproofing. In those applications, it is necessary to produce a more substantial char, with a quantifiable expansion pressure. In the case of the firestops, a melting, burning plastic pipe must be squeezed together and shut so that there will be no hole for fire to go through an opening in an otherwise fire-resistance rated wall or floor assembly. In the case of the exterior fireproofing, a hydrocarbon fire must be held off with quite potentially more kinetic energy than a house fire. Intumescents that produce hard chars are not typically used for interior spray fireproofing as they are not suitable for that application [8].

2.4 Material of Intumescent Coating

Intumescent coating consist of three main ingredients, acid source (such as ammonium polyphosphate, APP, mineral acid), carbon source(such as expandable graphite, sugar, starch) and blowing agent (such as melamine, carbon dioxide, water) and bound together by a binder (such as epoxy consist of Bisphenol A and Tetraethylene Amine). In recent research, additives such as boric acid, silica sand and zinc borate is added to improve fire protection properties.

2.5 Intumescent Coating Process

Process of making intumescent coating starts from treating graphite until applying the mixture of coating onto the substrate. Firstly, graphite is grinded by using grinder to get smaller particle size. Secondly, graphite is sieved to categorize its particle size (such as 300μm-200 μm). Next, graphite is treated with strong acid to soak and produce expandable graphite. Then, intumescent components which are acid source, carbon source, blowing agent and binder are mix well based on the weight percentage formulation and bound together by using binder. Lastly, mixture is put on substrate and let it dry in room temperature.

2.6 Expandable Graphite as Carbon Source

Graphite is a polymorph of the element carbon. Graphite has a sheet like structure where the atoms all lie in a plane and are only weakly bonded to the graphite sheets above and below [12]. While EG is a graphite intercalation compound (GIC). Compare with other pure carbon material like diamond and C60, the special layer lattice structure of flake graphite makes it possible to be intercalated by other chemicals. Flake graphite stems from nature graphite mine. The graphite ore is milled and then floated. By this processing, the purity of flake graphite can reach about 95%. To take out the remained organic and inorganic impurities and get higher purity, we have to use strong acid to soak the graphite and to bake the graphite together with alkali at high temperature. Then

after washing, we can get the graphite with purity up to 99.5%.
With flake graphite, the expandable graphite can be made through intercalate acid (sulphuric acid, nitric acid, and acetic acid are the commonly used acids) into the crystal layer of the graphite. To introduce such acid into the graphite layer, oxidants like H2O2, HNO3 or KMnO4 is necessary to be used. Or we can use electrochemical method to prepare GIC. After reaction, neutralizing, washing and drying are necessary.
For very long time, expandable graphite was mainly used as an intermediate. After expansion at 1000°C in oven, we get expanded graphite which could be used to make graphite sheet and graphite packing; an important sealing material in industry. As an intermediate product, expandable graphite had only a few specifications. Most important items were particle size, expansion volume, carbon content and volatiles. In some area, low sulphur content was also very important. Expandable graphite is halogen-free and works mainly in the condense phase. For this reason, expandable graphite can strongly reduced the smoke density in most cases. The expanded carbon layer works as an insulating layer to reduce the heat transfer [13].

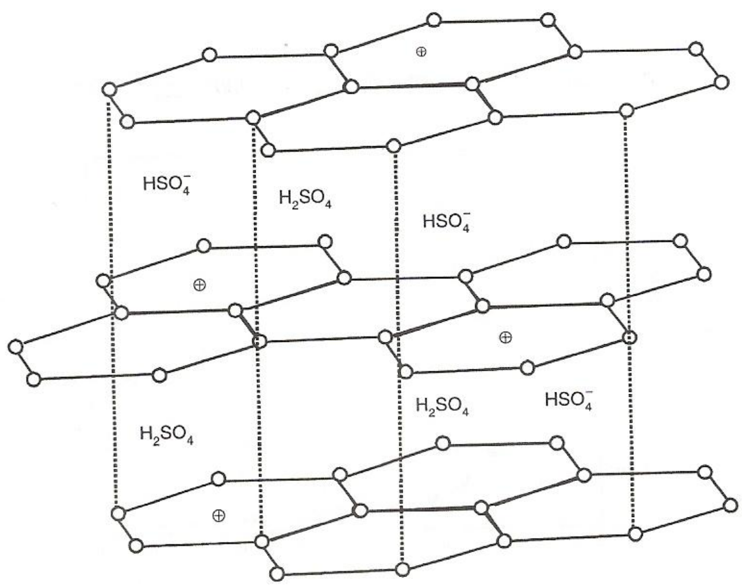


Figure 2.1: Structure of Expandable Graphite intercalated by H_2SO_4

2.7 Structural Steel as Substrate

Structural steel loses an appreciable part of its load carrying ability when its temperature exceeds 500^{o}C. The protection of metallic against fire has become an important issue in the construction industry. Indeed, prevention of the structural collapse of the building is paramount to ensure the safe evacuation of people from the building and is a prime requirement of building regulations in many countries. Exposure to fire will subject structural steel to thermally induce environmentally conditions that may alter its properties. Fire represents a transfer of energy from a stable condition to a transient condition as combustion occurs. During the process, steel temporarily absorbs a significant amount of thermal energy. Subsequently, the steel structure returns either to stable or unstable condition after cooling to ambient temperatures. During this cycle, individual structural steel may become badly bend or damaged without affecting the stability of the whole structure. It is possible to predict the range of temperature that a particular steel member of a building experienced during a fire using heat transfer theories. Besides of their relatively low carbon and other alloying content, structural steel usually regain close to 100 percent of their pre-heated properties provided the steel temperature did not exceed approximately 1330 degrees F. Steel protection means fireproofing and compartment ventilation conditions can affect the time-temperatue history. Theoretical studies by Iding (1977) that were calibrated with test results indicate that structural steel members in a building that had 2, 3 or more hour rated protection would not experienced any meaningful temperature change. Many procedures are available to assess structural steel integrity after fire exposure including visual observations, non destructive testing and destructive testing. The last method can incorporate chemical compound analysis obtaining physical properties such as ductility and toughness, residual stress determination and distribution, and metallographic observations. Each procedure will be assessed to examine its relative value in predicting structural steel conditions after fire exposure [17].

2.8 Synergistic Effect of Flame Retardancy

Tests showed that expandable graphite had good flame retardancy comparing with other halogen-free flame retardants. Recent research project "Flame retarded PP/Natural Fiber Compounds " showed that in polypropylene (PP), compare with other halogen-free flame retardants like ammonium polyphosphate (APP), zinc borate(ZB), melamine cyanurate (MC) , expandable graphite had a good effect in reducing the burning rate and increasing the low oxygen index (LOI). Cone calorimeter test is used to show peak heat release rate (pHRR). Recent research showed that expandable graphite combined with some halogen-free flame retardant additives, such zinc borate, phosphorus-nitrogen containing compound NP28, and microencapsulated red phosphorus, can apparently increase the limiting oxygen index value and improve the flammability properties of polyolefin blends. The data obtained from the Cone calorimeter test indicate that the heat release rate and effective heat of combustion decrease and the residues of carbonaceous chars increase remarkably with addition of expandable graphite and the other halogen-free flame retardant additives. The thermal analysis data give the positive evidence that the decreases of heat release rate and effective heat of combustion values of the flame retarded polyolefin materials are due to the increase of oxidative degradation temperature and the decrease of oxidization heat [13].

2.9 Fire Test

A fire test is a means of determining whether or not fire protection products meet minimum performance criteria as set out in a building code or other applicable legislation. Successful tests in laboratories holding national accreditation for testing and certification result in the issuance of a certification listing. Fire tests are conducted both on active fire protection and on passive fire protection items. Each has different test methods and scales. Active fire protection (AFP) is an integral part of fire protection. AFP is characterized by items and/or systems, which require a certain amount of motion and response in order to work. AFP includes manual or automatic fire detection and fire suppression. While passive fire protection (PFP) is an integral component of the three components of structural fire protection and fire safety in a building. PFP attempts to contain fires or slow the spread, through use of fire-resistant walls, floors, and doors. A fire test can also mean an ad hoc test performed to gather information in order to understand a specific hazard, such as a construction or storage configuration. Tests can be bench scale (flammable liquid flash point), medium scale (storage commodity classification), or full scale (replication of an entire rack storage configuration). Typical information gathered from full scale testing is heat release rate vs. time, smoke production and species composition, radiant heat, and interaction with fire control or suppression systems [14]. Bunsen Burner Test is used to do the fire test. This test has been used to characterize some properties of the char. A high temperature about 1000^{o}C is applied with a Bunsen Burner on to coated plate mounted vertically [16] as shown in Figure 2.2;

Figure 2.2; Bunsen Burner Test

A standardized procedure of fire test is described in ISO/TR 834-3 standard and a standardized thermal load in the standard is described by

$$T - T_o = 345 \log (8t + 1) \qquad (1)$$

Where T is the fire temperature (^{o}C), T_o is the temperature at the start of the fire (^{o}C) ant t is time of thermal load (minute). To determine the fire protection properties of coatings, the standardized fire test can be performed as the reference of the practical fire. In a fire test the coated test plates is placed on a hot source that was capable of providing the standardized thermal load. The standardized thermal load may be represented by time-temperature curve in Figure 2.3. The flame retardant coatings with a film thickness of 1mm were applied over the surface of bare steel plate and fire protection properties of the coatings were investigated. The back temperature of the back plate is recorded by using thermocouples. When the average back temperatures reached 300 ^{o}C, the time is defined as fire resistance time. The fire resistant time and thickness of char layer is used as judgment of fire resistant properties of coatings[16].

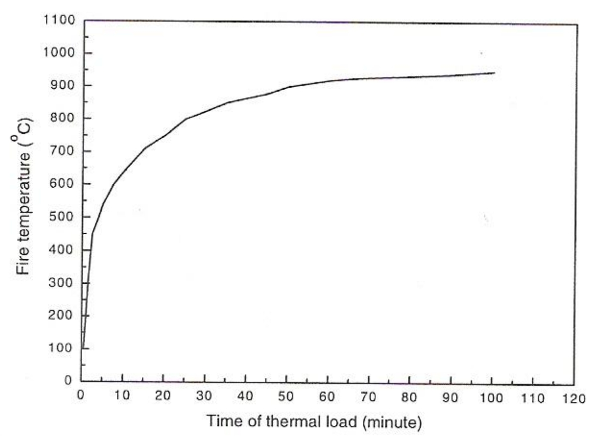


Figure 2.3: Time-temperature curve of ISO/TR 834-3 fire test

2.10 Equipment for Testing

The equipment used to check the char structure of the intumescent coating after fire test is Scanning Electron Microscopy (SEM). SEM is type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern [10]. The purpose is to observe the morphologies of char samples and analyze the concentration of elements in some parts of char samples [16].

CHAPTER 3

METHODOLOGY

3.1 Research

In pursuing this research project, Figure 3.1 illustrates the steps taken. Starting from grinding and sieving of graphite until analyzing the char after fire test.

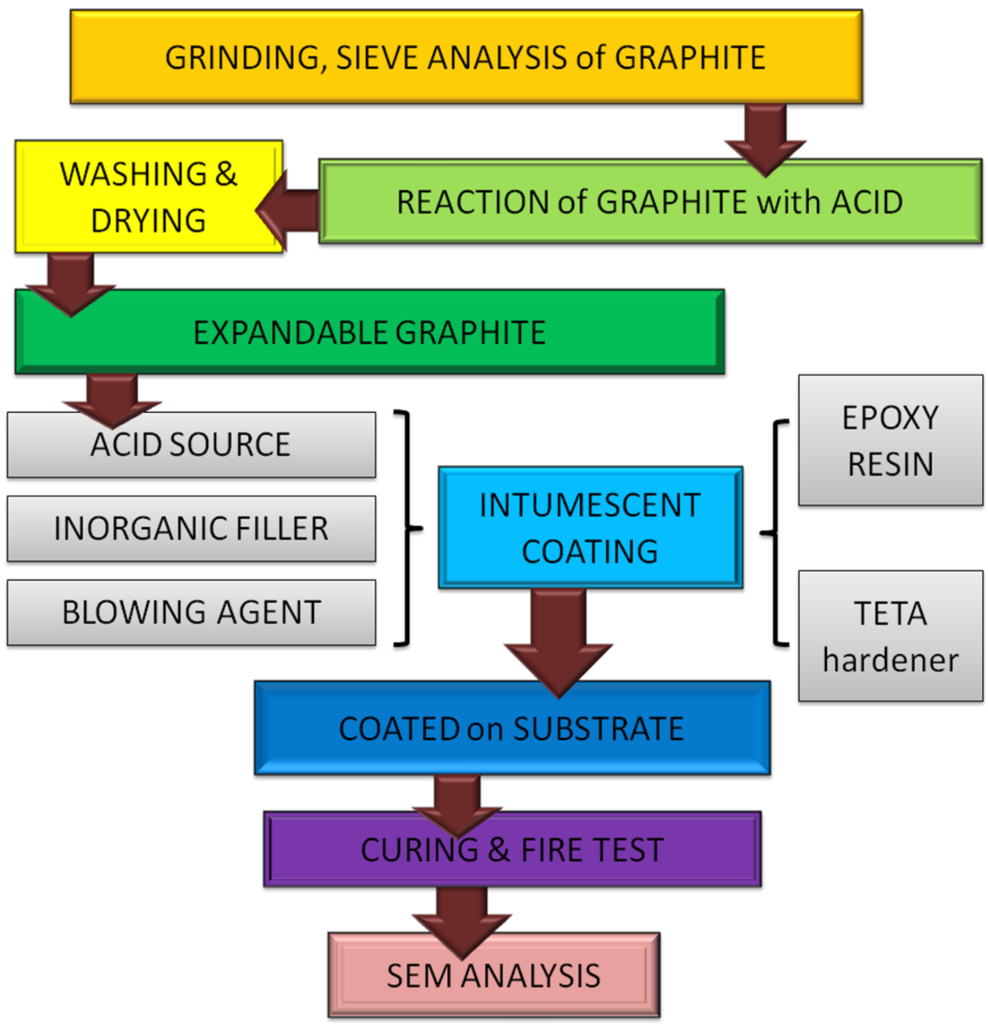


Figure 3.1 : Methodology

3.2 Project Gantt Chart

It is important to have timeline in doing a project. Figure 3.2 below shows the timeline for Final Year Project 2, from week one of study until week fourteen.

No.	Action/Plan	WEEK 1	2	3	4	5	6	7	MIDSEMBREAK	8	9	10	11	12	13	14
1	Preparation of coating															
2	Fire test and coating analysis with SEM															
3	Submission of Progress Report 1															
4	Project work continue															
5	Submission of Progress Report 2															
6	Seminar															
7	Preparation of coating															
8	Fire test and coating analysis with SEM															
9	Poster exhibition															
10	Submission of dissertation (final draft)															
11	Oral presentation															
12	Submission of dissertation															

Mile Stone			
Project Schedule		FYP Schedule	

Figure 3.2: Project Milestone

CHAPTER 4

RESULTS AND DISCUSION

4 .1 Preparation of Intumescent Fire Retardant Coating

In order to prepare intumescent coating, there are few basic steps need to be taken in order to develop the coating formulation. The steps are grinding, sieving and treating the graphite and then graphite is dried at room temperature for about one week. Next step is mix all components of intumescent coating based on the formulation developed.

4.1.1 Grinding Process

Graphite need to be grind first in order to get different particle sizes during sieving process. Figures below illustrate the process of grinding process.

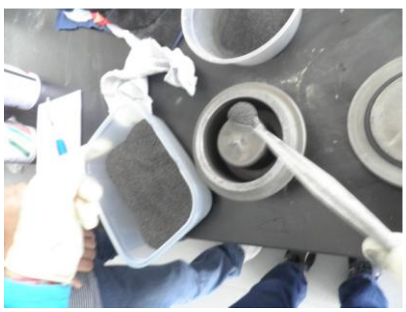


Figure 4.1 : Graphite is insert in the grinder case

Figure 4.2 : Grinder case in inserted in the grinder machine

Figure 4.2 : The time is set to 10 seconds

Figure 4.4 : Upon complete, graphite is poured in the container

4.1.2 Sieving Process

Next step is to sieve the graphite by using sieve machine in order to get six particle sizes which are 425 µm, 300 µm, 212 µm, 150 µm, 63 µm and below 63 µm. In this project, particle size of graphite used is 300 µm.

Figure 4.5: Graphite is poured into siever

Figure 4.6: Seiver is inserted into the sieve machine

Figure 4.7: The time is set to 10 minutes

Figure 4.8: All six particle sizes are ready and put in container

4.1.3 Process of Treating Graphite

Upon completing the sieving process, graphite for particle size of 300 µm is treated by using strong acid. Process of treating graphite is explained in section 2.4.

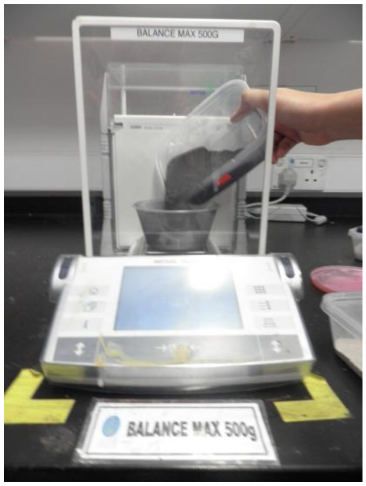


Figure 4.9 : All materials is weight by using weighing scale

Figure 4.10 : All material is mixed welll in 3 neck flask around 1 hour

Figure 4.11 : After 1 hour, the treated graphite is washed by using distilled water until colorless

Figure 4.12 : Treated graphite is then dried in the oven at 80^{o}C for 1 hour

4.2 Preparation of Intumescent Fire Retardant Coating formulation

Zinc Borate is used as additives in intumescent coating to replace Boric Acid. Boric Acid is used in the current market as additives in intumescent coating. Table 4.1 shows the formulation for Intumescent Coating. The formulation is based on weight percentage (%).

Table 4.1 : Formulation for Intumescent Coating

Coating Number	Expandable Graphite	Ammonium Polyphosphate	Melamine	Zinc Borate	Bisphenol-A	Tetraethylene Amine
1	0	11.76	11.76	5.88	47.00	23.60
2	5.56	11.11	11.11	5.56	44.44	22.22
3	5.56	11.11	8.33	8.33	44.44	22.22
4	5.56	11.11	5.56	11.11	44.44	22.22
5	5.56	8.33	11.11	8.33	44.44	22.22
6	5.56	5.56	11.11	11.11	44.44	22.22
7	8.33	11.11	8.33	5.56	44.44	22.22
8	11.11	11.11	5.56	5.56	44.44	22.22
9	8.33	8.33	11.11	5.56	44.44	22.22
10	11.11	5.56	11.11	5.56	44.44	22.22
11	8.33	8.33	8.33	8.33	44.44	22.22
12	5.56	8.33	8.33	11.11	44.44	22.22
13	11.11	8.33	8.33	5.56	44.44	22.22
14	8.33	11.11	5.56	8.33	44.44	22.22

4.2.1 Process of making formulation

All materials as shown in Table 4.1 are mix well based on their weight percentage. All formulations are labeled based on the coating number as shown in Table 4.1 above. Then, all formulation is dried at room temperature for a week.

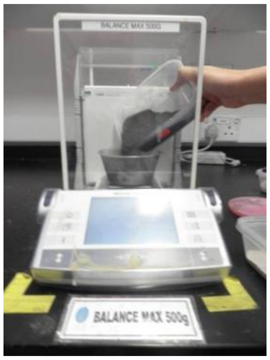


Figure 4.13 : All materials is weight using weighing scale

Figure 4.14 : All material is mix well by using mixer and spatula

Figure 4.15 : Mixture is put on substrate (mild steel)

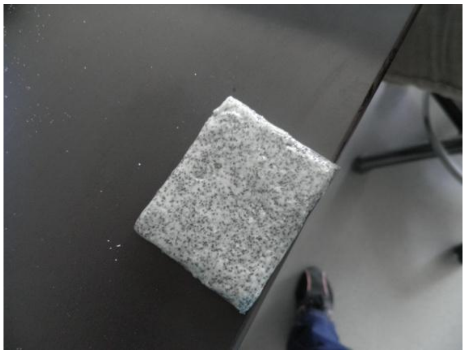
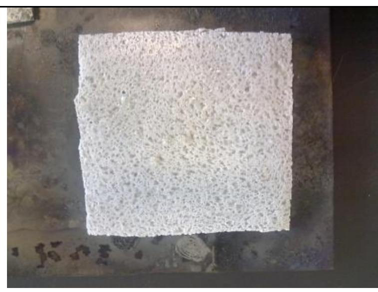


Figure 4.16 : Complete formulation is dried at room temperature for a week

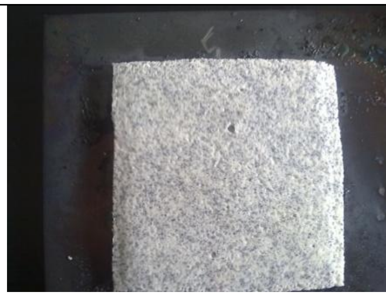
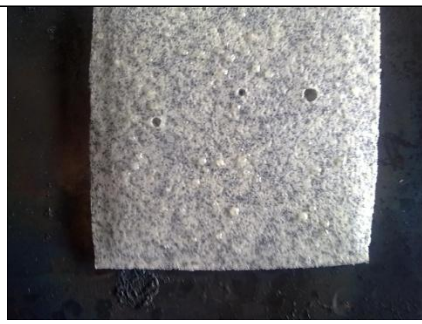
4.3 Fire Test by using furnace

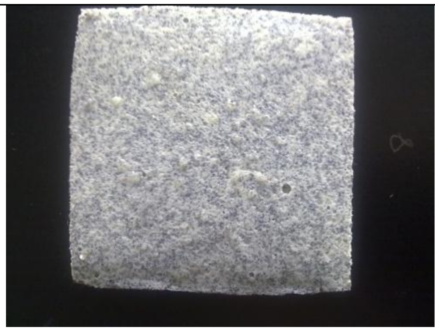
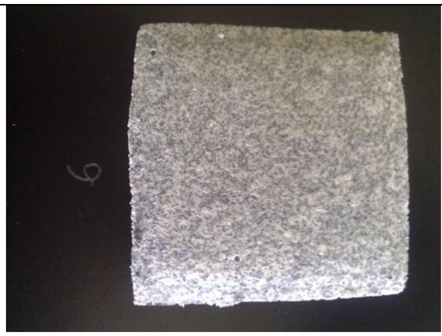
Each formulation is mixed well by using mixer. Completed formulation is put on mild steel; 5cm x 5cm. After drying process around one week at room temperature, the coating undergoes fire test. Furnace test is type of fire test. Furnace test has been done at temperature of 450^{o}C. Table 4.2 showed completed formulation before furnace test and after furnace test and its thickness. The thickness includes the thickness of mild steel which is 1.5mm.

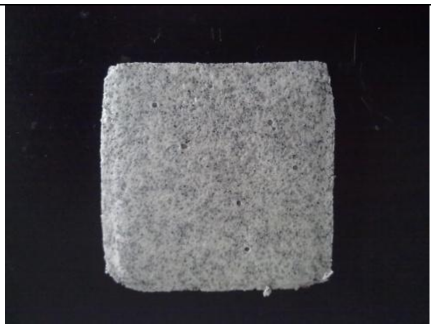
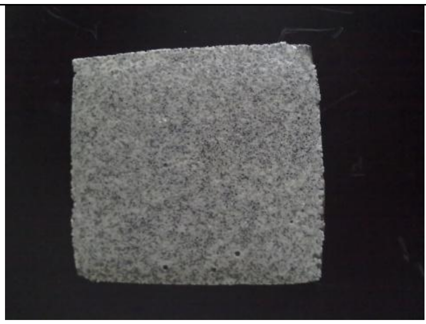
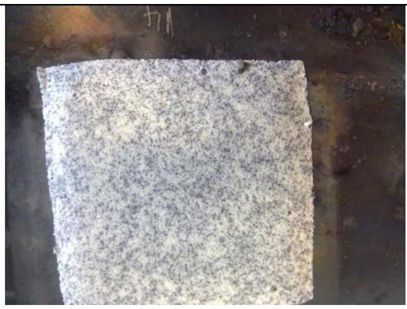
Table 4.2: Pictures of each formulation coating before and after fire test

No	Before Fire Test	After Fire Test
1	 Thickness of coating : 3.66 mm	 Thickness of char : 11.0 mm Expansion : 3 times Rough surface,hard and not detach from substrate
2	 Thickness of coating : 3.92 mm	 Thickness of char : 16 mm Expansion: 4.08 times Rough surface, hard and not detach from substrate

3	 Thickness of coating : 4.03 mm	 Thickness of char : 13 mm Expansion: 3.22 times Rough surface, cracks, not detach from substrate
4	 Thickness of coating : 4.30 mm	 Thickness of char : 14 mm Expansion: 3.26 times Rough surface, crack and detach from substrate
5	 Thickness of coating: 4.23 mm	 Thickness of char : 14 mm Expansion: 3.31 times Smooth surface, crack and detach from substrate

6	 Thickness of coating: 3.72 mm	 Thickness of char : 12 mm Expansion : 3.23 times Smooth surface, cracks and detach from substrate
7	 Thickness of coating : 3.98 mm	 Thickness of char : 17 mm Expansion : 4.27 times Rough surface, hard and not detach from substrate
8	 Thickness of coating: 3.86 mm	 Thickness of char : 22 mm Expansion : 5.70 times Rough surface, hard and not detach from substrate

9	 Thickness of coating : 4.17 mm	 Thickness of char : 15 mm Expansion : 3.6 times Smooth surface, cracks and detach from substrate
10	 Thickness of coating : 3.84 mm	 Thickness of charoating : 12.5 mm Expansion : 3.26 times Smooth surface, cracks and not detach from substrate
11	 Thickness of coating : 3.99 mm	 Thickness of char: 11 mm Expansion : 2.76 times Smooth surface, Cracks and not detach from substrate

12	 Thickness of coating : 3.82 mm	 Thickness of char : 7 mm Expansion : 1.83 times Rough surface, cracks and detach from substrate
13	 Thickness of coating : 4.16 mm	 Thickness of char : 17.5 mm Expansion 4.20 times Rough surface, hard and not detach from substrate
14	 Thickness of coating: 4.29 mm	 Thickness of char: 12 mm Expansion : 2.80 times Rough surface, hard and not detach from substrate

4.3.1 Discussion

During furnace test, the char will expand that indicates whether the coating can protect the substrate or not. High expansion shows good protection to the substrate, in this case; mild steel. Test sample of intumescent coating is placed in furnace chamber until the temperature reach $450^{o}C$. Time taken for the temperature to rise is about 30 minutes. Temperature of $450^{o}C$ will remain for about 1 hour and then it takes around 1 hour for the temperature to drop to $150^{o}C$. After that, test sample is taken out and the char expansion is measured and the physical properties are jotted down. Coating No8 shows five times expansion after fire test with hard char structure while coating No13 shows four times expansion. These two coatings show better expanding effect and fire protection properties. Coating No2 and No5 show four and three times expansion respectively. In a practical fire, inner temperature of the exposed mild steel will increase to $500\ ^{o}C$ and reach the critical strength of steel only after several minutes. The fast falling mechanical properties of the mild steel at high temperature will lead to poor resistance. Therefore, the coatings are applied to protect mild steel against fire and heat. The char structure provides a shield that insulates the metal substrate from radiant heat and direct contact with flame. Thus, high expansion of char is good to protect mild steel from failure. Table 4.3 showed side view of the best four expansion of intumescent coating.

Table 4.3: Char expansion of best four coating.

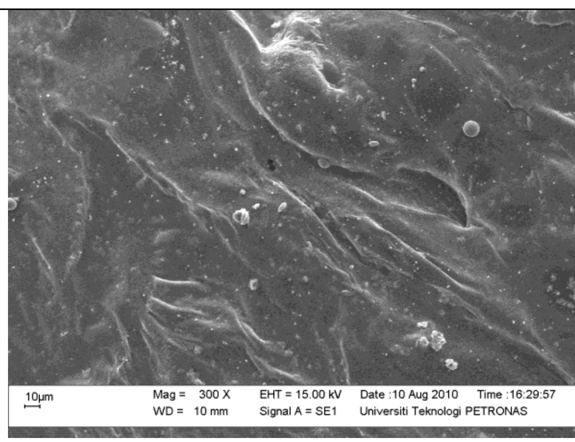
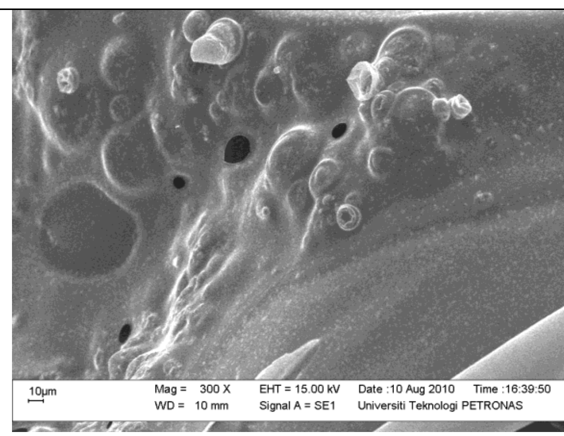
No	Side view of formulation	No	Side view of formulation
2		8	

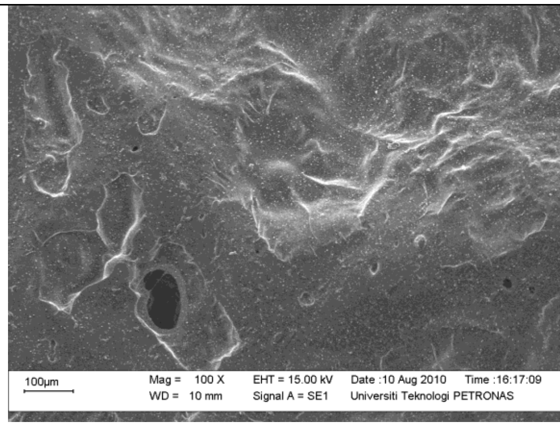
5		13	

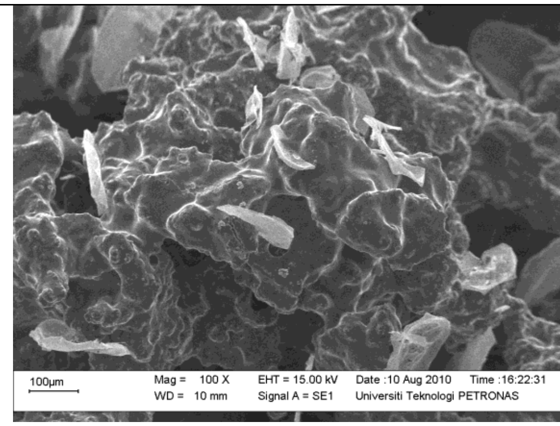
4.3.2 Morphology of intumescent chars for best four expansions

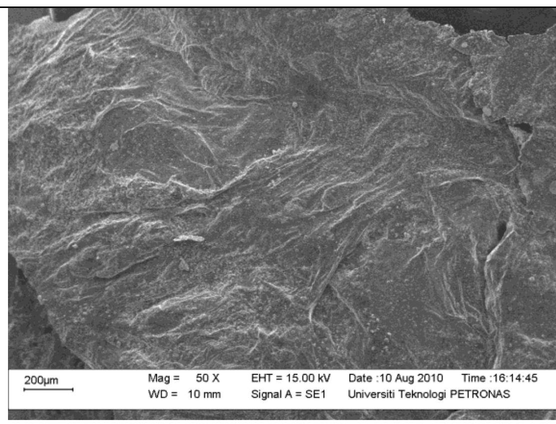
All four samples are sent for Scanning Electron microscopy (SEM). The micrographs of intumescent chars obtained from No2, No5, No8 and No13 coating are showed in Table 4.4. The micrographs are for outer surface and inner surface of the char.

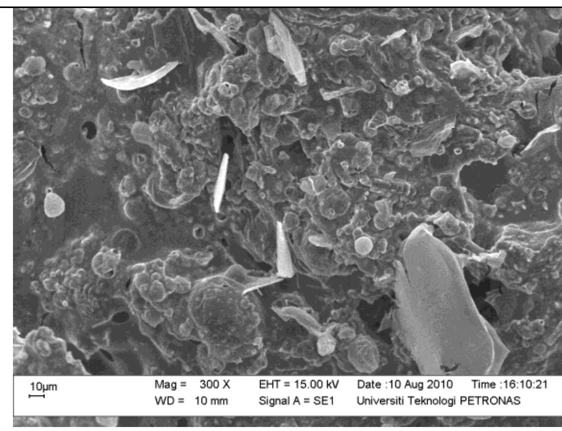
Table 4.4: SEM morphology of the char samples (inner and outer surface) obtained from coating No2, No5, No8 and No13

No	Outer Surface	Inner Surface (through section)
2	 *Scale : 10μm* *Magnifier : 300x* Smooth surface, few holes	 *Scale : 10μm* *Magnifier : 300x* Few holes appear ,less graphite flakes

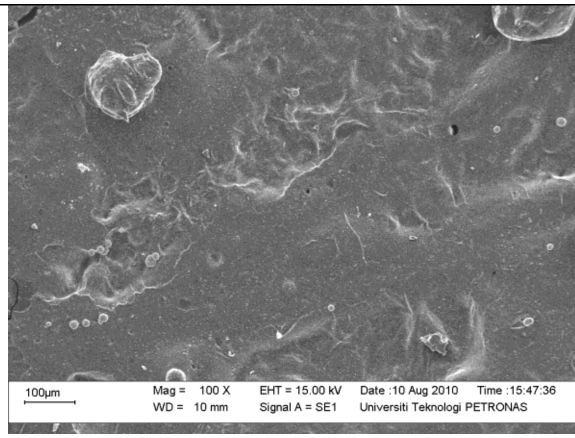
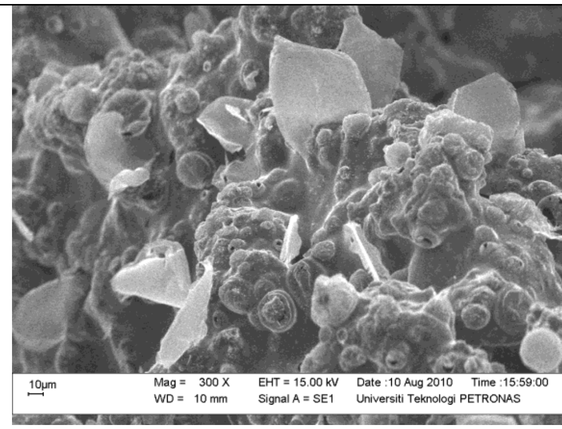
<table>
<tr>
<td>5</td>
<td>

Scale : 10µm
Magnifier : 300x
Good char structure, no cracks</td>
<td>

Scale : 10µm
Magnifier : 300x
Appearance of graphite flakes</td>
</tr>
<tr>
<td>8</td>
<td>

Scale : 10µm
Magnifier : 300x
Show uniform surface, no cracks that can cause heat to penetrate the surface, very small flakes of graphite.</td>
<td>

Scale : 10µm
Magnifier : 300x
Very hard inner surface, some flakes appear, holes appear due to dehydration.</td>
</tr>
</table>

13	 *Scale : 10µm* *Magnifier : 300x* Smooth surface ad appearance of bubbles	 *Scale : 10µm* *Magnifier : 300x* Appearance of graphite flakes

.

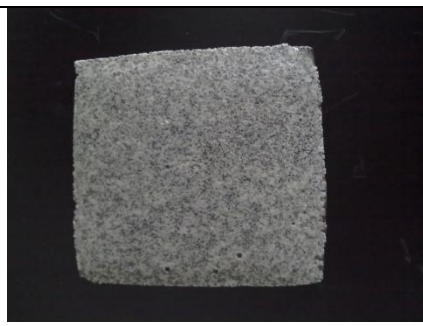
A smooth and uniform surface is observed in outer surface of coating No2, No5 No 8 and No13. The char structure provides shield that insulates the metal substrate from radiant heat and direct contact with flame. However, heat and fire may transfer to metal substrate through the cracks in the char structure, resulting decline of fire resistance. If cracks appear in outer surface of the char structure, it causes the releases of some gases; carbon dioxide, CO_2 ammonia, NH_4 and nitrogen, N_2 gas from graphite, melamine and bisphenol-A. Thus, char structure is not good. Inner surface of coating No2 is smooth and less appearance of graphite flakes. There are formations of graphite flakes observed in coating No5, No 8 and No 13. Graphite flakes act as heat insulator and reduce heat transfer to the substrate thus good fire protection properties. The embedded graphite, as a function of fiber, can enhance resistance of char structure to deformation. Lots of bubbles appear in coating No8 and No 13 due to dehydration during curing. The degraded matrix of fused carbonaceous compound can stick the wormlike expanded graphite formed and a large amount of expanded graphite is embedded in char structure as the high percentage of graphite in formulation No 8 and No 13 which is 11.11%   . All coating undergoes fast drying process by using oven at $100^{o}C$ causes dehydration. However, dehydration does not affect the char expansion but only microstructure of the char.

4.4 Fire test by using Bunsen burner

Best four formulations as stated in section 4.3.1 undergoes another fire test by using Bunsen burner test. The procedure is described in section 2.9. The test is done for at least 10 minutes depends on the temperature of the backside of mil steel plate. If the temperature exceeds 500 ^{o}C, the test should be stop.

Table 4.5 Pictures of coating No 2, No 5, No 8 and No 13 before and after fire test

No	Before Fire Test	After Fire Test
2	 Thickness of coating : 3.86 mm	 Thickness of char : 12 mm Expansion: 3.11 times Rough surface, hard and not detach from substrate
5	 Thickness of coating: 3.92 mm	 Thickness of char : 7 mm Expansion: 1.78 times Smooth surface, crack and detach from substrate

8	 Thickness of coating: 4.30 mm	 Thickness of char : 26 mm Expansion : 6.05 times Rough surface, hard and not detach from substrate
13	 Thickness of coating : 3.93 mm	 Thickness of char : 21 mm Expansion 5.34 times Rough surface, hard and not detach from substrate

4.4.1 Discussion

In practical fire, inner temperature of the exposed steel structural members will increase to 500 ^{o}C and reach the critical strength of steel only after several minutes. The fast falling mechanical properties of the steel structural members at high temperature will lead to the poor fire resistance. Therefore, flame retardant coatings are applied to protect steel structural members against fire and heat. The curves of fire protection are presented in Figure 4.17. After 25 minutes of the test, the back temperatures of test plates of coating No 2, No 5, No 8 and No 13 reach 344 ^{o}C, 334 ^{o}C, 287 ^{o}C and 253 ^{o}C respectively. From Figure 4.17, coating

No 13 show lowest value of back temperature of test plates after 25minutes followed by coating No 8. Thickness of char layer of coating No 2, No 5, No 8 and No 13 is 12mm, 7mm, 26 mm and 21 mm respectively. Compared with other three coating, coating No 8 has better expanding effect and fire protection properties. The expansion is 6.05 times. While the expansion of coating No 13 is 5 times. Therefore these two coatings, coating No 8 and No 13 show good fire protection properties with high expansion of char and low value of back temperature of test plates.

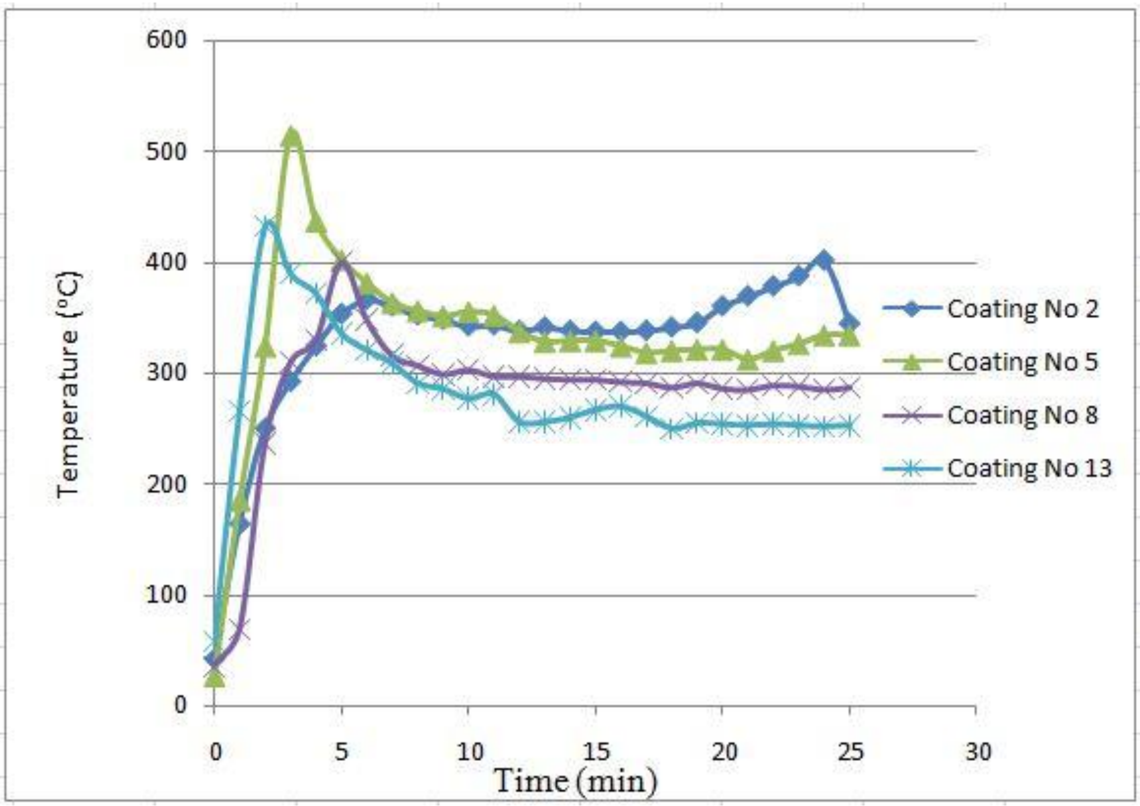


Figure 4.17 : Fire protection curves of flame retardant coatings

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The interaction of expandable graphite, ammonium polyphosphate, melamine, epoxy resin and zinc borate leads to formation of char structure. When heated,treated graphite undergoes oxidation by sulphuric acid with production of CO_2 and SO_2 that are responsible for the expansion process. During fire test in furnace, coating No 8 and coating No 13 shows high expansion of five and four times respectively. Both coatings have rough and hard char structure. The morphology of both chars shows good insulation to the substrate; mild steel. On heating using Bunsen burner, the back temperature of test plates of coating No 8 and No 13 is 287 ^{o}C and 253 ^{o}C respectively. Both coatings have good char structure and fire protection properties even after exposed to temperature of 450 ^{o}C.

5.2 Recommendation

In this research, the size of expandable graphite used is 300 µm. It is recommended to use lower particle size such as 150 µm, 212 µm or 63 µm. The reason is, lower particle size have higher surface area thus resulting in higher expansion during heating.

REFERENCES

References

[1] Titanium and titanium alloys, edited by C. Leyens and M. Peters, Wiley-VCH

[2] B. Ostman, A. Voss, A. Hughes, Fire Master. 25 (2001) 95

[3] W. Funke, Prog. Org. Coat 31 (1997) 5

[4] F.Kang T.Y Zhang, Y. Leng, J. Phys. Chem. Solid 6-8 (1995) 889

[5] G. Camino, L. Costal, L. Trossareli, Polym. Degrad. Stab. 8 (1984) 13

[6] Kirk-Othmer;Encyclopedia:Chemical Technology,John Wiley & Sons, 1992, Vol 4

[7] A Hasan, M.H.H 2008, Development of Intumescent Formulation for Fire Protection In Oil and Gas Industry, FYP Report, Universiti Teknologi PETRONAS, Malaysia

[8] Jimmy Wales,28 Oct 2010 < http://en.wikipedia.org/wiki/Intumescent>

[9] Jimmy Wales, 17 Dec 2009 <http://en.wikipedia.org/wiki/Thermogravimetry>

[10]W Jimmy , 1 Nov 2009 http://en.wikipedia.org/wiki/ScanningElectronMicroscopy

[11]Jimmy, 8 Oct 2010 <http://en.wikipedia.org/wiki/Differentialscanningcalorimetry>

[12] Jimenez, 28 Feb 2006, <http://www.galleries.com/minerals/elements/graphite>

[13] Kang Shen, 11 Oct 1999, http://www.nyacol.com/exgraphadv.htm

[14] NFPA 13 Standard for the Installation of Sprinkler Systems 2007 Edition, Annex C Explanation of Test Data and Procedures for Rack Storage

[16] Zhengyu Wang, Enhou Han, Wei Ke, Influence of Expandable Graphite of Fire Resistance and Water Resistance of Flame-Retardant Coatings, 2006, Institute of Metal Search, Chinese Academy of Sciences, China

[17] R.H.R Tide, Integrity if Structural Steel After Exposure to Fire, Engineering Journal, First Quarter, 1998