The Effects of Fiberglass on the Thermal Insulation Properties of Intumescent Coatings

by

Nor Sharifah binti Omar

Dissertation submitted in partial fulfillment of

the requirements for the

Bachelor of Engineering (Hons)

(Mechanical Engineering)

MAY 2011

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

The Effects of Fiberglass on the Thermal Insulation Properties of Intumescent Coatings

by

Nor Sharifah binti Omar

A project dissertation submitted to the

Mechanical Engineering Programme

Universiti Teknologi PETRONAS

in partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

(MECHANICAL ENGINEERING)

Approved by,

(Dr. Faiz bin Ahmad)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2011

CERTIFICATION OF ORIGINALITY

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

NOR SHARIFAH BINTI OMAR

ABSTRACT

The aim of this study is to investigate the influence of fiberglass addition into a basic intumescent coating formulation towards the enhancement of its thermal insulation properties. Intumescent coating has been an effective way to provide fire protection to the structural steel in the construction industry in the case of fire. The intumescent coating were formulated from expandable graphite (EG), ammonium polyphosphate (APP), melamine, boric acid, bisphenol A epoxy resin (BPA), triethylenetetramine (TETA) and fiberglass 3.00 mm. Eight different formulations were developed to study the effects of fiberglass addition on the thermal insulation performance of the coatings. The new developed intumescent coatings were tested for their fire performance by burning them at 450 °C, 650 °C and 850 °C in the furnace for 2 hours. The effects of each fire test at different temperatures; low and high temperature were evaluated. Scanning Electron Microscope (SEM), X-Ray Diffraction technique (XRD) and Thermogravimetric Analysis (TGA) were conducted to study the char morphology, the crystalline material structure and chemical components and the thermal analysis of the coatings. The FG07 and FG08 formulations with 6.0 grams and 7.0 grams fiberglass provide better results in terms of the enhanced thermal insulation properties of the coatings. The FG07 and FG08 coating recorded the best expansion during fire test at 450 °C and 650 °C, the highest back steel temperature during thermal insulation test and illustrate good coating characteristics after analysis using SEM. These coatings also recorded the presence of graphite, boron oxide and boron phosphate after XRD testing and with some residue left after degradation analysis using SEM. It can be concluded that the addition of fiberglass into the basic intumescent formulations has enhanced the thermal characteristic of the coating and helps to maintain the structure of buildings in longer time.

ACKNOWLEDGEMENT

First and foremost, I would like to thank Allah the Almighty who has given me strength and blessings to complete my Final Year Project entitled "The Effects of Fiberglass on the Thermal Insulation Properties of Intumescent Coating". The utmost gratitude goes to my supervisor, A.P.Dr. Faiz bin Ahmad for having his time to guide and assist me in my Final Year Project. His guidance, support and encouragement have kept the work on the right track with continually motivation. I would also like to give my greatest appreciation to Dr. Puteri Sri Melor bt Megat Yusoff who have supervised me during my Final Year Project I and Mr. Sami Ullah who has been helping me through many challenges during this project. My appreciations extended to the whole Mechanical Engineering Department staffs including the laboratory technicians, Mr Faizal, Mr Shairul, Mr Irwan, Mr Mahfuz and Mr. Paris who have guided and helped me using the laboratory equipments and handling the experimental works until the completion of this project. I would like to use this opportunity to express my deepest appreciation to everyone who has contributed towards this project directly or indirectly. Last but not least, not forgetting my family members and fellow friends who had gave me moral support and motivation.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDIES

The use of fire-retardant coatings is one of the easiest, oldest and most efficient ways to protect steel substrates against fire [1,2]. It is important to protect materials against fire in the construction and petrochemical industries to ensure safe evacuation of people from the building before the steel structures started to collapse when exposed to temperature above 550 °C [2]. The advantages of using intumescent coating is that it does not change the basic properties of the material (e.g. mechanical properties), easily processed and can be applied on several materials including metallic materials, polymers, textiles and wood [1,3].

Intumescent is defined as the swelling of certain substances when they are heated [3]. Intumescent coatings form an expanded multicellular layer upon heating; namely char, which acts as thermal barrier that effectively protect the substrate against rapid increase of temperature and thereby maintaining the structural integrity of the building [4,5]. The physical structure of the charring layer plays a very important role in the performance of flame retardant. Formation of homogeneous char (high residue amount and good thickness) will ensure longer fire-endurance time and better performance of the flame retardant coating [6,7]. In recent years price efficiencies and improvements in technology have created a situation where intumescent coatings have come to dominate the structural fire protection market [5,7].

The study on the thermal insulation property of the intumescent coating will allow determination of exact temperature at which the charring of the mixture begins. Fiberglass is used as reinforcement of the char strength [8]. It helps to maintain the char integrity and provide a higher mechanical resistance of the charring element. In terms of fire safety, fiberglass insulation is naturally noncombustible because it is made from sand and recycled glass [8]. The insulation requires no additional fire-retardant chemical treatments [9,10].

1.2 PROBLEM STATEMENT

Intumescent coating is among the various possible alternatives for fire prevention method which has been used for 50 years in the industry [8,9,10]. It is one of the methods to provide passive fire protection to structural steel used in modern architectural designs whilst at the same time maintain the aesthetic appearance of the structure [10,11]. Intumescent coatings are designed to perform under severe conditions and to maintain the integrity of the structural steel for 1-3 hours as the temperature of the surroundings is in access of 1100 °C; before the whole structure started to collapse [11,12]. Applying this type of coatings is one of the most efficient ways of providing fire retardancy to flammable materials [11,13]. Therefore, they might have a wide application for protecting metals, plastics, textiles and wood against fire [13]. Further, this coating can be applied in an economical and simple way such as by spray, brush or roller for the interior renovation of new or existing structural buildings.

Several studies have demonstrated the use of filler and binder as reinforcing agent helps to increase the efficiency of the intumescent coatings in terms of providing longer protection to the structural steel [12,13]. However, little studies focused on the effect of using fiberglass to increase the thermal insulation properties of the intumescent coatings. Thus, the aim of this study is to evaluate the thermal insulation properties of the intumescent coating when fiber glass is added in the formulations of intumescent coatings in various proportions.

1.3 OBJECTIVE AND SCOPE OF STUDIES

1.3.1 Objective

The objective of this study is to investigate the thermal insulation properties of intumescent coating with varying amount of fiber glass added into the basic intumescent formulations. The optimum intumescent formulation with varying fiberglass amount which provide the best thermal insulation properties against fire is determined.

1.3.2 Scope of Study

This study focused on the enhancement of the thermal insulation properties of the intumescent coating. Fiberglass of length 3.00 mm in various amount were added into the basic formulations to enhance the fire performance characteristics of the coating under fire. The entire laboratory works includes preparation of the intumescent coatings, thermal insulation and fire test at 450 °C, 650 °C and 850 °C and analysis of the coatings using Scanning Elecron Microscope (SEM), X Ray Diffraction (XRD) and Thermogravimetric Analysis (TGA).

1.3.3 Limitation of Study

This study focused only on the available expandable graphite of 300 μm particle sizes and fiberglass of 3.00 mm length. Expandable graphite is used as carbon source instead of other material since it has advantages of non-halogen, smoke reduction and non heavy materials. The amount of fiberglass added into the formulations ranging from 1.0-7.0 grams with only eight formulations developed.

CHAPTER 2
LITERATURE REVIEW

2.1 LITERATURE IN INTUMESCENT COATING FIELD

A large number of mechanistic studies have been performed over the past several years in the intumescent fire retardant fields. Review on early research and development of intumescent coatings was reported by Vandersall [14] and updated later by Kay et al. [15]. The first commercial patent for a foaming fire retardant system was issued to Tramm in 1938 [14,15]. A variety of coating formulations, in the forms of paints and mastics have been developed and pun into commercial use starting in the 1950's [15,16]. The increasing use of organic polymers, which are highly flammable in their natural state, has encouraged the development of intumescent additives systems [16,17].

Besides, studies using filler as an extender in intumescent coatings compositions and their effect on active performance under fire, related to char stabilizing properties was carried out later [13,14]. The materials created a coating layer on the organic material of the charring coating film, thus protecting the under laying material from collapse [16,18]. Polymeric binders have also identified to improve the efficiency of intumescent coatings as it provides better performance of the thermal efficiency of the intumescent coatings and therefore enhance the time of failure during fire cases [17,18].

Apart from that, studies on expandable graphite (EG); a new generation of intumescent additives has provides good fire retardancy to various materials [18]. EG helps to improve flame-retardant properties of the coatings [15,16]. The study of different intumescent formulations in the intumescent efficiency for steel protection

in hydrocarbon fire was also carried out previously in the field of intumescent coatings [16].

2.2 THEORY ON INTUMESCENT COATING

2.2.1 Intumescent Coating

There are two distinctly different types of intumescent coatings [15]. The first one is the traditional chemical intumescent fire retardant coatings with chemical fire retardant mechanism [15]. The second one is a new type of physical intumescent coatings containing expandable graphite (EG) with physical fire retardant mechanism [15,16]. The development of these coatings is focused on improving the fire retardant efficiency and weather durability of the products, and reducing the emission of smoke and volatile organic compounds (VOC) as well as toxic gases to provide safe and environmental-friendly products for end-users [11,16].

The four basic elements of intumescent coating are acid source such as ammonium polyphosphate (APP), carbon source such as pentaerythritol or dipentaerythritol, blowing agent such as melamine and binder such as epoxy resin. The acid source acts as a dehydrating or carbonizing agent which releases polyphosphoric acid at temperatures above 2000 °C, the carbon source which is an organic substance turns into a charring structure after burning processes, the blowing agent release N_2 and NH_3 gases under decomposition and expands the char. Binder will make the compounds contact with each other.

The intumescent concept allows a balance between the fire properties and the level of additives in the material [10,13]. The general intumescent ingredients consists of an a carbon source (e.g. polymer such as dipentaerythritol) which can be a carbon-rich polyhydric compound, an acid source (e.g. ammonium polyphosphate) as a dehydrating agent which is capable of promoting the formation of a carbonaceous char from the carbon source and a blowing agent (e.g. melamine)

bounded together by a binder with various formulations in order to form an efficient protective char [14,15]. Chemical interactions between these 'active' ingredients in the formulations lead to the formation of intumescent char [12,15].

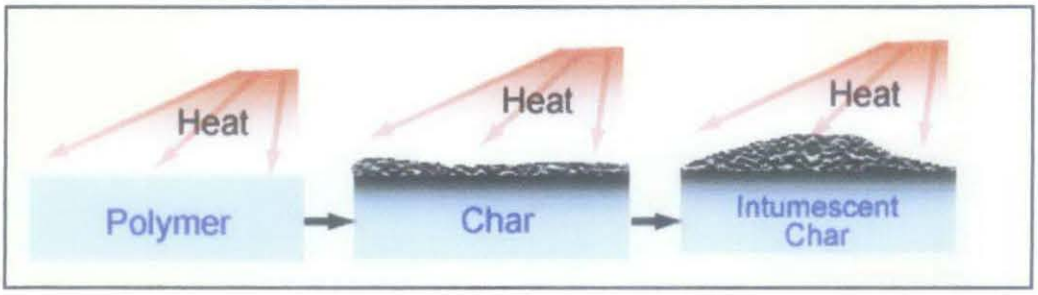


Figure 2.1: Char and intumescent formations

The reaction mechanism of the intumescent coating starts with the breakdown of the acid source to yield mineral acid, then it takes part in the dehydration of the carbonization source to yield the carbon char and finally the blowing agent decomposes to yield gaseous products. The latter causing the char to swell and produce the insulating multi-cellular protective layer [9,16]. Fig. 2.1 shows the schematic diagram of the formation of char during fire. During the burning of an intumescent system, the formation of effective char occurs via semiliquid phase, which coincides with gas formation and expansion of the surface [13]. Figure 2.1 shows the swelling process of intumescent coating as a result of a slow diffusion of the evolved degradation gases released into the degradation matrix [14,15,16].

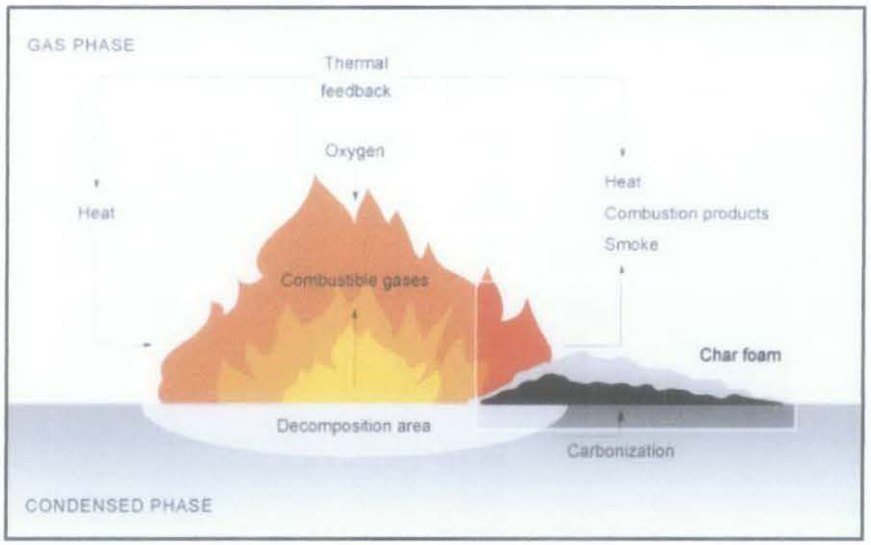


Figure 2.2: Schematic representation of intumescent coating

2.2.2 Expandable Graphite (EG) Intumescent Coatings

This type of intumescent coatings contains expandable graphite which is a new generation of fire retardant additives. The use of EG as an intumescent flame retardant in synthetic materials has attracted more and more interests in both research and industry circles in recent years. EG have the advantages of non-halogen, smoke reduction and non heavy materials which make it an important intumescent flame retardant.

EG is formed by treating crystalline graphite, which is composed of stacks of parallel planes of carbon atoms, with intercalates such as sulfuric acid and/or nitric acid [13,15]. EG is an intercalated compound formed by inserting sulfuric acid between the graphite layers EG is halogen-free and works mainly in the condense phase [11,15]. When the materials that contain EG were exposed to a heat source, EG expand and generate a voluminous insulative layer, providing the flame retardance of the polymeric matrix. The expanded carbon layer works as an insulating layer to reduce the heat transfer [14].

2.2.3 Fiberglass

Fiber glass is useful in terms of improving the thermal insulation property of the intumescent coatings due to its high thermal conductivity [13]. It has a low coefficient of thermal expansion combined with high thermal conductivity properties making the fiber glass a dimensionally stable material that rapidly dissipates heat as compared to asbestos or other organic fibers [13]. Fiberglass has high fire resistance property since it is an inorganic material and will not burn or support combustion [13,15]. Fiberglass retains approximately 25% of its initial strength at 540 °C. Besides, it is dimensionally stable; does not stretch or shrink upon exposure to extremely high or low temperatures. [14,15].

CHAPTER 3
METHODOLOGY

3.1 PROJECT WORK

The flow of the project activities from until the completion of the project is as below:

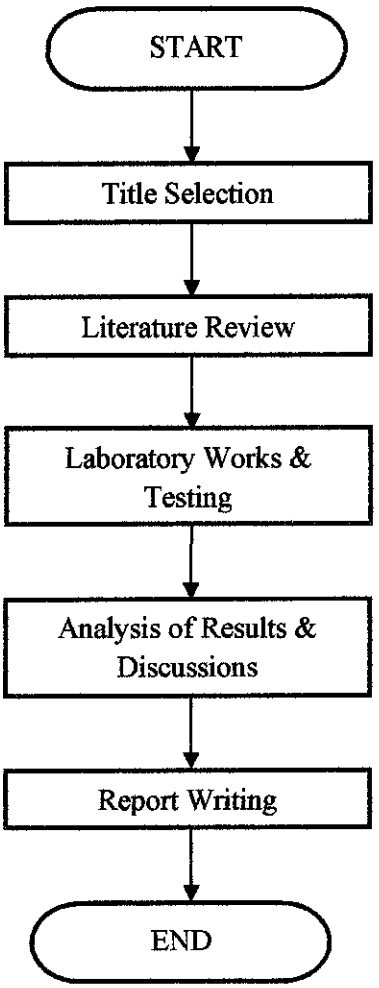


Figure 3.1: Project Activities Flow Chart

3.2 MATERIALS/ TOOLS

3.2.1 Materials

The materials used for these studies are bisphenol A epoxy resin (BPA) purchased from Chang Chun Plastic Co. Ltd. used as binder and is cured at ambient temperature. The fire retardant agents; ammonium polyphosphate (APP) purchased from Clariant (Exolit AP422), containing 20 percent phosphorus is used as the acid source, triethylenetetramine as hardener, melamine purchased from Sabic Chemical Industries as the blowing agent, boric acid, structural steel purchased from TSA Industries Sdn. Bhd., graphite purchased from Premier East West Malaysia Sdn. Bhd and short fiberglass of length 3.0 mm.

3.2.2 Equipments and Tools

The list of equipments used in the coating preparations are the sieve equipments, grinder machine, mixer, furnace, thermal testing equipments, Thermal Gravimetry Analyzer (TGA), X-ray Diffraction (XRD) and Scanning Electron Microscope (SEM).

3.3 MATERIALS PREPARATION

3.3.1 Expandable Graphite (EG)

The graphite required for the preparation of the EG is graphite of size 300 μm. The graphite powder is grinded using the grinder machine in order to obtain smaller particle size. Seive Analysis is performed on the graphite powder to classify the graphite into the desired particle size of 300 μm. A mixture of concentrated sulfuric acid (98 %, 37.5 g), acetic acid (150.0 g), graphite powder (75 g) and potassium permanganate (5.25 g) was stirred at room temperature for 1 hour.

The treated graphite was filtered, washed with distilled water until pH level closed to 6-7 and dried at 100-110°C in the oven for 4 hours. The formulations were prepared by mixing the components using Ultra Turrex mixer (600 rpm) and was compared to the virgin epoxy resin. The coating is then applied on at 3.5 mm thickness onto steel plates of dimensions 15 x 50 mm^2 and cured for one week under ambient conditions.

Figure 3.2: Seive analysis machine to classify the particle size of the graphite (left) and the equipments set up for the preparation of EG (right)

3.3.2 Intumescent Coating Formulations

In order to investigate the effects of adding fiberglass into the intumescent coating formulations, eight formulations have been developed based from the standard formulations developed earlier. The basic formulations consist of expandable graphite as carbon source, APP as acid source, MEL as blowing agent, boric acid, epoxy resin Bisphenol-A (BPA) as binder and triethylenetetramine (TETA) as hardener. Fiberglass of amount 1.00-7.00 g has been added into the formulations. Table 3.1 shows the eight formulations with using 300 μm particle size of expandable graphite (in grams):

Table 3.1: Formulation of intumescent coating with fiberglass

Units in grams (g)

Sample No	Expandable Graphite	APP	MEL	Boric Acid	Epoxy (BPA)	Hardener (TETA)	Fiberglass
FG01	5.50	11.10	5.50	11.10	44.60	22.20	0.00
FG02	5.50	11.10	5.50	11.10	44.60	22.20	1.00
FG03	5.50	11.10	5.50	11.10	44.60	22.20	2.00
FG04	5.50	11.10	5.50	11.10	44.60	22.20	3.00
FG05	5.50	11.10	5.50	11.10	44.60	22.20	4.00
FG06	5.50	11.10	5.50	11.10	44.60	22.20	5.00
FG07	5.50	11.10	5.50	11.10	44.60	22.20	6.00
FG08	5.50	11.10	5.50	11.10	44.60	22.20	7.00

3.3.3 Intumescent Coating Samples

Eight intumescent coating samples were developed based from the formulations. The amount of fiberglass added into the formulations ranging from 0.00-7.00 g. All the samples are left to cure at room temperature for about 1-2 weeks. Figure 3.3 below shows the process of preparing the intumescent coating.

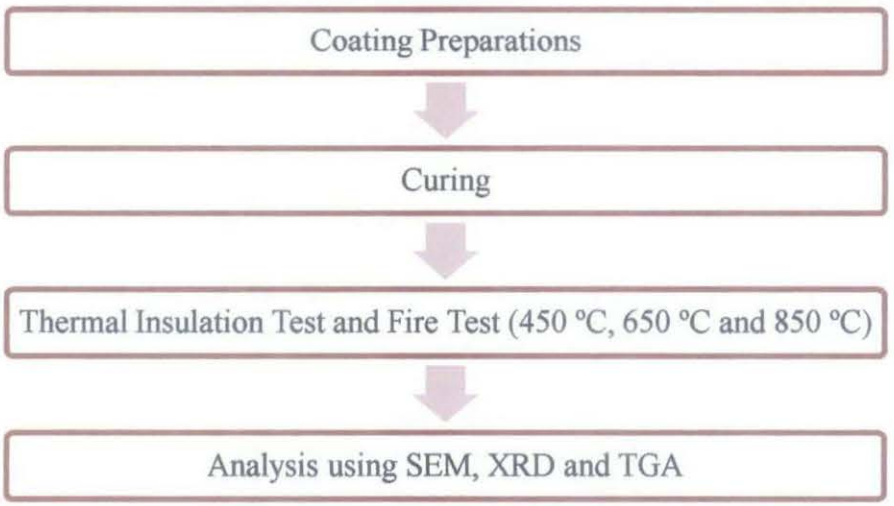


Figure 3.3: Intumescent coating preparations diagram

The preparations of the coating are done by weighting and grinding APP, MEL and boric acid into smaller particles. Grinding of the components is necessary to prevent from the presence of bubbles inside the coating. BPA and TETA is weight and mixed in the mixer until the mixture is homogenous. Next, expandable graphite and fiberglass is added into the mixture and is stirred well until they become homogenous again. The coating were then applied on steel structure and left for curing at room temperature for 1-2 weeks. Figure 3.4 shows the diagram of preparation of the coating:

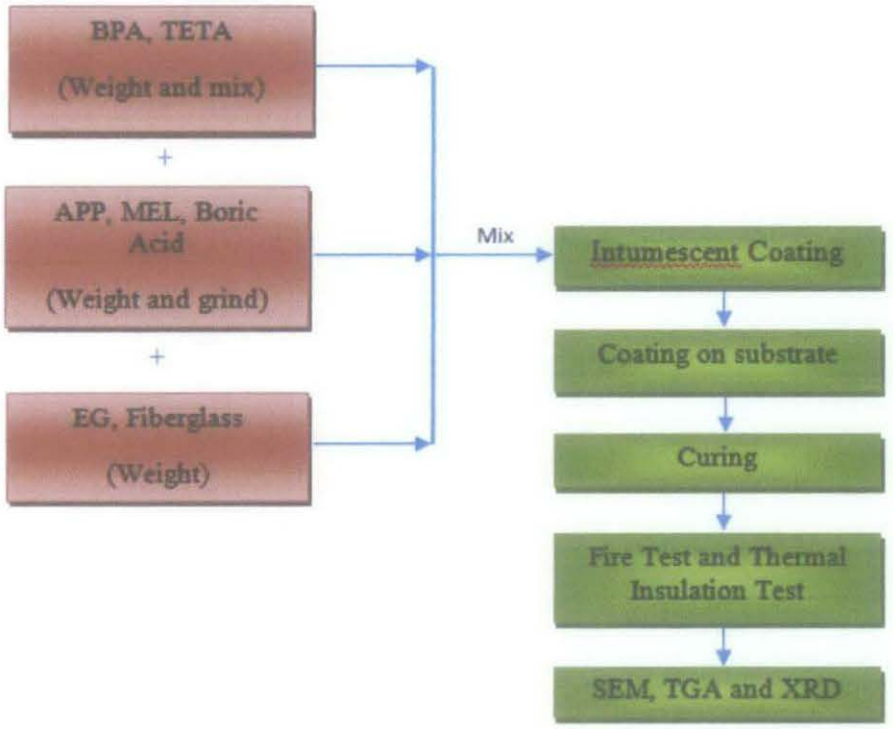


Figure 3.4: Diagram of intumescent coating preparation

3.4 ANALYSIS AND CHARACTERIZATION

3.4.1 Fire Test

The intumescent coating samples were burnt at three different temperatures; 450 °C, 650 °C and 850 °C for two hours in the furnace to measure the expansion rate of each sample. The char morphology characteristics and microstructure of each

sample were then determined using SEM and XRD. Figure 3.5 below shows the temperature profile of the burning processes:

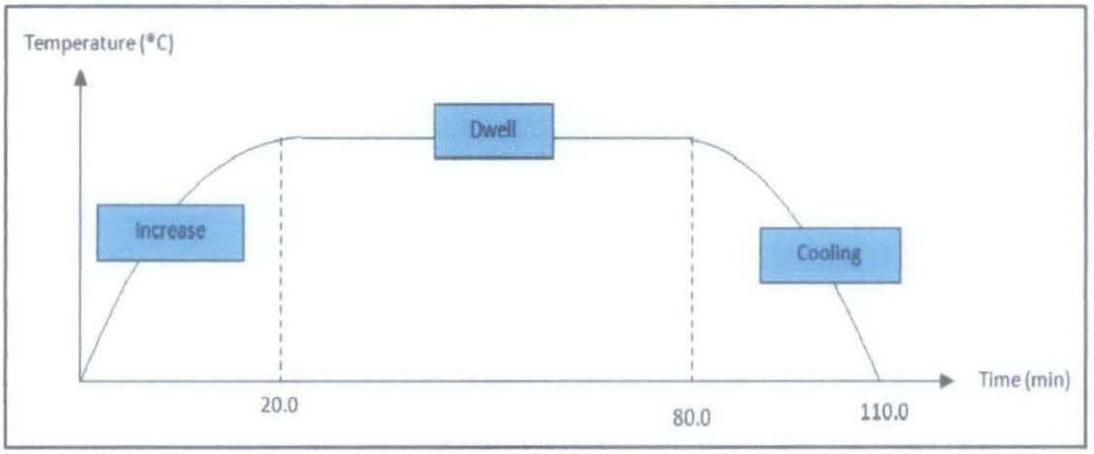


Figure 3.5: Burning of samples temperature profile

3.4.2 Thermal Insulation Test

A Bunsen burner was used as a fire source to fire up the intumescent samples using the standard UL 94. The coated steel were attached to a digital thermocouple and fired up using a Bunsen burner. The measurements were taken using a data logger.

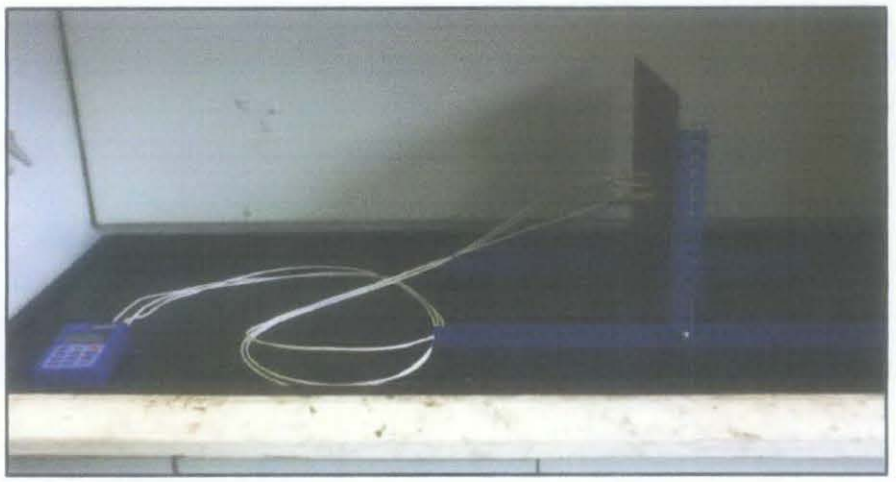


Figure 3.6: The thermal insulation test equipment setup

3.4.3 Scanning Electron Microscopy (SEM)

The charring layer and the morphological structures of the inside and outside coating were observed and analyzed using AMARY 1000.

3.4.4 X-Ray Diffraction (XRD)

The presence of boron oxide, boron phosphate and graphite in the intumescent coating after fire test of the coating were determined using XRD technique.

3.4.5 Thermogravimetric Analysis (TGA)

The Thermogravimetric analysis of samples (approximately 10mg) were carried out at 10 °C/min under N_2 over the whole range of temperature (50 °C – 800 °C) using TGA Q50.

3.5 PROJECT GANTT CHART

Table 3.2 shows the Gantt chart for FYP I and FYP II project activities:

Table 3.2: The Gantt chart for project activities

No	Action/ Plan	Week														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Preparation of Expandable Graphite (EG) - Grind graphite powder - Seive graphite powder - Treat graphite powder	Done in FYP I														
2	Preparation of coating sample															
3	Curing of coating sample															
4	Analysis of coating samples using TGA															
5	Fire test at 450 °C, 650 °C and 850 °C															
6	Submission of Progress Report															
7	Analysis of coating sample using SEM and XRD															
8	Submission of Poster and Pre EDX															

9	Submission of Draft Report												●			
10	Submission of Dissertation (Soft Bound)													●		
11	Submission of Technical Paper													●		
12	Oral Presentation														●	
13	Submission of Project Dissertation (Hard Bound)															●

● Project Milestone

■ Project Activities

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 FIRE TEST

4.1.1 Fire Test at 450 °C

The height of each sample before and after burning process is measured. The differences in the height after the burning indicate the percentage of expansion of the coatings. The results are tabulated in Table 4.1:

Table 4.1: Results of fire test at 450 °C

No	Samples	Height Before (cm)	Height After (cm)	Expansion Ratio/ Descriptions
1	FG01	 H_{before} = 0.35 cm	 H_{after} = 1.35 cm	3.88 times (Rough surface, little cracks & detachable)
2	FG02	 H_{before} = 0.35 cm	 H_{after} = 1.25 cm	3.57 times (Rough surface, major cracks & detachable)

3	FG03	 H_{before} = 0.35 cm	 H_{after} = 1.35 cm	3.88 times (Rough surface, little cracks & detachable)
4	FG04	 H_{before} = 0.35 cm	 H_{after} = 0.95 cm	2.71 times (Very rough surface, little cracks & detachable)
5	FG05	 H_{before} = 0.35 cm	 H_{after} = 1.65 cm	4.71 times (Rough surface, minor cracks & detachable)
6	FG06	 H_{before} = 0.35 cm	 H_{after} = 1.85 cm	5.29 times (Rough surface, no cracks & detachable)
7	FG07	 H_{before} = 0.35 cm	 H_{after} = 2.35 cm	6.71 times (Very rough surface, major cracks & detachable)

8	FGO8	 H_{before} = 0.35 cm	 H_{after} = 1.85 cm	5.29 times (Rough surface, little cracks & detachable)

Based from Table 4.1, the samples which show the greatest amount of expansion are sample FG07 followed by sample FG06 and FG08. After the fire test, the coatings swelled and expanded into charring elements that protects the substrate steel from collapse. The char had a very rough surface with major cracks and detachable towards the steel substrate. The microstructure of samples FG07 and FG08 were examined using SEM and XRD. Figure 4.1 shows the graph of expansion ratio shown by each sample:

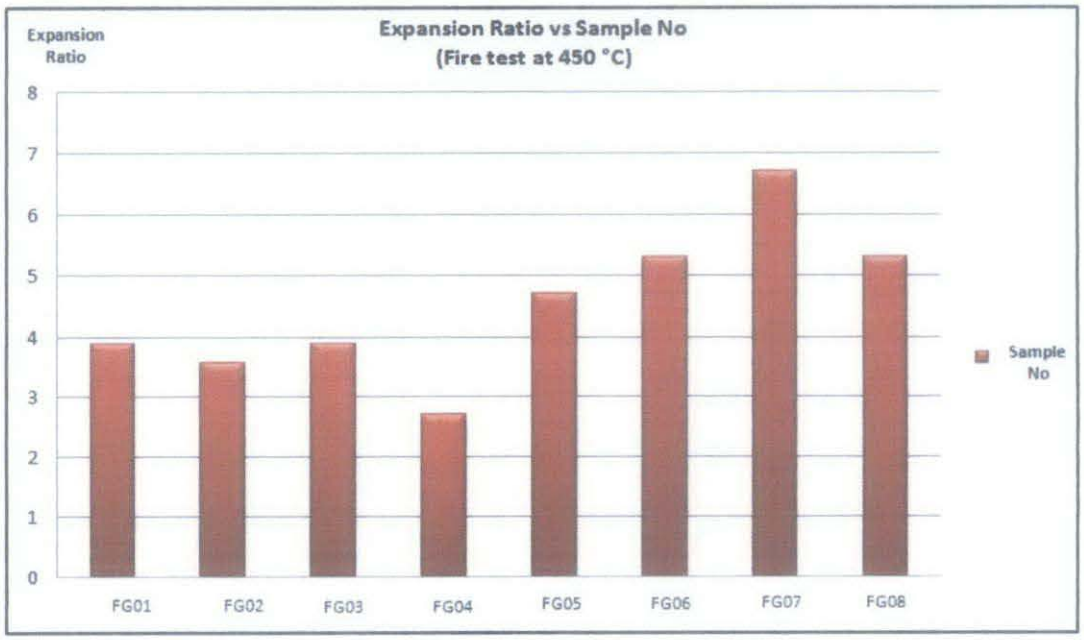


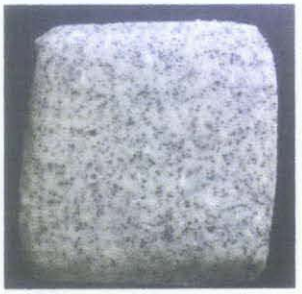
Figure 4.1: Graph of expansion ratio by each sample no (at 450 °C)

4.1.2 Fire Test at 650 °C

The height of each sample before and after burning process is measured. The differences in the height after the burning indicate the percentage of expansion of the coatings. The results are tabulated in Table 4.2 below:

Table 4.2: Results of fire test at 650 °C

No	Samples	Height Before (cm)	Height After (cm)	Expansion Ratio/ Descriptions
1	FG01	 $H_{before} = 0.35$ cm	 $H_{after} = 1.30$ cm	3.71 times (Rough surface, no cracks & detachable)
2	FG02	 $H_{before} = 0.35$ cm	 $H_{after} = 1.30$ cm	3.71 times (Rough surface, little cracks & detachable)
3	FG03	 $H_{before} = 0.35$ cm	 $H_{after} = 1.70$ cm	4.88 times (Rough surface, little cracks & detachable)

4	FG04	 H_{before} = 0.35 cm	 H_{after} = 1.80 cm	5.14 times (Rough surface, no cracks & detachable)
5	FG05	 H_{before} = 0.35 cm	 H_{after} = 1.80 cm	5.14 times (Very rough surface, little cracks & detachable)
6	FG06	 H_{before} = 0.35 cm	 H_{after} = 2.00 cm	5.71 times (Rough surface, little cracks & detachable)
7	FG07	 H_{before} = 0.35 cm	 H_{after} = 2.40 cm	6.88 times (Very rough surface, little cracks & detachable)
8	FGO8	 H_{before} = 0.35 cm	 H_{after} = 2.50 cm	7.14 times (Very rough surface, little cracks & detachable)

Based from Table 4.2, the samples which show the greatest amount of expansion are sample FG08 followed by sample FG07. The char had a very rough surface with little cracks and detachable towards the steel substrate. The microstructure of each sample will be examined using SEM and XRD. Figure 4.2 shows the graph of expansion ration by each sample:

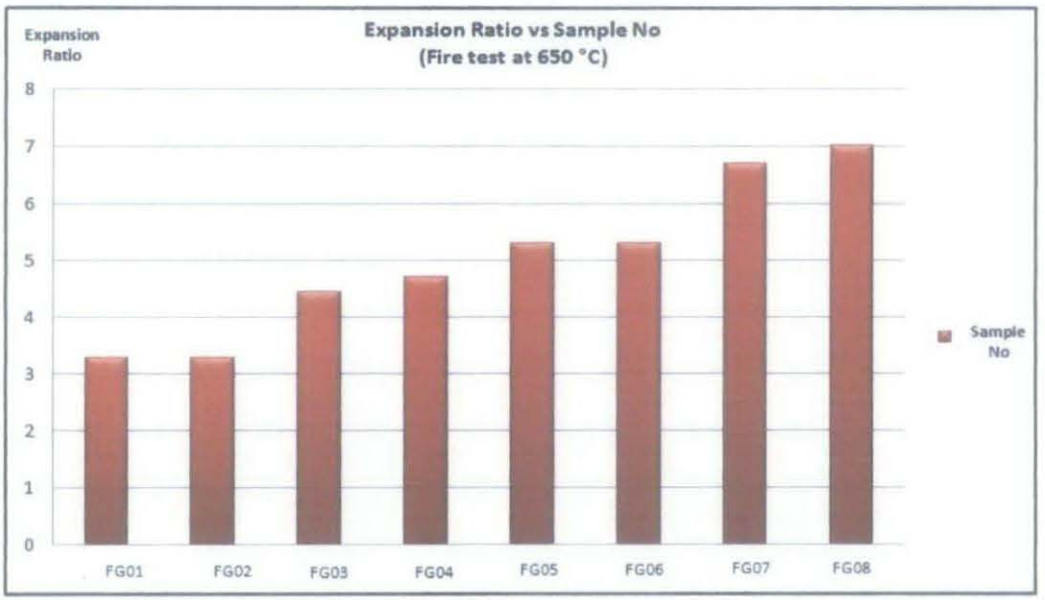
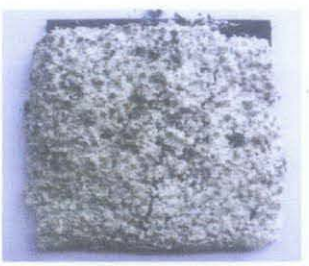


Figure 4.2: Graph of expansion ratio by each sample no (at 650 °C)

4.3.3 Fire Test at 850 °C

The expansion ratio of the intumescent coating samples were not measured before and after the fire test at 850 °C since the coatings oxidized at temperature of 850 °C. The results are tabulated in Table 4.3 below:

Table 4.3: Results of fire test at 850 °C

No	Samples	Before	Height After
1	FG01		
2	FG02		
3	FG03		
4	FG04		

5	FG05		
6	FG06		
7	FG07		
8	FGO8		

The results from fire test conducted at temperature 850 °C were shown as in Table 4.3. The charring layer formed upon burning has turned into white powder (ashes) due to a very high fire temperature of the furnace.

4.2 THERMAL INSULATION TEST

The thermal properties of the coating with fiberglass were then compared to the thermal properties of a coating without fiberglass as in Figure 4.3. The coating without fiberglass was observed to reach the highest back steel temperature of 210 °C while coating FG07 and FG08 with 6.0 and 7.0 grams of fiberglass recorded the highest temperature of <300 °C compared to the coating with less amount of fiberglass. It can be concluded that the use of fiberglass in the intumescent coating formulations helps to increase the thermal insulation properties of the coating, prolong the lifetime of steel structures and improved the strength of the char.

The results of each samples recorded using the data logger are shown in Figure 4.4 until Figure 4.11. The figures show the influence of the presence of fiberglass of 1.0 grams - 7.0 grams on the protective properties of the char layer with expandable graphite.

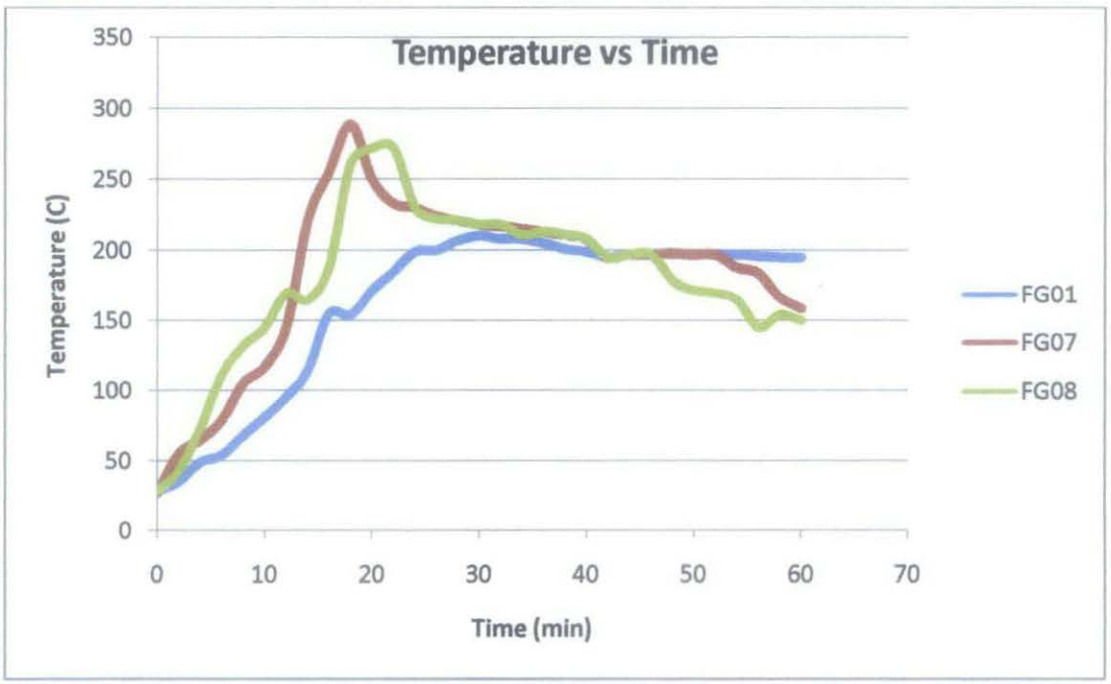


Figure 4.3: Comparison between FG01, FG07 and FG08

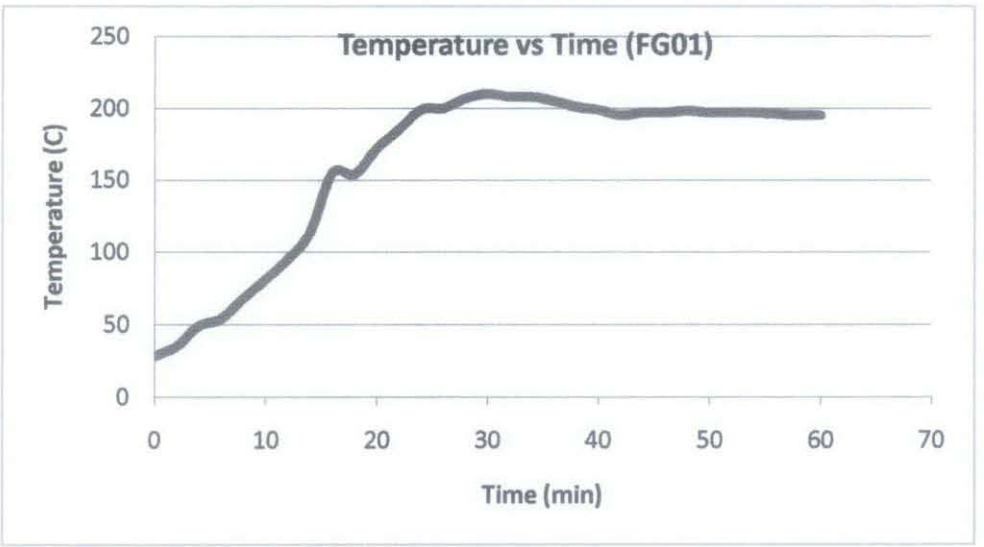


Figure 4.4: Back value temperature of sample FG01

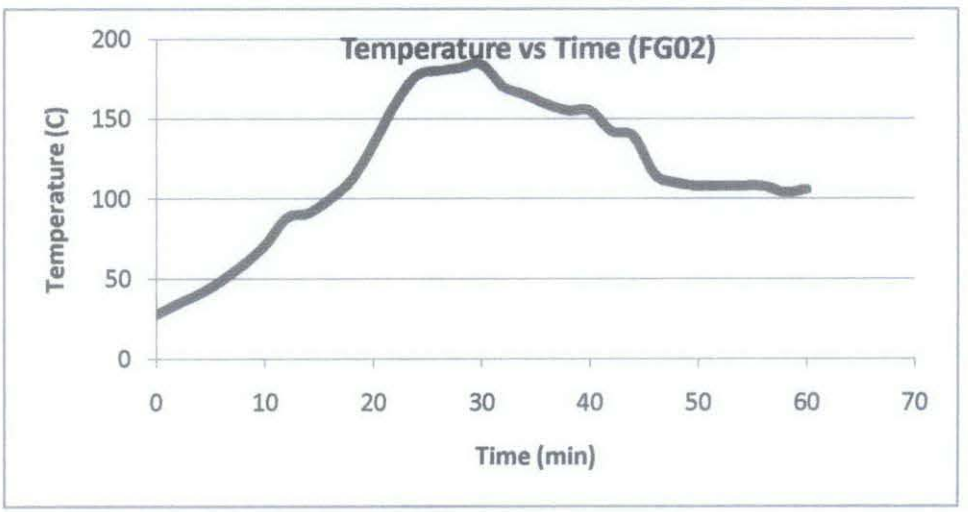


Figure 4.5: Back value temperature of sample FG02

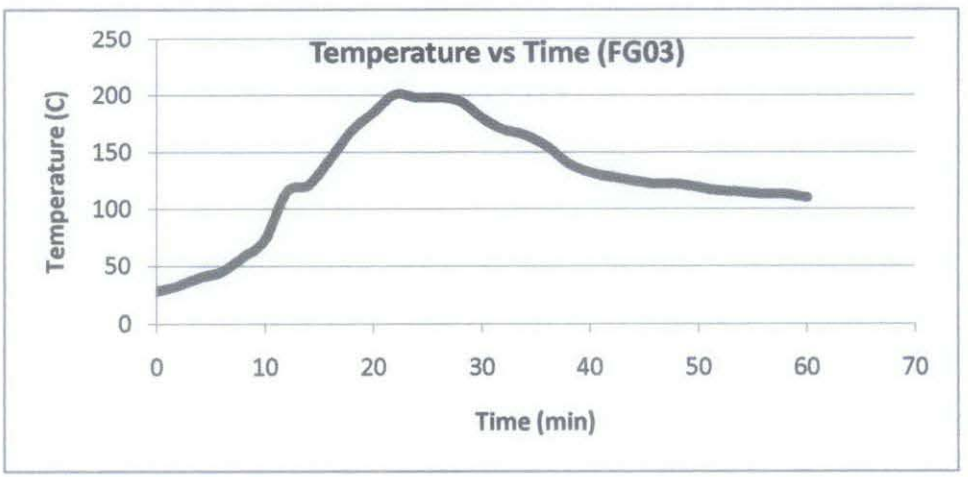


Figure 4.6: Back value temperature of sample FG03

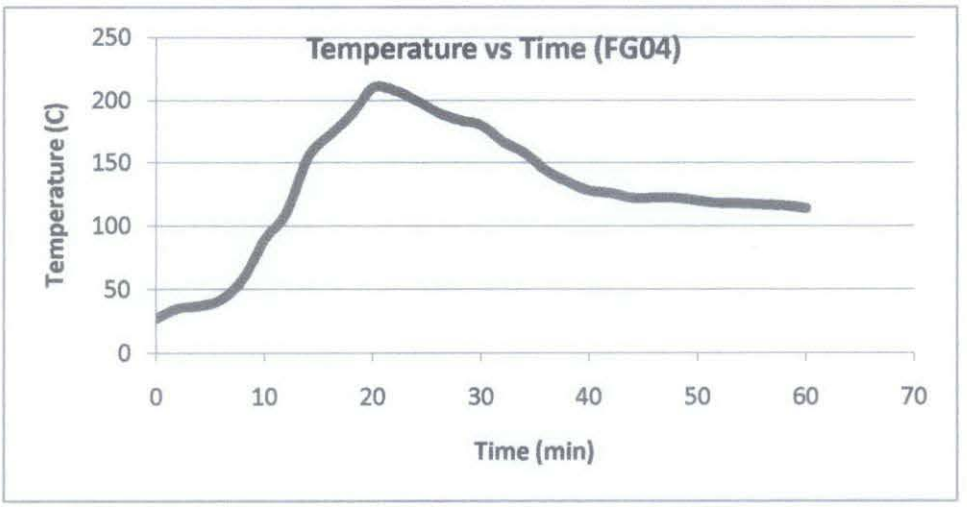


Figure 4.7: Back value temperature of sample FG04

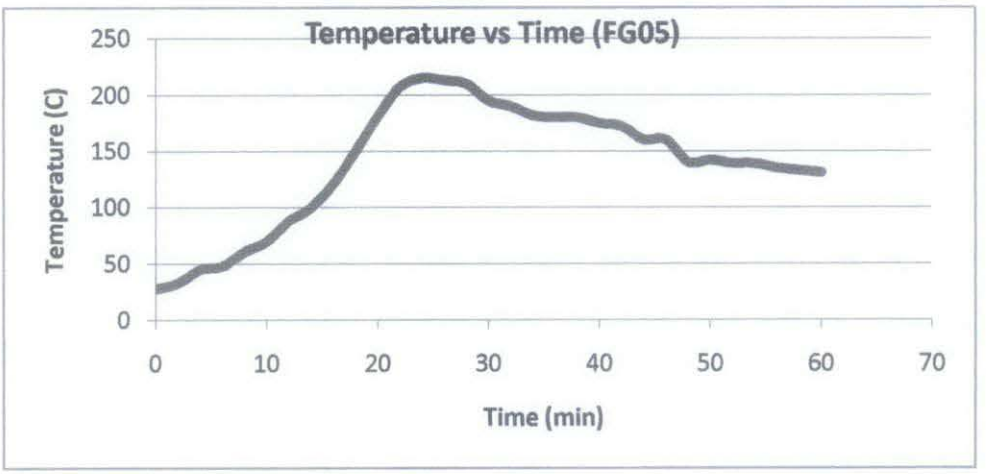


Figure 4.8: Back value temperature of sample FG05

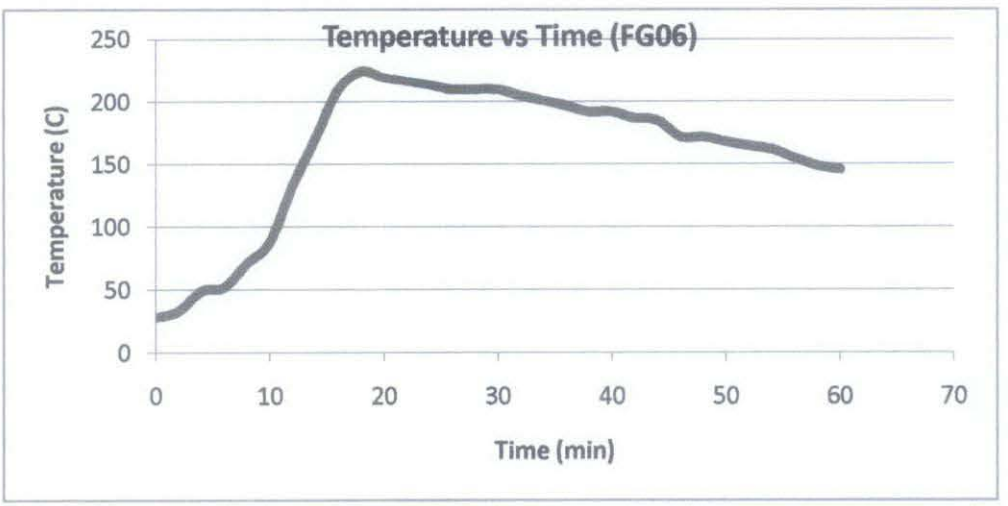


Figure 4.9: Back value temperature of sample FG06

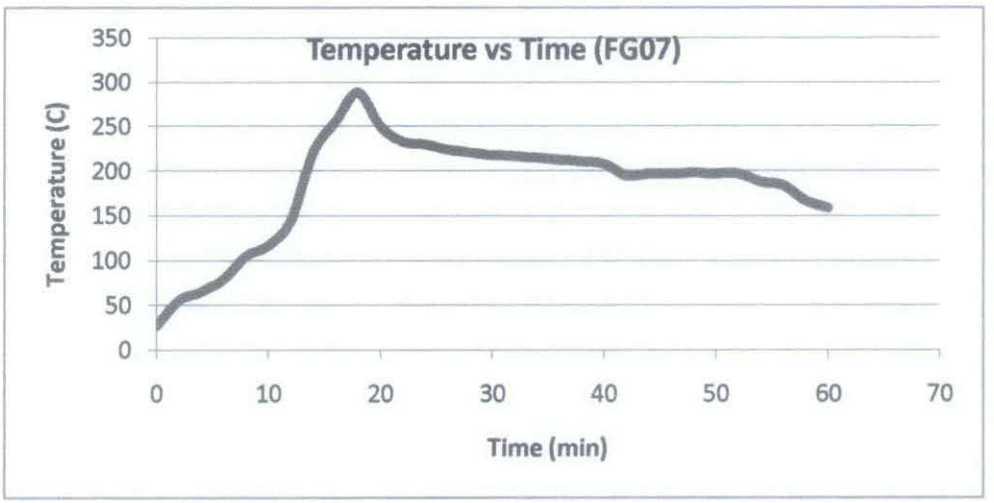


Figure 4.10: Back value temperature of sample FG07

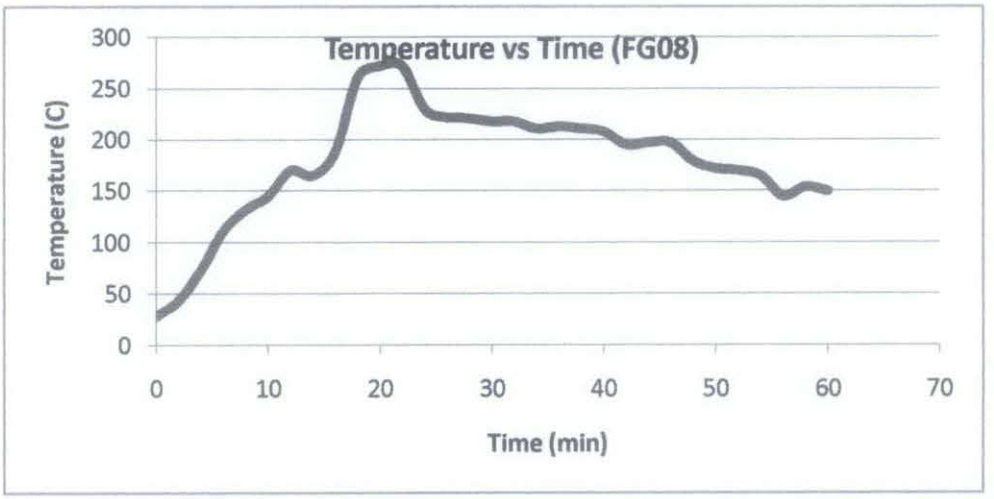


Figure 4.11: Back value temperature of sample FG08

4.3 SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

The SEM micrograph images of chars for outer and inner surface for sample FG07 and FG08 are shown in the figures below using magnifications of 50x and 500x (outer surface) and 100x and 500x (inner surface).

4.4.1 Fire Test at 450 °C

i) FG07 Coating Sample

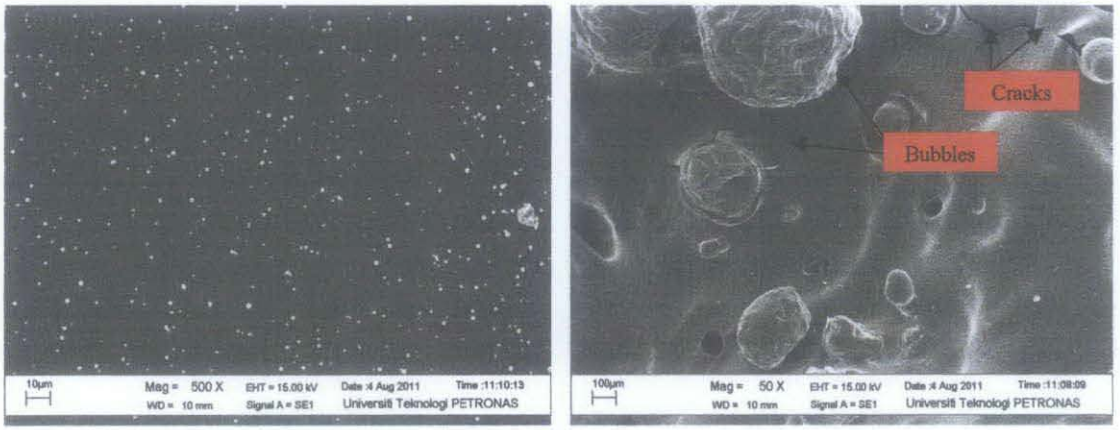


Figure 4.12: SEM Micrograph of FGO7 coating for outer surface with 500x (left) and 50 x (right) magnifications

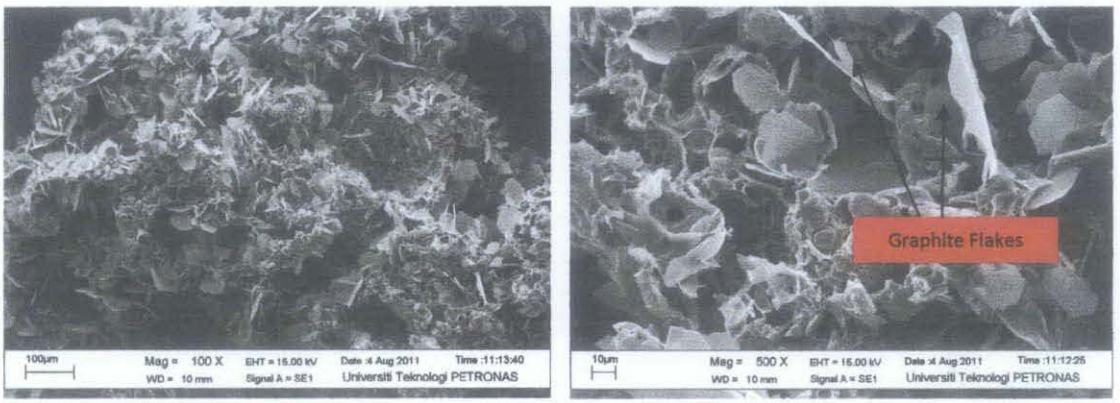


Figure 4.13: SEM Micrograph of FGO7 coating for inner surface with 100x (left) and 500x (right) magnifications

From Figure 4.12, the outer surface of the coating was smooth with bubbles, holes and cracks observed on the charring layer. The bubbles are formed due to the emission of N_2 and ammonium gases during burning process. The FG07 coating demonstrates a good intumescent behavior. Inside the coating occurs the emission of N2, NH3 and CO2 gas and dehydration of water. The graphite flakes appear in the inner surface and produce the heat barrier to protect the steel (substrate). Small holes observed are due to the heat dissipation that occurs which prevent heat from transferring to the surface.

ii) FG08 Coating Sample

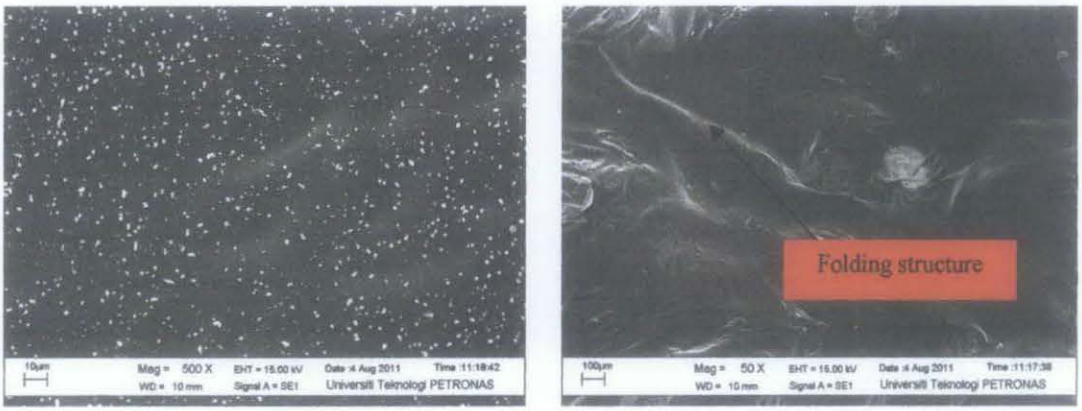


Figure 4.14: SEM Micrograph of FGO8 coating for outer surface with 500x (left) and 50 x (right) magnifications

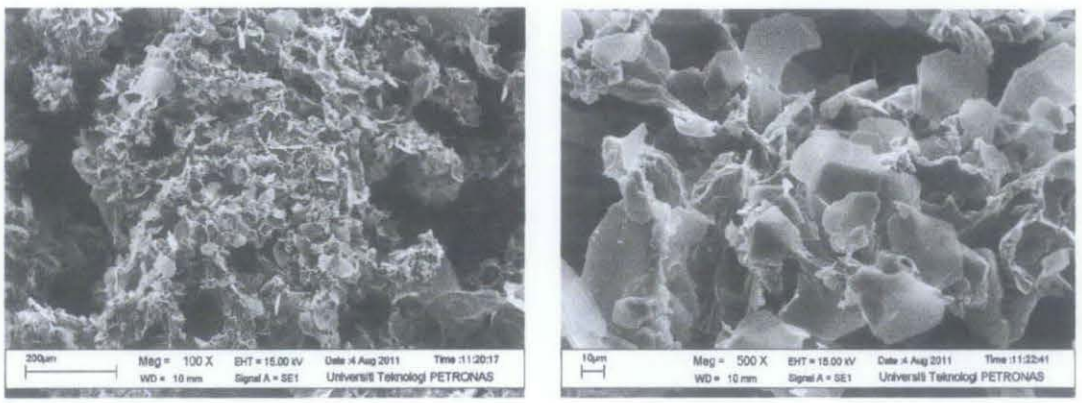


Figure 4.15: SEM Micrograph of FGO8 coating for inner surface with 100x (left) and 500x (right) magnifications

From the SEM images results in Figure 4.14 and Figure 4.15, the outer surface of the coating was smooth with bubbles, little cracks and folding structures. The formations of bubbles are due to the emission of N_2 and ammonium gases during burning process. The FG08 coating demonstrates a good intumescent behavior and swells nicely since there are emissions of N2, NH3 and CO2 gas and dehydration of water which occurred inside the charring layer. The graphite flakes appear in the inner surface and produce the heat barrier to protect the steel (substrate).

4.4.2 Fire Test at 650 °C

i) FG07 Coating Sample

Figure 4.16: SEM Micrograph of FGO7 coating for outer surface with 500x (left) and 50 x (right) magnifications

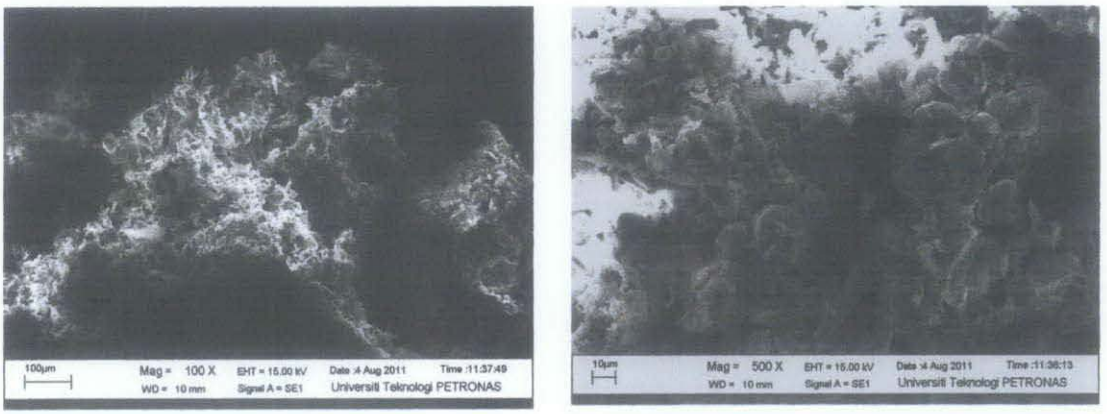


Figure 4.17: SEM Micrograph of FGO7 coating for inner surface with 100x (left) and 500x (right) magnifications

The SEM micrographs of chars for sample FG07 are shown in Figure 4.16 and Figure 4.17. There are large holes and white powder formations observed on the surface with cracks in the inner surface of the charring layer. The presences of white powder (ashes) on the surface was as a result of high burning temperature of 650 °C of the coatings which turned little portions of the coating into ashes.

The surface of the coating swells properly due to dehydration of water and emission of gas from the holes. Large holes dissipated heat from inside and prevent the heat from transferring to the surface.

ii) FG08 Coating Sample

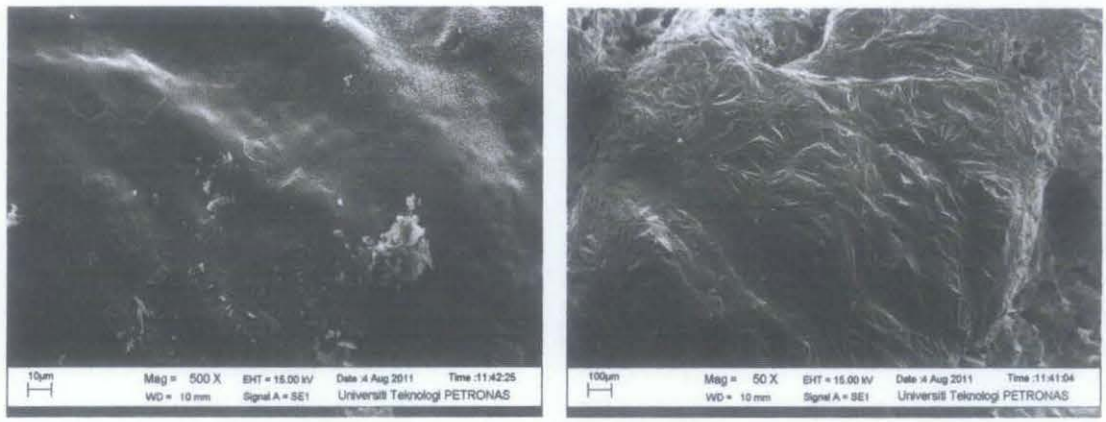


Figure 4.18: SEM Micrograph of FGO8 coating for outer surface with 500x (left) and 50 x (right) magnifications

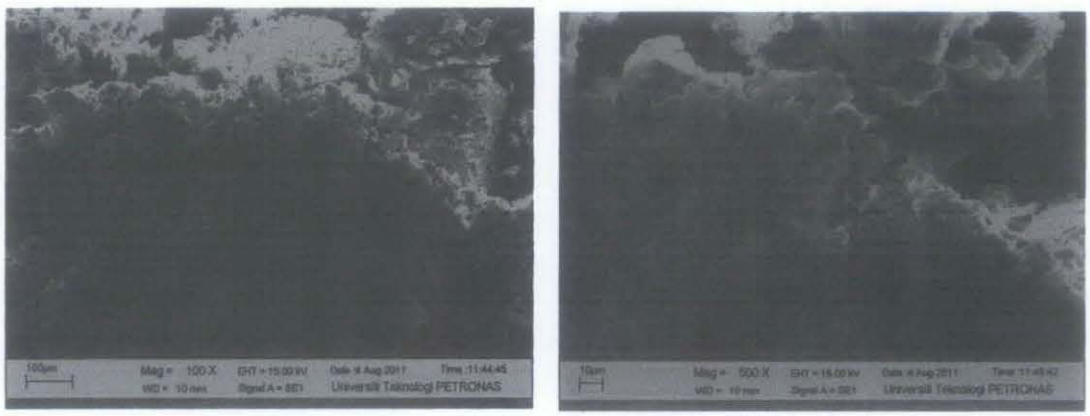


Figure 4.19: SEM Micrograph of FGO8 coating for inner surface with 100x (left) and 500x (right) magnifications

The SEM micrograph of chars for outer and inner surface for sample FG08 is shown in Figure 4.18 and Figure 4.19 where holes, folding structure and white powder formations observed on the charring layer. The outer surface of the coating was smooth with small holes and the presence of white powder (ashes) due to very high temperature of the fire test at 650 °C.

The FG08 coating illustrates a good intumescent behavior. Inside the coating occurs the emission of N2, NH3 and CO2 gas and dehydration of water. The graphite flakes appear in the inner surface and produce the heat barrier to protect the steel (substrate). Small holes can be observed too where the heat dissipated from and prevent the heat from transferring to the surface.

4.4 X-RAY DIFFRACTION ANALYSIS

After the residue char of the intumescent coating was oxidized at high temperature, only some amorphous carbon and inorganic materials remained. The inorganic materials might be the main protecting layer at later stages of burning. XRD analysis is carried out to investigate the presence of graphite, boron oxide and boron phosphate in the coating after fire test at 450 °. The facial residue of sample FG07 and FG08 were analyzed using XRD and the results are shown in Figure 4.20 and 4.21.

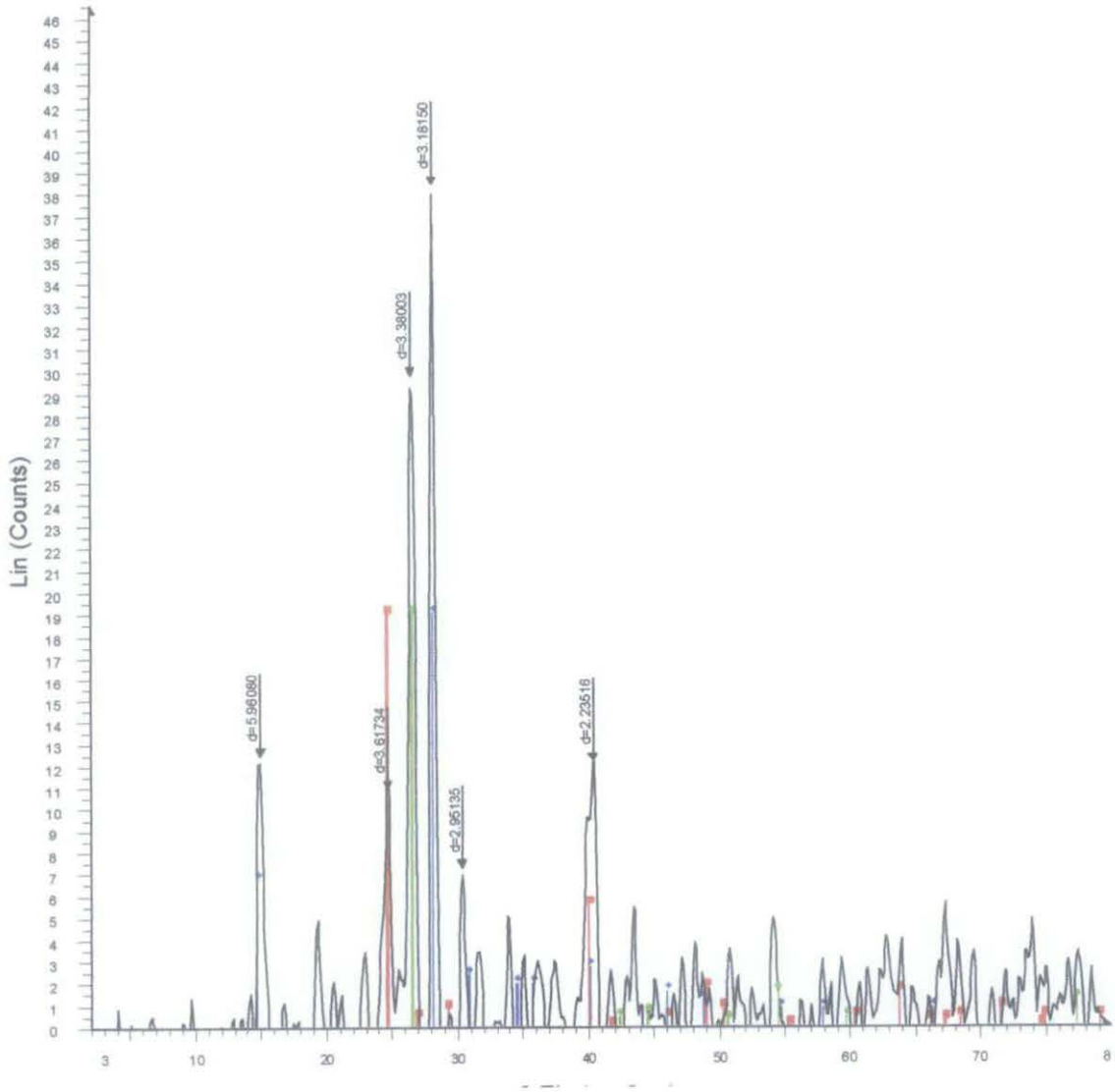


Figure 4.20: XRD curve of the facial residue char of sample FG07 at 450 °C

In Figure 4.20, several XRD peaks of the residue char at 2.95135 and 3.18150 were assigned to boron oxide and peaks at 2.23516 and 3.61734 were assigned to boron phosphate as the result of dehydration of boric acid. The reaction between APP and boron oxide yield some boron phosphate in the charring element. The graphite peak was found at 3.38003.

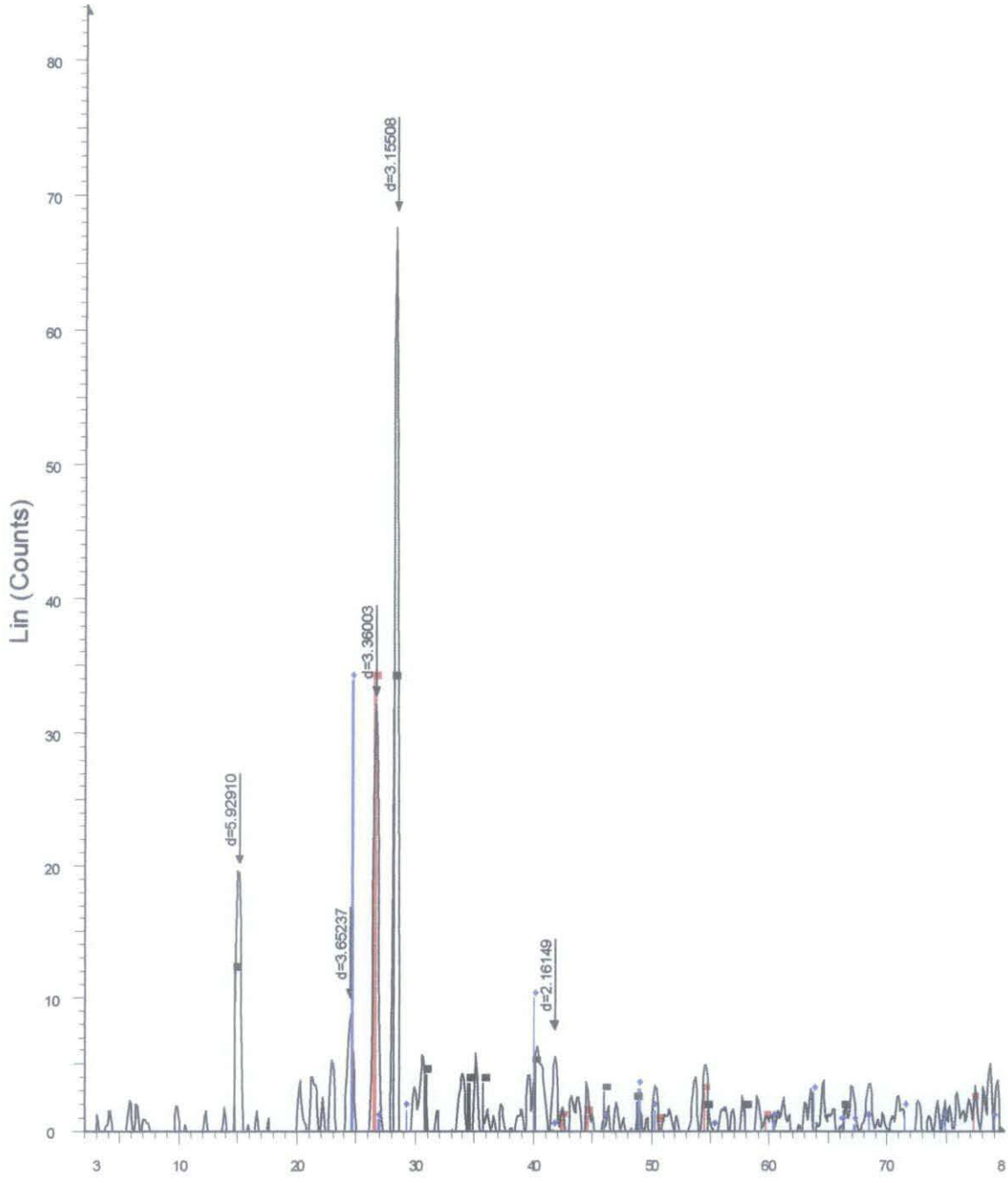


Figure 4.21: XRD curve of the facial residue char of sample FG07 at 450 °C

Figure 4.21 shows the facial residue curves of sample FG08 analyzed using XRD. Several peaks of the residue char at 3.65237 and 2.16149 were assigned to boron oxide and peaks 3.36003 were assigned to boron phosphate. The dehydration of boric acid yield boron oxide while the reaction between APP and boron oxide yield some boron phosphate in the charring element. No graphite peak was found in the FG08 sample.

4.5 THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermogravimetric analysis of samples carried out under controlled air and temperature conditions gives an overview of the degradation process of the coating. The weight loss is plotted against the temperature to see if effective intumescent occurs. The aim is to obtain a high level of homogenous char at the end of the experiment with high amount of residue and homogenous char. This residue will limit the heat transfer to the substrate and will limit the gases feeding combustion process. A slow degradation rate will lead to a more homogenous char.

Sample FG07 and FG08 with 6.0 grams and 7.0 grams of fiberglass left some residue at 800 °C which may not be enough charring to allow a good expansion. There should be higher amount of residue left at temperature higher than 800 °C in order for the coating to protect the steel. Figure 4.22 and Figure 4.23 shows the TG curves of sample FG07 and FG08 carried out at 10 °C/min under N_2 over the whole range of temperature of 50 °C – 800 °C. Some residues were observed after degradation over 800 °C.

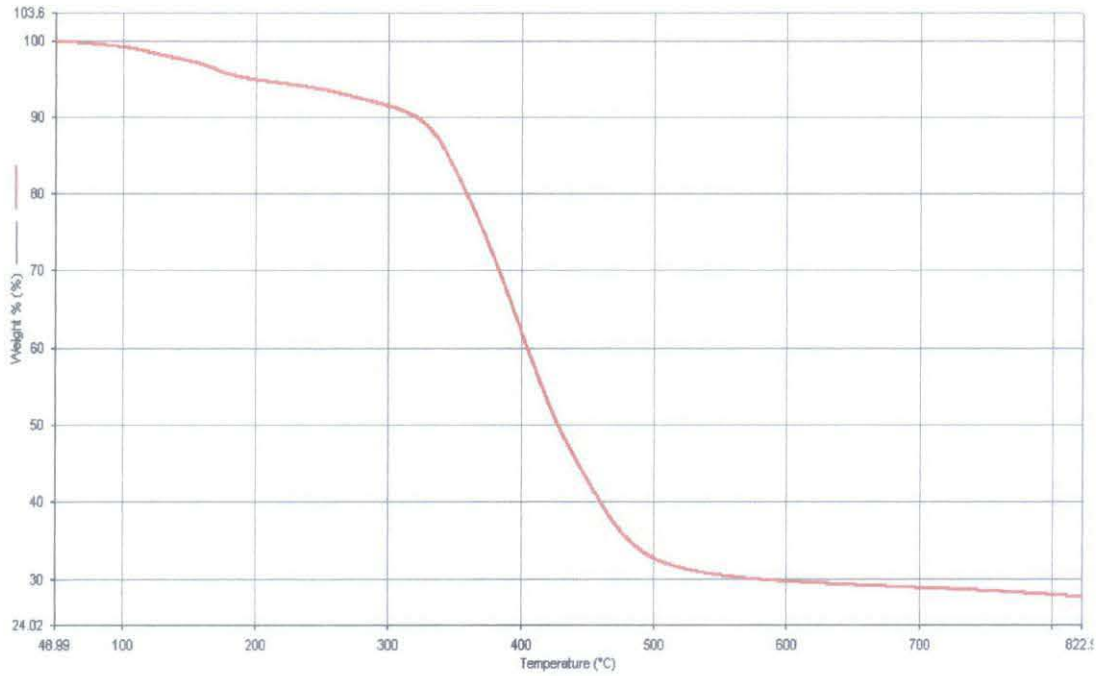


Figure 4.22: TG curves of sample FG07

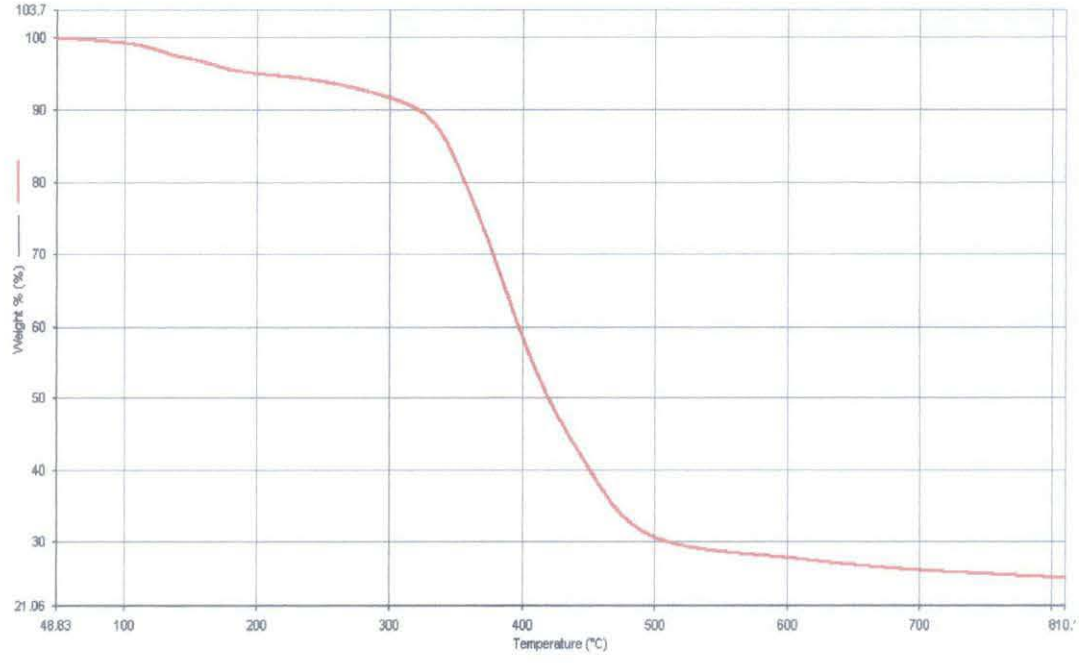


Figure 4.23: TG curves of sample FG08

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSION

Intumescent chemical systems are designed to swell into thick, strong foam upon exposure to heat, protecting the underlying material from fire by providing a physical barrier to heat and mass transfer [9]. The fire resistant capabilities of intumescent materials, which respond to heat by swelling into an insulating char of thickness between 5 to 100 times of the original material [11,14]. This study have focused into the preparation of the expandable graphite as char former and fiberglass as an insulating agent in enhancing the thermal insulation property of the coating. Various amount of fiberglass ranging from 1.0-7.0 grams were added into the basic intumescent coating formulations. Sample FG07 (with 6.0 grams fiberglass) and FG08 (with 7.0 grams of fiberglass) recorded the best results in terms of the enhanced thermal insulation properties of the coatings. The FG07 and FG08 coating recorded the best expansion during fire test at 450 °C and 650 °C with 5-7 times expansion than the original thickness of the coating before burnt, the highest back steel temperature approaching 300 °C during thermal insulation test and illustrate good coating characteristics after analysis using SEM. These coatings also recorded the presence of graphite, boron oxide and boron phosphate after XRD testing and with some residue left after degradation analysis using SEM. Thus, it can be concluded that the addition of fiberglass into the basic intumescent formulations helps to enhance the thermal characteristic of the coating and helps to maintain the structure of buildings in longer time.

5.2 RECOMMENDATIONS

Intumescent flame retardant systems using expandable graphite (EG) as the carbon source is good due to its advantages of non-halogen, smoke reduction and non-heavy materials. It is recommended to use EG that are manufactured and treated from a trusted supplier rather than treating graphite manually. The reason is because the EG from a trusted supplier provide better char structure and expansion than the EG that was manually treated. For further studies in the intumescent coating field, other high resistance fibre could be used as reinforcement since it is a good reinforcement agent in improving the thermal properties of intumescent coating. Other testing such as DSC can be performed to study more on the thermal performance of the intumescent coatings.

REFERENCES

[1] Jimenez. M, Duquesne. S, Bourbigot. S, 2008, *"Multiscale Experimental Approach for Developing High-Performance Intumescent Coatings"*, **45**: 4500 – 4508.

[2] Gu. Jun-wei, Zhang. Guang-cheng, Dong. Shan-lai, Zhang. Qiu-yu, Kong. Jie, 2007, *"Study on Preparation and Fire-Retardant Mechanism Analysis of Intumescent Flame-retardant Coatings"*, **201**: 7835 – 7841.

[3] Olcese. T, Pagella. C, 1999, *"Vitreous Fillers in Intumescent Coatings"*, **36**: 231 – 241.

[4] Reshetnikov. Igor S, Yublokova. Marina Yu, Khalturinskij. Nikolay A, 1997, *"Influence of Surface Structure on Thermoprotection Properties of Intumescent Systems"*, **115**: 199 – 201.

[5] Duquesne. S, Magnet. S, Jama. C, Delobel. R, 2004, *"Intumescent Paints: Fire Protective Coatings for Metallic Substrate"*, **180 – 181**: 302 – 307.

[6] Duquesne. S, Magnet. S, Jama. C, Delobel. R, 2005, *"Thermoplastic Resin for Thin Film Intumescent Coatings – Towards a Better Understanding of their Effect on Intumescent Efficiency"*, **88**: 63 – 69.

[7] Jiminez. M, Duquesne. S, Bourbigot. S, 2006, *"Characterization of the Performance of an Intumescent Fire Protective Coating"*, **201**: 979 – 987.

[8] Guoxin. Li, Junfen. Yang, Tingshu. He, Yonghua. Wu, Guozheng. Liang, 2008, *"An Investigation of the Thermal Degradation of the Intumescent Coating Containing MoO_3 and Fe_2O_3"*, **202**: 3121 – 3128.

[9] Wang, Z, 2007, *"Application of EG to Flame-retardant Coatings for Steel Structure and Influence of EG on Corrosion Resistance"*, **49**: 2237 – 2253.

[10] Wang. Et Al, 2007, *"Corrosion Science"*, **49**: 2237-2253.

[11] Przybylak-Wladyka. M, Kozlowski. M, 1999, *"Thermal Characteristic of Different Intumescent Coatings"*, **23**: 330-350.

[12] Bugajni. M, Le Bras. M, Bourbigot. S, 1999, *"New Approach to the dynamic Properties of an Intumescent Materials"*, **23**: 49.

[13] Kang Shen, Schilling. Bernd, *"Recent Advances with Expandable Graphite in Intumescent Flame Retardant Technology"*, **34**: 205.

[14] Bourbigot. S, Flambart. X, 2002, *"Heat Resistance and flammability of High Performance Fiber"*, **26**: 155.

[15] Tramm. H, 1998, U.S Patent 2, 106, 938.

[16] J. Hao, W.K. Chow. 2003, *"A Brief Review of Intumescent Fire Retardant Coatings"*, **46**: 300-440.

[17] Sami Ullah, Faiz Ahmad, P.S.M. Megat-Yusoff. *"Effect of Boric Acid with Kaolin Clay on Thermal Degradation of Intumescent Fire Retardant Coating"*.

[18] Kang Shen, Bernd Schilling. *"Recent Advances with Expandable Graphite in Intumescent Flame Retardant Technology"*.

APPENDIX A

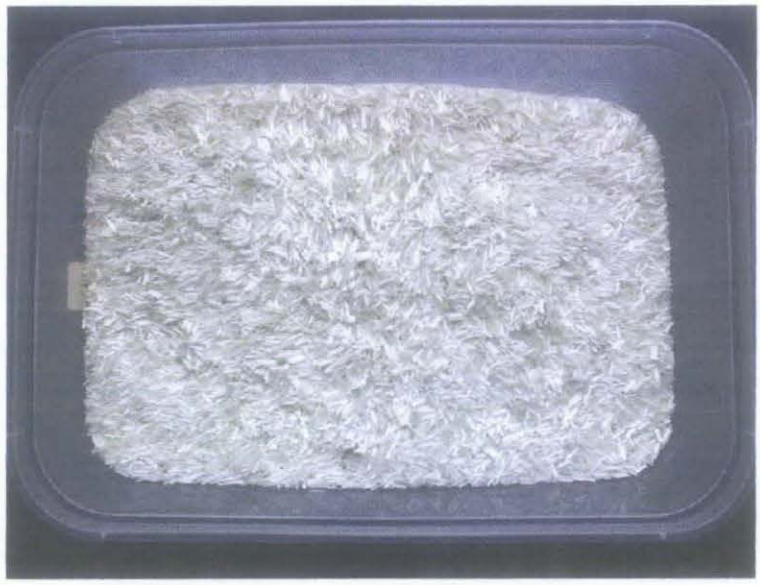


Figure 6.1: Short fiberglass of length 3.0 mm

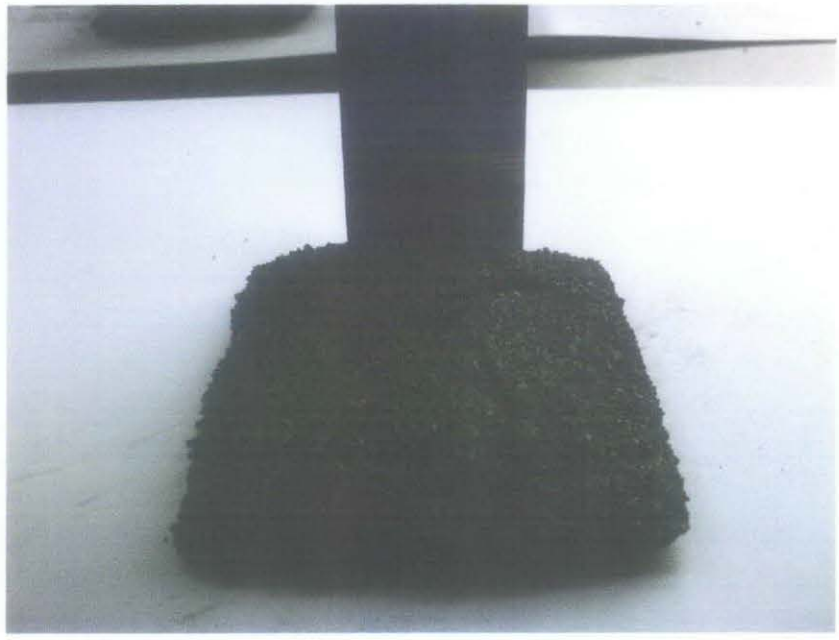


Figure 6.2: Coating thickness measurement technique

APPENDIX B

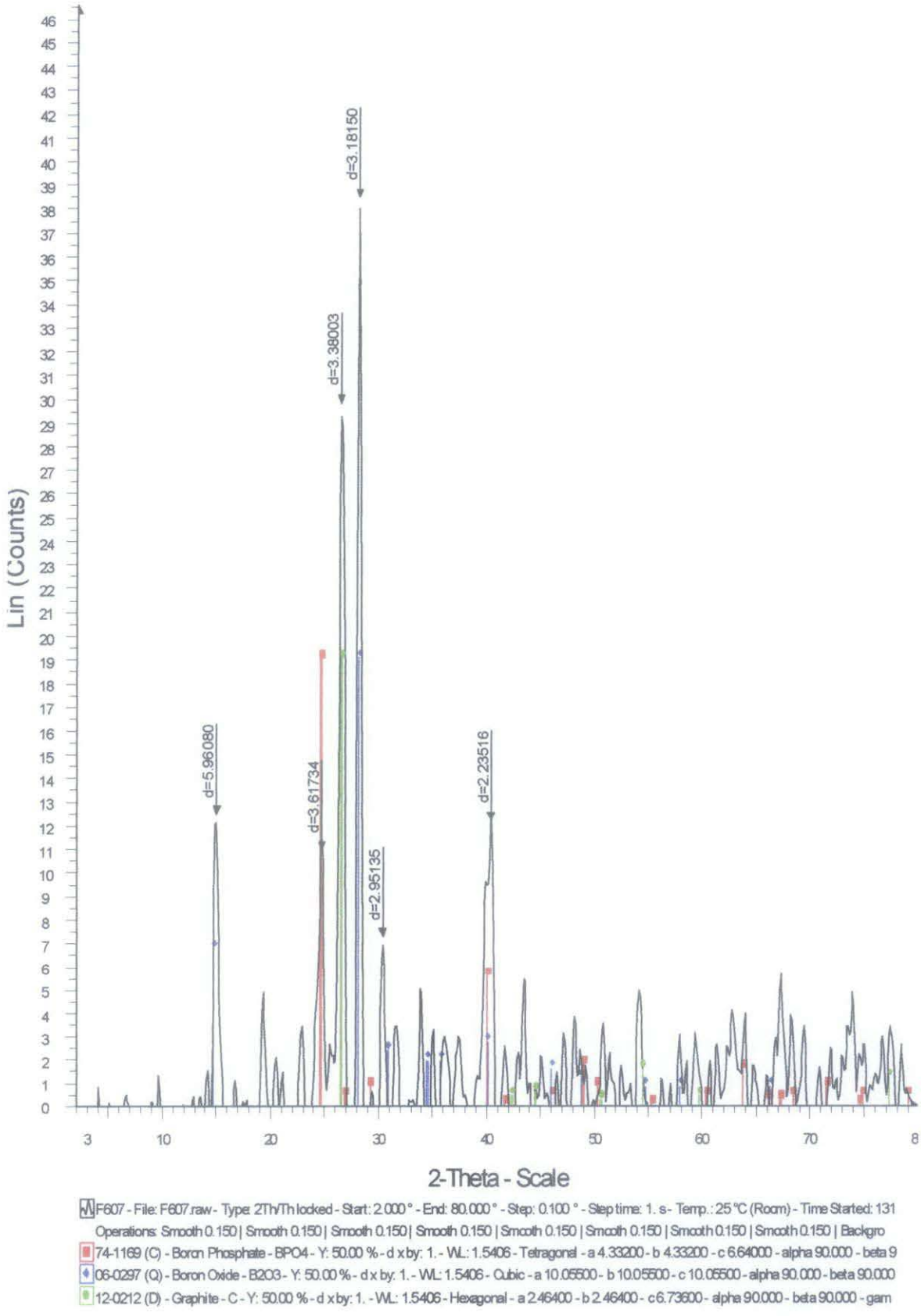


Figure 6.3: XRD curve of the facial residue char of sample FG07 at 450 °C

APPENDIX C

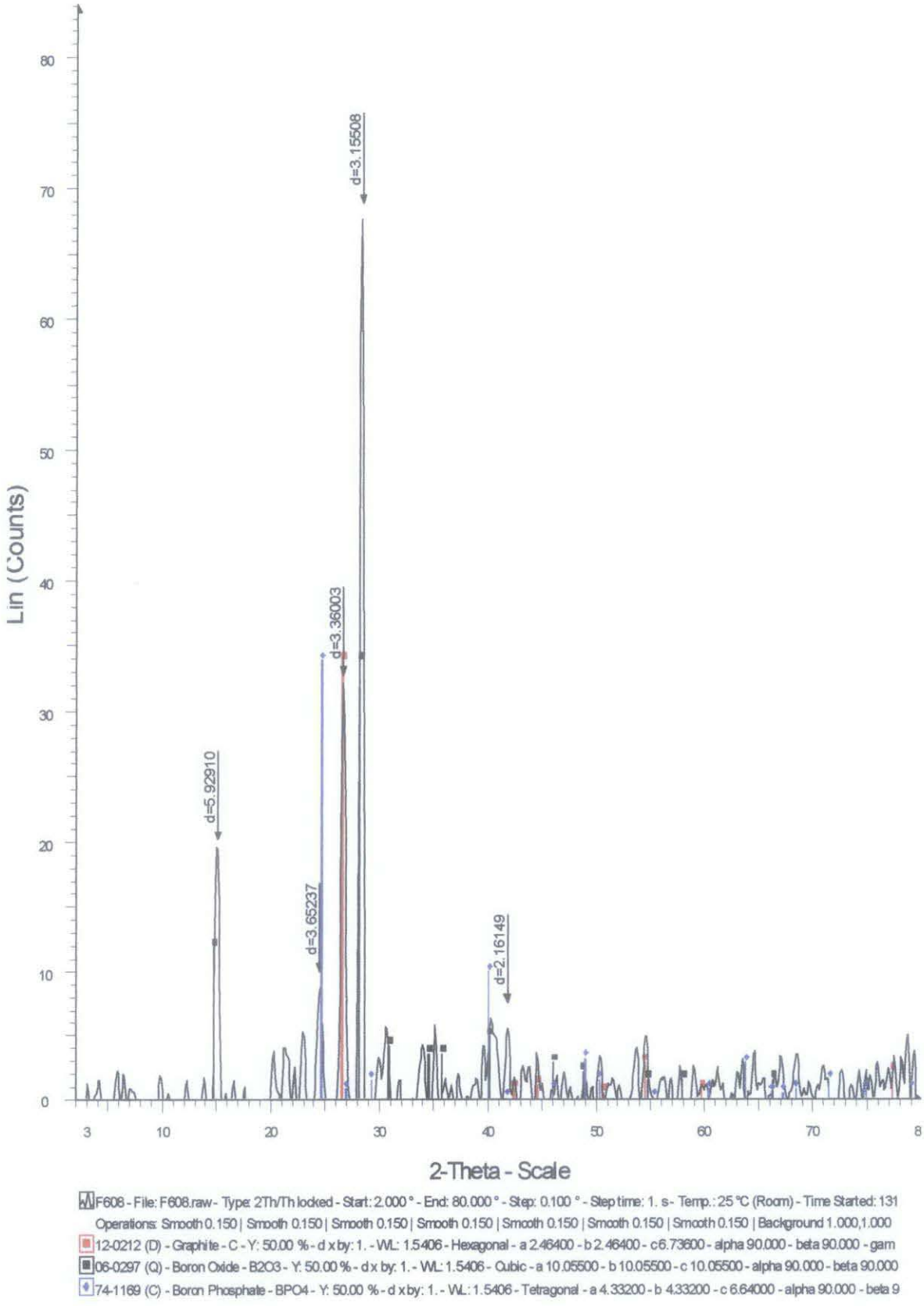


Figure 6.4: XRD curve of the facial residue char of sample FG08 at 450 °C