STUDY ON CHAR EXPASION AND HEAT SHIELDING OF INTUMESCENT COATING BASED ON INORGANIC FILLERS

BY

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CERTIFICATION OF APPROVAL

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Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

September 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.

(HARNAMDAV SINGH A/L HARBINDER SINGH)

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ABSTRACT

Fire Accidents are constant threat to man and materials. When a fire occurs it takes a long time from hours to days to put it off depending on the burning material and other circumstances. This can be reduced in term of risks with the help of findings on Intumescent coating. Traditional fire retardants are not very effective especially when there is a huge breakout of fires in plants and platforms. These traditional fire retardants also contain halogen and releases toxic vapours thus leading to a severe threat to life and environment. By contrast, intumescent coatings are relatively thin-film products that expand rapidly in a fire to insulate the steel. They come in various 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 the steel. In general, steel loses half its strength at 1,100 °F and begins to degrade as well as starts to loose its properties. Consequently leads us to the aim of this project which is to study the details of the expansion of char and heat shielding performance with respect to two main inorganic fillers, (Fume Silica & Aluminium Tri-Hydrate). In addition, researcher will develop an intumescent coating formulation with inorganic fillers to get the optimal performance for char expansion and heat shielding performance. Studies will focus mainly on the expansion of char that is influenced by inorganic fillers. Firstly, an Intumescent coating is to be made by the provided formula. Expandable graphite is to be grinded and sieved into various sizes. Inorganic fillers (Fume Silica and Aluminium Tri-hydrate) are also added as part of the mixture for the Intumescent coating. There are also other additives involve in developing the coating such as, Melamine, APP, Epoxy (Bisphenol A), Boric Acid, ACR Polyamide Hardener, etc. Once the coating had been completed, it is than applied onto steel plates and left cured under room temperature. The experiment is further carried out by heating the Intumescent coating using Bunsen burner to a certain temperature. Expansion of char is to be thoroughly observed as well as also the performance of heat shielding. Results obtained are discussed and further studied.

CHAPTER 1: INTRODUCTION

1.1 Background of study

The protection of metallic materials 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 [1]. Steel usually begins to lose its structural properties above 500°C. Intumescent coatings are designed to perform under severe conditions and to maintain the steel integrity for between 1 and 3 h in some cases when the temperature of the surroundings is in excess of 1100 °C.[2-4] Upon heating, foamed cellular charred layers are formed on the surface, which protect the underlying material from the action of the heat and/or flame.

Intumescent thick films are usually based on epoxy, vinyl, or other elastomeric resins and contain ingredients providing intumescence upon heating [5]. They are available as solvent-free systems, permitting the application of up to 8-10 mm thick coating. They are hard and durable, and some of them can provide excellent protection from corrosion [6]. They exhibit very high adhesion to the substrate and resistance to impact, abrasion, and vibration damage. High tensile and compressive strengths can be obtained, and weather resistance is excellent.

These intumescent coatings are high-value products for industries, and the competition in developing new products is high. But the development of new intumescent coatings depends on many parameters: the Intumescence concept requires a balance between the fire properties and the level of additives in the material. The formulation of the coating has first to be optimized in terms of physical and chemical properties (char strength, expansion, and viscosity, etc.) in order to form an effective protective char. This latter should limit both the heat transfer from the heat source to the substrate and the mass transfer from the substrate to the heat source, resulting in conservation of the underlying material.

1.2 Problem statement

- Fire Accidents are constant threat to man and materials.
- Fire fighting process takes time, from hours to days, depending on the burning material and other circumstances.
- Traditional Fire retardants are not very effective.
- These also contain halogens and release toxic vapours, a severe threat to life and environment.

1.2.1 Problem Identification

Different inorganic fillers react differently onto the Intumescent coating thus resulting in difference of expansion and heat shielding performance.

1.2.2 Significant of Project

The aim of this project is to observe the expansion of char with respect to use of different inorganic fillers (Fumed Silica and Aluminium Tri-Hydrate) as well as testing its heat shielding ability.

There were reasons to why the two fillers (Fumed Silica and AluminiumTri-Hydrate were selected as the properties and behavior and advantages of each were studied. Hence producing a better coating is the aim with respect to some parameters.

1.3 Objective

- The aim of this project is to study the details of the expansion of char and heat shielding performance with respect to two main inorganic fillers, (Fume Silica & Aluminium Tri-Hydrate).

- To Characterize (chemical) intumescent coating formulation with inorganic fillers to get the optimal performance on heat shielding and char expansion.

1.4 Scope of Study

Studies will focus mainly on the expansion of char that is influenced by inorganic fillers. Firstly, an Intumescent coating is developed by the provided formula. Expandable graphite is to be grinded and sieved into 300, 200 & 150 Micron Meter. Inorganic fillers (Fume Silica and Aluminium Tri-hydrate) are also added as part of the mixture for the Intumescent coating. There are also other additives involve in making the coating such as, Melamine, APP, Epoxy (bisphenol A), Boric Acid, ACR Polyamide Hardener, etc. Once the coating had been completed, it is than applied onto steel plates and left to dry. The experiment is further carried out by heating the Intumescent coating using Bunsen burner to a certain temperature. Expansion of char is to be thoroughly observed as well as also the performance of heat shielding. Improvisation on the formula is recorded down and further studies.

1.4.1 Relevancy of the Study

This project discusses on the effects of Inorganic fillers onto the performance of Intumescent coating. The project as per stated has its relevancy to the course that the author is undertaking, Mechanical Engineering. Under this course there are a few subjects that will help contribute information as well as better understanding to help make this project a successful one. The subjects are :-

1) *Engineering Materials*
2) *Manufacturing Technology*
3) *Advanced Polymer Engineering*

1.4.2 Feasibility of the Project within the Scope and Time Frame

The project is divided into three sections. Section one will basically be on finding, collecting, and reading of journals, technical papers, and books of the research topic. In this section one, the author should understand the characteristics of the inorganic fillers and other related materials in producing the coating.

The second section of the project will be where the developing of the intumescent coating with proper formulation that relates other related materials inclusive of :-

- Expandable Graphite
- Melamine
- Ammonium Polyphospate
- Epoxy Resin Bisphenol A
- Triethlyene-Tetramine
- Fumed Silica and Aluminium Tri-Hydrate
- Graphite
- Sulphuric Acid
- Boric Acid
- Acetic Acid

The third section of the project involves experimental activities such as furnace test and Bunsen burner test for heat shielding effect as well as also to monitor on the expansion of char.

Final section will be analytically observing the results obtained from experiments and further documenting the data to show findings. Data and findings are formally presented in written format as well as presentation slides with the help of visual and graphical aids.

CHAPTER 2: LITERATURE REVIEW

Intumescent flame retardant (IFR) is well known as an efficient flame retarding system for polymer materials especially polyolefin (i.e, polypropylene, polyethylene) [7–9]. Generally, the Intumescent flame retardant system mainly consists of three ingredients, namely acid sources (acids, ammonium salts, phosphates, etc), blowing agents (melamine, etc) and carbonization agents (polyols. etc) [10,11].

Currently, the barrier effect of carbonaceous char existing not only in condensed phase but also gaseous phase is involved in intumescence mechanism [12,13]. In brief, the flame retardation process of IFR involves the following steps; the carbonization agent dehydrates to form melted char precursor under the catalysis of acid source which can form acid or polyacid in the initial combustion; inert gases released from the blowing agent on heating expand the melted char precursor simultaneously and form a swollen char layer. The formed non-flammable and swollen char layer can provide a shielding and barrier for the polymer against the direct contact with flame, and inhibit heat and oxygen transfer between the flame zone and the underlying material. Based upon the above analysis, it is of great importance to modify the barrier formed in intumescent flame retarded systems so as to achieve a greater degree of flame retardation [14].

Nowadays, synergistic flame retarding performance [15] of polyolefins has attracted much attention from both academia and industry, among which synergistic effect between intumescent flame retardants and inorganic nanofillers (clay minerals, layered double hydroxides, carbon nanotubes, etc.) is one of the most important research fields. Layered double hydroxides (LDHs), also known as bucite-like compounds, are a large class of anionic clays with general formula of [M2þ 1_xM3þ (OH)2]xþ[An_]x/n$nH2O (whereM2þ andM3þ represent metallic cations and An_ represents the interlayer anion). As a subgroup of two dimensional nanostructure materials, similar structure for layered double hydroxide and clay mineral leads to common characters when being used as fillers in polymer composites, for example, as potential flame retardant [16–18].

Manzi-Nshuti et al. [19] investigated a few polymers modified by a series of LDHs with various compositions that showed improved fire properties for PMMA by the introduction of LDHs. Study on the effect of various loading of both undecenoate intercalated ZnAl-LDH and/or melamine demonstrated that fire performance of PMMAwas significantly modified in terms of reduction in PHRR and the average smoke generated by the combination of the two additives [20]. Although the enhancement of flame retardancy is mainly attributed to the promoting effect of inorganic fillers on charring of composites, different degradation pathways lead to various performances of flame retarded composites upon pyrolysis and combustion. Among several mechanisms proposed to annotate the flame retarding action modes of polymer/ layered silicate composites, barrier effect [21–23] of carbonaceous char catalyzed by clay particles is a dominant one. According to Lewin et al. [24,25], free migration of clay nanofoils taking place before pyrolysis and combustion due to different surface energy between clay and polymer matrix and which will be propelled by bubbles generating from degradation of polymer provides a novel and convincing idea to interpret the flame retardancy mechanism for layered silicates. Whereas, enhanced flame retardancy of polymer/ LDH nanocomposites is mainly attributed to water released from LDH, which can dilute and cool the combustible products in the flame zone as well as improve char formation [26,27].

However, the conversion of LDH from layered structure to mixture of metal oxides occurred upon heating seems to result in reverse action, especially being used along with intumescent flame retardant as synergistic agent in comparison with their cationic counterparts to modify the protective carbonaceous char, and which is rarely studied till now.

Internationally most countries are interested in establishing and sustaining high level of fire safety in consumer products. The role of flame retardants as a part of the solution is well recognized. Majority of the flame-retardants are still halogenated chemicals and they operate in the gas phase by diluting combustible gases. During flame retardant action, there is a loss in the mass of the polymer, which is very high and is responsible for producing toxic gases like carbon monoxide and other harmful gasses These gases are considered to be harmful and contaminate the fireplace and its surroundings due to its high toxicity. Present need is to reduce the toxic gases during fire, this is possible by using non-halogenated flame retardant. However, non-halogenated flame-retardants also have some problem like high level of loading in to the polymer, which deteriorate the original properties of polymer. Although suitable solutions have not been found in all cases. However, it is possible in flexible polyurethane foam by the proper selection of expandable graphite as a non-halogenated flame retardant.

When expandable graphite is exposed to heat, it expands to more than 100 times its original volume and covers the entire burning surface by "worm" like structure of expanded graphite.

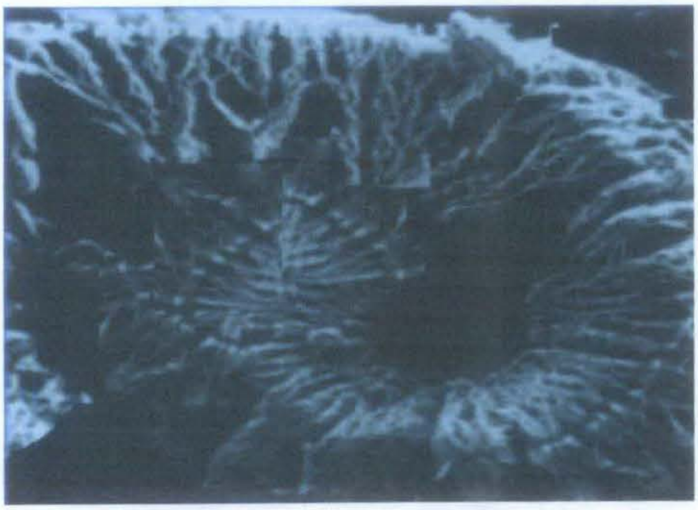


Figure 1 : Worm like structure of expandable graphite

Expanded graphite acts as a char former and also as an insulating agent due to the formation of small air gaps between the graphite layers8. It dramatically reduces the heat release, mass loss, smoke generation and toxic gas emission. However, all expandable graphite does not act as flame-retardant, only low temperature expandable graphite act as flame retardant. The expansion must occur at "critical temperature" where decomposition, exothermal reaction and ignition occur spontaneously. This critical temperature range depends upon the chemical composition of polymer. Critical temperature in flexible polyurethane is 300°C to 500°C. [28]

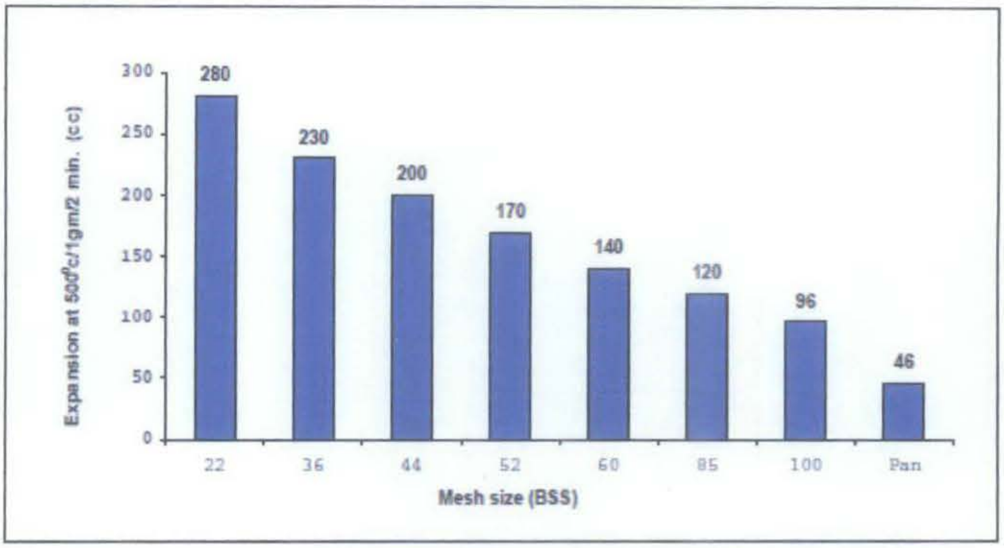


Figure 2 : Expansion of expandable graphite with respect to various particle size

CHAPTER 3: METHODOLOGY

3.1 Project Work

Different findings and methodologies are gathered from the research work of other researchers and to be incorporated in this project. First and foremost, various journals and technical papers were read through the get the general understanding on the project. It is also needed to identify the objective of this project and to come up with an organized strategy on conducting experiment next semester.

Below is a brief approach taken by the author in planning a successful project and aims for success.

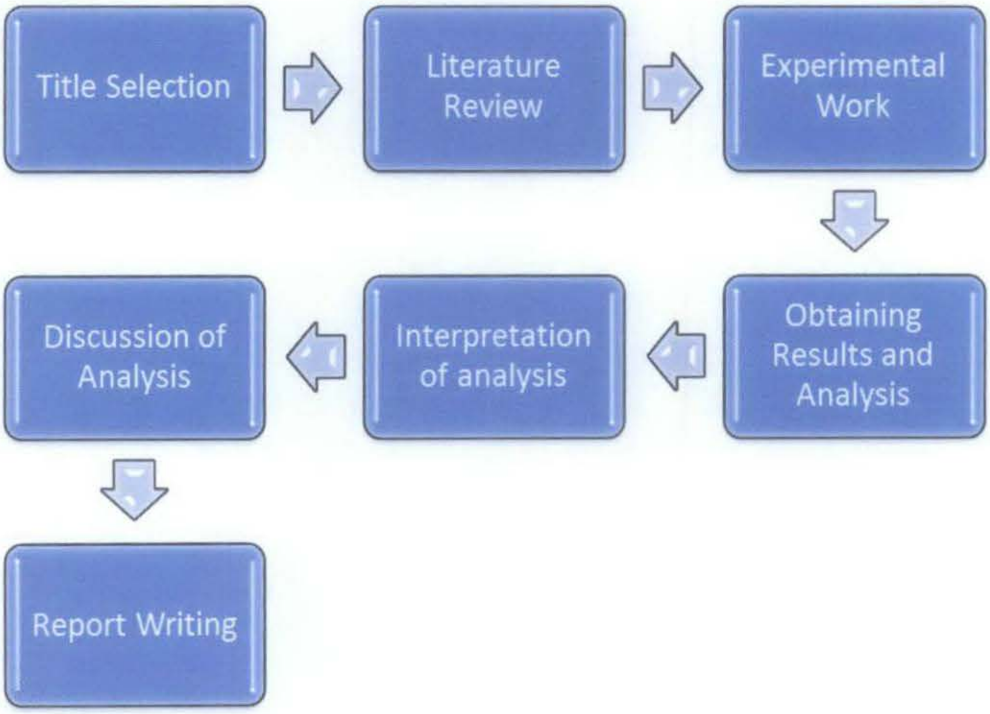


Figure 3: Project Activities Flow Chart

3.2 Project Activities

The project of producing the coating is as in figure 4. Just after the coating is produced using two different types of inorganic fillers (Fumed Silica and Aluminium Tri-Hydrate). It is than coated/applied onto steel plates. These steel plates are left to cure naturally for 7 days with room temperature to ensure that it is properly dried. Substrate is experimented with Bunsen burner as well as furnace heating. Results after experimented is viewed and further studied using TGA microscope.

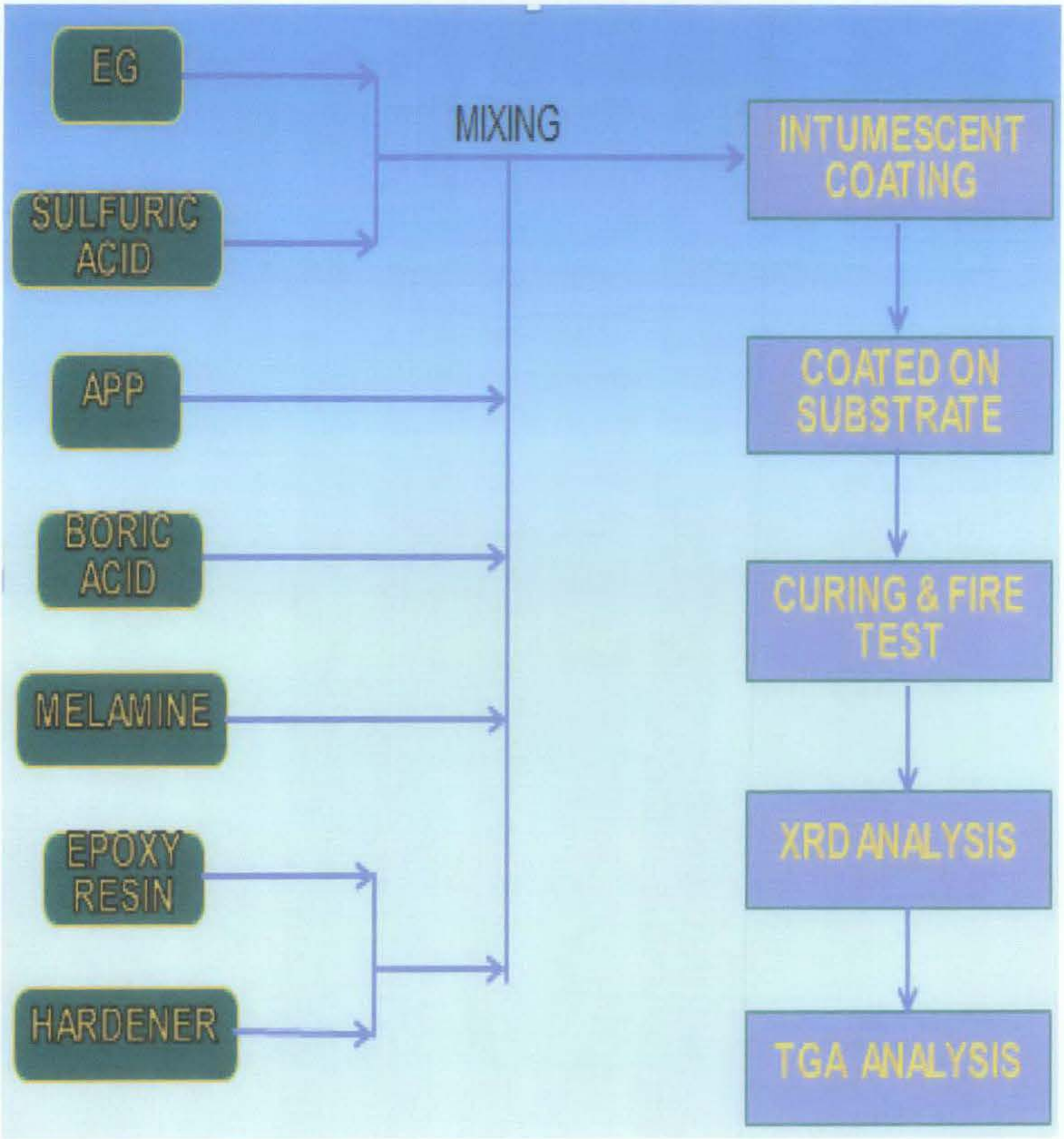


Figure 4 : Experimental process overview upon developing Intumescent Coating

NO	*SAMPLE*	*BPA (g)*	*TETA (g)*	*APP (g)*	*EG (g)*	*MEL (g)*	*BA (g)*	*FILLER (g)*
0	**ATH & FS**	44.44	22.22	11.11	5.56	5.56	11.11	0
1	**ATH 1**	43.94	21.72	11.11	5.56	5.56	11.11	1
2	**ATH 2**	43.44	21.22	11.11	5.56	5.56	11.11	2
3	**ATH 3**	42.94	20.72	11.11	5.56	5.56	11.11	3
4	**ATH 4**	42.44	20.22	11.11	5.56	5.56	11.11	4
5	**ATH 5**	41.94	19.72	11.11	5.56	5.56	11.11	5
1	**FS 1**	43.94	21.72	11.11	5.56	5.56	11.11	1
2	**FS 2**	43.44	21.22	11.11	5.56	5.56	11.11	2
3	**FS 3**	42.94	20.72	11.11	5.56	5.56	11.11	3
4	**FS 4**	42.44	20.22	11.11	5.56	5.56	11.11	4
5	**FS 5**	41.94	19.72	11.11	5.56	5.56	11.11	5

ATH	**ALUMINIUM TRI-HYDRATA**
FS	**FUMED SILICA**

Table 1: Formulation of mixture for Intumescent coating

3.2.1 Procedure of preparing Intumescent Coating

1. Weigh the materials in the table above using a proper weighing scale.
2. Each sample prepared is grinded using a grinder.
3. Once properly grinded, Epoxy (BPA) and Hardener (TETA) is mixed in a separate container according to the listed weight.
4. Epoxy and Hardener is mixed using mixer for approximately 5 minutes. Make sure that the two substrates are properly mixed.
5. Add the grinded mixture of sample into the mixed epoxy and hardener slowly using the mixer with speed increasing from 20Rpm – 50Rpm.
6. Substrate is left mixed for about 15 minutes to ensure a proper mixing coating.
7. Completed mixture is coated on steel plates with specific dimension and left for curing naturally under room temperature for approximately 7 days.

3.2.2 Task Completed (FYP 1)

- Identification of Title and Project Objective
- Research Works
- Methodology
- Preparation of sample for Inorganic filler (Aluminium Tri- Hydrate)
- Grinded mixed and coated sample (0-5 for ATH) onto steel plated and left for drying naturally under room temperature.

3.2.3 Task Completed (FYP 2)

- Completed grinding, mixing, and coating the samples onto steel plates.
- Completed furnace test at 500 and 800 degree Celsius for ATH and FS filler.
- Recorded results of heat shielding test for all substrates.
- Sent selected substrates for XRD testing.
- Sent selected substrates for TGA testing.
- Measured Char expansion for all substrate for both heat shielding and furnace test.
- Discussing the results obtained.

3.2.4 Tools Required

- Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD) and FTIR analysis - charring layer and their morphological structures.
- Thermo gravimetric analysis (TGA) - thermal stability of coating.
- Bunsen burner
- Grinder
- Thermologger (Temperature Gauge)
- Furnace

3.3 Gantt Chart and Key Milestone

No	Detail / Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic	■	■						Mid Semester Break							
2	Preliminary research work		■	■	■	■										
3	Submission of Extended Proposal						☆									
4	Proposal Defense									■	■					
5	Experimental works, familiarize with the equipments											■	■	■		
6	Experimental works regarding with materials (preliminary)											■	■	■		
7	Submission of Interim Draft Report														☆	
8	Submission of Interim Report															☆

Figure 5: Gantt chart for the first semester project implementation

■ Processes ☆ Milestones

CHAPTER 4: RESULTS & DISCUSSION

4.1 Results

4.1.1 Measurement for IFR coating with/without filler and Char Expansion

Thickness of the coating coated onto the steel substrate is taken at four points on each side using a vernier caliper and the average is recorded. Once the sample completes the furnace testing, the expansion of char is measured by using a ruler. Measurement is taken at the peak of the char expansion for maximum expansion record on each samples. The highlighted data is for samples Without filler.

Measurement Technique :-

Average Thickness = (Thickness 1 + Thickness 2 + Thickness 3 + Thickness 4) / 4

Figure 6 : Thickness measurement

Maximum Expansion = Peak expansion of Char

Figure 7 : Char Expansion Measurement

4.2 Experiment Results

4.2.1 Furnace Test Results for ATH and FS Substrates

SAMPLE	TEMPERATURE	NOTE	PICTURE
ATH 0.1	500 DEGREE CELCIUS	DETACHABLE	
ATH 1.1		NOT DETACHABLE	
ATH 2.1		NOT DETACHABLE	
ATH 3.1		NOT DETACHABLE	
ATH 4.1		NOT DETACHABLE	
ATH 5.1		NOT DETACHABLE	

Figure 8 : ATH Char Expansion at 500 Degree Celsius

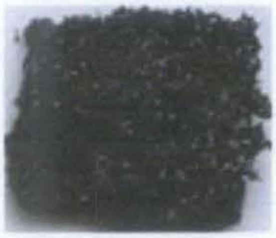
SAMPLE	TEMPERATURE	NOTE	PICTURE
ATH 0.2	800 DEGREE CELCIUS	DETACHABLE	
ATH 1.2		DETACHABLE	
ATH 2.2		DETACHABLE	
ATH 3.2		DETACHABLE	
ATH 4.2		DETACHABLE	
ATH 5.2		DETACHABLE	

Figure 9 : ATH Char Expansion at 800 Degree Celsius

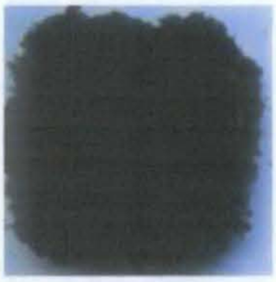
SAMPLE	TEMPERATURE	NOTE	PICTURE
FS 1.1	500 DEGREE CELCIUS	NOT DETACHABLE	
FS 2.1		NOT DETACHABLE	
FS 3.1		NOT DETACHABLE	
FS 4.1		NOT DETACHABLE	
FS 5.1		NOT DETACHABLE	

Figure 10 : FS Char Expansion at 500 Degree Celsius

SAMPLE	TEMPERATURE	NOTE	PICTURE
FS 1.2	800 DEGREE CELCIUS	DETACHABLE	
FS 2.2		DETACHABLE	
FS 3.2		DETACHABLE	
FS 4.2		NOT DETACHABLE	
FS 5.2		NOT DETACHABLE	

Figure 11 : FS Char Expansion at 800 Degree Celsius

4.2.2 Furnace tests data for ATH and FS Substrate

TEMPERATURE	SAMPLE	THICKNESS		EXPANSION
		BEFORE	AFTER	
500 CELCIUS	0.1	0.447	2.05	6.40
	ATH 1.1	0.446	2.5	7.94
	ATH 2.1	0.45	3	9.50
	ATH 3.1	0.4	2.1	7.80
	ATH 4.1	0.442	3.05	9.93
	ATH 5.1	0.443	3.1	10.07

Table 2 : Char expansion of ATH substrates exposed to 500 degree Celsius

TEMPERATURE	SAMPLE	THICKNESS		EXPANSION
		BEFORE	AFTER	
800 CELCIUS	0.2	0.285	1.95	6.84
	ATH 1.2	0.2475	1.85	7.47
	ATH 2.2	0.27	1.95	7.22
	ATH 3.2	0.335	2.45	7.31
	ATH 4.2	0.3525	2.45	6.95
	ATH 5.2	0.2825	2.35	8.32

Table 3 : Char expansion of ATH substrates exposed to 800 degree Celsius

TEMPERATURE	SAMPLE	THICKNESS		EXPANSION
		BEFORE	AFTER	
500 CELCIUS	FS 1.1	0.51	2.6	6.81
	FS 2.1	0.497	2.4	6.48
	FS 3.1	0.502	2.78	7.47
	FS 4.1	0.501	2.4	6.41
	FS 5.1	0.51	2.3	5.97

Table 4 : Char expansion of FS substrates exposed to 500 degree Celsius

TEMPERATURE	SAMPLE	THICKNESS		EXPANSION
		BEFORE	AFTER	
800 CELCIUS	FS 1.2	0.610	2.82	5.80
	FS 2.2	0.500	2.34	6.26
	FS 3.2	0.510	2.2	5.69
	FS 4.2	0.502	2.4	6.39
	FS 5.2	0.504	2.9	7.77

Table 5 : Char expansion of FS substrates exposed to 800 degree Celsius

4.2.3 Heat Shielding Results for ATH Substrate

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 0	16.36	29	NOT DETACHABLE	
	16.46	138		
	16.56	134		
	17.06	117		
	17.16	130		
	17.26	142		
	17.36	148		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 1	12.46	27	NOT DETACHABLE	
	12.56	105		
	1.06	101		
	1.16	104		
	1.26	121		
	1.36	114		
	1.46	111		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 2	14.28	27	DETACHABLE	
	14.38	133		
	14.48	137		
	14.58	158		
	15.08	163		
	15.18	183		
	15.28	264		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 3	10.42	27	NOT DETACHABLE	
	10.52	129		
	11.02	127		
	11.12	145		
	11.22	155		
	11.32	158		
	11.42	166		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 4	12.07	35	DETACHABLE	
	12.17	154		
	12.27	144		
	12.37	145		
	12.47	142		
	12.57	137		
	1.07	129		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
ATH 5	15.03	30	DETACHABLE	
	15.13	102		
	15.23	99		
	15.33	107		
	15.43	106		
	15.53	104		
	16.03	101		

Figure 12 : Heat Shielding Results for ATH Substrate

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
FS 1	15.17	34	NOT DETACHABLE	
	15.27	129		
	15.37	133		
	15.47	123		
	15.57	123		
	16.07	115		
	16.17	113		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
FS 2	14.20	32	NOT DETACHABLE	
	14.30	144		
	14.40	145		
	14.50	142		
	15.00	144		
	15.10	148		
	15.20	141		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
FS 3	15.38	34	NOT DETACHABLE	
	15.48	139		
	15.58	134		
	16.08	138		
	16.18	136		
	16.28	140		
	16.38	130		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
FS 4	12.51	30	NOT DETACHABLE	
	13.01	106		
	13.11	112		
	13.21	109		
	13.31	108		
	13.41	109		
	13.51	118		

SAMPLE	TIME	TEMPERATURE	NOTE	PICTURE
FS 5	15.03	36	DETACHABLE	
	15.13	130		
	15.23	126		
	15.33	120		
	15.43	114		
	15.53	116		
	16.03	112		

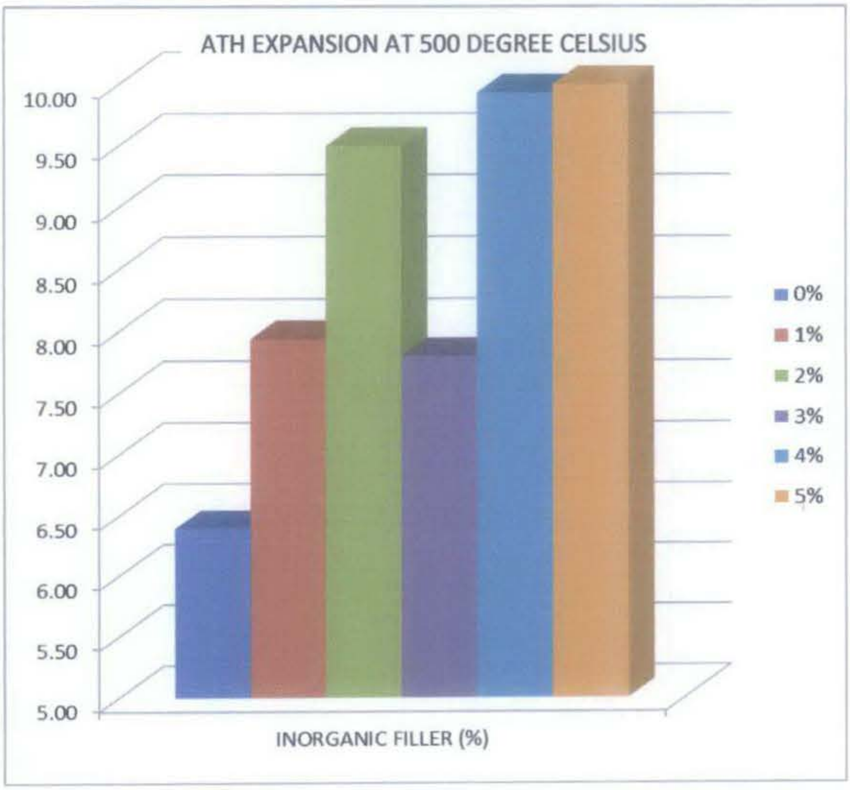
Figure 13 : Heat Shielding Results for FS Substrate

4.2.4 Summary of Heat Shielding Effect Data

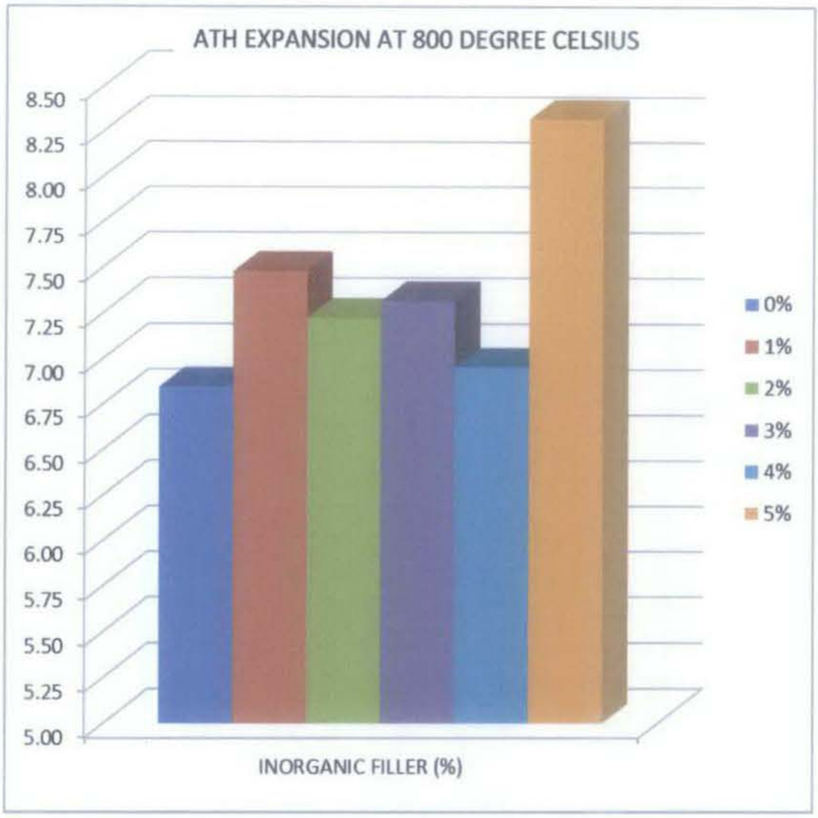
	SAMPLE	FINAL TEMPERATURE
HEAT SHIELDING	0	148'C
	ATH 1	111'C
	ATH 2	264'C
	ATH 3	166'C
	ATH 4	129'C
	ATH 5	101'C
	FS 1	113'C
	FS 2	141'C
	FS 3	130'C
	FS 4	108'C
	FS 5	112'C

Table 6 : Heat Shielding Effect Data

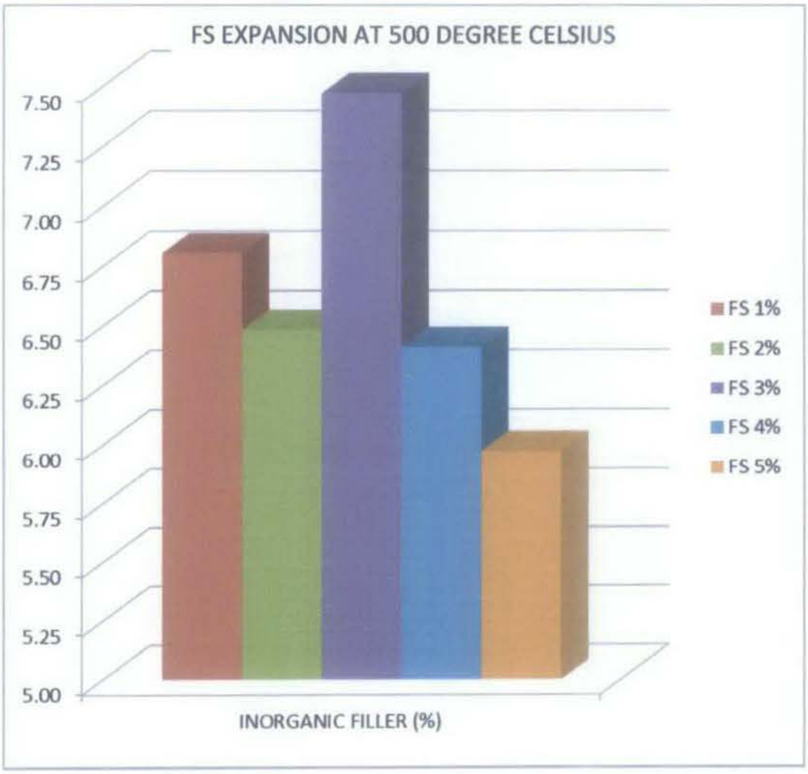
4.2.5 Graphical View of Furnace Tests Result



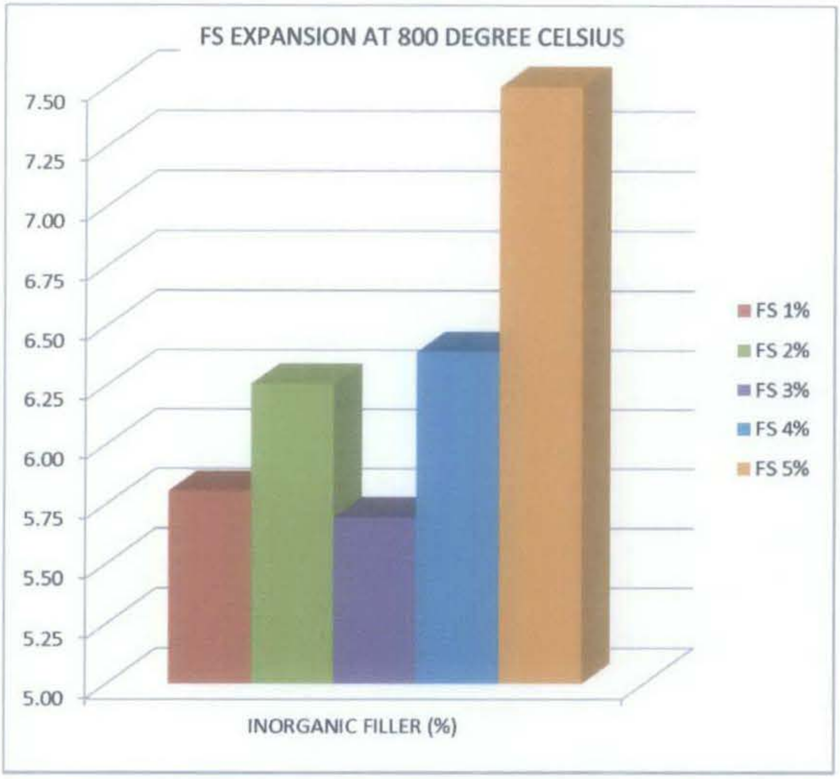
Graph 1 : Furnace test results for ATH at 500 Degree Celsius



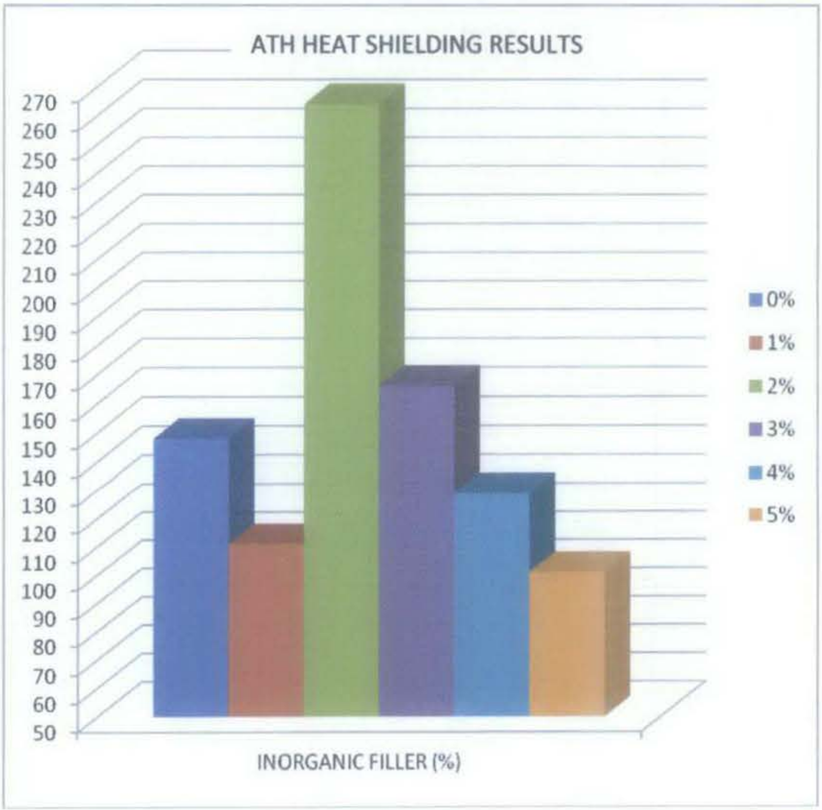
Graph 2 : Furnace test results for ATH at 800 Degree Celsius



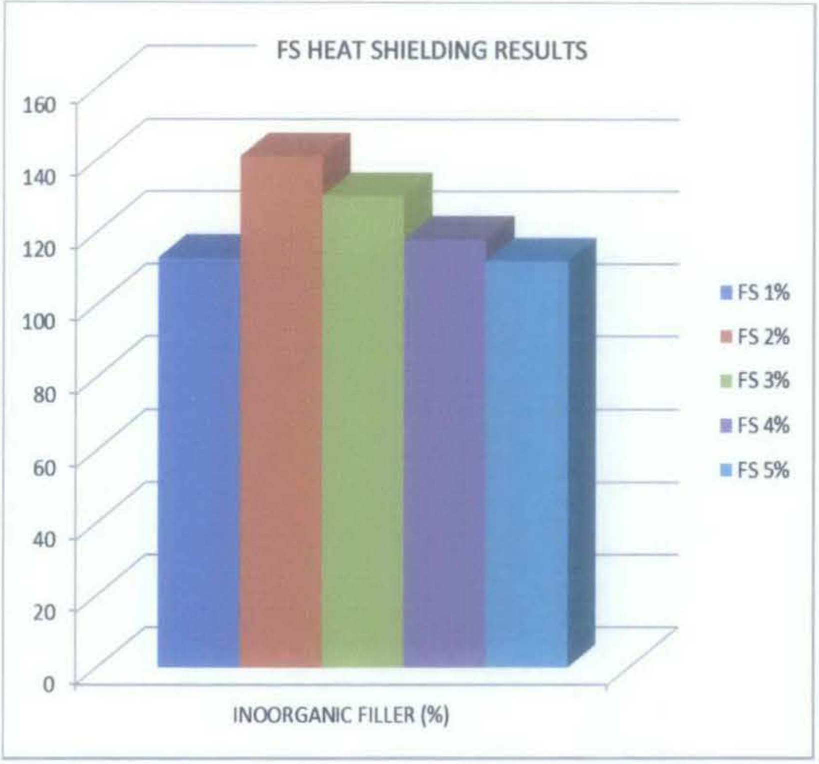
Graph 3 : Furnace test results for FS at 500 Degree Celsius



Graph 4 : Furnace test results for FS at 800 Degree Celsius

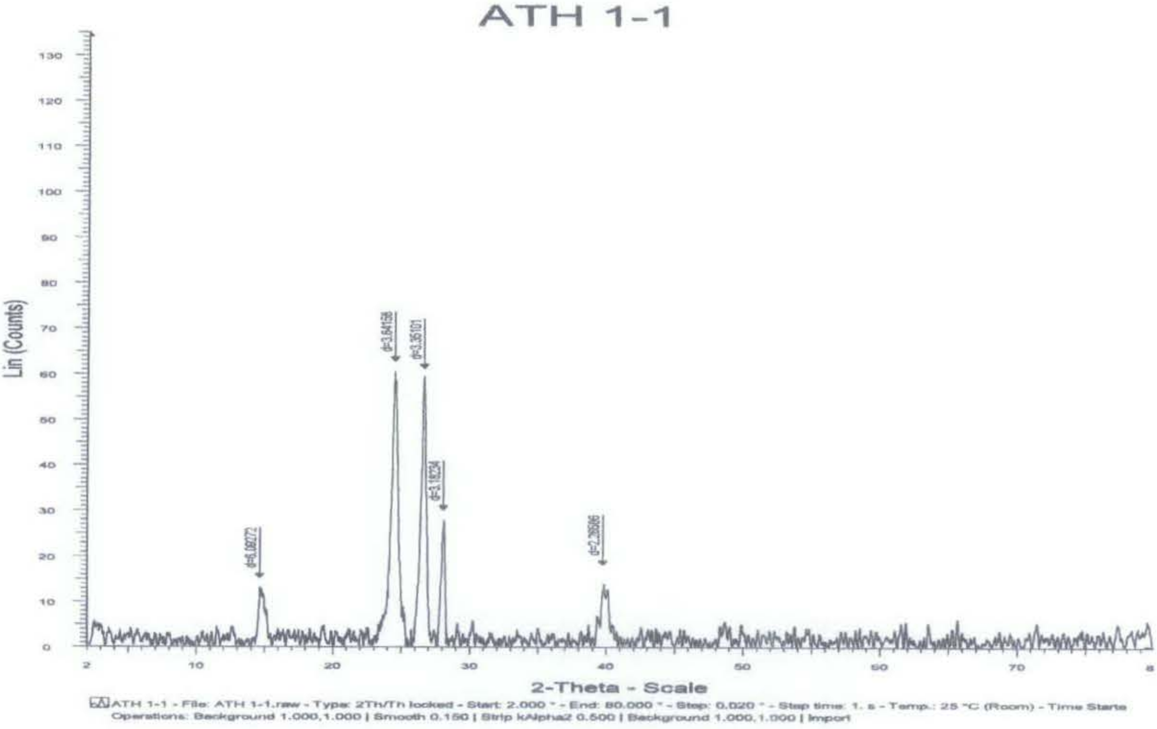


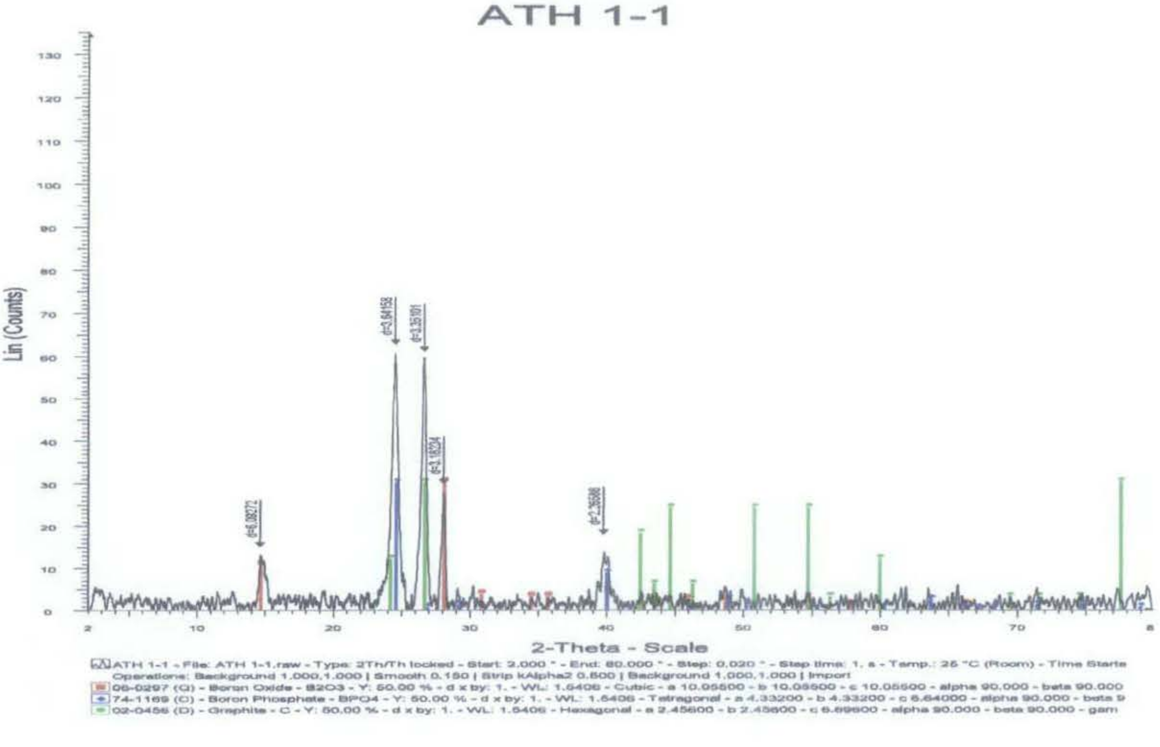
Graph 5 : Heat Shielding Temperature results for ATH Substrate



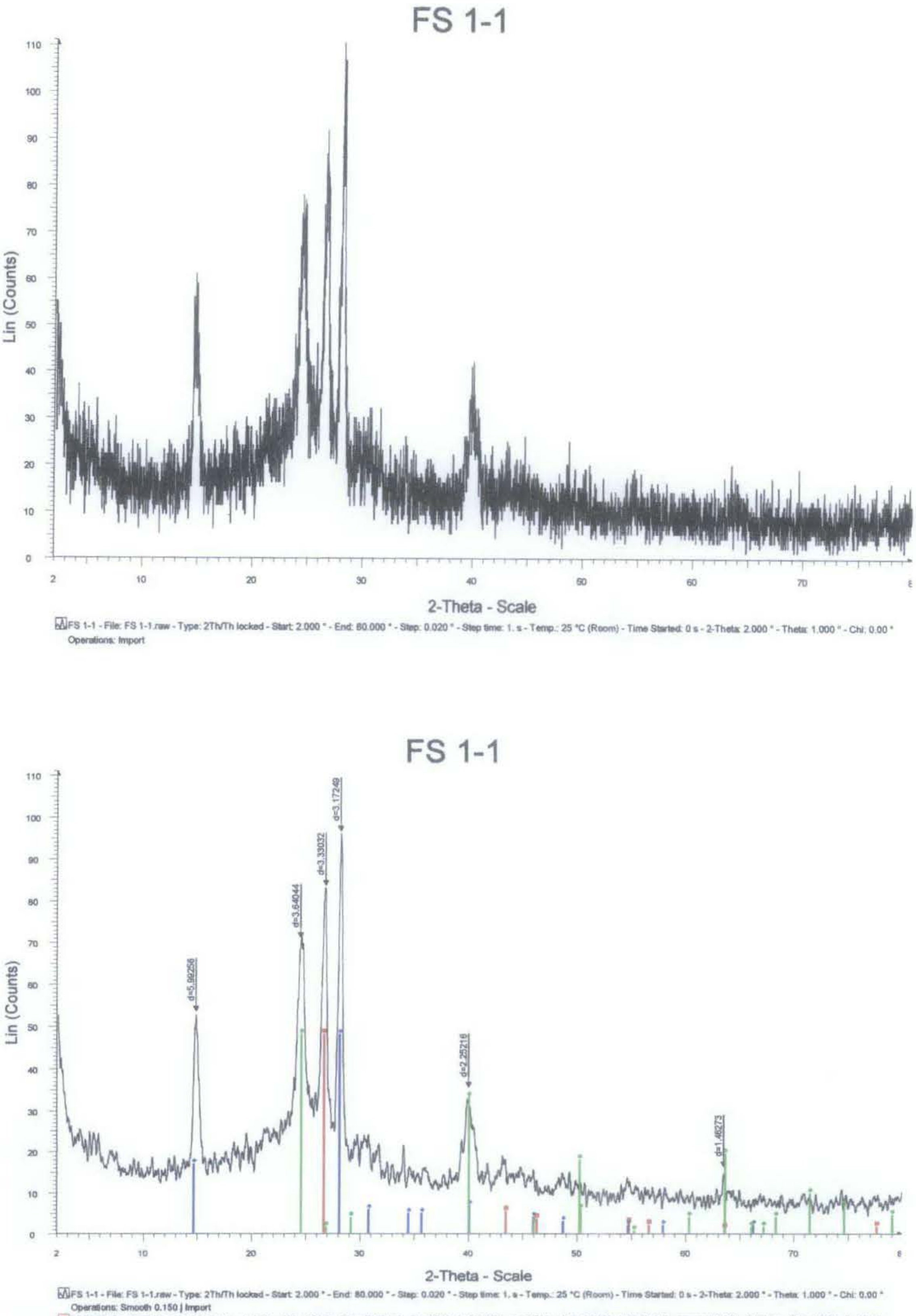
Graph 6 : Heat Shielding Temperature results for FS Substrate

4.2.6 XRD Results for ATH and FS Substrate at 500'C

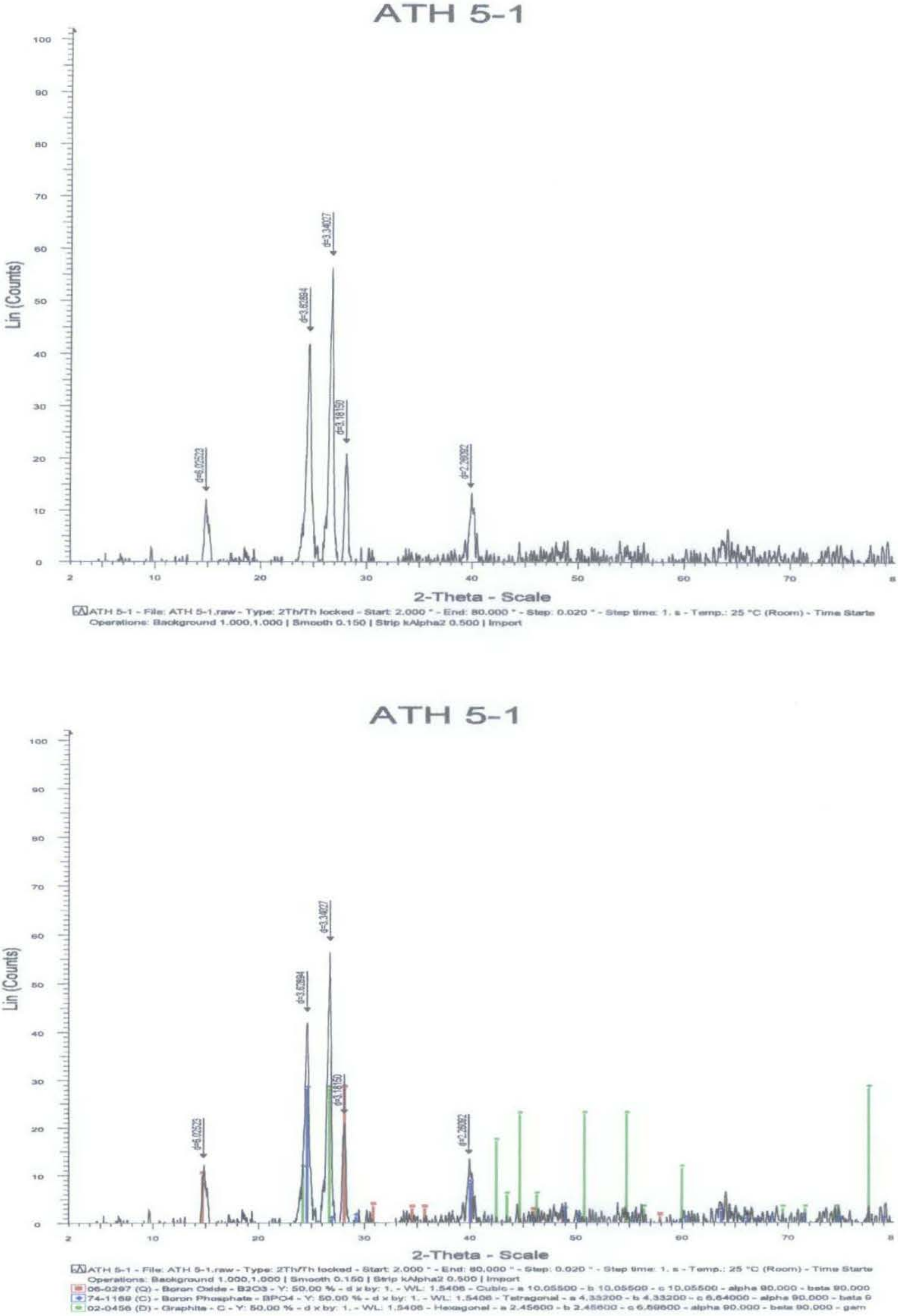




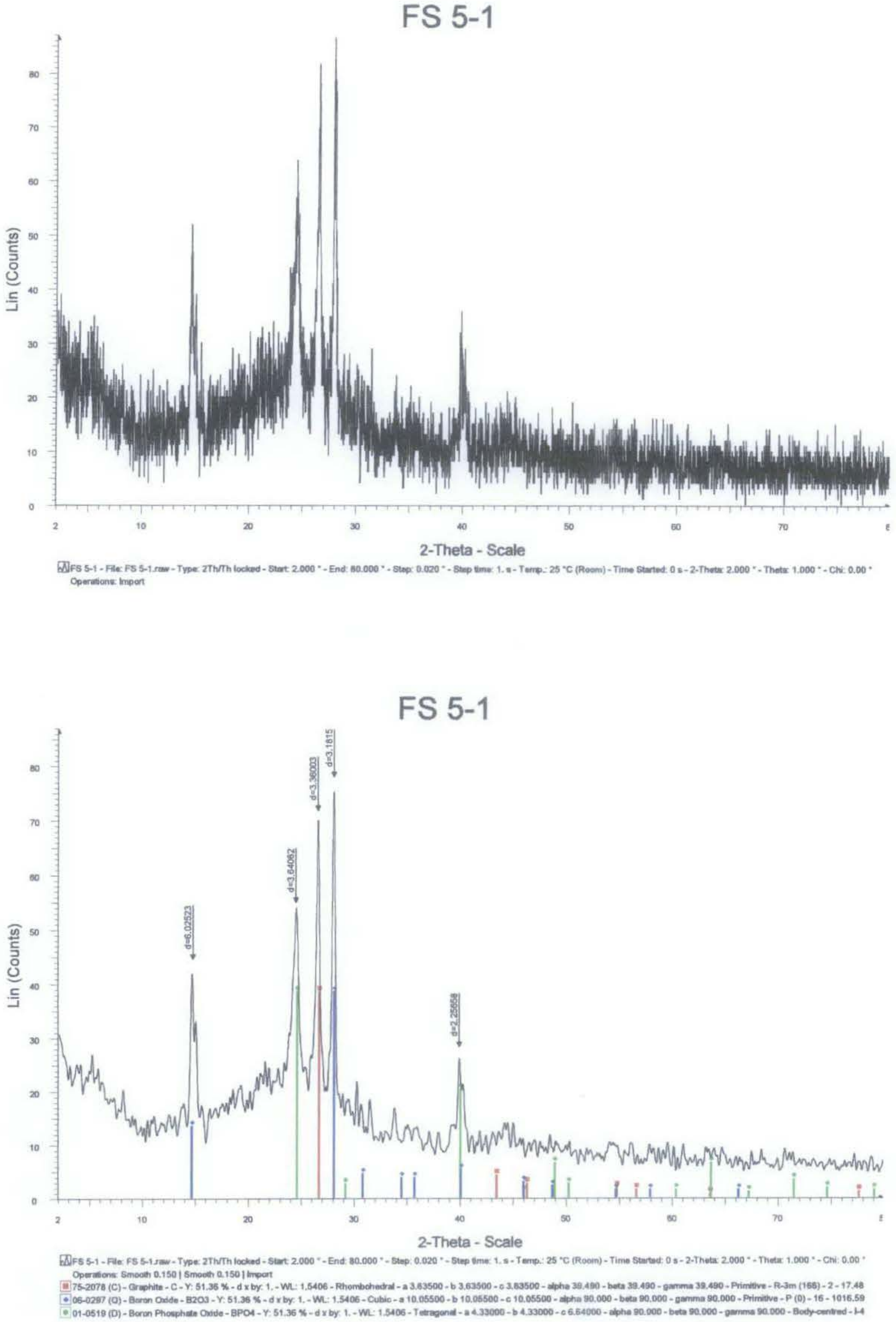
Graph 7 : XRD result for ATH 1% at 500 Degree Celsius



Graph 8 : XRD result for FS 1% at 500 Degree Celsius

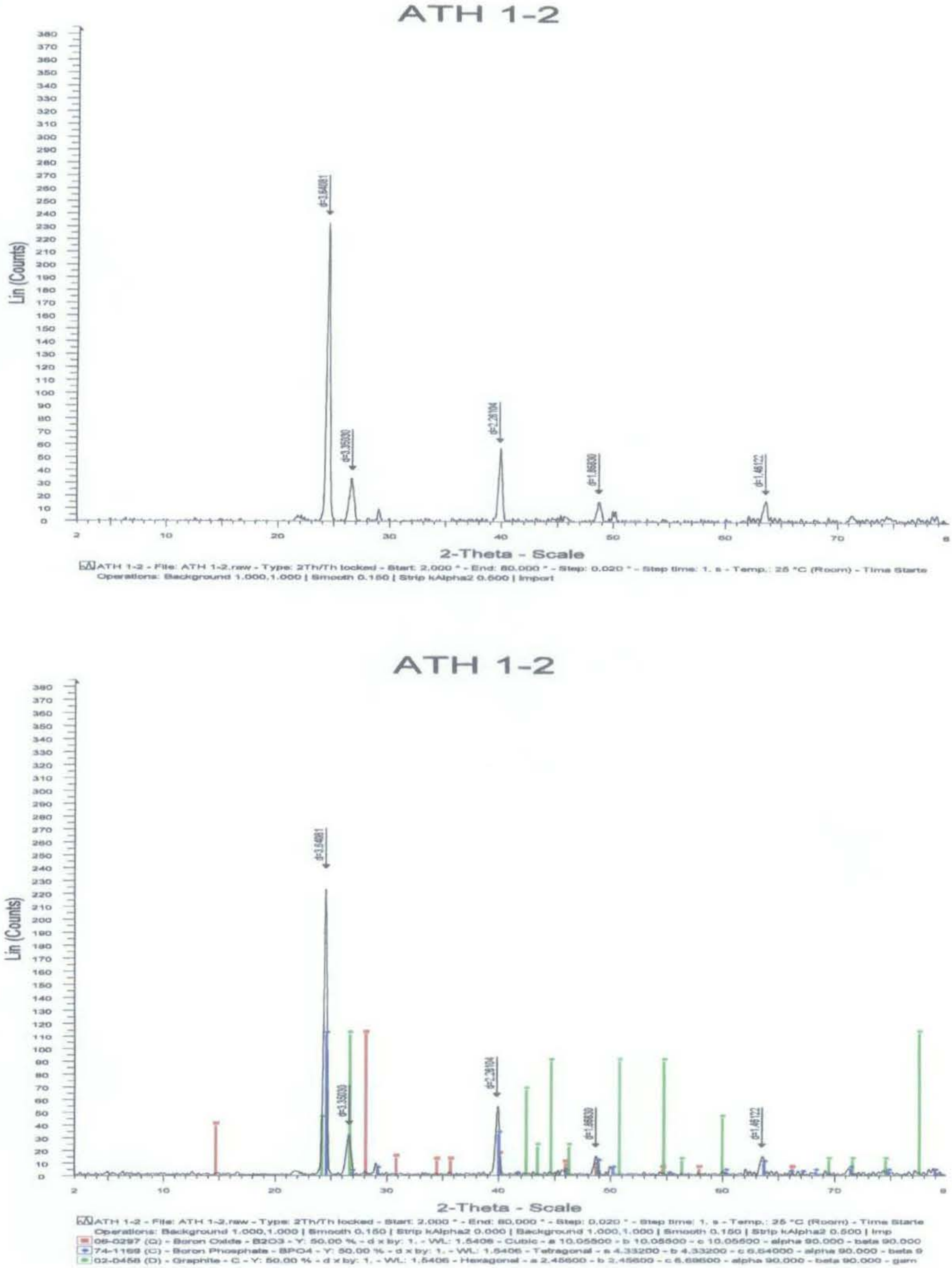


Graph 9 : XRD result for ATH 5% at 500 Degree Celsius

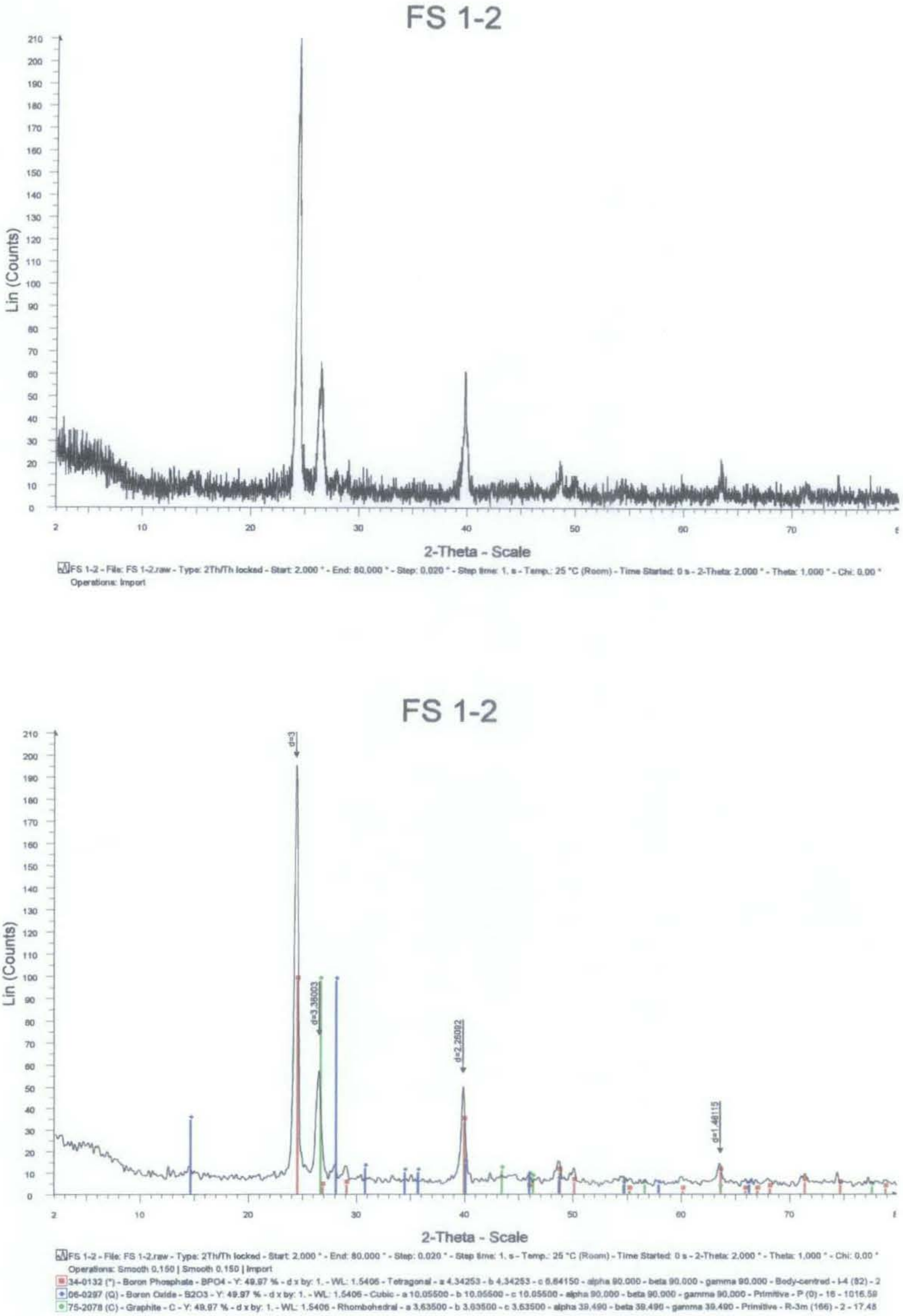


Graph 10 : XRD result for FS 5% at 500 Degree Celsius

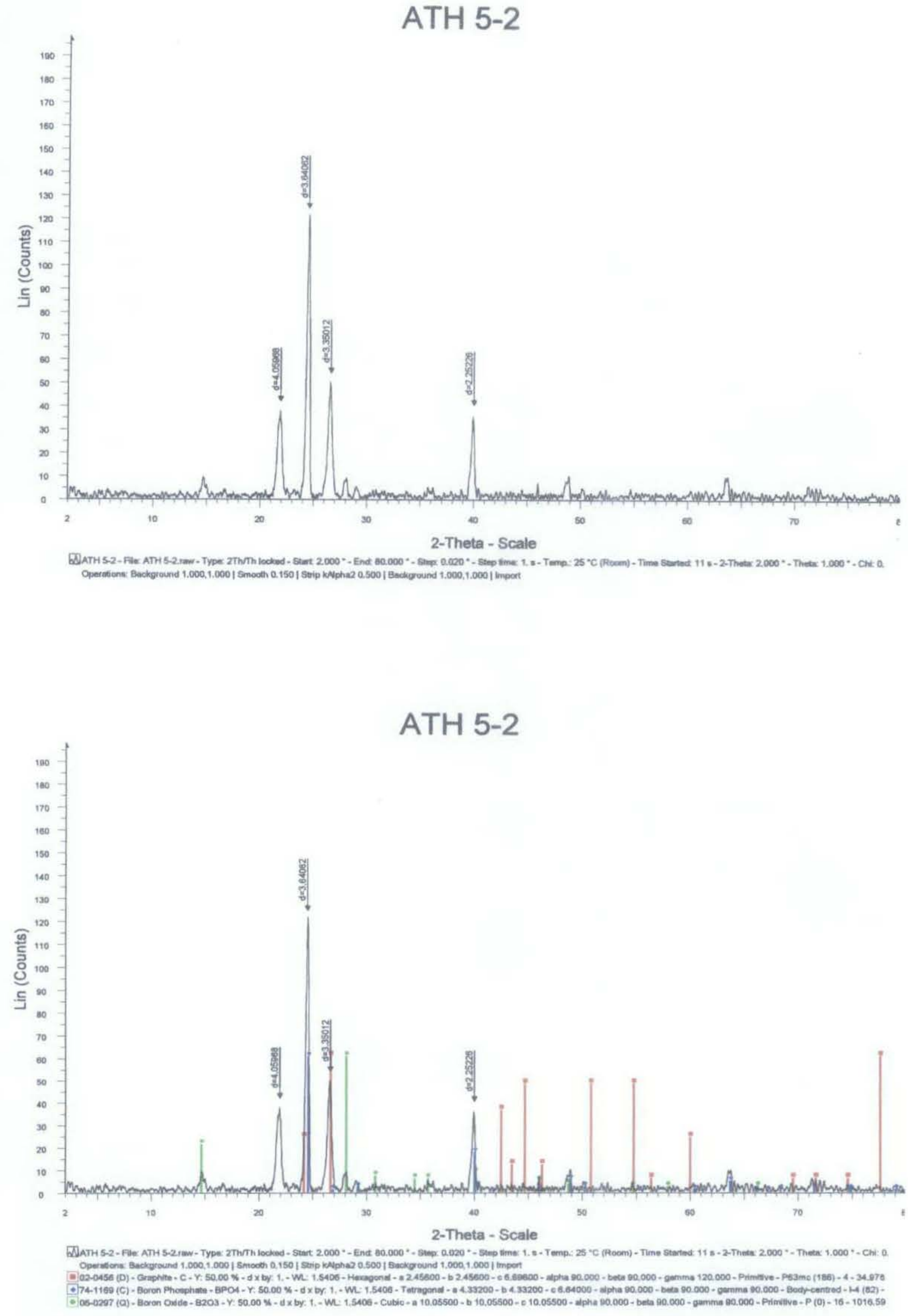
4.2.7 XRD Results for ATH and FS Substrate at 800'C



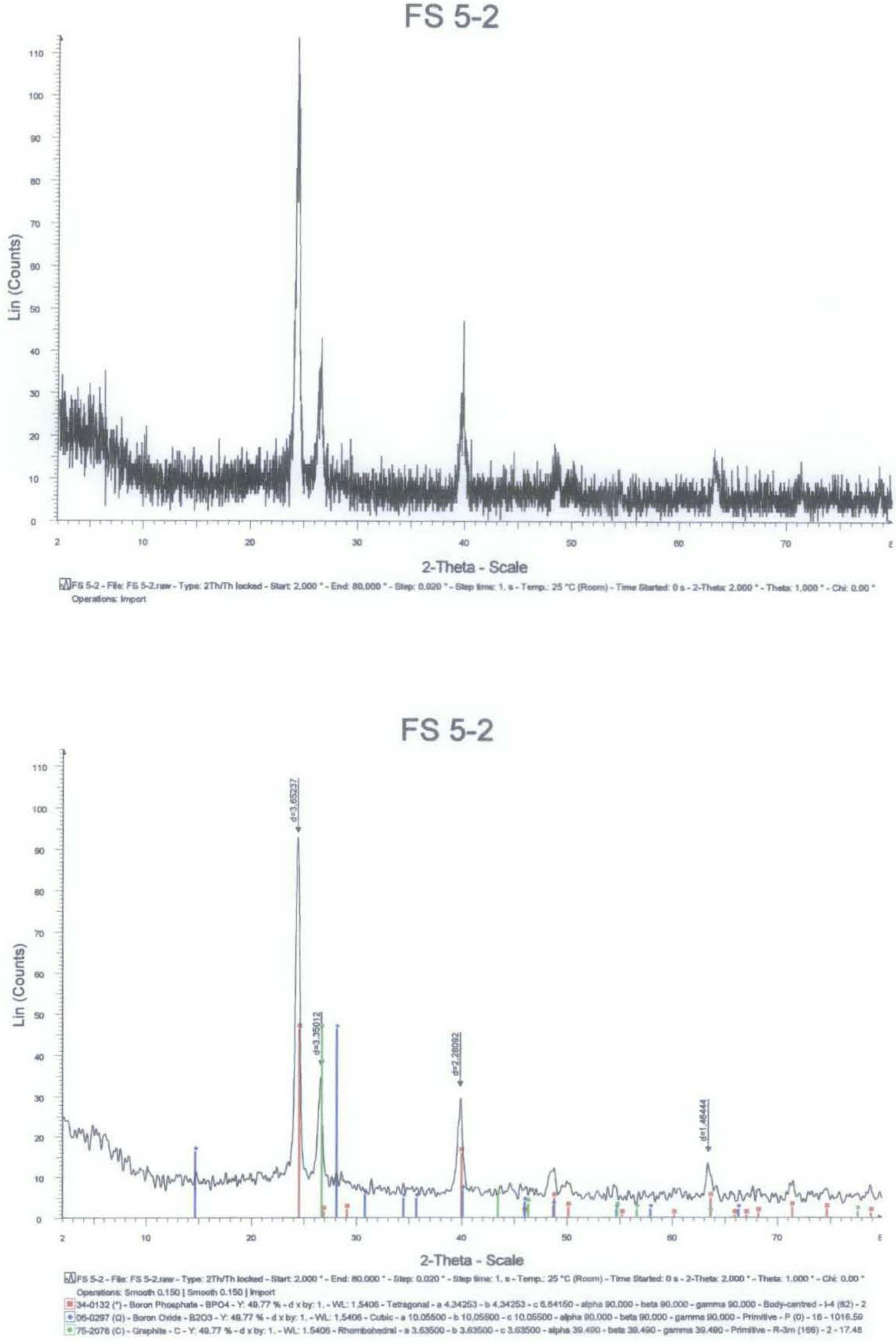
Graph 11 : XRD result for ATH 1% at 800 Degree Celsius



Graph 12 : XRD result for FS 1% at 800 Degree Celsius

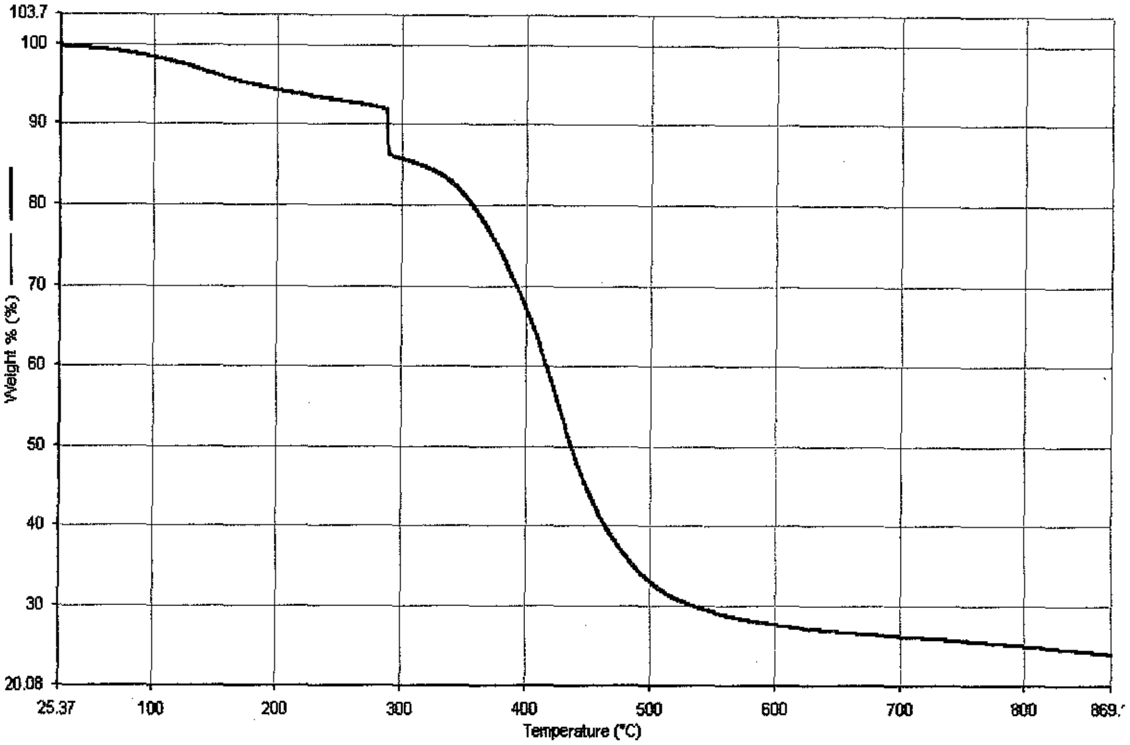


Graph 13 : XRD result for ATH 5% at 800 Degree Celsius

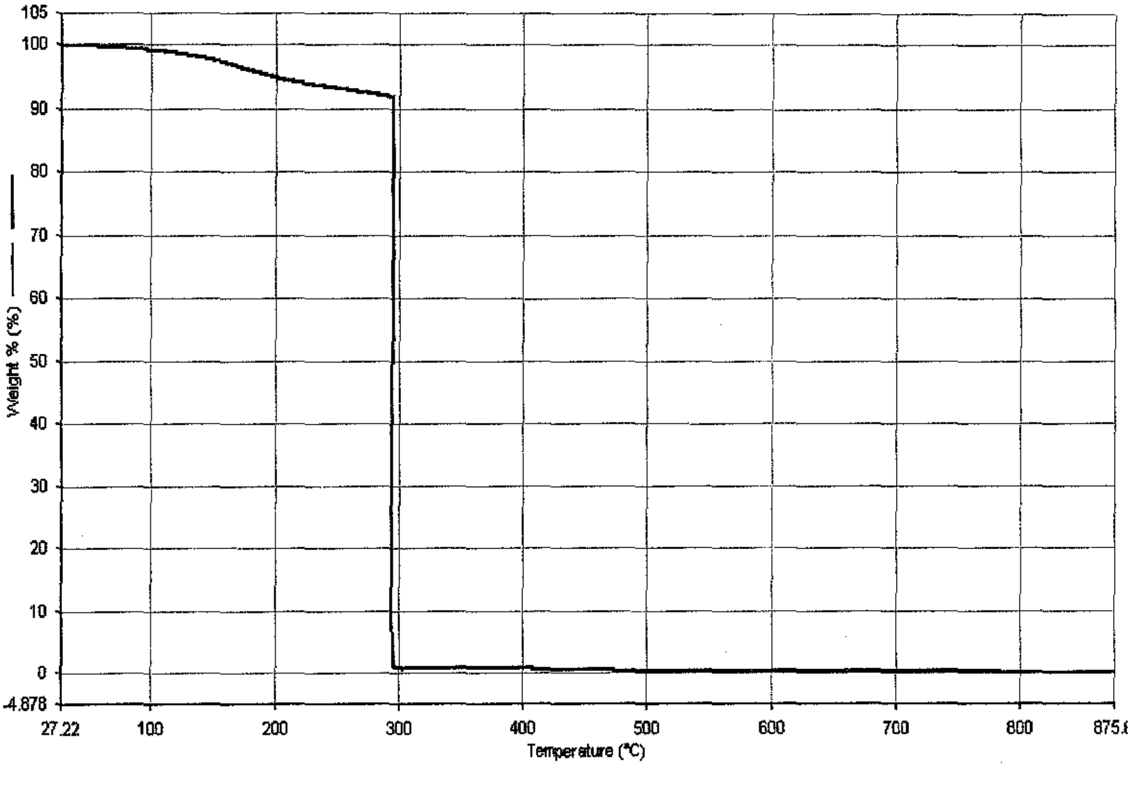


Graph 14 : XRD result for FS 5% at 800 Degree Celsius

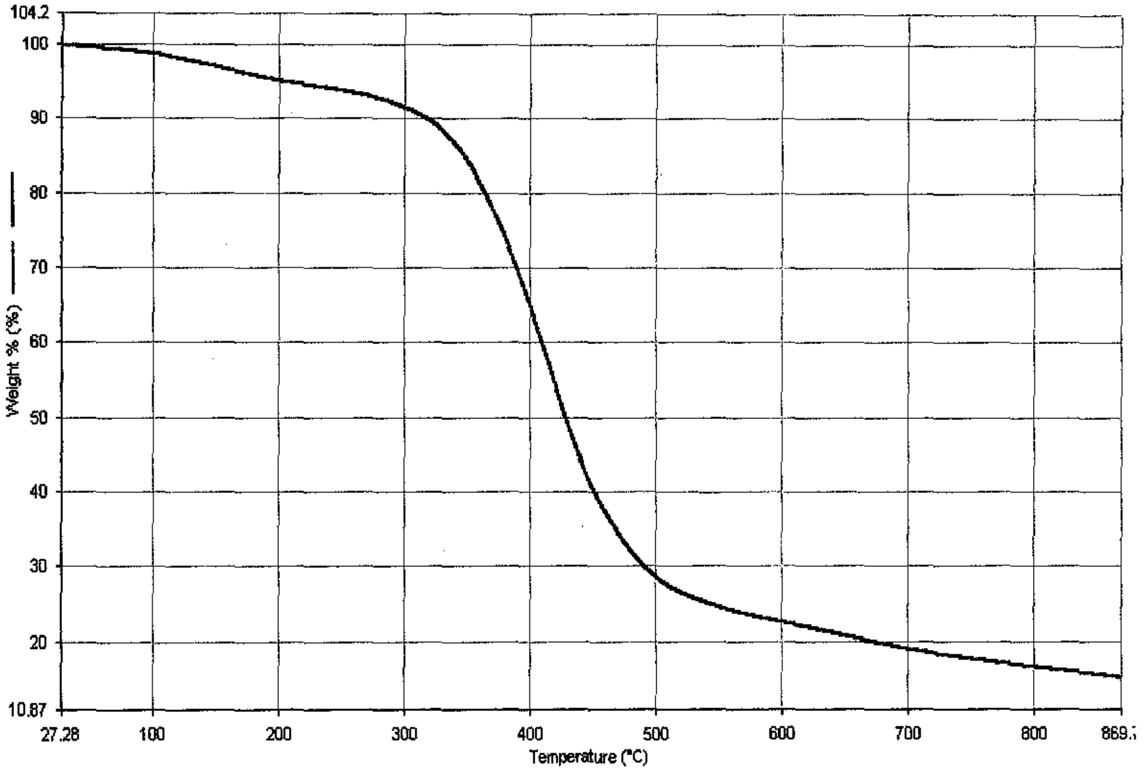
4.2.8 TGA Results for ATH and FS Substrates



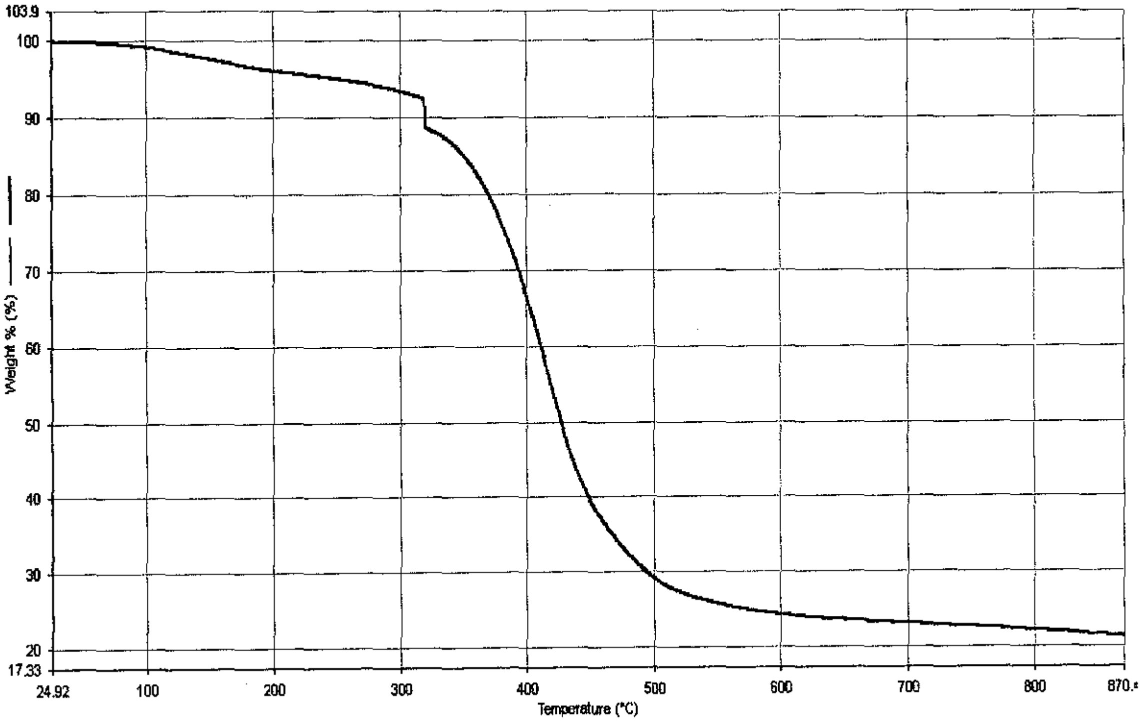
Graph 15 : TGA result for ATH 1%



Graph 16 : TGA result for ATH 5%



Graph 17 : TGA result for FS 1%



Graph 18 : TGA result for FS 5%

4.3 Discussion

4.3.1 Expansion Graphical Interpretation for Furnace Test

As for the furnace test for ATH samples, the result for the experiment at 500 degree Celsius shows that the maximum expansion is showed by substrate with 5% ATH. ATH 4% shows the second highest in char expansion. It can be seen from graph that the increase percentage of ATH fillers impacts in char expansion. Furnace tests were also conducted for ATH substrates at 800 degree Celsius and from graphical plotted results, we can interpret that the expansion is maximum with 5% ATH filler.

NOTE : Notice that the expansion comparison for both 500 and 800 Degree Celsius differ in maximum expansion. The 500 degree shows better expansion as per compared to 800 degree for ATH substrate which is 10.07 times compared to 8.32 times. This happens because at 800 degree Celsius the substrate have fully degraded and oxidized. The substrate is extremely brittle and dust like. The bonding strength at 500 degree Celsius is far better compared to substrate tested at 800 degree Celsius.

Similarly for FS substrates at 500 degree Celsius, maximum expansion is showed at 3% filler which expands up to 7.47 times its original size followed by 1% filler that expands up to 6.81 times its original size. FS substrates tested at 800 degree Celsius showed its highest expansion at 5% filler expanding 7.77 times its original size followed by 4% that expands 6.39 times its original size.

NOTE : Notice that the expansion comparison for both 500 and 800 Degree Celsius differ in maximum expansion. The 800 degree shows better expansion as per compared to 500 degree for FS substrate which is 7.77 times compared to 7.47 times. This behavior is closely related to the behavior of Fumed Silica itself of where the melting temperature of fumed silica is higher compared to Aluminium Tri-Hydrate. It is also observed when there is an increase in FS% the char becomes lesser brittle and it gives better bonding.

At an overall view the best char expansion is showed by ATH substrate which is at 5% at 500 degree Celsius whereby the substrate expands up to 10.07 times its original size. Thus, making this the most ideal formulation for char expansion as per compared to Fumed Silica as filler.

4.3.2 Heat Shielding Effect

The second part of the experiment consist of fire/Bunsen burning experiment. The experiment is left exposed to a burner for an hour and the end temperature is recorded every 10 minutes from the temperature gauge reader attached at the back of the steel.

Figure 14 : Fire Test with Thermologger used for Experiment

From graphical plotted data, substrate with 5% ATH showed the best heat shielding results as per compared to other percentage of ATH fillers recording a final back temperature of 101 degree Celsius. FS substrate with 4% filler showed best heat shielding effect within its range recording final back temperature of 112 degree Celsius. After an overall graphical view analyzing, it can be stated that ATH shows best heat shielding effect amongst both the filler used with the lowest back temperature recorded.

NOTE : Sample ATH 0%, 2% and 3% showed very high temperature (low heat shielding effect), this is because the fire had penetrated the coating and touched the steel therefore the temperature increased rapidly.

4.3.3 XRD Result

There were 2 samples each from 500 degree and 800 degree Celsius from both the fillers used (ATH &FS) sent for this test namely sample :

- ATH 1-1 (500 Degree Celsius)
- ATH 1-2 (800 Degree Celsius)
- FS 1-1 (500 Degree Celsius)
- FS 1-2 (800 Degree Celsius)
- ATH 5-1 (500 Degree Celsius)
- ATH 5-2 (800 Degree Celsius)
- FS 5-1 (500 Degree Celsius)
- FS 5-2 (800 Degree Celsius)

From Graphical interpretation of the XRD result we observe that the ATH substrates under 500 Degree Celsius contain obvious elements of :-

- Boron Oxide
- Boron Phosphate
- Graphite

From Graphical interpretation of the XRD result we observe that the ATH substrates under 800 Degree Celsius contain obvious elements of :-

- Boron Phosphate
- Graphite

NOTE : The result from 800 degree Celsius showed extremely low content of Boron Oxide as per compared to the 500 Degree Celsius results.

4.3.4 TGA Result

Generally, In the range of 0-200°C the weight loss is usually because of the removal of hydroxide group from polyamide hardener . In the range of 200-350°C the expandable graphite oxidized by releasing CO_2 and H_2O. In the range of 350-500°C the weight loss is due to the decomposition of APP to release gas of NH_3 and H_2O. In the range of 500-700°C the weight loss further contributed to the releasing of phosphoric acid, poly phosphoric acid and ploy metaphosphoric acid. At 800°C the weight is due to, poly metaphosphoric acid is vaporized with APP decomposing.

Note : As from the TGA results obtained it can be observed that there are different temperatures that influence on degradation of the various ingredients used. It can be seen from graph also that when there is increased in % of filler, the residual weight also increases proportionally. This explains the incremental in scale of the XRD results as well as also the

CHAPTER 5 : CONCLUSION & RECOMMENDATION

5.1 Conclusion

Intumescent coating consists of 3 main components which are acid source, carbon source and blowing agent. The right amount of those 3 components results in a good sample of intumescent coating. Other than that, using inorganic fillers to improve the properties of coating are very useful as every inorganic filler is unique in its own way and shows different results when tested. In this particular project, there are two inorganic fillers used, Fumed Silica and Aluminium Tri-Hydrate. These two inorganic fillers have different properties and are tested in expansion of char as well as the heat shielding effects. Having completed the entire project, it is obvious that ATH filler showed the best results for both heat shielding and char expansion.

As for this project, there are three parts that are majorly concentrated..

- One, the preparation of formulation of Fumed Silica samples as well as mixing grinding and coating it onto steel plates.

- Secondly the applied coating for both the different inorganic fillers (Fumed Silica & Aluminium Tri-Hydrate) are experimented with exposing it to fire using Bunsen burner and applying heat using furnace. This test is for the purpose of testing the heat shielding as well as the char expansion for both the inorganic fillers used.

- Finally, the experimented specimens are thoroughly studied with the aid of some technological equipments for better view and data collection for plotting of graph or understanding the pattern of effects and cause.

Technological visual equipments and its functions :-

Thermo gravimetric analysis - is a type of testing performed on samples that determines changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues.

X-ray scattering techniques - are a family of non-destructive analytical techniques which reveal information about the crystal structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy.

5.2 Recommendations

There are various angles to look at this project for room of improvement for expansion and continuation. The project can be further experimented by using different inorganic fillers that might help increase the strength of char and heat shielding effects. Improvisation on increasing the heat shielding effect so that it could be used not just onto steel products but also other metal products that degrades at different temperature.

Although steel structures are commonly used for construction of most industries in particular offshore structures, once this experimental study achieves its objective, a proper study in detail can be carried out as a new way of protecting other metal related materials that is able to withstand higher percentage of heat as well as the char expansion. Various tests can be conducted and this particular study will be very beneficial to not only the Oil and gas industries but also to other industries which can help minimize the risk and work hazard as well as increasing the safety of workplace and workers.

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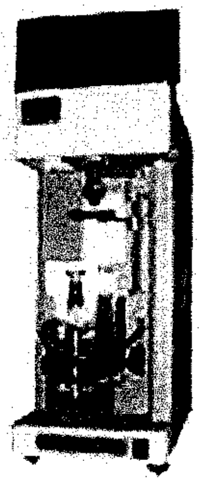
APPENDICES

*THERMO*OGRAVIMETRIC ANALYSIS

Thermogravimetric analysis or **thermal gravimetric analysis (TGA)** is a type of testing performed on samples that determines changes in weight in relation to change in temperature. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. As many weight loss curves look similar, the weight loss curve may require transformation before result>s may be interpreted. A derivative weight loss curve can identify the point where weight loss is most apparent. Again, interpretation is limited without further modifications and deconvolution of the overlapping peaks may be required.

TGA is commonly employed in research and testing to determine characteristics of materials such as polymers, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation.

Simultaneous TGA-DTA/DSC measures both heat flow and weight changes (TGA) in a material as a function of temperature or time in a controlled atmosphere. Simultaneous measurement of these two material properties not only improves productivity but also simplifies interpretation of the results. The complementary information obtained allows differentiation between endothermic and exothermic events with no associated weight loss (e.g. melting and crystallization) and those that involve a weight loss (e.g. degradation).

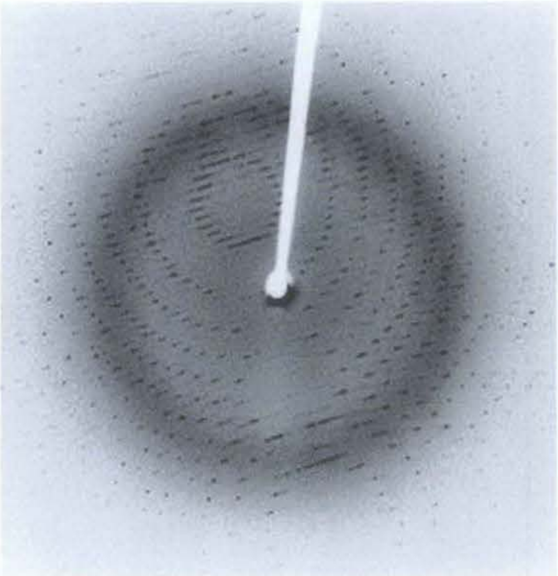


A typical TGA system

X-RAY DIFFRACTION TECHNIQUES

X-ray diffraction (XRD) yields the atomic structure of materials and is based on the elastic scattering of X-rays from the electron clouds of the individual atoms in the system. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction.

- Single-crystal X-ray diffraction is a technique used to solve the complete structure of crystalline materials, ranging from simple inorganic solids to complex macromolecules, such as proteins.
- Thin film diffraction and grazing incidence X-ray diffraction may be used to characterize the crystallographic structure and preferred orientation of substrate-anchored thin films.
- High-resolution X-ray diffraction is used to characterize thickness, crystallographic structure, and strain in thin epitaxial films. It employs parallel-beam optics.
- X-ray pole figure analysis enables one to analyze and determine the distribution of crystalline orientations within a crystalline thin-film sample.
- X-ray rocking curve analysis is used to quantify grain size and mosaic spread in crystalline materials.



This is an X-ray diffraction pattern formed when X-rays are focused on a crystalline material, in this case a protein. Each dot, called a reflection, forms from the coherent interference of scattered X-rays passing through the crystal.