Study the Effect of Temperature on Crack Propagation in Ceramics

By

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirements for the
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Approved by,

____
(AP. Dr. Othman Mamat)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
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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.

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(NUZUL HAZWANI BT MOHAMAD HANAFI)

ABSTRACT

The failure of most brittle components can usually be traced to the presence of flaws either within the component or, more frequently, on the surface of the component. Generally, the internal flaws are usually a result of the manufacturing route whereas surface flaws result from component machining, handling, and deployment. Ceramic materials are being used for high temperature application and structural applications. When the ceramic material is exposed to high temperature, it could change the behavior of the ceramic material. The aim of this study was to test the hypothesis that the crack of ceramic specimens will be affected or propagates by increasing of the temperature. The scope of work for this project is to characterize crack of ceramic specimens, measure them and fire at high temperature at different holding time by using the chamber furnace. The specimens been fired at temperature ranges from 800°C - 1400°C. The crack propagation length was measured by using Vernier caliper. The X-ray Fluorescence analysis was performed to support the experiment with the explanations from elemental field. From the experiment done, the crack propagates when the specimens were fired. As the temperature increase, the length of crack is increased.

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Nuzul Hazwani Mohamad Hanafi.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The word *ceramic* comes from the Greek word κεραμικός (*keramikos*) meaning pottery, which is said to derive from the Indo-European word *ker*, meaning heat. The earliest ceramics were pottery objects made from clay, either by itself or mixed with other material [1]. Ceramics can be defined as heat-resistant, nonmetallic, inorganic solids that are made up of compounds formed from metallic and nonmetallic elements. Although different types of ceramics can have very different properties, in general ceramics are corrosion-resistant and hard, but brittle. Most ceramics are also good insulators and can withstand high temperatures [1].

The two main categories of ceramics are traditional and advanced. Traditional ceramics include objects made of clay and cements that have been hardened by heating at high temperatures. Traditional ceramics are used in dishes, crockery, flowerpots, and roof and wall tiles. Advanced ceramics include carbides, oxides, nitrides other materials, can act as superconductors. Advanced ceramics require modern processing techniques, and the development of these techniques has led to advances in medicine and engineering [1].

Ceramic materials are brittle, hard, and strong in compression, weak in shearing and tension. A material is brittle if it is liable to fracture when subjected to stress. The material has little tendency to deformed (or strain) before fracture. Brittle material is generally applied to materials that fail in tension rather than shear, or when there is little or no evidence of plastic deformation before failure. When a material has reached the limit of its strength, it usually has the option of either deformation or fracture [2].

The brittle fracture of ceramics limits its applications. It occurs due to the unavoidable presence of microscopic flaws (micro-cracks, internal pores, and atmospheric contaminants) that result during cooling from the melt [3]. The flaws lead to crack formation, and crack propagation (perpendicular to the applied stress) along cleavage planes. The flaws cannot be closely controlled in manufacturing; this leads to a large variability (scatter) in the fracture strength of ceramic materials.

There are four general classes of failure causing flaws (listed in the overall from most severe to least severe, based on the most severe members of each classes) are: (1) processing defects, (2) handling / service flaws, (3) machining flaws, and (4) basic micro-structural sources of failure [4]. When ceramic materials are used for structural applications, it is of paramount importance to know of the existing of any faults in the structure of the material. By predicting the failure, it will remain in service resulting in substantial cost savings.

1.2 Problem Statement

Ceramic material is known as hard and brittle material. The brittle fracture of ceramics limits its applications. It occurs due to the unavoidable presence of microscopic flaws. The flaws cannot be closely controlled in manufacturing; this leads to a large variability (scatter) in the fracture strength of ceramic materials. As brittle materials by definition possess low fracture toughness, it follows that high strength brittle material may fail due to the presence of flaw sizes on the micron scale.

Ceramic materials are being used for high temperature application and structural applications. Being exposed to high temperature could change the behavior of the ceramic material. The change of behavior of the material is unpredictable thus can leads to any fault and failure. Therefore, it is important to know the behavior of cracks when exposed to high temperature.

1.3 Objectives

The objective of this study is to test the hypothesis that the crack of ceramic specimens will be affected or propagates by increasing its temperature.

1.4 Scope of Study

The scope of work for this project is to characterize crack of ceramic specimens and measure them before being put at high temperature at different holding time. Ceramic materials always faced a little or no evidence of plastic deformation. Due to the increasing application of porcelain and ceramics, the study regarding ceramics is at paramount importance and valuable to the development of ceramic application in future. This project mainly involves experimental and data gathering and calculation. Cracks length data will directly use for the failure prediction. All of this factor is time consuming and will require a timeline of one year as given.

CHAPTER 2

LITERATURE REVIEW

Through out the stages of this research, there are a few journals, books and websites related to this project. Basically, spot to be highlighted for the study consists of the method for firing the specimen, flexural test and crack measurement.

2.1 The Manufacturing Process of Ceramic Products.

The ceramic mug manufacturing process start with granular powder made up of a base material such as Alumina or Zirconia, mixed with other stabilizers and binders that give each ceramic body its own unique characteristics [5]. The manufacturing process for ceramic mug is shows in **Figure 1**.

Figure 1: Manufacturing Process for Ceramic Mug [5].

The ceramic tile manufacturing process consists of a series of successive stages, which can be summarized as follows:

- Raw materials preparation
- Pressing and drying of the green body
- Firing, with or without glazing
- Additional treatments
- Sorting and packing

Depending on whether the product to be made is glazed or not and whether single fire, twice fire or third fire is involved, the tile will or will not be glazed in a given process, or the order of the glazing and firing stages will be suitably rearranged [6]. The manufacturing process of ceramic is shows in **Figure 2**.

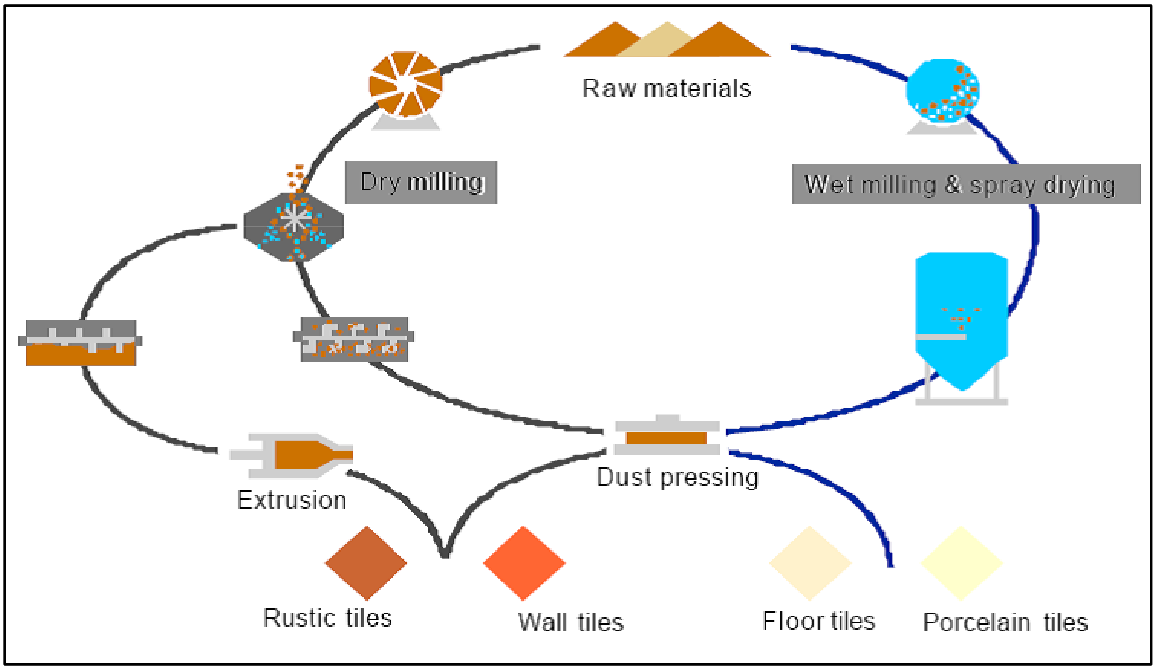

Figure 2: Manufacturing Process of Ceramic Tile [6].

2.2 Residual Stress Acting on Crack Propagation

Cracks usually result from stress in the clay. There is always some stress in clay because it shrinks as it dries, when it is fired and it also expands and contracts during firing. Sometimes the stress is too much for the clay to handle it cracks. Different clays can handle different amounts of stress without cracking.

The shape of the pottery or ceramic may cause the cracking. Sharp corners concentrate stress and are more prone to cracking. Fast drying will tend to cause more stress than slow drying. Over firing pottery or ceramics or firing it multiple times will tend to make it more susceptible to cracking.

Differences in thickness of the pottery or ceramic will also set up stresses, since the thin areas will dry faster than the thick and the stress won't be evenly distributed. This is very common when the base of pottery is thicker or thinner than the walls. Sometimes this will show up as a circular crack around the bottom edge of the pottery.

A thick layer of glaze on the inside of a pottery or ceramic and a thin or no layer on the outside will cause stress which will result in a spiral crack up the sides. If glaze pools on the inside of a pot, tension is created and the pottery or ceramic may crack or split across the base [11].

The strength of the fired ceramic is increased at an accelerating rate with firing temperature. The strength increased is correlated with the development of a glassy bonding phase at temperatures about 800°C, and the amount of bonding phase is increases with temperature. The ceramic thus become stronger as it is fired to higher temperature. When the clay is fired at higher temperature and above, the glassy phase is sufficiently fluid and presents at a high enough volume percentage that the ceramic will deform or slump [10].

2.3 Typical Type of Cracks Found in Ceramic Products

The failure of most brittle components can usually be traced to the presence of flaws either within the component or, more frequently, on the surface of the component. Generally, the internal flaws are usually a result of the manufacturing route whereas surface flaws result from component machining, handling, and deployment. The typical types of cracks found in ceramic products are shivering, crazing, dunting and S-crack.

2.3.1 Shivering

Sometimes a piece of glaze will crack off, normally near a rim or at edges. Some clay may be attached to the glaze piece that cracks off. This occurs because stress has built up between the clay and glaze that can't be absorbed.

It is often caused by over sponging which takes away the fine clay particles and leaves behind the groggier clay particles which are not elastic enough to absorb the stress [11].

2.3.2 Crazing

This is a network of very fine cracks in the glaze. It is caused by a mix-match between the clay and glaze. It often will not show up until the pottery or ceramic is cooled or sometimes even until it has been heated and cooled a few times. Some people believe slow cooling will prevent crazing, but the stress still exists and eventually the crazing will occur [11].

2.3.3 Dunting

This is a special type of crack which occurs from stresses caused during firing and cooling. This stress primarily occurs during two critical points of firing called silica inversions which occur at 573˚ C and 226˚ C. At these inversion points, the structure of the silica molecules rearranges. Most dunting however is caused during cooling. These cracks appear as long, clean, body cracks with sharp edges. If the pottery or ceramic is glazed, the glaze edges are sharp. They can be vertical, horizontal or spiral.

There are three main reasons why cooling dunts occur.

(i) The first reason occurs as the material was cool through the first silica inversion at 1063˚ F. At this inversion the body contracts suddenly. The more silica and quartz in the material, the more contraction. Since different parts of the pottery or ceramic reach this temperature at different times, it doesn't all contract together and that causes stress which can crack. Take for example a tall pot. The top will cool much faster than the bottom, because the bottom has the whole temperature of the kiln shelf keeping it warm. The top will cool faster than the bottom, causing a crack around the bottom wall.

(ii) The second reason occurs as the material was cool through the 439˚ F inversion. A similar thing happens as above. But, pottery and ceramic artists sometimes like to open their kilns at about this temperature to see their pieces and this will make it much worse.

(iii) The third reason of cooling dunt occurs months or even years after firing. Sometimes the pottery or ceramic might split right in half after three months. This is usually the result of thermal shock. In this case the clay and glaze expand at different rates when exposed to temperature variations and this change causes the object to crack. To be more specific, the body contracts more than the glaze. If the glaze is weaker it will shiver. If the clay is weaker the object will crack.

2.3.4 S Cracks

One of the most common cracks found in pottery, is the S crack, which occurs at the bottom of a pot, in the shape of an s, usually on thrown pieces, but can also happen to a poured ceramic. The most important thing to remember is that you should keep the bottom of the pottery or ceramic as dry as possible while throwing or pouring and compress the bottom during throwing and trimming [11].

2.5 Journals Summary

Reviews from the journals related to this project are as in **Table 1**.

Table 1: Summary of the Journals

Journal	Methodology	Conclusion
[7]	1. Fabricate the specimens. 2. Test the bending fixture. 3. Stress and fracture analysis applied to the specimen. 4. Applied different temperature and the procedure number 3 is repeated.	Either bulk ceramic material or the adhesive / glass interface may fail depending on the applied temperature and the notch length. The curved of the crack growth path inside the ceramic can be predicted by fracture mechanics.
[8]	1. Fabricate the specimens and fired below than recommended firing temperatures. 2. Polishing and induced cracks with a Vickers microhardness indenter. 3. Fire the specimens at 30°C more, measure the crack and data recorded.	The crack propagation data indicate that the higher the thermal shock resistance of one of the ceramics -as measured previously by a water-quench technique- may be due to its grater resistance to stress corrosion at the initial stage of crack propagation.
[9]	1. Specimens are prepared. 2. Each group was fractured in water using one of four target stressing rates ranging on a log scale. Group 5 were tested in inert environment (oil). 3. The effect of stressing rate and environment on flexural strength, critical flaw size and fracture being	The flexural strength of the ceramic bars will decrease with slower stressing rates in a water testing environment was verified but their fracture toughness will remain the same. The flexural strength, critical flaw sizes, and the fracture patterns of the ceramic loaded in flexure were governed by the testing

	determined.	environment and stressing rate, but the fracture toughness is a material property, which is independent of the experimental factors in this study.
[10]	1. Specimens are prepared (uniform circular cross section by extrusion) 2. Fire the bars at temperature high enough to achieve high strength by development of a glassy bonding phase. 3. Fire the bars at temperatures: 650, 750, 850, 950 and 1050°C. (Hold the specimen at that temperature for 1.5 hours, then allow furnace to be cool) 4. 3-point bend test is used for fracture toughness testing. Load <1000N used.	The strength of the fired ceramic increased at an accelerating rate with firing temperature. The amount of glassy bonding phase is increases with temperature. When the clay is heated at 1150°C, the glassy phase is sufficiently fluid and present at a high enough volume percentage that the ceramic will deform or slump.

CHAPTER 3

METHODOLOGY

3.1 Experiment Flow

Before proceeding with the study itself, steps were drawn out diagrammatically to clear out on the flow to ensure the study can be completed within the given time (**Figure 3**). A key milestone for the project is attached in Appendix I.

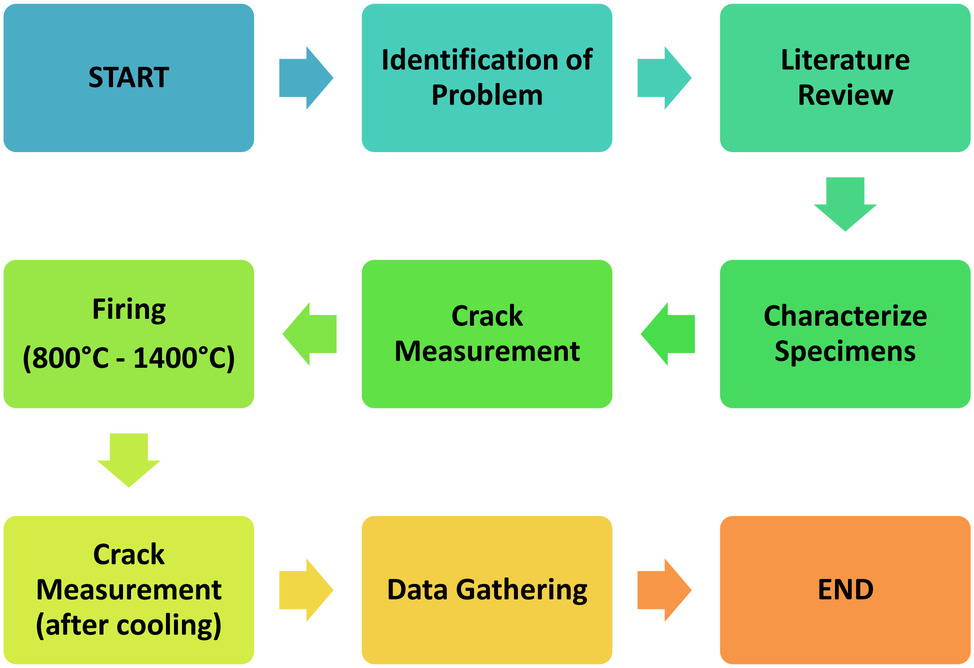

Figure 3: Experiment Flow

Based of the experiment flow shown in **Figure 3**, the crack measurement is done after cooling the specimens at room temperature. The specimens are being fired at temperature range 800°C - 1400°C. Different holding times are set which are one hour and two hours. The holding time is when the furnace heated at the desired temperature, it was hold for one hour or two hour at that temperature before cooling down to room temperature.

3.2 Background Information of the Experiment Specimens

There are five different specimens used in this study which are manufactured from different manufacturer. The types of specimens used are different which is specimen A, B, C, and E are the ceramic mug and specimen D is a heavy duty tile. The background information of the experiment specimens is show in **Table 2**.

Table 2: Background Information of the Specimens

Specimen	Manufacturer	Failure Type	Type of Specimen
A	Air Hitam Pottery	Manufacturing failure	Glazed
B	Air Hitam Pottery	Manufacturing failure	Unglazed
C	Air Hitam Pottery	Manufacturing failure	Unglazed
D	Alpha Tiles Sdn Bhd	Manufacturing failure	Glazed
E	Air Hitam Pottery	Handling failure	Glazed

All specimens are claimed reject due to fail to reach the standard from the manufacturer. The specimens also have different type of failure such as cracks, porosity and inclusion.

Air Hitam Pottery put the standard that the products is claimed reject if the flaw size can be detected using a naked eyes. Since the product coming out from this company are household and art products, the standard are quite lenient. Alpha Tiles Sdn. Bhd. has put a very high standard for their product since the products are being used for heavy duty usage.

3.3 Firing and Cooling Temperature Process Flow

The specimens were firing at different temperature and different holding time. The firing and cooling process is show in **Figure 4**.

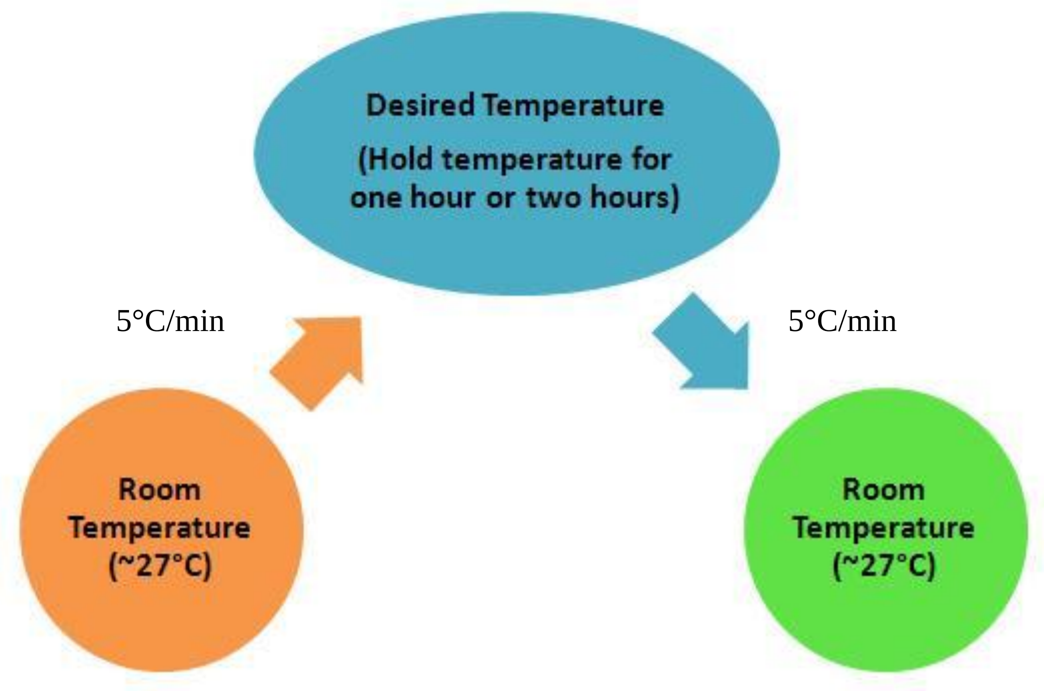

Figure 4: Firing and Cooling Process

The specimens were fired at desired temperature at 5°C/min and held for one hour (or two hour) before cooled at rate 5°C/min to the room temperature. For the one hour holding time (specimen A, B and C), the temperature was varies from 800°C until 1400°C. For two hours holding time (specimen D and E), the temperature was varies from 800°C until 1300°C.

3.3 Tools and Equipments Required

The tools and equipments required are based on the journals related to this project. The tools and equipments required for this project listed as below:

3.3.1 Furnace

The specimens were fired using chamber furnace at the desired temperature. The furnace is referring to a direct fired heater, used in boiler applications in chemical industries or for providing heat to chemical reactions for processes like cracking, and are part of the standard names for many metallurgical furnaces worldwide. The furnace used is a Carbolite chamber furnace (**Figure 5**) which is an electrical furnace and the temperature can be controlled until maximum temperature which is 1500°C [12].

Figure 5: Carbolite Chamber Furnace

3.3.2 X-Ray Fluorescence Machine

X-ray fluorescence is the emission of fluorescent X-rays characteristic from a material that has been excited by bombarding with high-energy X-rays or gamma rays. The phenomenon is widely used for elemental analysis and chemical analysis, particularly in the investigation of metals, glass, ceramics and building materials, and for research in geochemistry, forensic science and archaeology.

The machine used for XRF analysis is Brunker S4 Pioneer (**Figure 6**) which is the most compact wavelength dispersive X-ray fluorescence spectrometer (WDXRF). The machine together with advanced excitation technology provides highest sensitivity especially for light elements and traces due to optimized beam geometry. It is provided with improved analytical performance for light elements and guaranteed with the very thin Beryllium tube window in combination with optimized excitation parameters [13].

Figure 6: Brunker S4 Pioneer XRF Machine

The X-ray fluorescence principle is depicted in **Figure 7**. An inner shell electron is excited by an incident photon in the X-ray region. During the de-excitation process, an electron is moving from a higher energy level to fill the vacancy. The energy difference between the two shells appears as an X-ray, emitted by the atom. The X-ray spectrum acquired during the above process reveals a number of characteristic peaks. The energy of the peaks leads to the identification of the elements present in the sample (qualitative analysis), while the peak intensity provides the relevant or absolute elemental concentration (semi-quantitative or quantitative analysis) [14].

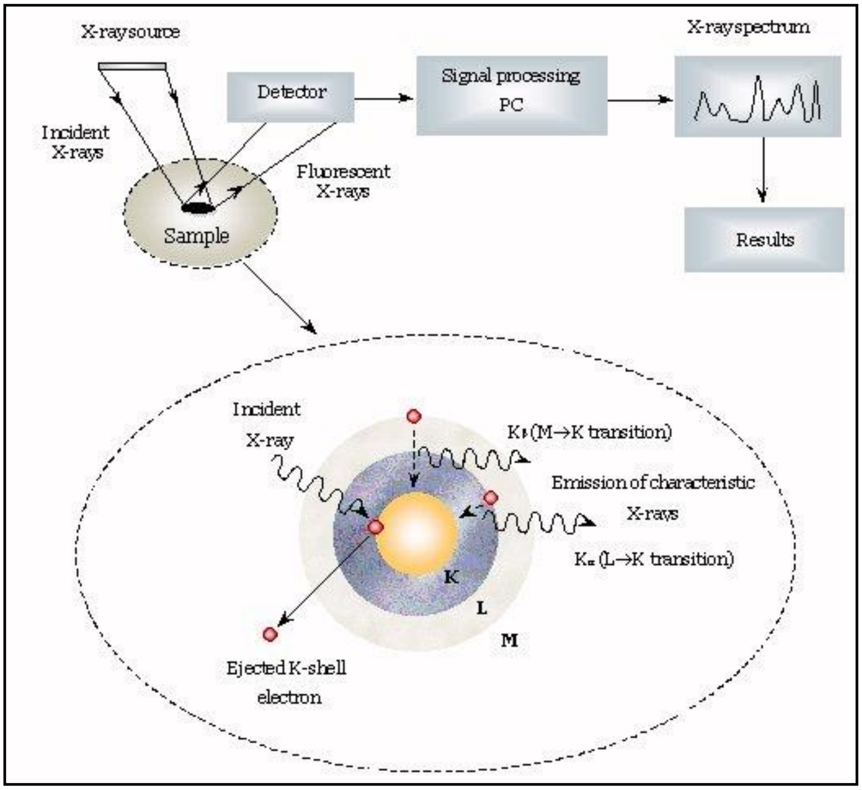

Figure 7: The Principle of XRF and the Typical XRF Detection Arrangement [14].

3.3.3 Measurement Tool

A caliper is a device used to measure the distance between two symmetrically opposing sides. A caliper can be as simple as a compass with inward or outward-facing points.

Since the cracks on the specimens are in millimeter readings, a vernier caliper with two decimal places is used for measurement (**Figure 8**).

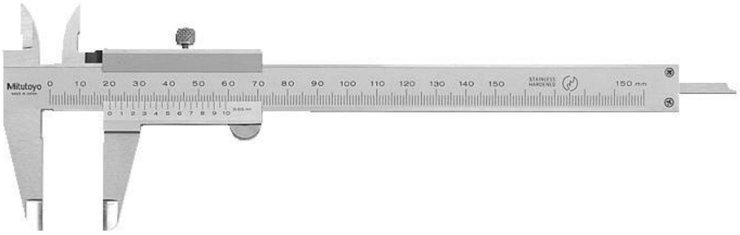

Figure 8: Mitotuyo Vernier Caliper

CHAPTER 4

RESULTS AND DISCUSSION

All of the experimental works on firing the specimens under desired temperature under different holding time and the elemental analysis by using X-ray Fluorescence has been done.

4.1 Crack Propagation (One hour holding time)

There are three specimens (Specimen A, B and C) tested under the first part of the experiment which is the desired temperature is held at one hour before cooling down. **Figure 9, 10** and **11** shows the picture of specimens before firing at room temperature.

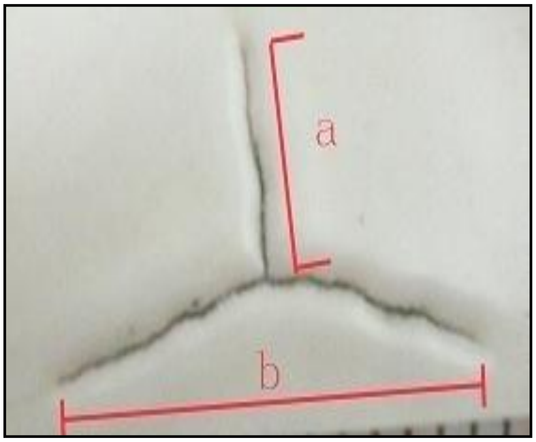

Figure 9: Specimen A (Before Firing).

Crack measurement points are referred as (a) and (b).

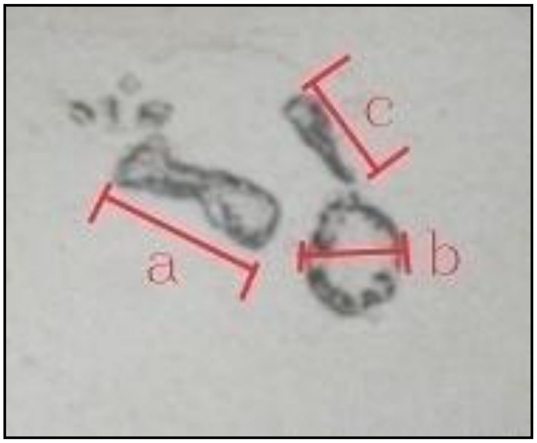

Figure 10: Specimen B (Before Firing).

Crack measurement points are referred as (a), (b) and (c).

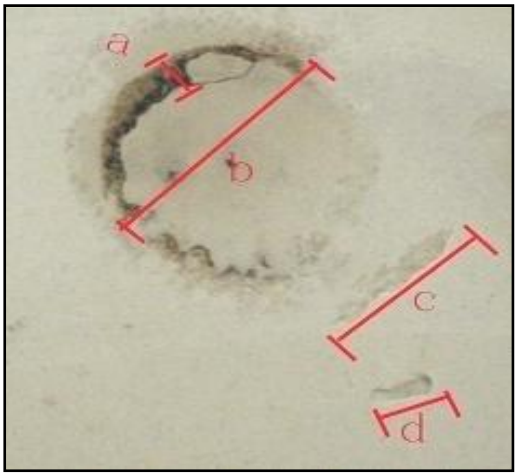

Figure 11: Specimen C (Before Firing).

Crack measurement points are referred as (a), (b), (c) and (d).

All three specimens were fired inside the furnace chamber which is heated at a rate of 5°C/min and held at 800°C for one hour. The furnace is subsequently cooled down to room temperature at a rate of 5°C/min. After the cooling down completed, the specimen's cracks were measured using Vernier caliper.

The firing process is repeated at different temperatures of 900°C, 1000°C, 1100°C, 1200°C, 1300°C and 1400°C. **Figure 12, 13** and **14** shows the appearance of the specimens after fired. All photos are in the scale of 1:4cm.

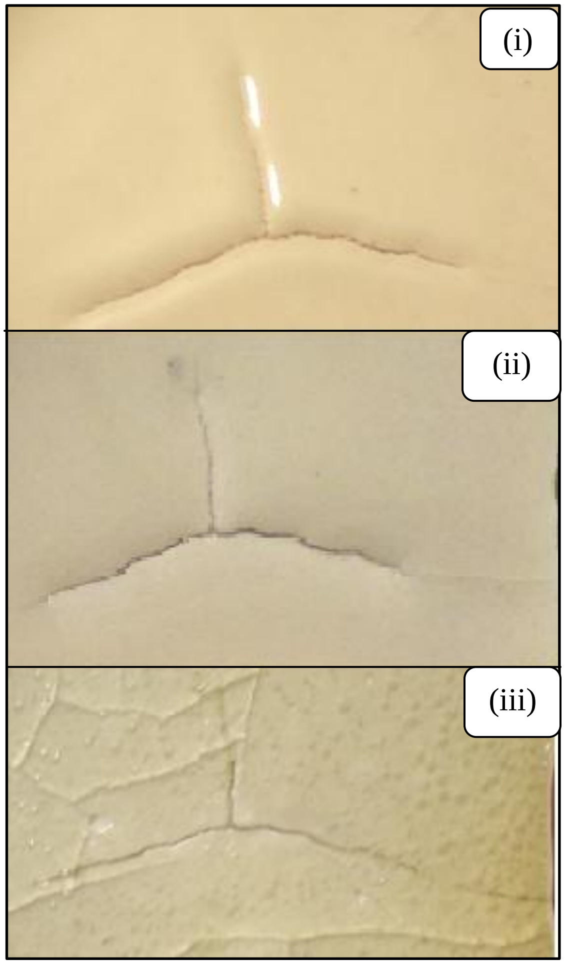

Figure 12: Specimen A after Firing at Different Temperatures.

(i) 800°C, (ii) 1200°C, and (iii) 1400°C.

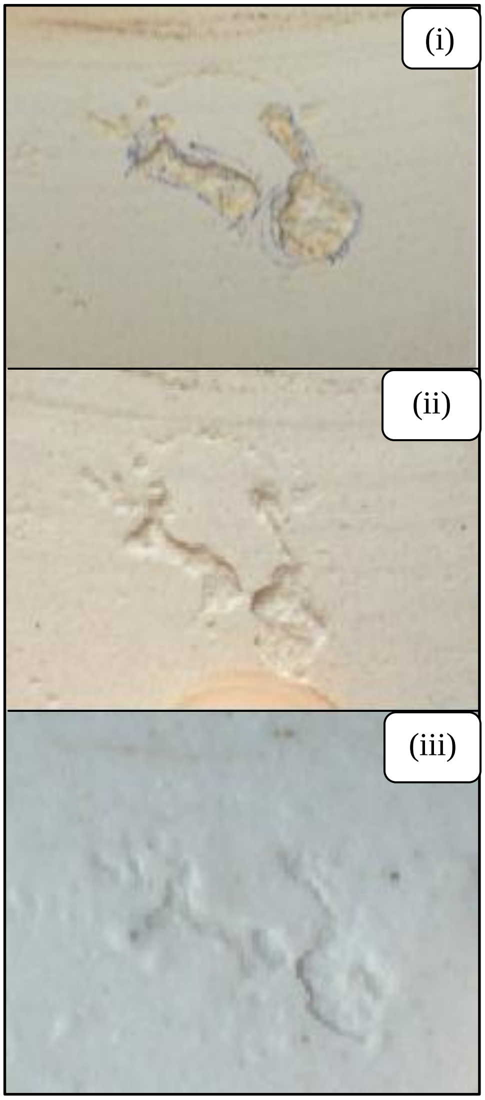

Figure 13: Specimen B after Firing at Different Temperatures.

(i) 800°C, (ii) 1200°C, and (iii) 1400°C.

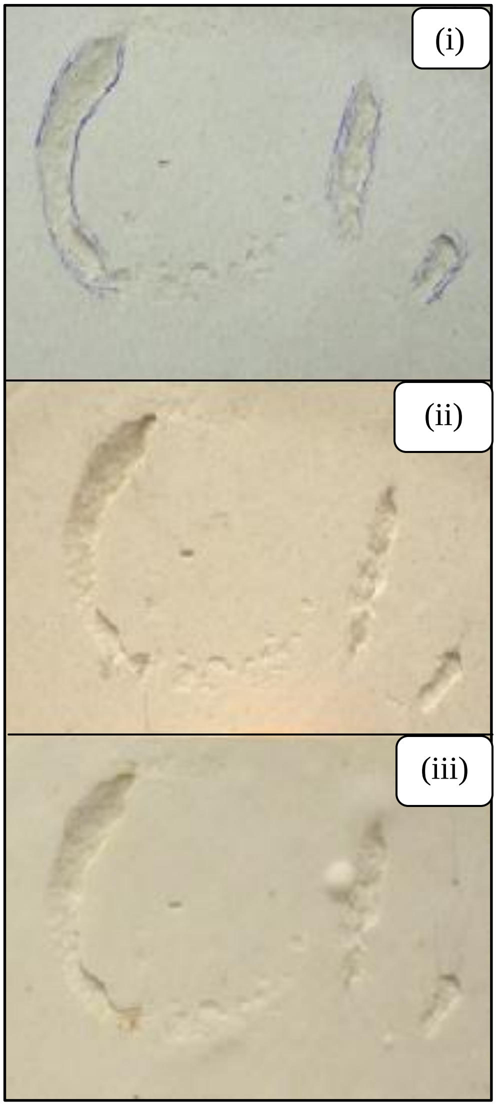

Figure 14: Specimen C after Firing at Different Temperatures.

(i) 800°C, (ii) 1200°C, and (iii) 1400°C.

4.1.1 Cracks Measurement

After the cooling down process of the specimens is completed, the cracks were measured by using Vernier caliper. **Table 3** shows data gathered from specimen A, B and C after firing.

Table 3: Crack Propagation Data for Specimen A, B and C.

Specimen	**Crack Point/ Area**	**Crack Length (mm)**							
		Room Temp ~27°C	**800°C**	**900°C**	**1000°C**	**1100°C**	**1200°C**	**1300°C**	**1400°C**
A	a	10.00	10.00	10.00	10.00	10.04	10.09	10.24	11.26
	b	22.14	22.16	22.00	22.36	22.56	31.38	31.68	31.72
B	a	5.21	5.22	5.32	5.40	5.46	5.66	5.82	5.92
	b	3.02	3.08	3.54	3.80	3.92	3.96	3.98	3.98
	c	2.92	2.96	2.98	3.04	3.24	3.32	3.48	3.52
C	a	2.00	2.00	2.06	2.36	2.40	2.42	2.43	2.46
	b	11.72	11.78	11.98	12.02	12.36	12.40	12.44	12.66
	c	6.76	6.78	6.78	7.18	8.08	8.10	8.66	9.12
	d	2.82	2.84	3.34	3.34	3.38	3.38	3.38	3.40

Based on the **Table 3** and **Figure 15**, specimen A-a (specimen A, crack measurement point a) shows no propagation but after 1100°C, the crack start propagates slowly until temperature 1200°C. The crack propagates rapidly after that until the firing temperature stop at 1400°C. The propagation length for specimen A-a is 1.26mm. For specimen A-b, crack propagates as the firing temperature added and propagates rapidly after temperature 1100°C. The propagation length for specimen A-b is 9.58mm.

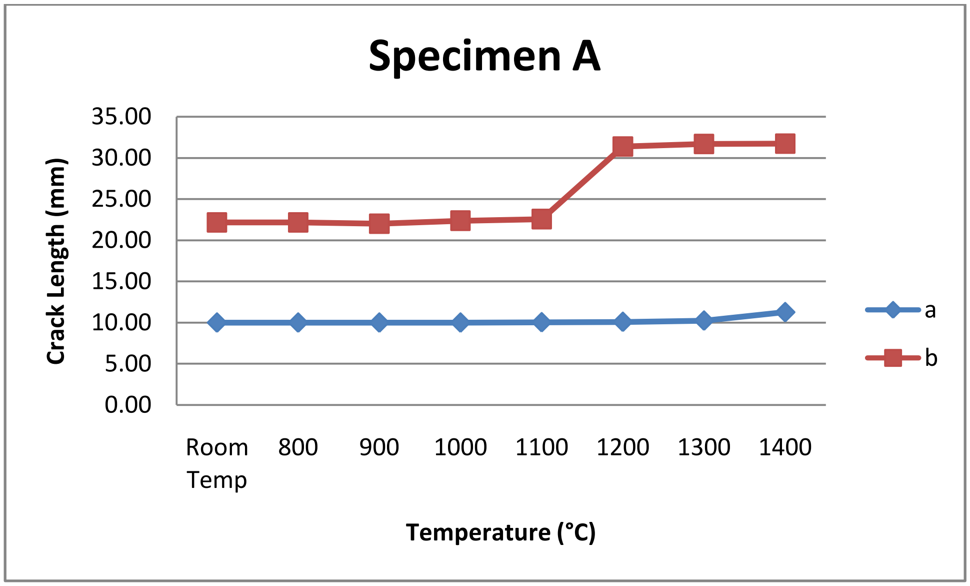

Figure 15: Graph of Crack Propagation for Specimen A

Based on the **Table 3** and **Figure 16**, specimen B-a shows a slow propagation as the temperature increase until the firing temperature stop at 1400°C. The propagation length for specimen B-a is 0.71mm. For specimen B-b, crack propagates rapidly after 900°C and propagates stable until reach temperature 1400°C. The propagation length for specimen B-b is 0.96mm. Specimen B-c shows a linear propagation data as the temperature increased. The total crack length measured for specimen B-c is 0.60mm.

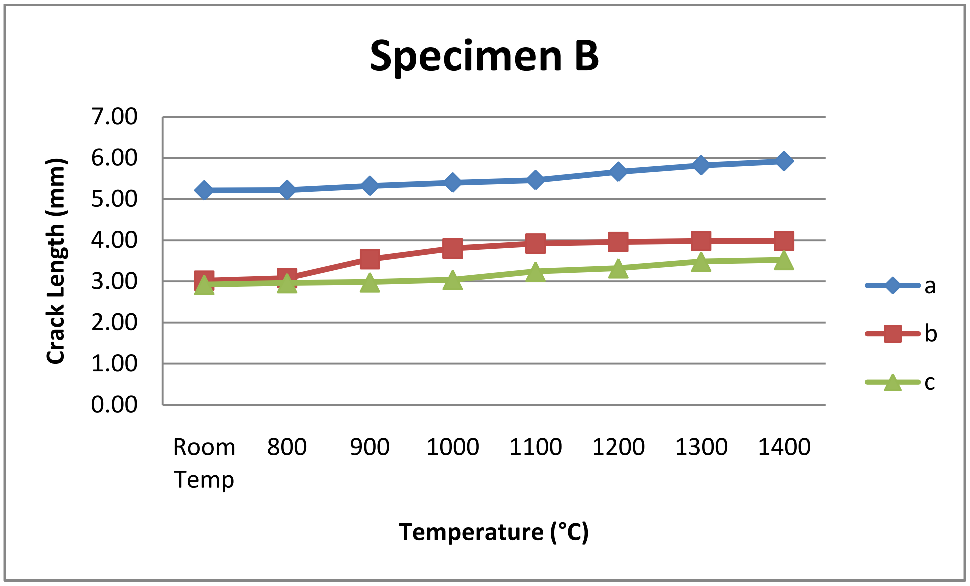

Figure 16: Graph of Crack Propagation of Specimen B

Based on the **Table 3** and **Figure 17**, specimen C-a shows no propagation but after 900°C, the crack start propagates slowly until temperature stop at 1400°C. The propagation length for specimen C-a is 0.46mm. Specimen C-b shows that crack propagates slowly as the temperature increase. The total crack propagation length for specimen C-b is 0.94mm. For specimen C-c, crack propagates as the firing temperature increase and propagates rapidly after temperature 900°C. The total crack propagation length for specimen C-c is 2.36mm. Specimen C-d shows small propagation but after 900°C, the crack start propagates slowly until temperature stop at 1400°C. The total crack propagation length for specimen C-d is 0.52mm.

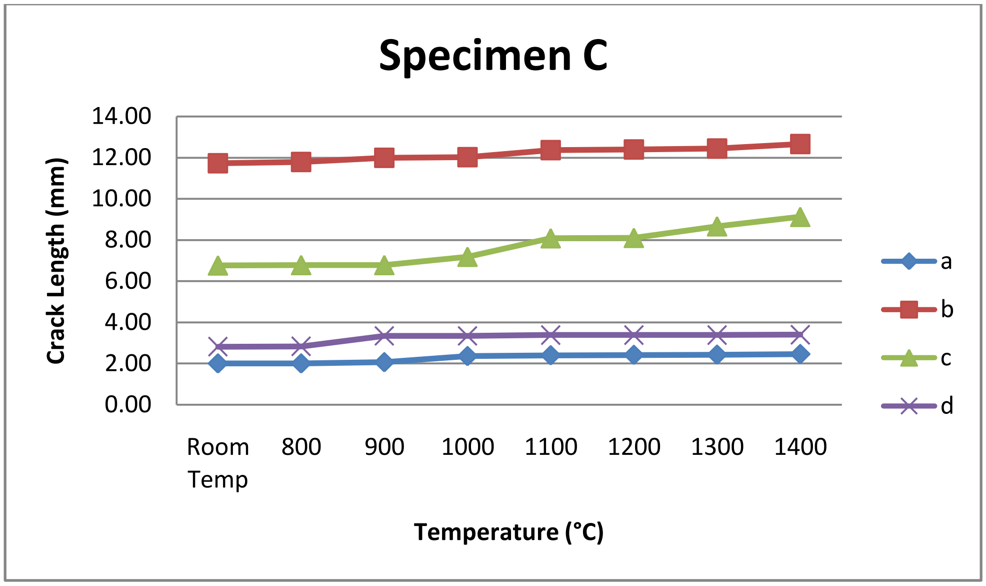

Figure 17: Graph of Crack Propagation of Specimen C

4.2 Crack Propagation (Two Hours Holding Time)

There are two specimens (Specimen D and E) tested under the second part of the experiment which is the desired temperature is held at two hour before cooling down. **Figure 18** and **19** shows the picture of specimens before firing at room temperature.

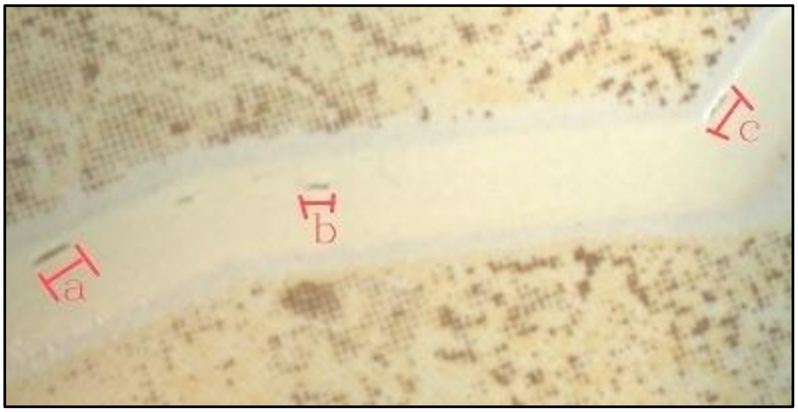

Figure 18: Specimen D (Before Firing).

Crack measurement points are referred as (a), (b) and (c).

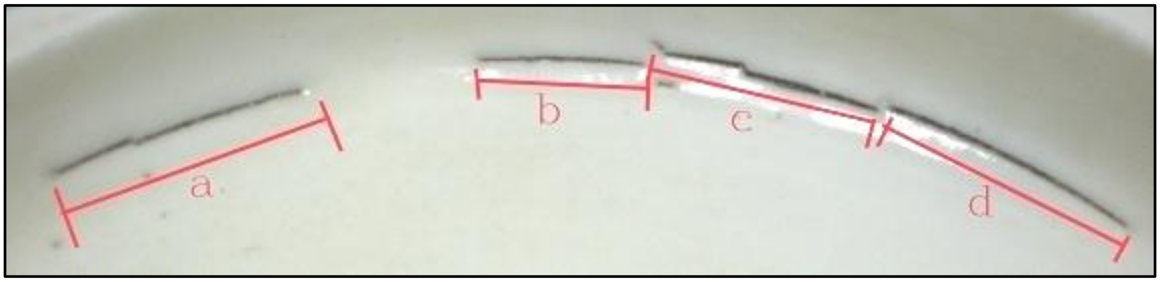

Figure 19: Specimen E (Before Firing).

Crack measurement points are referred as (a), (b), (c) and (d).

All the specimens were fired inside the furnace chamber which is heated at a rate of 5°C/min and held at 800°C for two hour. The furnace is subsequently cooled down to room temperature at a rate of 5°C/min. After the cooling down completed, the specimen’s cracks were measured using Vernier caliper.

The firing process is repeated at different temperatures of 900°C, 1000°C, 1100°C, 1200°C and 1300°C. Figure 20 and 21 are the appearances of the specimens after fired. All photos are in the scale of 1:4cm.

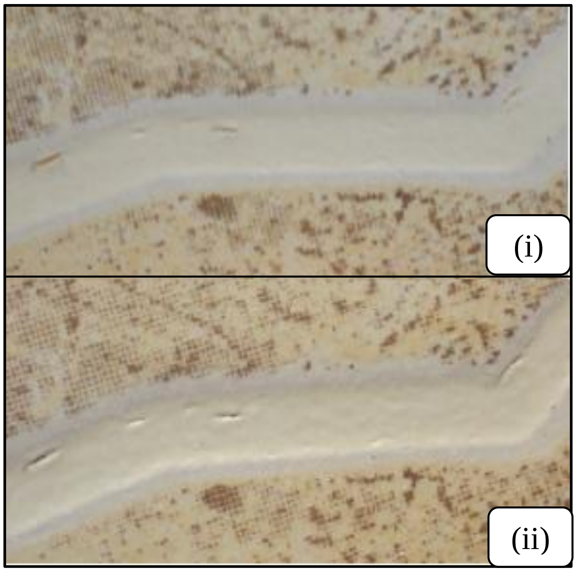

Figure 20: Specimen D after Firing at Different Temperatures.

(i) 900°C and (ii) 1100°C

Figure 21: Specimen E after Firing at Different Temperatures.

(i) 900°C and (ii) 1100°C.

4.2.1 Crack Measurement

After the cooling down process of the specimens is completed, the cracks were measured by using Vernier caliper. **Table 4** shows data gathered from specimen D and E after firing.

Table 4: Crack Propagation Data for Specimen D and E.

Specimen	Crack Point/ Area	Crack Length (mm)						
		Room Temp ~27°C	800°C	900°C	1000°C	1100°C	1200°C	1300°C
D	a	2.10	2.16	2.20	2.34	2.40	2.44	∞
	b	1.32	1.74	1.76	1.72	1.78	1.84	∞
	c	1.85	1.92	2.06	2.08	2.10	2.13	∞
E	a	12.58	13.34	13.54	14.12	14.16	14.56	14.59
	b	8.58	8.74	8.76	8.82	9.00	9.22	9.31
	c	12.15	12.50	12.52	12.53	12.53	12.58	12.73
	d	13.38	13.70	14.26	14.32	14.36	14.45	14.47

Based on the **Table 4** and **Figure 22**, specimen D-a (specimen D, crack measurement point a) shows slow propagation but after 900°C, the crack start propagates rapidly until temperature 1200°C. The propagation length for specimen D-a is 0.34mm. For specimen D-b, crack propagates rapidly as the firing temperature added. The propagation length for specimen D-b is 0.52mm. Specimen D-c shows slow propagation but linearly the crack propagates. The propagation length for specimen D-c is 0.28mm. At temperature 1300°C, the specimen fails as the specimen burn out. This will be covered in the X-ray Fluorescence analysis.

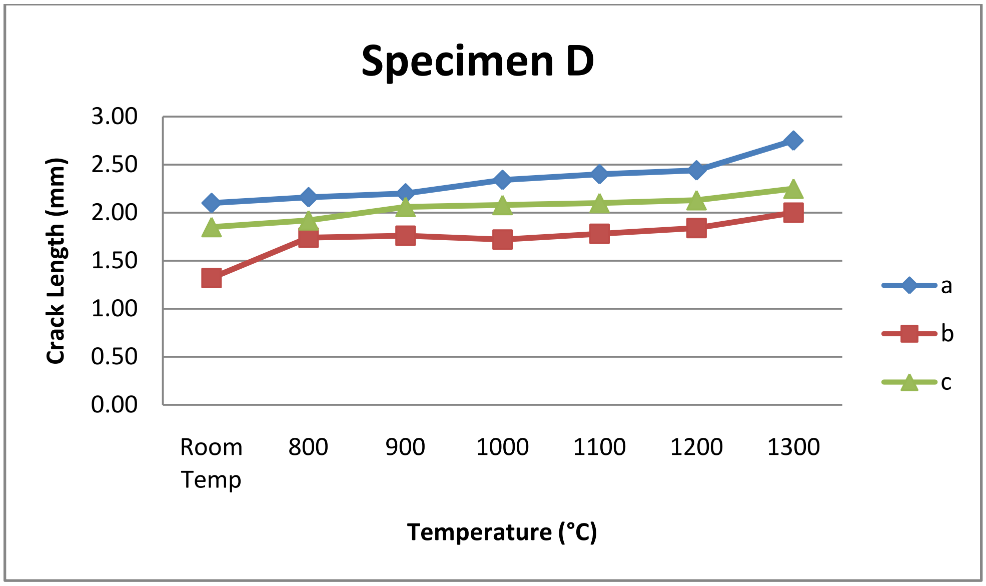

Figure 22: Graph of Crack Propagation of Specimen D

Based on the **Table 4** and **Figure 23**, specimen E-a shows a rapid propagation along the temperature increased. The propagation length for specimen E-a is 2.01mm. For specimen E-b, it shows a small propagation but after 1100°C, the crack start propagates slowly until temperature stop at 1200°C. The total crack propagation length for specimen E-b is 0.73mm. Specimen E-c shows a small propagation as the temperature increased. The propagation length for specimen E-c is 0.58mm. For specimen D-d, the crack propagates rapidly as the temperature increased but slowly propagates at temperature 900°C until temperature stop. The propagation length for specimen A-a is 1.09mm.

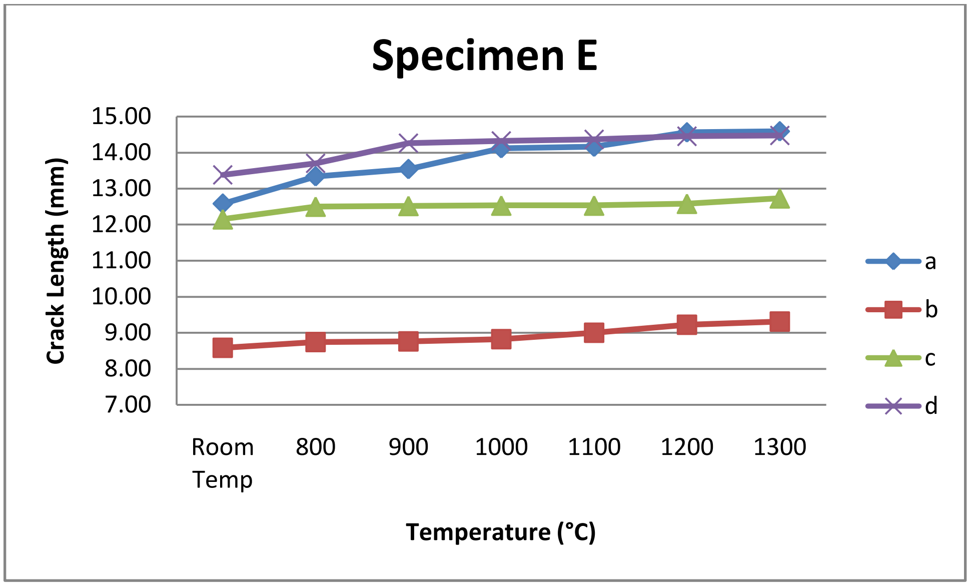

Figure 23: Graph of Crack Propagation of Specimen E.

4.4 X-ray Fluorescence Analysis.

The X-ray Fluorescence analysis was done for all specimens before the firing experiment and after had been firing at 1400°C for specimen A, B and C, and at 1300°C for specimen D and E. The behavior of specimens before and after firing could give useful information about the strength of the material and why the crack propagates.

4.4.1 XRF Analysis for Specimen A and B.

Quantitative XRF analysis of the specimen A and B is as given in the **Table 5**. There are changes in the elemental analysis before and after the firing process. After firing at 1400°C, elements such as Al, P, K, Ca, Zn, Rb, Sr, Zr, Si and Ba is decreasing in terms of KCps (kilo counts per second). Elements such Ti and Fe is increasing in KCps. New elements such as TiO2, Ar and Cu were found exist after the firing process. Element such Cr, K2O and V2O5 were extinct after the firing process.

Table 5: XRF Analysis for Specimen A and B.

(Left table indicates XRF analysis of the specimen before firing process. Right table indicates XRF analysis of the specimen after firing at 1400°C)

Element	**Sample A and B**
	KCps
O	-1000.0
Mg	0.6
Al	59.0
P	3.0
K	58.5
Ca	51.3
Ti	12.9
Cr	1.0
Fe	88.0
Cu	
Zn	170.1
Rb	9.9
Sr	38.1
Zr	127.5
Si	199.1
Ba	4.5
Ar	
K2O	51.3
V2O5	1
TiO2	

Element	**Sample A and B**
	KCps
O	-1000.0
Mg	
Al	55.1
P	2.9
K	55.1
Ca	39.5
Ti	14.1
Cr	
Fe	98.2
Cu	21.9
Zn	124.2
Rb	7.6
Sr	29.3
Zr	29.2
Si	191.0
Ba	3.2
Ar	
K2O	
V2O5	
TiO2	14.1

Note: Specimen A and B are from the same material type.

4.4.2 XRF Analysis for Specimen C

Quantitative XRF analysis of the specimen C is as given in the **Table 6**. There are changes in the elemental analysis before and after the firing process. After firing at 1400°C, elements such as Al, P, K, Ca, Ti, Zn, Rb, Sr, Zr, Si and Ba is decreasing in KCps (kilo counts per second). Fe element was found increase after the firing process. New elements such as TiO2, Zn, Ar and Cu were found exist after the firing process. Element such Ba, K2O and V2O5 were extinct after the firing process.

Table 6: XRF Analysis for Specimen C

(Left table indicates XRF analysis of the specimen before firing process. Right table indicates XRF analysis of the specimen after firing at 1400°C)

Element	**Sample C**
	KCps
O	-1000.0
Al	73.4
P	2.9
K	55.6
Ca	43.7
Ti	22.6
Cr	1.0
Fe	86.6
Cu	
Zn	
Rb	13.5
Sr	24.2
Zr	144.2
Si	190.9
Ba	3.1
Ar	
K2O	55.6
V2O5	2.2
TiO2	

Element	**Sample C**
	KCps
O	-1000.0
Al	58.7
P	2.7
K	41.4
Ca	20.9
Ti	21.4
Cr	0.9
Fe	81.0
Cu	12.4
Zn	2.5
Rb	8.2
Sr	15.3
Zr	48.2
Si	161.9
Ba	
Ar	
K2O	
V2O5	
TiO2	21.4

4.4.3 XRF Analysis for Specimen D

Quantitative XRF analysis of the specimen D is as given in the **Table 7**. There are changes in the elemental analysis before and after the firing process. After firing at 1400°C, elements such as Al, K, Ca, Ti, Mn, Fe, Zn, Rb, Sr and Zr is decreasing in KCps (kilo counts per second). P element was found increase after the firing process. New elements such as V2O5, K2O, Si and Cu were found exist after the firing process. Element such Cr, Sc2O3 and MnO were extinct after the firing process.

Table 7: XRF Analysis for Specimen D

(Left table indicates XRF analysis of the specimen before firing process. Right table indicates XRF analysis of the specimen after firing at 1300°C)

Element	**Sample D**
	KCps
O	-1000.0
Na	0.2
Mg	1.1
Al	44.2
P	3.0
K	79.0
Ca	18.9
Ti	16.1
Cr	1.0
Mn	2.4
Fe	630.2
Cu	
Zn	21.1
Rb	13.5
Sr	15.7
Zr	223.1
Si	
K2O	
V2O5	
Sc2O3	0.0
MnO	2.4

Element	**Sample D**
	KCps
O	-1000.0
Na	
Mg	0.9
Al	35.0
P	3.1
K	71.7
Ca	17.9
Ti	15.3
Cr	
Mn	2.3
Fe	610.2
Cu	8.3
Zn	17.6
Rb	11.7
Sr	14.3
Zr	209.6
Si	195.1
K2O	71.7
V2O5	1.4
Sc2O3	
MnO	

4.4.4 XRF Analysis for Specimen E

Quantitative XRF analysis of the specimen D is as given in the **Table 8**. There are changes in the elemental analysis before and after the firing process. After firing at 1400°C, elements such as Mg, Al, P, K, Ca, Zn, Rb, Sr, Si, Ba and Zr is decreasing in KCps (kilo counts per second). Mn and Fe element was found increase after the firing process. New elements such as Ti, Cu and CaO were found exist after the firing process. Element such CoO, Pb, S and V2O5 were extinct after the firing process.

Table 8: XRF Analysis for Specimen E

(Left table indicates XRF analysis of the specimen before firing process. Right table indicates XRF analysis of the specimen after firing at 1300°C)

Element	**Sample E**
	KCps
O	-1000
Mg	21.7
Al	17.2
P	2.9
K	29.2
Ca	111.8
Ti	
Mn	3.1
Fe	20.5
Cu	
Zn	55.9
Rb	41.6
Sr	11.3
Zr	514.7
Si	181.4
Ba	7.4
V2O5	0.2
S	1.3
Pb	0.6
CoO	0.5
CaO	

Element	**Sample E**
	KCps
O	-1000.0
Mg	19.6
Al	15.7
P	2.9
K	18.7
Ca	87.9
Ti	0.7
Mn	3.3
Fe	23.4
Cu	14.7
Zn	32.1
Rb	40.0
Sr	9.1
Zr	206.4
Si	155.3
Ba	2.6
V2O5	
S	
Pb	
CoO	
CaO	87.9

4.5 Discussion

Understanding the thermal behaviour of ceramic is important because it can explain the consequences of ceramic usage at high temperature. From the data and graphs above, it can be concluded that crack propagates when exposed to high temperature. The crack propagation of the specimen is not equally same to each other, depends on the initial crack size, crack shape, type of cracks and also the type of specimens.

Specimen A has the largest crack propagation length which is 9.58mm. This is due to the specimen size and crack shape. The specimen has no equal thickness which is one side is much thinner than the other one. The crack propagates towards the small thickness of the specimen. The residual stress acting at the tip of the crack helps the propagation of the crack is easier at the least thickness since the stress is not evenly distributed at the ceramic layer.

Kaolin function is for strengthen and developing a stronger structure inside the ceramic. Referring to the XRF analysis above, kaolin element which is contribution of the alumina and silicate element, kaolin value is decreasing after the firing process at high temperature. With the decreasing of kaolin, it will results in lower the strength of the ceramic specimens.

Specimen D was burn out after firing at temperature 1300°C. Therefore the specimen is declared fails. In firing a glaze specimen, the glaze is softened to the point where the glaze settles onto the softened surface and becomes a part of that surface. Over firing a glaze specimen will cause colors to sink too deeply into the glaze resulting in distortion, fade-out or burn-out. Vanadium pentoxide, V2O5 is function as a colorant. After firing at high temperature, V2O5 was found exist.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The work reported shows that the crack will propagates when firing at high temperature. Apart from that, the crack propagation in ceramic is depend on the type of specimen, the thickness of the specimen, failure type of the specimen, shape of the crack and also the stress acting on the crack tip. From XRF analysis, the extinction on kaolin elements results in low strength of the specimen.

5.2 Recommendation

As for recommendation and future works, large numbers of the specimens should be used. The specimens must from the same type and the intentional defects will be introduced into the specimens. Detail study on the elemental analysis and the surface appearance changes of the specimen result from the firing process can be done. Detail of elemental analysis should be done in order to understand the behaviour of ceramics when firing at high temperature.

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APPENDIX I

Action Plan	**Semester Jan 2010**													
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Problem Definition														
Literature Review	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦	🟦
Lab Work														
Firing Process	🟦	🟦	🟦	🟦										
XRF					🟦	🟦								
Analysis														
Crack Measurement		🟦	🟦	🟦	🟦	🟦	🟦							
XRF Analysis							🟦	🟦	🟦					
FYP II														
Submission of Progress Report I				🟥										
Submission of Progress Report II								🟥						
Seminar								🟥						
Poster Exibition										🟥				
Submission of Project Dissertation (soft bound)													🟥	
Oral Presentation														🟥
Submission of Project Dissertation (hard bound)														🟥