Middle East Technical University

Department of Metallurgical and Material Engineering

Mete215 – Materials Processing Laboratory

Experiment 2: Fabrication of Ceramics by Pressing and Firing

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ABSTRACT

Ceramics were produced with the powder forming process and the steps of sieving, granulation, consodilation, drying and firing were observed. Consodilation was done using the die pressing method. The die was exposed to 240 bar of pressure for 30 seconds. In the firing process, firing temperature of 900 ^{O}C and 1250 ^{O}C were used. The differences such as the color, texture and dimensions were observed and explained.

INTRODUCTION

Ceramics are a type of material that has been around for a very long time. Infact, there have been founding's of a ceramic that has been dated back 25.000 years to prehistoric age (Geopolymer Institute, 2006). Ceramics are also categorized into two categories: Modern ceramics and Traditional ceramics.

Traditional Ceramics

From the tiles of our house to, porcelain and china as well as earthenware, traditional ceramics are all around us. They are mainly composed of clay, quartz and feldspar which makes up 90% of the mass of traditional ceramics (lab handout, 2019). The materials that makes the traditional ceramics are ones that can be found in nature in a raw and earthly form. Clays and silica can be considered examples of such materials. It should also be noted that while clay, quartz and feldspar makes up most of the composition of traditional ceramics, materials such as talc and limestone makes up the remaining part of the composition and they are used to make certain changes in the end product (lab handout, 2019).

Modern Ceramics

Ceramics can be much more than what meets the eye. It can be said as such because from synthetically produced raw materials, ceramics or what we call modern ceramics, with desired properties can be created. The properties could be named as hardness, strength, electrical conductivity and many more. Therefore, it can be said that they play a very important role in the advancement on technology. It being used in the areas such as, electronics, space exploration, transportation and manufacturing (Science learning hub, 2010) further supports this claim. To give some examples of compositions and the places that they could be used: titanate compositions are used for capacitors while materials such as alumina, zirconia can be used in places where wear and corrosions happen (Pfeifer, 2009).

One of the important topic that needs to be mentioned is the processing of ceramics. Modern ceramics were defined as a category of ceramics where desired properties could be engineered. This is made possible by the advanced techniques that are used in the production which makes it possible to manipulate the grain size as well as the chemistry of the said material. On the other hand, in the case of traditional ceramics the processes are relatively more simple as it won't be used in places where high technology would be used.

The general processes can be put under three main categories. Namely, powder forming, melt processing and chemical processing. In the experiment powder forming process was used. Figure 1 shows the steps of the ceramic production done under the powder forming process.

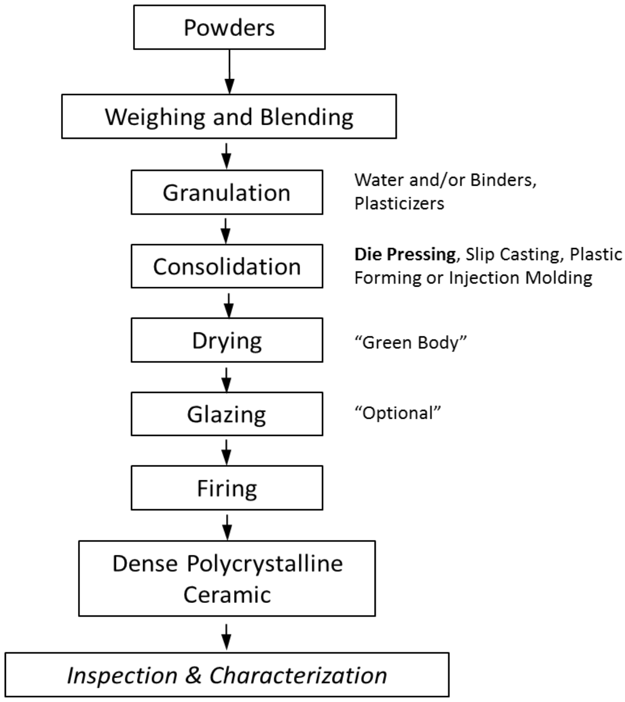

Figure 1. Production steps of a polycrystalline ceramic starting from powder form (Lab Handout, 2019)

In the case of producing ceramics from the powder form, the size and distribution of the particles that makes up the powder is of importance. The difference can be observed in the comparison of traditional ceramics and modern ceramics. Traditional ceramics might contain particle sizes ranging from 5 microns to 1 micron, where else modern ceramics generally consists of particles sizes smaller than 1 micron. Therefore, while producing ceramics in the first stage which is the shaping stage, the powder must go through a sieving process to get the desired particle size distribution.

After the end of the sieving process, granulation must be done. It is done by adding water or binders to the powder. It is done as to let the powders achieve good flow which also gives the ability for the powder to fill the die. When the powder is able to fill the die than it is ready for the consolidation step where the powder takes form. This step can be done in a number of ways. In the experiment, the method of die pressing was used and in this method the powder takes form of the die that it has been put into. Die pressing method also allows the compressed powder to obtain a desired porosity level and strength. It has been mentioned of the addition of water in the granulation phase. However, water is not wanted in the end product hence in the drying phase the compressed ceramic powder is left to dry. Afterwards glazing could be done. Which results in the surface of the ceramics body being coated with a thin layer of glass.

At the end of all the steps mentioned above, the only step left is the firing step. This step can be considered as the most crucial step in the production of ceramics as not only most of the cost depends on this step, the physical properties of the end product are the consequences of this step. The firing process is done in the temperatures ranging from 700 ^{o}C to 2000 ^{o}C (Lab

Handout, 2019). In the experiment firing process was done in temperatures of 900 ^{o}C and 1200 ^{o}C. The change in the smoothness of the surface and the edges as well as the color and the density was observed.

Experimental

Materials that were used;

- Ceramic powder mixture
- A mixture of clay, feldspar and quartz
- Water
- Zinc stearate

Equipment that were used in the experiment;

- Mechanical balance,
- Mortar and pestle
- Laboratory glass ware like burette and beakers
- Sieve (100 mesh)
- Electrical balance,
- Steel dies, (with circular or rectangular die cavities)
- Hydraulic press (max capacity 5 tons)
- Drying oven (max 250 °C)
- Firing furnace (max 1250 °C)
- Digital Caliper
- Density measurement kit

Procedure

The experiment started with the measurement of ceramic powder that consisted of clay, feldspar and quartz. 10 grams of the powder was measured and put in the mortar. Next came the addition of water to the mortar. 1 gram of water was needed and it was obtained from the density relationship where 1 gram of water was equated to 1 ml of water. Therefore, 1 ml of water was added into the mortar. Afterwards, the mixture was pestled until it was sufficiently homogeneously moistened. To get rid of the particles bigger than 150 µm a sieving process where the mixture was put through a 100 mesh sieve. Before the resulting mixture could be put inside the die, zinc stearate mixed with acetone was put on the insides of the die. This was done for the ease of removal of the powder after the die pressing processing was over. Then, a pressure of 60 bar was read on the hydraulic press and such pressure was hold on for 30 seconds. At the end of this the ceramic powder mixture was compressed and took on a cylindrical shape. After this the ceramic was left to dry overnight at a temperature of 50 ^{o}C. Dimensions of the ceramic body was taken and it was labeled "Dry Body" as it can be seen in figure 2. For firing stage, the ceramic body was put on a sintering furnace where it was heated to 900^{o}C with a heating rate of 4 ^{o}C/min. With the same heating rate of 4 ^{o}C/min, in the second run it was heated till 1250 ^{o}C. The samples both stayed 2 hours in the temperatures stated above and after the furnaces were cooled by themselves to room temperature their dimensions as well as their weights were measured and noted.

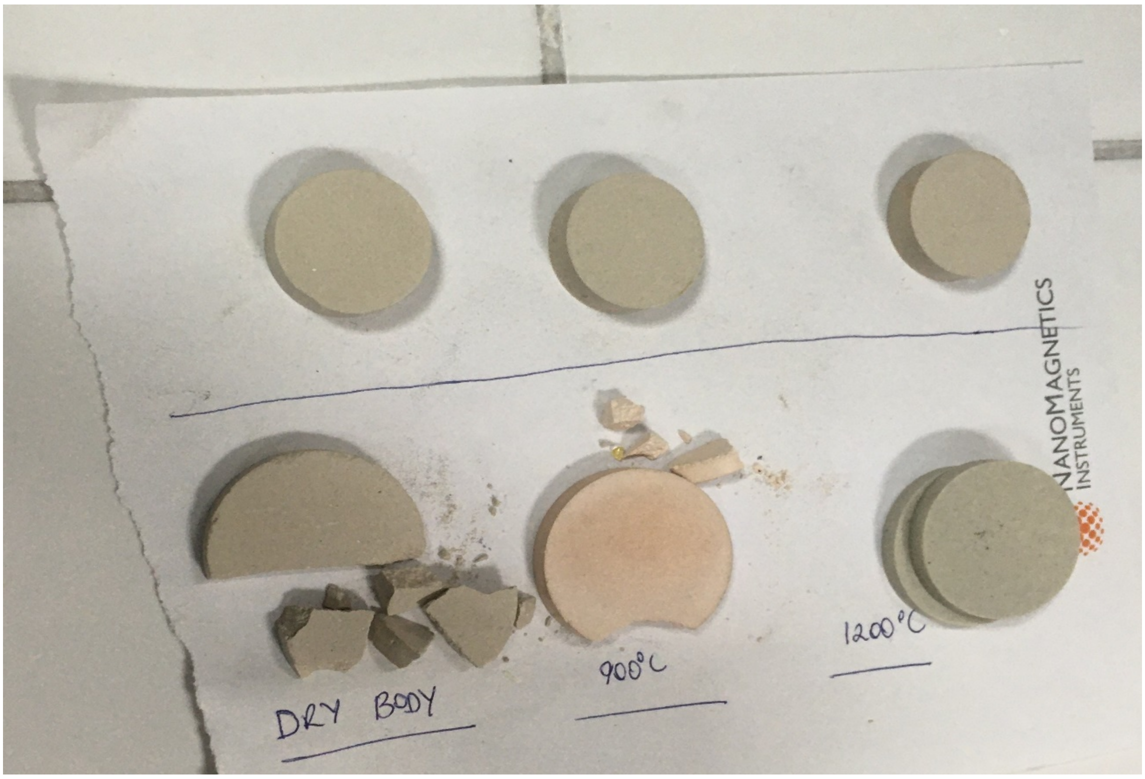

Figure 2. Ceramic Bodies After various processes

Results

Dimensions	*Dry Body*	*900 ^{0}C*	*1250 ^{o}C*
Mass(gram)	9.21	8.61	8.57
Diameter(millimeter)	35.2	33.5	33.0
Thickness(millimeter)	5.00	4.80	4.68

Table 1. Dimensions of the ceramic body after various processes

Table 1 shows the dimensions of the ceramic body;

- After leaving it overnight to dry at 50 ^{o}C under the "Dry Body".
- After the firing process at 900 ^{o}C labeled as "900 ^{o}C".
- After the firing process at 1250 ^{o}C labeled as "1250 ^{o}C".

- In the case of the ceramic body labeled "Dry Body" the bottom and top of it were rough but the edges were smoother when compared to the top and bottom. It also was in greyish color.
- In the case of the ceramic body labeled "900 ^{o}C" It had a light pinkish color with a smooth surface.
- In the case of the ceramic body labeled "1200 ^{o}C" It was of a similar color to the "Dry Body", a greyish color. It was also very rough on the surfaces.

Ceramic Bodies	*Dry Body*	*900 ^{0}C*	*1250 ^{o}C*
Roughness	Middle roughness	Smooth	Very rough

Table 2. Roughness comparison of the ceramic bodies relative to each other

Table 2 compares the ceramic bodies surfaces according to its roughness.

Properties	*Dry Body*	*900 ^{0}C*	*1250 ^{o}C*
Volume(cm^3)	4.870	4.230	4.00
Density(g/cm^3)	1.90	2.04	2.14

Table 3. Computed values of ceramic bodies

Table 3 are the volume and density of the ceramic bodies. When compared, it is clear that the volume decreases and density increases as the firing temperature increases. Figure 3 also shows this graphically.

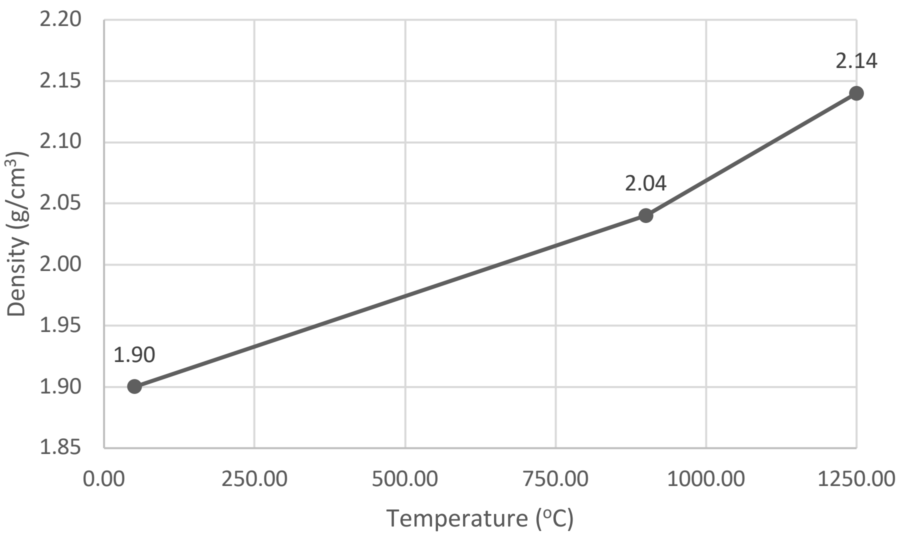

Figure 3. Graph of Temperature vs Density

Discussion

There are 4 main questions that should be answered which are;

1. Why does the end products color changes as the firing temperature changes?
2. Why is the roughness of the surfaces different in different ceramic bodies?
3. In the case of “Dry Body” why is the edges of the ceramic body is smoother than the top and bottom surfaces?
4. Why does density increase as the temperature increases?

Before the questions are answered, it should be mentioned of why Zinc stearate was mixed with acetone instead of water and also on why the pressure value that we read on the hydraulic press is not the actual pressure exerted to the die. The first of the two question is an easy one to answer. Zinc stearate repels water therefore acetone was used instead of water. As for the second question, the hydraulic press gave the value of 60 bar assuming the cross section of the hydraulic press that has a radius of 5 cm. However, the die that was used has a diameter of 2.5cm meaning that the hydraulic press cross section has 4 times the area than the cross section of the die. From this relation we can compute that 240 bar of pressure was exerted on the die which is 4 times as much.

To answer the main 4 questions;

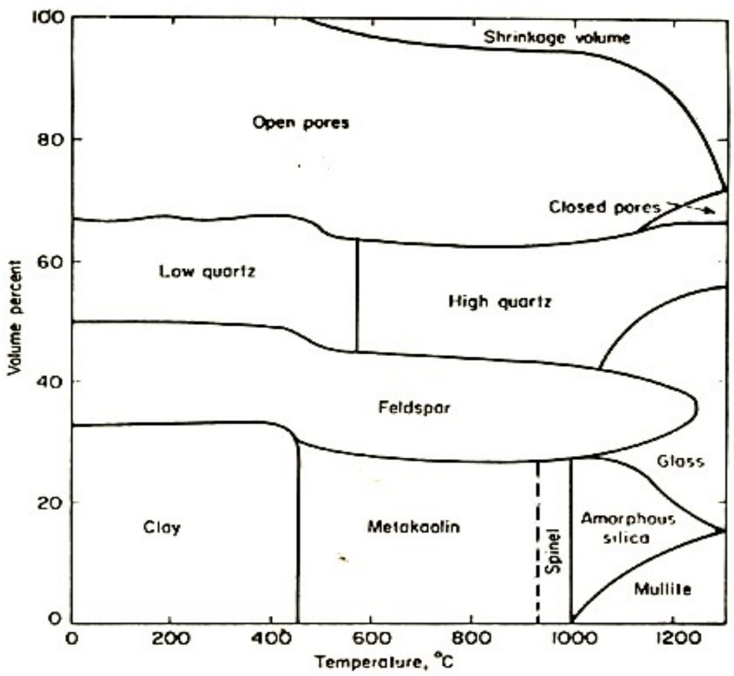

Figure 4. Graph showing the composition of typical clayware in different temperatures (Lab Handout, 2019)

1) In the experiment we have concluded that the ceramic body produced in a firing temperature of 900 °C had a pinkish color where the other two samples had a similar greyish color. From figure 4 we can determine that the component of melakaolin is the

material that differs from the "Dry body" and "1200 °C" therefore we can say that it is the reaseon for the pinkish color we observe.

2) If we look at figure 4 again in the "Dry Body", It can be seen that it a considerable amount of pores and it has a rough surface. As the firing temperature is increased the percentage of open pores also decreases and it is known that "900 °C" has a smooth surface. In this aspect, "1200 °C" is expected to be the smoothest but it is the roughest. We can correlate this with the formation of glass that is seen in figure 4. Glass may result in the rough surface that has been observed.
3) In question 2 we concluded that porosity caused the roughness of the surface. In "Dry Body" the reason for the edges to be smoother can be attributed to the process in which the ceramic body was obtained. The method that was used was the die pressing method. This method resulted in the edges having lesser porosity density therefore resulting in them having smoother surface.
4) Density is equal to mass divided by volume. It is known that shrinkage occurs as the temperature increases. Therefore, if the volume decreases while mass approximately stays the same than the density must increase.

Study Questions

1) The main difference between two ceramic bodies sintered at two different temperatures are that of their compositions. To give an example from "Dry Body" to "900 °C" from figure 4 it can be determined that the porosity and the components making up the ceramic body has changed.
2) From figure 4 it is observed that for "900 °C" Melakaolin, Feldspar High quartz and open pores makes up the ceramic body. While for "1200 °C", mullile, glass, high quartz and closed pores are the components.
3) In the case that the clay is used just after firing it would be still very hot and prone to damage therefore one can break it or as it is hot it might be a potential safety hazard. Also if the glazing hasn't been done water can damage it.
4) It is possible to paint clay after firing therefore it is an advantage on creating esthetically pleasing clay.
5) With firing ceramics with low amount of porosity and a densified body as well as a durable body is desired. Therefore, from the texture and the durability it can be determined whether the firing resulted in good quality clay.

Conclusion

To summarize and conclude it can be said that ceramics are part of our daily lives. They are also used in many areas where high technology is needed. In this experiment the effect of firing rate was observed on the end products. It was concluded that with the change in firing rate, change in color could be observed. The change in texture of the surfaces as well as the composition of the ceramic is also affected by firing rate. In the progression of the experiment there were small details that should be noted such as the use of zinc stearate. It should also be noted that there are many methods on consolidating the ceramic powder and in this experiment the method of die pressing was used.

Reference

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Appendix

Volume of Dry Body, 900 °C Body and 1200 °C Body was calculated by the formula;

V = h π r2 where;

V: Volume

h: Height of the cylindrical shape

r: Radius of the cylindrical shape

Volume of Dry Body: $(5.00) * \pi * (17.6)^2 = 4.870$ cm^3

Volume of 900 °C Body: $(4.80) * \pi * (16.75)^2 = 4.230$ cm^3

Volume of 1200 °C Body: $(4.68) * \pi * (16.5)^2 = 4.00$ cm^3

Density of Dry Body, 900 °C Body and 1200 °C Body was calculated by the formula;

ρ = m/V where;

ρ: Density

m: mass of the ceramic body

V: Volume of the ceramic body

Density of Dry Body: (9.21/4.870) = 1.90 g/cm^3

Density of 900 °C Body: (8.61/4.230) = 2.04 g/cm^3

Density of 1200 °C Body: (8.57/4.00) = 2.14 g/cm^3

Temperature(^{o}C)	Density(g/cm^3)
50	1.90
900	2.04
1250	2.14

Table 2. Data of figure 3