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PHYSICAL CHARACTERIZATION OF ALUMINA (Al_2O_3) BASED REFRACTORY APPLIED ON INDUCTION FURNACE LINING

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ABSTRACT

The broad variety of pyro-processing applications across industry demands great diversity in the supply of refractory materials. In fact, many of these materials have been developed specifically to meet the service conditions of a particular process. The characteristic properties of each refractory class are a function of both their raw materials base and the methods used to manufacture the refractory products. This study aims to characterize alumina (Al_2O_3) based refractory materials applied on induction furnace lining based on its physical properties such as: the shape and grain size; chemical composition; and density of the specimens which sintered at various temperature. From the X-ray Diffraction Test compared with specification data from the manufacturer, the main composition of the base refractory material used in this study is alumina (Al_2O_3) with a low amount (less than 10%) of magnesia (MgO) and silica (SiO_2). Alumina refractory material consists of coarse grains (with its size larger than 0,85mm) and fine grains (size is equal to 0,15mm or smaller). Grains shape of the aggregates are mostly angular with its sharp edges. Values of density at temperature 110^0C, 1000^0C, 1300^0C, 1500^0C in sequence are 2,92 g/cm^3; 2,66 g/cm^3; 2,80 g/cm^3; 2,98 g/cm^3. Density of the refractory will increase as the increase of sintering temperature.

Keywords: *Alumina Refractory, density, microstructure, sintering, X-Ray diffraction.*

1. INTRODUCTION

Researches in material are widely developing thus the knowledge of the experimental result are very beneficial for the use in engineering and technology field nowadays. The invention of the latest materials (e.g., ceramic, composite, polymer, refractory, etc.) shows human intention of finding a proper material to be used in high-operation conditions such as high-temperature; high-stress; corrosive environment etc. The fabrication of these latest materials has a purpose to produce good and reliable product with an easy processing and a low cost operation.

Refractory is one of the ceramic varieties which have the ability to withstand its physical and chemical condition on a relatively high temperature. Because of its ability, refractory material suits the industrial factory condition on the utilization of furnace or melting-pot, as widely seen on metal-casting industry. The main function of this refractory material in metal-casting is to insulate the high-temperature product from the outside environment or as a container to place the melted product. Therefore, the knowledge of refractory material is necessary in metal-casting industry.

As the widely application of pyro-proccessing in metal industries, the needs of refractory is highly demanded. Important knowledge in refractory material is needed by the industries to select the most appropriate type of insulating and/or lining material for their foundries. A good selection of refractory material may affect the usage a high-temperature unit to keep working efficiently. Based on these needs, researches in this material are needed to fulfill the demand of this material to supply the developing metal industries nowadays.

Main objective of this thesis is to characterize the physical properties of Alumina (Al_2O_3) based refractory applied on induction-furnace lining, which are: to find out the size and shape of refractory grains; to finds the main chemical composition of refractory; to finds the optical microstructure of refractory; to measures the bulk density of refractory.

2. MATERIALS AND METHODOLOGY

2.1. Alumina Refractory

Primarily, refractories are classified as basic, high-alumina, silica, fireclay and insulating. There are also classes of “special refractories” which include silicon carbide, graphite, zircon, zirconia, fused cast and several others. Most refractory materials are supplied as preformed shapes. However, they also are manufactured in the form of special purpose clays, bonding mortars, and monolithics, such as hydraulic setting castables, plastic refractories, ramming mixes and gunning mixes. A variety of processed refractory grains and powders are also available for certain applications [1].

Base material used in this research is a high-alumina powder refractory (with 87% alumina content) which commonly used in metal-casting induction furnace. Specimens are formed using hollow-cylindrical dies with d = 2.78 cm and t = 8 cm. Specimens are formed using hollow-cylindrical dies with d = 2.78 cm and t = 8 cm. Sintering are executed up to temperature 1000°C, 1300°C, 1500°C. Test on material: meshing, XRD (X-Ray Diffraction), optical microscope, and bulk density. Chemical reactions during testing process are not discussed. Heat transfers during testing process are not discussed.

The coreless induction furnace is a refractory lined vessel with electrical current carrying coils surrounding the refractory crucible. A metallic charge consisting of scrap, pig iron and ferroalloys are typically melted in this vessel. The coreless induction furnace is used when a quick melt of one alloy is desirable, or it is necessary to vary alloys frequently. The coreless furnace may be completely emptied and restarted easily, makes it perfect for one-shift operations [2].

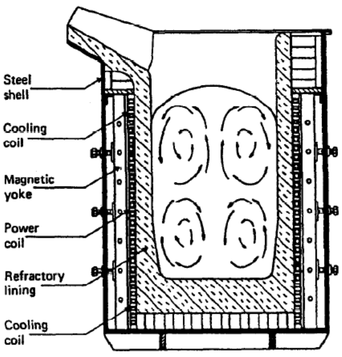


Figure 1. Schematic of induction furnace (coreless)

Metal casting company PT Suyuti Sido Maju, Ceper, Klaten using Induction furnace coreless type to smelt steels, as in Figure 1. The specification of the furnace are shown below:

- Type : coreless
- Furnace height : 75-80 cm
- Furnace diameter : 50 cm
- Electric Current : 350-400 kVA
- Frequency : 50-1000 Hz
- Refractory Thickness
  Side : 10 cm
  Bottom side : 15-20 cm
- Furnace capacity : ± 1000 kg
- Application : steel melting, iron casting.

Alumina (Al_2O_3) is one of the ceramic material which has an important role in casting industry today. Alumina (Al_2O_3) has the physical stability such as melting temperature, hardness, resistance to abrasion, high mechanical strength and its polimorph characteristic, that is the material has the same chemical composition but have a different crystal structure which is alpha α- Al_2O_3 and γ- Al_2O_3. Alumina melting point is at a temperature of 2054^0C. Alumina possessed a high strength due to the strong chemical bond between aluminum ions and oxygen ions in the Al_2O_3 structure. Mechanical strength of alumina at room temperature is very high, but the strength decreases at a temperature of 800^0C-1100^0C. This is because alumina has a small thermal coefficient however other limitations that have a low fracture toughness value ranging from 4,18 MPa- 5,9 MPa. Al_2O_3 have a stable chemical properties and a high corrosion resistance.

In the metal casting industry, alumina is used for furnace linings or other installations such as CCM (Continuous Casting Machine), ladle, funnel and rotary kiln. High alumina refractory is classified based on the content percentage of alumina, 50%, 60%, 70%, 80%, 85%, 90% and 99%. Other types of high alumina refractories are mullite, alumina-spinel, alumina-chrome and alumina-carbon products.

Corundum

Based on the forming process, alumina derived from pure material containing bauxite and andalusite. The bauxite ore is a major source of high alumina refractory material, bauxite containing gibbsite, ɣ-$Al(OH)_3$. Dominating mineral phase is corundum and mullite. Corundum (α-Al_2O_3) is a stable phase of alumina at various temperature. In the form of crystalline structure, corundum is called sapphire. Solid polycrystalline alumina formed from alumina powder after sintering. Corundum has a very high melting temperature if the refractories containing alumina more than 80%. Corundum will form its liquid phase at temperature of 2020^0C. Sintering method is basically to form a ceramic powder into specimen, dry it at room temperature (110^0C) then heat it at calcining temperature (900^0C -1100^0C), and burn the specimen at a relatively high temperature (for alumina is above 1.400^0C). [3]

Alumina-Silica (mullite)

Mullite is classified as special refractories because its two forming compounds Al_2O_3 and SiO_2. The chemical formula of mullite is $3Al_2O_3.2SiO_2$ with considerable alumina content between 60% -78% and a silica content of about 28.4% [4]. Mullite has a melting point at a temperature of 1850^0C. Content of alumina in mullite will determine many factors such as sintering temperature, heating time, particle size and others. Basically, high alumina refractories with silica content of more than 20% will decrease the strength of refractory at high temperatures because the silica content will erode the surface of alumina.

Alumina-Spinel

Basically the content of alumina-spinel is almost similar with the content of MgO-Magnesium Alumina (MA). Al_2O_3-spinel is melt at a high melting temperature so that it is considered having a high refractoriness. Therefore, alumina-spinel refractory usually has better thermal shock resistence than MgO-

MA. Similar with MgO-MA refractories, refractory Al_2O_3-spinel is formed into bricks or monolithic castable. In recent years, intensive research and development on alumina-spinel refractory castable has been used in many applications such as steel ladles, furnace linings, etc. The effect of adding spinel to the physical properties of spinel-alumina refractory castable is affecting its mechanical strength. A study conducted by experts in the field of refractory states that, alumina-spinel with 15 wt% spinel content, 15 wt% CA cement, and 70 wt% alumina and do sintering at temperatures 1.400^{0}C or 1.500^{0}C has a higher strength than alumina castable refractories without the addition of spinel. The increasing value of mechanical strength of alumina-spinel castable influenced by the addition of spinel and the reaction of CaO to Al_2O_3 cement binder.

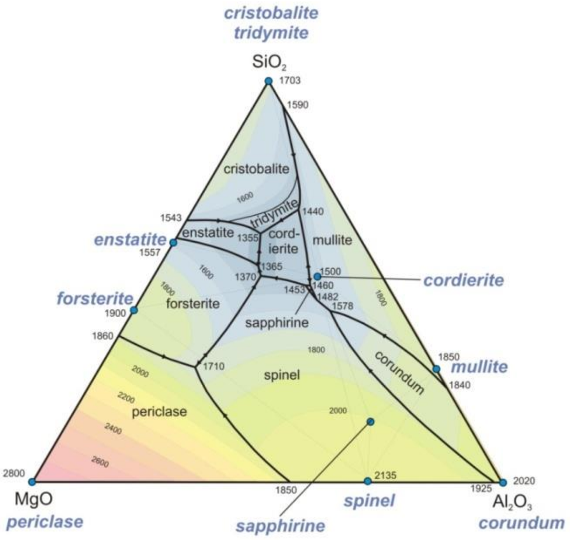


Figure 2. Ternary Phase Diagram of $MgO\text{-}Al_2O_3\text{-}SiO_2$

2.2. Methodology

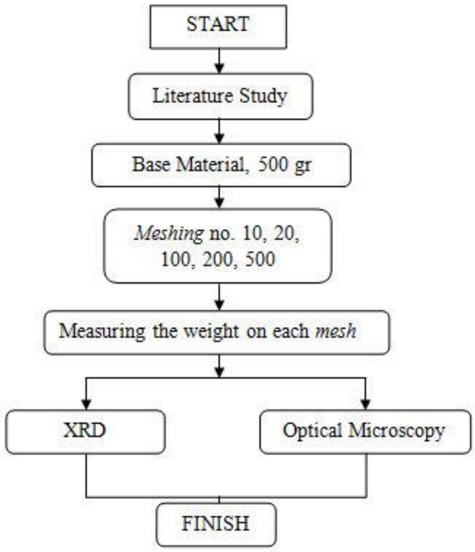


Figure 3. Flowchart diagram of Base Material.

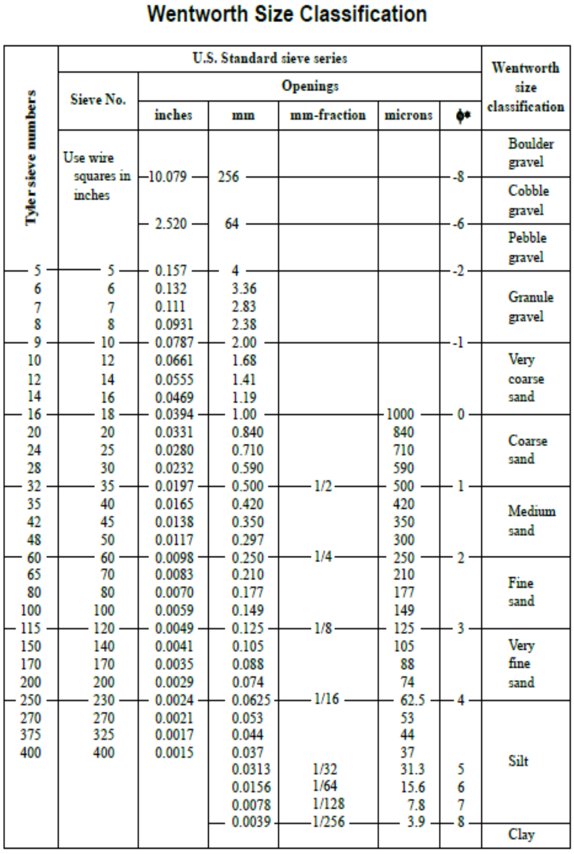
500 grams of Alumina (Al_2O_3) powder material is sieved using a mesh tool to separate big aggregates to the smaller one. Each of the following aggregates is observed to find out the size and the structure of the material. During this preparation process, make sure the equipments are well-cleaned to prevent foreign materials present on basic material. Take the sieved material sufficiently for further experiment.

The size of the grains plays a major influence on the strength and mechanical properties of refractory. Small and finer grains will increase the strength and toughness due to its increase of density at certain temperature and lowering its porosity.

Roundness is the measure of the sharpness of a particle's edges and corners of the grains or defined as the degree of smoothing due to abrasion of sedimentary particles. It is expressed as the ratio of the average radius of curvature of the edges or corners to the radius of curvature of the maximum inscribed sphere. Roundness can be seen in many variations of rock forms. The following comparison are:

a. Wellrounded: All surfaces are convex, mostly equidimensional, spheriodal.
b. Rounded: Generally rounded surfaces, rounded edge.
c. Subrounded: Generally flat surface with rounded ends.
d. Angular: Concave surface with sharp edges

Table 1. Wentworth Size Classification [6]



Wentworth Size Classification

Tyler sieve numbers	U.S. Standard sieve series						Wentworth size classification
	Sieve No.	Openings					
		inches	mm	mm-fraction	microns	ϕ*	
	Use wire squares in inches						Boulder gravel
		10.079	256			-8	Cobble gravel
		2.520	64			-6	Pebble gravel
5	5	0.157	4			-2	Granule gravel
6	6	0.132	3.36				
7	7	0.111	2.83				
8	8	0.0931	2.38				
9	10	0.0787	2.00			-1	Very coarse sand
10	12	0.0661	1.68				
12	14	0.0555	1.41				
14	16	0.0469	1.19				
16	18	0.0394	1.00		1000	0	Coarse sand
20	20	0.0331	0.840		840		
24	25	0.0280	0.710		710		
28	30	0.0232	0.590		590		
32	35	0.0197	0.500	1/2	500	1	Medium sand
35	40	0.0165	0.420		420		
42	45	0.0138	0.350		350		
48	50	0.0117	0.297		300		
60	60	0.0098	0.250	1/4	250	2	Fine sand
65	70	0.0083	0.210		210		
80	80	0.0070	0.177		177		
100	100	0.0059	0.149		149		
115	120	0.0049	0.125	1/8	125	3	Very fine sand
150	140	0.0041	0.105		105		
170	170	0.0035	0.088		88		
200	200	0.0029	0.074		74		
250	230	0.0024	0.0625	1/16	62.5	4	Silt
270	270	0.0021	0.053		53		
375	325	0.0017	0.044		44		
400	400	0.0015	0.037		37		
			0.0313	1/32	31.3	5	
			0.0156	1/64	15.6	6	
			0.0078	1/128	7.8	7	
			0.0039	1/256	3.9	8	Clay

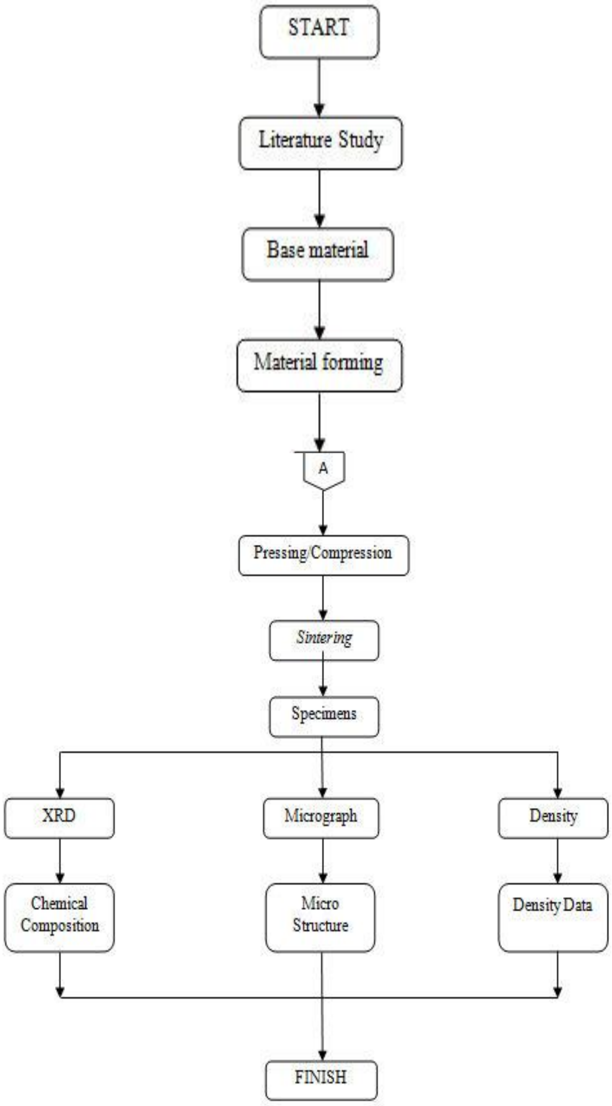


Figure 4. Flowchart diagram of Sintered Specimens

Compressed specimen is heated in a furnace up to different temperatures (1000°C; 1300°C; 1500°C). In the sintering operation, the pressed powder compacts are heated in a controlled-atmosphere environment to a temperature below the melting point but high enough to permit solid-state diffusion, and held for sufficient time to permit bonding of the particles. Most metals are sintered at temperature of 70 to 80% of their melting points, while certain refractory materials may require temperatures near 90%. When the product is composed of more components, the sintering temperature may even be above the melting temperature of one or more components. The lower-melting-point materials then melt and flow into the voids between the remaining particles.

Most sintering operations involve three stages, and many sintering furnaces employ three corresponding zones. The first operation, the burn-off or purge, is designed to combust any air, volatilize and remove lubricants or binders that would interfere with good bonding, and slowly raise the temperature of the compacts in a controlled manner. Rapid heating would produce a high internal pressure from air entrapped in close pores and will result in swelling or fracture of the compacts. When the compacts contain appreciable quantities of volatile materials, their removal increase the porosity and permeability of the pressed shape. The second, or high-temperature stage is where the desired solid-state diffusion and bonding between the powder particles takes place. The time must be sufficient to produce the desired density and final properties. Finally, a cooling period is required to lower the temperature of the products while maintaining them in a controlled atmosphere. This feature serves to prevent both oxidation that would occur upon directing discharge into the air and possible thermal shock from rapid cooling [5]. All three stages of sintering must be conducted in a protective atmosphere. At elevated temperatures, rapid oxidation would occur and significantly impair the quality of interparticle bonding.

The most widespread use of x-ray powder diffraction is for the identification of crystalline compounds by their diffraction pattern. Some specific uses of x-ray diffraction are to identify single-phase materials – minerals, chemical compounds, ceramics or other engineered materials. To identify multiple phases in microcrystalline mixtures (i.e., rocks). To determine the crystal structure of identified materials. Identification and structural analysis of clay minerals Recognition of amorphous materials in partially crystalline mixtures [7].

Bulk Density is the ratio of weight (or mass) to volume and it is expressed in pound per cubic foot (lb/ft^3) or grams per cubic centimeter (g/cm^3). The density of refractories is an indirect measurement of their heat capacity or ability to store heat. This is particularly important in applications such as regenerator installations.

The bulk density is generally considered in conjunction with apparent porosity. For many refractories, the bulk density provides a general indication of the product quality; it is considered that the refractory with higher bulk density (low porosity) will be better in quality. An increase in bulk density increases the volume stability, the heat capacity, as well as the resistance to abrasion and slag penetration.

The value of Bulk Density can be measured by using this formulation below [8]:

$$B = \frac{d}{l \text{ x } w \text{ x } t}$$

B = bulk density (gr/cm^3)
d = dry weight (gr)
l = length (mm)
w = width (mm)
t = thickness (mm)

3. RESULTS AND DISCUSSIONS

3.1. Size and Shape characterization

Table 2. Base Material under-sieved

Sieve No	***Opening (mm)***	**Weight (gram)**	**Percentage (%)**	**Aggregate**
10	2	96,25	19,25	Coarse
20	0,85	71,25	14,25	Coarse
100	0,15	188,85	37,77	Fine
200	0,075	46,65	9,33	Fine
500	0,025	26,45	5,29	Fine
< 500	<0,025	61,45	12,29	Fine
	Total	490,90		

We can find out the grain size of the material by sieving the powder material. From the data shown at Table 2, we can classify the shape and size of the grains based on its meshing number (10, 20, 100, 200 and 500). To determine the class of the grains on each meshing number, a classification table is needed. This study used the Wenthworth Size Classification as the reference.

A total 500 grams of base material was taken as sample than sieved using From the table above we can determine the grain size of the alumina powder. Small aggregates are dominating with 65,88% and large aggregates with 34,12%.

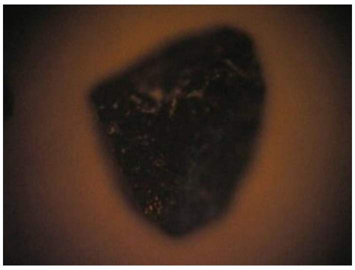


Figure 5. Grain on *mesh* number 10

Figure 6. Grain on *mesh* number 20

Figure 7. Grain on *mesh* number 100

Shape of the grains can be divided based on its roundness starts from very-angular to well-rounded. In this study, large aggregates can be observed through it macrostructural image, whilst for small aggregates, shape of the grains can be observed through optical microscopy.

Due to limited scale of magnification of the optical microscope, small aggregates are difficult to be observed. Classification could be obtained only from the data of large aggregates. Grains sieved under meshing number 10 are generally classified into angular due to its concave surface with its sharp edge. Grains sieved under meshing number 20 are classified into sub-angular due to its high spherity and its sharp edges. Grains sieved under meshing number 100 are classified into angular because of the concave surfaces with sharp edges.

3.2 Refractory Composition

Base material used in this research is an alumina (Al_2O_3) based refractory applied in metal-casting induction furnace with its high alumina content and other composition.

Table 3. Composition of Base Material

Composition	**Weight (%)**
Al_2O_3	86,7%
MgO	8,1%
SiO_2	2,7%
TiO_2	1,5%
Other	1,0%
TOTAL	100%

Table 3 provides informations about the composition of base material from the manufacturer. This is product is registered with DRI-VIBE registered name for coreless induction furnace working linings, channel induction furnace inductors, as well as channel induction furnace throat and uppercase linings. Formulations are available for melting all grades of iron, steel, copper, and aluminum. Also used as working and safety linings in blast furnace troughs and runners. This type of material, based on its composition, is classified as high-alumina (Al_2O_3 > 60%) with a slight amount of magnesia which commonly called as alumina-spinel. This material are mostly used in steel and iron smelting with its relatively high temperature resistance and high melting point at approximate 1920^0C, good slag resistance and corrosion resistance.

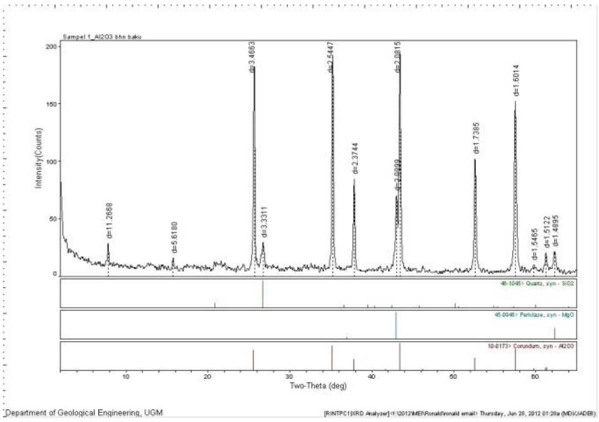


Figure 8. X-ray Diffraction of Base Material

Graphic result of X-ray difractometer is compared to the data in Table of Key Lines in X-ray Diffractometer Patterns of Minerals [appendix A]. The pattern shows that most dominant composition of the base refractory material is corundum (Al_2O_3), with a small amount of quartz (SiO_2) and periclase (MgO), as shown at Figure 10. Alumina content based on the refractory composition given by the manufactures is 86,7%, so it is considered as high-alumina refractory. Corundum has a very high melting temperature if the refractories containing alumina more than 80%. Corundum is a stable phase of alumina at various temperature. Content of alumina in the refractory material will determine factors in sintering process.

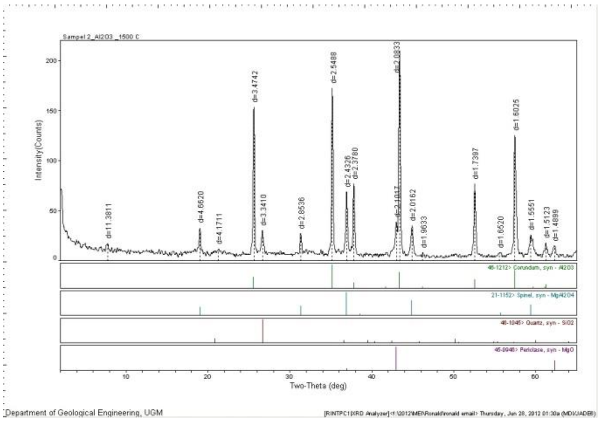


Figure 9. X-ray Diffraction of Sintered Specimen at 1.500^{0}C.

There is difference between the XRD pattern of the base material with the XRD pattern of the specimen sintered at 1.500^{0}C. The pattern shows that most dominant composition of the alumina refractory after sintering process at 1.500^{0}C is corundum (Al_2O_3), with a small amount of quartz (SiO_2), periclase (MgO) and a newly formed Spinel ($MgAl_2O_4$) added. Spinel is formed due to sintering process where magnesia (MgO) and alumina (Al_2O_3) unified. Spinel is considerably more resistant to slags and fluxes, especially alkalis, than pure alumina. The effect of adding spinel to the physical properties of the refractory is affecting its mechanical strength

The explanation above may indicate that the process of sintering in high-alumina refractory may formed new composition without affecting the main compound of the materials. Thus, it can be said that main composition of the materials is remained stable.

3.3. Bulk Density

Table 4. Density Value

No.	Temperature (^{0}C)	Density (g/cm^3)
1	110	2,92
2	1.000	2,66
3	1.300	2,80
4	1.500	2,98

From the table, density values at temperature 110^{0}C, 1000^{0}C, 1300^{0}C and 1500^{0}C in sequence are 2,92 g/cm^3; 2,66 g/cm^3; 2,80 g/cm^3; and 2,98 g/cm^3. This graphic shows the relationship between the increase of sintering temperature with the value of density. Temperature is expressed in Celsius and density in gram per cubic centimeter.

To remove its water content, specimens are heated at 110^{0}C. Specimens heated at 110^{0}C has an average density value of 2,92 g/cm^3. Compacting process during the making of the specimens affects this density value. Loose powder is compressed and densified into a shape to make a specimen. The compacts should posses sufficient strength to maintain the shape of the specimen when sintered at high temperature.

After heated at 110^{0}C, specimens were burnt at sintering temperature above 1000^{0}C. In this study, specimens are sintered at various temperature of 1000^{0}C, 1300^{0}C and 1500^{0}C. Density value is decreasing when the specimen is heated up to 1000^{0}C. This is a stage where the powder is melt and shrinked due to calcination and resulting in porosity reduction. Density at 1000^{0}C is 2,66 g/cm^3. The desired solid-state diffusion and bonding between the powder particles are not happened yet at this temperature. It is predicted due to a high melting point of alumina, so that a higher level of temperature needed to perform a sintering process.

From the graphic in Figure 12, density value is increasing at 1300^{0}C and 1500^{0}C. Density of the specimens at 1300^{0}C is 2,80 g/cm^3 and at 1500^{0}C is 2,98 g/cm^3. Density value is predicted to keep increasing up to temperature 1700^{0}C before reaching its melting point at 2000^{0}C (high-alumina).

Compaction process densifies the grains, but the desired solid-state diffusion and bonding between the powder particles occurs when material is sintered at high temperature. Sintering process plays role in the binding process between the grains where bonding process between grains is increasing due to heat treatment. The dimension shrinkage will occur accompanied by a porosity reduction, then the density will increase, therefore the material which been through a sintering process will having more strength and more solid material.

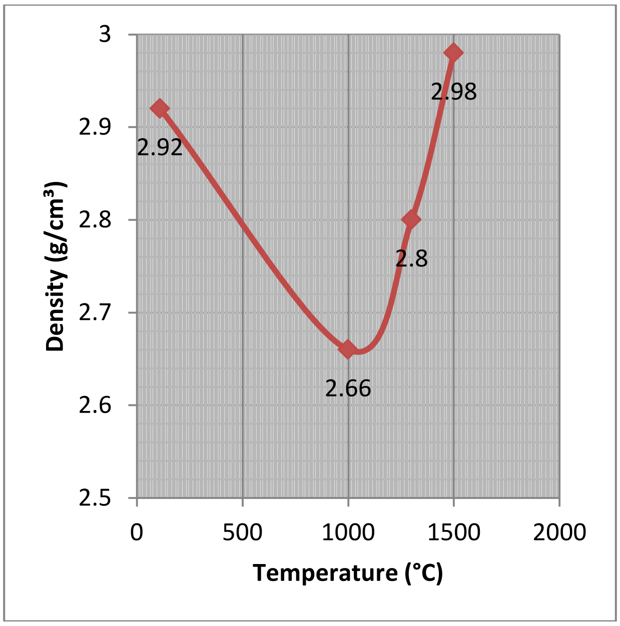


Figure 10. Graphic correlation of temperature and density of alumina refractory.

3.4 Microstructure of Refractory

During the sintering operation, a number of changes occur in the structure of the material. Metallurgical bonds form between the powder particle as a result of solid-state atomic diffusion occurs. In addition, the bond formation results in increase of density value. By observing the microstructure of the specimens, sintering process of the material can be seen. The figures show the microstructures of the speciemens heated at 110^0C, 1000^0C, 1300^0C and 1500^0C.

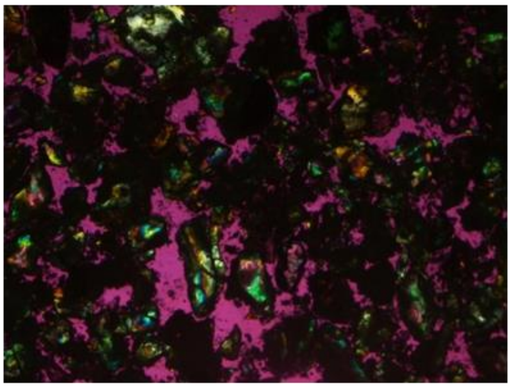


Figure 11. Microstructure of Heated Specimen at 110^0C

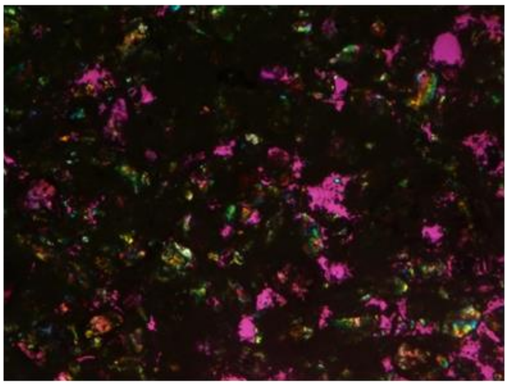


Figure 12. Microstructure of Sintered Specimen at 1000^0C

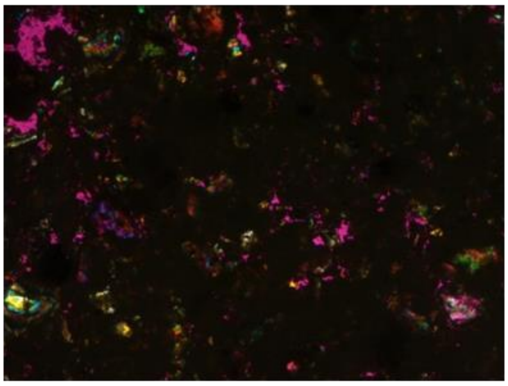


Figure 13. Microstructure of Sintered Specimen at 1300^0C

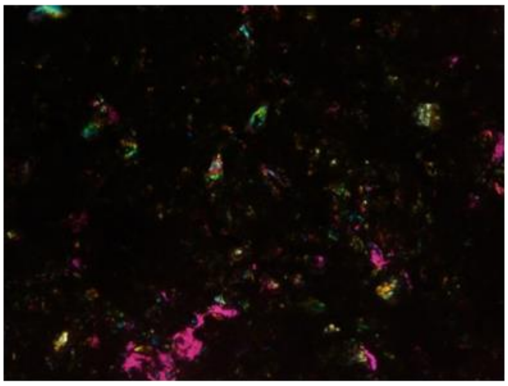


Figure 14. Microstructure of Sintered Specimen at 1500^oC

From those figures above, it can be seen that the increase of the sintering temperature affects the density of the refractory material at the temperature of 110^0C, 1000^0C, 1300^0C and 1500^0C. Purple color in the microstructure images shows the porosity of the material and dark color shows the density of the material. As can be seen on the picture, as the increase of temperature, the microstructure of the materials is getting darker. It proofs that the density value is increasing along with the increase of temperature.

4. CONCLUSIONS

From the X-ray Diffraction Test compared with specification data from the manufacturer, the main composition of the base refractory material is alumina (Al_2O_3) with a low amount (less than 10%) of magnesia (MgO) and silica (SiO_2). Alumina refractory material consists of coarse grains (large aggregates) with its size larger than 0,85 mm and fine grains (small aggregates) with its size equals to 0,15 mm or smaller. Grains shape of the aggregates are mostly angular with its sharp edges. Values of density at temperature 110^0C, 1000^0C, 1300^0C, 1500^0C in sequence are 2,92 g/cm^3; 2,66 g/cm^3; 2,80 g/cm^3; 2,98 g/cm^3. Density of the refractory will increase as the increase of sintering temperature.

Microstructure of the refractory at different temperature shows that the grains are getting darker as the increase of the temperature. This is the proof where atomic-diffusion occurs and density of the material increases.

Keep the base material on its good condition to maintain the powder quality of refractories when being treated during the study.

5. REFERENCES

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