# **OPTIMIZING THE COMPOSITION OF BASALT AND HEAT TREATMENT OF FLY ASH-BASED MULLITE CERAMICS USING THE TAGUCHI METHOD**

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# **ABSTRACT**

The development of industrial activities impacts the increase in waste produced, and fly ash, aluminum dross, and basalt dust are no exception. This research studies the potential of these three materials as ceramic materials. Basalt dust from East Lampung was used as filler. The effect on the physical and mechanical properties of mullite  $(3Al_2O_3-2SiO_2)$  ceramics was studied. The manufacture of mullite ceramics based on Taguchi design includes fly ash compositions of 40%, 50%, and 60% and basalt content of 0%, 5%, and 10%. sintering temperatures of 600°C, 900°C, and 1,200°C. Taguchi and Anova were used to determine the effect of independent variables on hardness and density. In addition, macro and microphoto tests were carried out to determine mullite ceramics' physical and topographic changes. Chemical composition tests with X-ray fluorescence were carried out on raw materials and ceramics that have been formed. Changes in the crystal phase in mullite ceramics were studied through the Xray diffragment test. XRF test results obtained for raw fly ash  $(SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>)$ : 84.84 wt%. The composition of raw basalt is predominantly  $48.42\%$  SiO<sub>2</sub>,  $18.82\%$  Al<sub>2</sub>O<sub>3</sub>, 12.60%  $Fe<sub>2</sub>O<sub>3</sub>$ , and raw aluminum dross with 67.821%  $Al<sub>2</sub>O<sub>3</sub>$  content. The mullite ceramic specimen consists of 38.24–45.30% SiO<sub>2</sub>, 34.72–48.73% Al<sub>2</sub>O<sub>3</sub>, 6.3–9.99% Fe<sub>2</sub>O<sub>3</sub>, and 2.31–5.31% CaO. The crystal phases formed are mullite, pyroxene, and diopside. Analysis of variance shows that sintering temperature significantly affects hardness, with a P-value of 0.013 and a contribution of 93.77%. This modeling is acceptable with an error value of 1.26%, or R-sq: 98.74%. Adding basalt increases mullite ceramics' density, with a P-value of 0.033 and a contribution of 96.05%. Adding basalt as a filler cannot increase the hardness significantly, but it affects the higher ceramic density value. However, mullite formation is interesting to study further as a refractory material.

Keywords: Fly Ash; Aluminum Dross; Basalt; Ceramics; Mullite; Hardness.

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#### **INTRODUCTION**

The rapid development of industry in Indonesia has led to an increase in the amount of waste generated. The accumulation of industrial waste will cause environmental damage and threaten public health  $\left[1\right]$ . The continued dependence of state electricity companies on coal raw materials has left fly ash (FA). Although FA is no longer hazardous waste, its utilization is still limited. Each coal combustion process produces 5% solid pollutants, consisting of 80–90% fly ash and 10–20% bottom ash  $^{[2]}$ . This is also the case in the aluminum industry,

where every ton of primary aluminum processing produces at least 200 to 450 kg of aluminum dross. It is estimated that 100 tons of aluminum waste are discharged into the surrounding environment without further treatment [3].

Previous research has successfully used aluminum and fly ash waste to produce mullite ceramics. Research conducted by Foo et al. (2019) on the effect of sintering temperature, acid dissolution, and the  $Al_2O_3/SiO_3$  ratio on the properties of mullite ceramics has been investigated. In this study, the sintering process was carried out at high temperatures, namely 1200  $\degree$ C and 1500  $\degree$ C <sup>[4]</sup>. However, this research does not study the mechanical properties of the resulting ceramics. Physical and mechanical properties are important characteristics of mullite ceramics. Romero et al. (2021) reviewed the production of mullite ceramics using mining waste materials, such as waste rock  $[5]$ . However, this article has not reviewed the effect of basalt addition on the characteristics of mullite ceramics.

Basalt rocks in Lampung Province are scattered around the west side of Mount Tanggamus, through the bay around Semangko, eastwards to Mount Rajabasa, and the highland basalt area in Sukadana, East Lampung [6]. This basalt is resistant to chemical reactions, can withstand high temperatures, and can increase the hardness of composites <sup>[7]</sup>. Currently, basalt is only used as an ornamental rock or foundation stone. This activity of breaking basalt into smaller sizes produces basalt dust, which is only used as a road pavement material and asphalt mixture. Not many have examined the potential of basalt dust in manufacturing mullite ceramics. This study studied the effect of basalt dust composition and FA on hardness, density, chemical composition, and crystal phases formed in mullite ceramics. The experimental design used Taguchi orthogonal array L9 with the help of Minitab 17 software. Basalt composition and sintering temperature parameters were optimized using Taguchi analysis and analysis of variance (Anova).

#### **METHOD**

#### **Materials**

Mullite ceramics are made using aluminum dross, fly ash, and basalt rock from Sukadana, Lampung Province, Indonesia, as shown in Figure 1.



**Figure 1.** (a). Aluminum Dross, (b). Fly Ash, (c). Basalt Lampung Timur – Indonesia

The mullite ceramic material is composed of aluminum dross, fly ash, and 325 μm-sized basalt powder, which is leached with 2M HCl at a speed of 300 rpm for 1 hour. The sintering process is carried out at temperatures of 600℃, 900℃, and 1,200℃ for 4 hours. The numbering of specimens and experimental parameters are shown in Table 1.

<b>Tuble 1:</b> The taggern design of experiment to produce munite certainles	Level Values				
Name					
FA Content (wt%)		50	60		
Basalt Content (wt%)					
Sintering Temperature $^{\circ}C$ )	600	900	$-200$		

**Table 1.** The taguchi design of experiment to produce mullite ceramics

#### **Characterization**

The X-Ray Fluorescence (XRF) characterization uses the XRF Epsilon 4 XRF Spectrometer from Malvern Panalytical. X-Ray Fluorescence is one of the methods used to analyze the elemental composition of a material or sample by irradiating it with X-rays, which are absorbed and reflected by the material. The X-Ray Diffraction characterization is performed using the PAN analytical X'Pert3 Powder instrument. X-Ray Diffraction is a tool used to examine the crystal structure of fine materials or substances. This method can analyze specific types and properties of minerals by observing the diffraction patterns produced by minerals. The analysis method used is the application of the High Score Plus Crystallography Open Database  $(COD)$ <sup>[8]</sup>. The Scanning Electron Microscope is characterized using the SEM Phenom Pro X instrument with magnification of up to 150,000 times.

# **Hardness Testing HV**

Hardness testing uses a Vickers instrument of the Zwick Roell Emco Test brand. The equation to obtain the Vickers hardness value is shown in Equation 1.

$$
VHN = \frac{(1,854)P}{D^2}
$$
 (1)

Where VHN is Vickers hardness (Hv), P is the load (N), and D is the indenter diameter (mm). Hardness results are analyzed using Taguchi analysis and ANOVA software Minitab. Taguchi is used to analyze the influence of parameters on hardness value. Meanwhile, ANOVA determines significant influence with P-Value  $< \alpha = 5\%$ .

#### **RESULTS AND DISCUSSION**

Mullite ceramics are characterized by shades of gray, light brown, and dark brown. The color characteristics are influenced by the sintering temperature and the percentage of added basalt. At a basalt percentage of 10% and a sintering temperature of 1,200℃, the ceramics exhibit a dark brown color, while a basalt percentage of 5% results in a light brown color. Ceramics without added basalt have a gray color, as shown in Figure 2.



**Figure 2.** Mullite ceramics based on basalt rock from East Lampung-Indonesia

#### **The Chemical Content of Raw Materials**

The chemical analysis of the raw materials used to make mullite ceramics reveals that the dominant elements are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, and CaO, as indicated in Table 2.





Based on the XRF test, it was found that the composition of FA consists of a total of 84.84%  $(SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>)$ , and 8.602% CaO. This FA is F type which is characterizsed by  $SiO_2 + Al_2O_3 + Fe_2O_3$  content > 70% and a little CaO<sup>[9]</sup>. Aluminum dross as a raw material has a dominant chemical composition of 17.284%  $SiO_2$ , 67.821%  $Al_2O_3$ , 5.81% Fe<sub>2</sub>O<sub>3</sub>, and 3.364% CaO. Furthermore, basalt as a raw material has a dominant chemical composition of 48.418%  $SiO_2$ , 18.82%  $Al_2O_3$ , 12.595% Fe<sub>2</sub>O<sub>3</sub>, and 9.761% CaO. SiO<sub>2</sub> plays a role as a silicon source in the formation of mullite ceramics. When  $SiO<sub>2</sub>$  is heated at high temperatures, it reacts with  $A_1Q_3$  to form the mullite phase  $(A_1Q_3 \cdot 2SiO_2)$ , which is the main component of mullite ceramics  $[10]$ . SiO<sub>2</sub> also helps improve the mechanical properties, thermal stability, and corrosion resistance of mullite ceramics.  $Al_2O_3$  serves as an aluminum source in the formation of mullite ceramics. Aluminum is a key element in mullite and enhances the mechanical and thermal properties of mullite ceramics. Additionally,  $A1_2O_3$ plays a role in forming a stable mullite phase, controlling the size and distribution of the mullite phase within the ceramic structure  $^{[11]}$ . As for Fe<sub>2</sub>O<sub>3</sub>, although it is undesirable in large amounts in forming mullite ceramics, it can function as an additive in limited quantities.  $Fe<sub>2</sub>O<sub>3</sub>$  can help reduce firing temperature and improve the mechanical properties of mullite ceramics, but it must be controlled appropriately to avoid the formation of undesired phases in the ceramic structure [12].

#### **The Chemical Content of Mullite Ceramics**

The dominant chemical elements found in mullite ceramics are  $SiO<sub>2</sub>$ ,  $Al<sub>2</sub>O<sub>3</sub>$ ,  $Fe<sub>2</sub>O<sub>3</sub>$ ,  $CuO$ , and CaO, as shown in Table 3.

Comp. (% )	Specimen									
	1	$\overline{2}$	3	$\overline{4}$	5	6	7	8	9	
SiO <sub>2</sub>	38,23	40,15	39,81	38,79	41,01	42,47	43,19	42,72	45,29	
$Al_2O_3$	48,73	43,43	40,43	44,32	38,82	38,49	37,08	36,76	34,71	
TiO <sub>2</sub>	0,97	1,18	1,34	1,19	1,39	1,35	1,32	1,38	1,47	
Fe <sub>2</sub> O <sub>3</sub>	6,30	7,50	9,53	7,68	9,99	8,97	10,04	9,64	9,86	
CuO	1,18	1,32	1,42	1,52	1,27	1,12	1,05	0,80	0,73	
CaO	2,30	3,75	4,64	3,89	4,78	4,64	4,21	5,30	5,23	
MnO	0,17	0,18	0,23	0,19	0,22	0,17	0,20	0,18	0,17	
ZnO	0,10	0,14	$\overline{\phantom{a}}$	0,14	$\overline{\phantom{0}}$	0,11	$\overline{\phantom{0}}$	0,12	0,10	
K <sub>2</sub> O	0,72	0.88	0,88	0,84	0,99	0,90	0,99	0,95	1,03	
$P_2O_5$	0.70	0,75	0,77	0.76	0,73	0.73	0.75	0.73	0.72	

**Table 3.** The composition of oxide in mullite ceramics

The chemical composition of mullite ceramics is presented in Table 2. The chemical content of mullite ceramics with basalt after the sintering process shows significant changes in the dominant elements  $SiO<sub>2</sub>$  and  $Al<sub>2</sub>O<sub>3</sub>$  among different specimens, according to the composition of the base materials. The percentage increase or decrease in the chemical content of  $SiO<sub>2</sub>$  and  $Al<sub>2</sub>O<sub>3</sub>$  is influenced by the composition of the base materials used in the production of mullite ceramics. In specimens 1, 2, and 3, the dominant base material composition is aluminum dross, resulting in a dominant composition of Al<sub>2</sub>O<sub>3</sub>. Furthermore, in specimen 4, the base material composition is a combination of fly ash and aluminum dross, resulting in a dominant value of  $A<sub>2</sub>O<sub>3</sub>$ . Subsequently, specimens 5 to 9 have a dominant base material composition of fly ash and basalt, resulting in a dominant composition of SiO<sub>2</sub>. The content of Al<sub>2</sub>O<sub>3</sub> ranges from  $34.715\%$  to  $48.731\%$ . The compositions of  $SiO<sub>2</sub>$  and  $Al<sub>2</sub>O<sub>3</sub>$  are essential elements in the formation of mullite ceramics <sup>[13]</sup>. Meanwhile, the SiO<sub>2</sub> content ranges from 38.235% to 45.295%, which is a crucial factor during melting and casting processes.  $SiO<sub>2</sub>$  content below 50% indicates lower melt viscosity compared to values above 50%, which indicate higher melt viscosity than those below 50%.

#### **The Crystall Phase of Mullite Ceramics**

The comparison of X-ray diffraction  $(X-RD)$  phases of mullite ceramics  $(1-9)$  is show in Figure 3.



**Figure 3.** Mullite ceramics based on basalt rock from East Lampung-Indonesia

Mullite ceramics consist of alumina  $(A<sub>2</sub>O<sub>3</sub>)$  and silica  $(S<sub>i</sub>O<sub>2</sub>)$  and are commonly used in industrial applications due to their high temperature resistance and corrosion resistance. A phase is a solid crystal formed from the combination of alumina and silica, with a stable and strong crystal structure. According to Chen et al (2020), the phase is formed in mullite ceramics through a high-temperature firing process <sup>[13]</sup>. During the firing process, alumina and silica react to form mullite phase with a stable crystal structure. The dominant mullite phase in ceramics exhibits strong mechanical properties, abrasion resistance, and resistance to corrosion and high temperatures. Therefore, mullite ceramics with mullite phase are often used in applications that require these properties, such as in the refractory industry, cement industry, and catalyst manufacturing. In the journal  $[14]$ , it is also mentioned that factors such as alumina-silica ratio, firing temperature, firing time, and additives can influence the formation of mullite phase in ceramics. In this study, a dominant mullite phase was obtained at a temperature of 1,200 ℃, which affected the mechanical properties of the material, resulting in higher hardness.

The quartz phase in mullite ceramics is a solid crystalline phase formed from silicon dioxide  $(SiO<sub>2</sub>)$  in the mullite crystal structure. Mullite is a ceramic compound consisting of alumina  $(Al<sub>2</sub>O<sub>3</sub>)$  and silicon dioxide  $(SiO<sub>2</sub>)$  in a molar ratio of 3:2. The quartz phase in mullite ceramics has significant importance as it can affect the mechanical, thermal, and chemical properties of mullite ceramics. The presence of quartz phase can cause pores and cracks in ceramics, which can reduce their mechanical strength and thermal resistance. However, if the content of quartz phase remains low, its influence can be neglected [15].

Furthermore, the presence of diopside phase with its main function in mullite ceramics is to enhance strength and thermal resistance [16]. Diopside has a relatively low coefficient of thermal expansion, which can help reduce stress and cracks in ceramics when subjected to temperature changes. This is important because rapid or extreme temperature changes can cause cracking and damage to ceramics. Moreover, the diopside phase can also improve the dimensional stability of mullite ceramics. When exposed to high temperatures, ceramics tend to undergo significant volume changes. The addition of diopside phase helps control these dimensional changes, allowing the ceramics to maintain their shape and integrity. The diopside phase can also influence the mechanical and thermal properties of mullite ceramics. Diopside mineral has high hardness, which can enhance the strength of ceramics. However, if the content of diopside phase remains low and forms at low temperatures, its influence can be neglected <sup>[17]</sup>. Additionally, diopside also has good thermal conductivity, which aids in regulating heat distribution within the ceramics.

In mullite ceramic materials, there is also a labradorite phase that can help improve the strength and wear resistance of mullite ceramics. With the presence of the labradorite phase, mullite ceramics can exhibit better mechanical properties, including resistance to pressure, fracture, and deformation. The labradorite phase contributes to the increase in the hardness of mullite ceramics. The existence of labradorite helps fill the intergranular gaps of mullite, thereby increasing the overall density and hardness of the ceramics <sup>[18]</sup>. Furthermore, the labradorite phase has a relatively high coefficient of thermal expansion compared to mullite. This can help reduce the thermal expansion mismatch between the labradorite and mullite phases, thereby reducing the potential for cracking and thermal damage in mullite ceramics during rapid or extreme temperature changes <sup>[19]</sup>. The labradorite phase can also enhance resistance to corrosion in mullite ceramics. Its presence can form a protective layer that inhibits the penetration of corrosive substances and protects mullite from chemical damage. In sample 1, there is a different phase compared to other samples, namely melilite, which has high hardness, thus it can increase the overall hardness of mullite ceramics but can be ignored if the content of melilite phase remains low  $[20]$ . In sample 2, there is also the appearance of a different phase compared to other samples, namely gahlenite, which has high temperature stability, good mechanical strength, and good chemical resistance [21]. Furthermore, in sample 5, a new phase emerges, namely cordierite, which has a stable tetragonal crystal structure at high temperatures. This crystal structure is characterized by the arrangement of magnesium (Mg), aluminum (Al), and silicon (Si) atoms in a regular crystal lattice. This phase has favorable properties for ceramics, such as high temperature resistance, resistance to rapid temperature changes, and resistance to corrosion [12].

The formation of pyroxene in basalt-based mullite ceramics shows an increase in mechanical and physical changes. Research comparing Enstatite-pyroxene basalt in its raw form with ceramic basalt has been conducted. Pyroxene basalt from Peg. Vrelo-Kopaonik, Serbia, was then processed to form mullite ceramics and subjected to cavitation erosion testing. Analysis of mass loss and surface erosion during cavitation processes showed different damage effects between raw basalt and mullite ceramics. Cavitation erosion of ceramic samples is characterized by small holes located in the basalt bubble structure and their number increases at low speeds during testing. Mass loss during testing indicates that mullite ceramics have lower mass loss compared to raw basalt, and the mass loss of ceramics tends to be constant and unaffected by the duration of testing. Ceramic samples show lower surface damage coverage compared to raw basalt samples. There is almost no change in dimension in the pits on the sample surface before testing. The profile lines of ceramic samples are uniform with individual peaks at the same locations on the sample surface. The presence of individual holes caused by bubbles in the structure is identified before testing  $[22]$ .

The crystal phase of pyroxene consists of augite and diopside. Both crystal phases provide high hardness to ceramics. The diopsidic-augite phase provides superior wear resistance and chemical resistance. It is explained that the formation of basalt-based mullite ceramics does not require nucleating agents due to the presence of FeO and  $Fe<sub>2</sub>O<sub>3</sub>$  oxides. It is stated that FeO and Fe<sub>2</sub>O<sub>3</sub> in basalt are oxidized to Fe<sub>3</sub>O<sub>4</sub>, which acts as a nucleating agent and the site of crystal growth formation. XRD analysis shows that higher crystallization temperatures result in longer diopsidic-augite peaks, indicating that increasing the heat treatment temperature leads to the formation of a large number of crystal phases. The higher the crystallization temperature and the longer the treatment time, the harder the basalt-based mullite ceramic layer becomes. The hardness of mullite ceramics slightly decreases due to deformation and grain growth of the crystal phase with increasing heat treatment temperature.

#### **The Microhardness (Hv) of Mullite Ceramics**

The highest hardness was obtained from the testing of specimen 7 with an average hardness value of 171.33 HV. This sample was obtained from the production of samples using the material parameters of 60% fly ash, 40% aluminum dross, 0% basalt, and sintering temperature of 1,200℃. Based on the highest hardness results obtained from sample 7, it can be inferred that the hardness value is influenced by the sintering temperature and the composition of mullite ceramics production. Sintering temperature refers to the temperature at which mullite ceramics are transformed into a solid and hard form through heating. A higher sintering temperature can result in larger mullite crystals, which can enhance the strength and hardness of the ceramic. However, excessively high sintering temperature can also lead to deformation or cracking of mullite ceramics. The base material used in the

production can also affect the hardness of mullite ceramics. The results of the analysis of variance for the hardness data using Minitab can be seen in Table 4.

Material imperfections are highly possible in experiments, but based on Table 4, the measurement accuracy is at 98.74%, indicating an error rate of 1.107%. Table 4 also shows a p-value of 0.013 for the temperature parameter, indicating that the temperature parameter significantly affects the hardness of mullite ceramics. The hardness of mullite ceramics tends to increase with the increase in sintering temperature  $[23]$ . Sample 7 exhibited the highest hardness with a sintering temperature of 1,200 ℃, while sample 6 had the lowest hardness with a sintering temperature of 600℃.

During the sintering process, mullite ceramics undergo shrinkage as the ceramic particles move and bind together. Higher sintering temperatures tend to result in greater shrinkage because ceramic particles become more active and bind more strongly. High shrinkage can produce a dense ceramic structure with low porosity, which in turn can increase the hardness of mullite ceramics [14].

Source			DF Adj SS Adj MS F-Value P-Value
FA and Al Wt 2 2,648 1,3242 2,39			0,295
<b>Basalt Wt</b>		2 1,712 0,8559 1,55	0,393
Temperatur		2 82,357 41,1785 74,37 0,013	
Error		2 1,107 0,5537	
Total	8 87,824		

**Table 4.** Analysis of Variance on the Influence of Parameters on Hardness (HV).

Sintering temperature also affects the microstructure of mullite ceramics, which refers to the arrangement and size of grains within the ceramic material. At a sintering temperature of 1,200 ℃, mullite ceramic particles tend to melt and join together, forming larger grains. Larger grains can result in longer grain boundaries and potentially reduce weaknesses in the ceramic structure, thereby increasing the hardness of mullite ceramics [11]. Sintering temperature affects the quantity and size of pores and cracks in mullite ceramics. At lower sintering temperatures, mullite ceramics tend to have more pores and cracks because the ceramic particles have not fully bonded to each other. Pores and cracks can provide easy paths for crack initiation and propagation, which can reduce the hardness of ceramics. However, at higher sintering temperatures, the number and size of pores and cracks tend to be lower and smaller because the ceramic particles have bonded well. This can enhance the hardness of mullite ceramics.

The results of the analysis of variance can be confirmed by the Taguchi analysis based on the Signal-to-Noise ratio larger is better, as shown in Figure 4 and Figure 5.



Figure 4. The mean value of mullite ceramic hardness.



**Figure 5.** The Signal to Noise Ratio of mullite ceramic hardness.

The best average hardness value was obtained at a temperature of 1,200°C with 0% basalt, which amounted to 171.33 Hv, while the lowest hardness value was at a temperature of 600°C with 10% basalt, which amounted to 19.36 Hv as shown in Figure 4. Based on the Signal to Noise ratio, larger is better, the highest hardness will be obtained from the process parameter of 1,200°C temperature with 0% basalt. The higher the temperature, the higher the hardness value. On the other hand, with the addition of basalt, the hardness of mullite ceramic decreases as shown in Figure 5.

#### **The Density of Mullite Ceramics**

Density testing was carried out using the simple principle of Archimedes' law, where the weight of the object per unit volume was measured based on the volume of water after the object was immersed in a tube filled with water. The density of basalt ceramics ranges from 2.1 to 3.8 g/cm<sup>3</sup>. According to Lima et al. (2020), the density of ceramics is influenced by the treatment temperature and crystallization formed in the ceramic phase. Mullite ceramics made from basalt rocks of the Serra Geral Formation from Brazil obtained a bulk density

value of 2.39 and 2.38  $g/cm^3$ , depending on the treatment of basal crystallization  $[24]$ . The analysis of density testing results using Analysis of Variance yielded results as shown in Table 5.

Source		DF Seq SS Contribution Adj SS		Adj MS F-Value P-Value		
FA wt%	2 0.00039	$0.04\%$	0.000387	0.000194	0.01	0.989
Basalt wt%	2 0.96530	96.05%	0.965296	0.482648	28.90	0.033
Sintr. Temp. oC $2 \quad 0.00588$		0.58%	0.005877	0.002939	0.18	0.850
Error	2 0.03340	3.32%	0.033396	0.016698		
Total	8 1.00496	100.00%				

**Table 5.** Analysis of Variance on the Influence of Parameters Used on Density.

In the analysis of variance, the percentage measurement error was 3.32%. Table 5 shows a P-value of 0.033 for basal content, which indicates that the parameter has a significant effect on density. While the parameters of FA concentration and sintering temperature did not have a significant effect because the P-value was above 5%, The results of the analysis of variance can be confirmed by Taguchi analysis based on the signal-to-noise ratio, which is greater the better, as shown in Figures 6 and 7.



**Figure 6.** The mean value of mullite ceramic density



**Figure 7.** The Signal to Noise Ratio of mullite ceramic density.

The Taguchi analysis uses the "smaller is better" equation with the aim of obtaining a material that is lightweight but still strong. As shown in Figure 6, the lowest average density was obtained with 60 wt% fly ash, 0 wt% basalt, and a sintering temperature of 1200  $\degree$ C, namely in specimen 7. Specimen 7 has a density of 1.63  $g/cm<sup>3</sup>$ . From Figure 7, it appears that the basalt concentration parameter has a significant impact on density. It is marked by a drastically decreasing graph, while the other two parameters, namely fly ash composition and sintering temperature, do not experience much change in value.

# **The Microstructure of Mullite Ceramics**

The microstructure of mullite ceramics was performed on solid (bulk) materials. There are differences in the topographical shape of mullite ceramics between sample 1 and other samples, as shown in Figure 8.



**Figure 8.** Microstructure Mullite Ceramics

In sample 8, there are bubbles of various sizes filled with bubbles, and these bubbles on the surface cause surface roughness. The sintering process of fly ash, aluminum dross, and basalt raw materials into mullite ceramics causes shock energy. Shock energy causes the bubbles in this liquid to melt and be absorbed by the contacting solid surface <sup>[22]</sup>. Dominasi Si dan Al dari ketiga bahan membentuk fasa mullite  $(AI_{4.76}Si_{1.24}O_{9.54})$  in each sample from 1 to 9. In sample 7, there is the highest peak of the Mullite phase  $(Al_{4.76}Si_{1.24}O_{9.54})$  because of the sintering temperature used.

# **CONCLUSION**

The optimal and significant contribution to hardness is sample 7 with a composition of 60% fly ash, 40% aluminum dross, and 0% basalt at a sintering temperature of 1,200 °C, resulting in a hardness value of 171.33 HV. Anova showed a correctness of 98.74% for the parameters used, indicating that the parameters used significantly affect the hardness of mullite ceramics because the error is less than 5%. The parameter that has a significant effect on mullite ceramics is temperature, with a p-value of 0.013. The fly ash content has a P-value of 0.295, and the basalt parameter has a p-value of 0.393. Mullite ceramics with the same treatment also produced the lowest density of  $1.63$  g/cm<sup>3</sup>. Therefore, this study has succeeded in making lightweight but hard mullite ceramics produced from sintering raw

materials of type F fly ash, aluminum dross, and basalt dust from Lampung Province, Indonesia.

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