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Effects of Ifon clay and sintering temperature on the phase evolution and physicomechanical properties of mullite-carbon ceramic composites

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Effects of Ifon clay and sintering temperature on the phase development and physico-mechanical properties of mullite-carbon ceramic composite was investigated. Powders of Ifon clay, kaolin from Okpella and graphite of known mineralogical composition were thoroughly blended in a ball mill for 3 hrs at a speed of 60 rev/min using a predetermined ratio. From the blended powders, standard samples were produced by uniaxial compression. This was followed by sintering in an electric furnace at 1400 °C, 1500 °C and 1600 °C for 1 hr. The sintered samples were characterized for various physical and mechanical properties. It was observed that; Ifon clay addition to the sample favours the formation of microcline in preference to mullite at temperature between 1400 °C and 1500 °C. As the sintering temperature increases to 1600 °C there is the formation of mullite phase and pores. These pores allowed the graphite content of the samples to be oxidized leading to reduction in the graphite content of the samples. The addition of Ifon clay improved on some of the mechanical properties of the samples like the cold crushing strength and Young's Modulus compared to the earlier reported samples without Ifon clay. The sample with optimum physico-mechanical is considered to be the sample sintered at 1400 °C.

Key words: Sintering temperature, Mullite, Graphite, Ceramic composite, Phase evolution.

Introduction

Carbon based composites find increasing applications as refractories in the context of recent developments in ceramic industry for high temperature applications [1]. There is an ever increasing interest in ceramic based materials which offer improved high temperature mechanical behaviour for structural applications. Recently, materials production and utilization has shifted from monolithic to composite materials in a view to adjust to the global need for reduced weight, low cost, quality, and high performance in structural materials [2, 3]

In the past decade a lot of research have been focussed on development of more ceramic materials and ceramic matrices composites (CMC) for utilization in the industries because of their low density, hardness, wear resistance, toughness, mechanical strength and physical properties [4-8]. This is because unreinforced ceramic fails catastrophically when subjected to around 0.1% tensile strain [9] but fiber reinforced ceramic (composites) could withstand more than 0.5% tensile strain.

Furthermore, most monolithic ceramics are characterized by low intrinsic fracture toughness which limit their roles for structural applications [10], for this cause, ceramic

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are subjected to toughening processes in order to improve on their fracture toughness. This is usually achieved by phase-transformation toughening or by fiber synthesis within the ceramic matrix [11, 12]. Monolithic ceramics have the disadvantage of being brittle. A reinforcing phase can improve the toughness of these materials, while still taking advantage of the matrix's other properties such as wear resistance, hardness, corrosion resistance and temperature resistance [13].

Many researcher have worked on carbon or mullite based ceramic composite from using clay as one of the constituents. Kurovics and Gömze [14], reported their findings on the development of high-tech ceramic composite from conventional kaolinite minerals and some bio-additives. They discovered that the bulk shrinkages, density and pore structures of pre-sintered specimens depend strongly on quantity of used bioorigin additives. Rendtorff et al. [15] prepared zirconiamullite composites through reaction sintering of alumina with zircon and direct sintering of mullitezirconia grains by slip casting and sintered at 1600°C for 2 hrs. Badiee et al. [16] studied the effect of CaO, MgO, TiO₂ and ZrO₂ on mullitization of the Iranian clay and alusite located in Hamedan mines. They discovered that the first three of these additives favoured mullite formation from the clay and alusite. Ebadzadeh and Ghasemi, [17] produced zirconiamullite composites using a-alumina and aluminium

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nitrate and zircon powder with TiO_2 as additive. Aramide et al [18] reported on in-situ synthesis and characterization of mullite-carbon refractory ceramic composite from Okpella kaolin and graphite. They discovered that the investigated samples possess low cold crushing strength and Young moduli. The aim of the work is to improve on the physic-mechanical properties of the ceramic composite produced from the same raw materials.

Materials and methods

Materials

The kaolin sample was sourced from Okpella, Edo State, Nigeria. The Ifon clay was sourced from Ifon, Ondo State, Nigeria and graphite was sourced from (Pascal Chemicals, Akure).

The Ifon clay and kaolin samples were first separately soaked in water for three days to dissolve the deleterious materials in them and at the same time to form slurry. The slurries were then sieved to remove deleterious materials and other foreign substances. The sieved slurries were then allowed to settle down for three days after which the clear floating water was decanted. The dispersed fine clays in water (clay slurries) were then poured into plaster of Paris (P.O.P) moulds and left undisturbed for three days in order to allow the remaining water present to drain out completely. The resulting plastic clay masses were sun dried and subsequently dried in a laboratory oven at 110 °C for 24 hrs. The resulting dried clay samples were crushed and milled in a Rawwley Sussex grinder to an average particle size of 300 um.

The mass percent of the composition below was computed using Eqs. (1) and (2) using the densities of the various constituents and the individual powders volume fraction in each composition. The powders were weighed per batch of 100.00 g on a sensitive electronic weighing balance to five (5) decimal places. The individual (batch) composition was thoroughly mixed in a ball mill for 3 hrs at 60 rev/min. The compositions of the blended samples were shown in Table 1

If

$$M_t = V_k \rho_k + V_G \rho_G + V_{Ifon} \rho_{Ifon}$$
(1)

Where $V_{k,}\,V_{C}$ and V_{Ifon} are respectively the volume fraction of kaolin, graphite and Ifon clay, $\rho_{k,}\,\rho_{C}$ and

Table 1. Constitution of the sample.

Sample designation	SampleGraphiteesignation(vol. %)		Ifon clay (vol. %)		
A	30	40	30		

 ρ_{Ifon} are respectively the density of kaolin, graphite and Ifon clay. M_t is the total mass contribution of all the components. And if M is the mass of each batch, then the mass contribution of each component could be calculated from:

$$Mc = \frac{Vc\rho c \times M}{Mt}$$
(2)

Mc is the mass contribution of a component in a batch (kaolin or graphite or Ifon clay), Vc, ρ_c the respective volume fraction and density of the component.

The resulting homogenous powder mixtures were compacted uniaxially into standard sample dimensions for the various analyses. The compacted samples were fired (sintered) at the temperature of 1400 °C, 1500 °C and 1600 °C.

Apparent porosity

Test samples from each of the ceramic composite samples were dried out for 12 hrs at 110°C. The dry weight of each fired sample was taken and recorded as D. Each sample was immersed in water for 6 hrs to soak and weighed while being suspended in air. The weight was recorded as W. Finally, the specimen was weighed when immersed in water [19, 20]. This was recorded as S. The apparent porosity was then calculated from the expression:

% apparent porosity =
$$\frac{(W-D)}{(W-S)} \times 100$$
 (3)

Bulk Density

The test specimens were dried out at 110°C for 12 hrs to ensure total water loss. Their dry weights were measured and recorded. They were allowed to cool and then immersed in a beaker of water. Bubbles were observed as the pores in the specimens were filled with water. Their soaked weights were measured and recorded. They were then suspended in a beaker one after the other using a sling and their respective suspended weights were measured and recorded [19, 20]. Bulk densities of the samples were calculated using the formula below:

Bulk density =
$$\frac{D}{(W-S)}$$
 (4)

where D represents weight of dried specimen, S represents weight of dried specimen suspended in water, and W represents weight of soaked specimen suspended in air.

Water Absorption

The test sample was dried out in an oven till a constant weight of the sample was obtained. The sample was then placed in a vessel containing water in order to be completely submerged without touching the bottom of the vessel in which it is suspended. The vessel was then heated slowly so that the water boileds after heating. After boiling for about an hour with the evaporated water replaced, the sample was allowed to cool at room temperature for 24 hrs. The sample was then renamed, blotted and then reweighed. The percentage water absorption was calculated as showed below:

Water Absorption =
$$\frac{\text{(Soaked-Dried wt)}}{\text{(Dried wt)}} \times 100$$
 (5)

Cold compression strength, modulus of elasticity and absorbed energy

Cold compression strength test is to determine the compression strength to failure of each sample, an indication of its probable performance under load. The standard ceramic samples were dried in an oven at a temperature of 110 °C, allowed to cool. The cold compression strength tests were performed on INSTRON 1195 at a fixed crosshead speed of 10 mm min⁻¹. Samples were prepared according to ASTM C133-97 [19, 20] cold crushing strength, modulus of elasticity and absorbed energy of standard and conditioned samples were calculated from the equation:

Cold Crushing Strength =
$$\frac{\text{(Load to fracture)}}{(\text{Surface area of samle})}$$
(6)

Qualitative and quantitative (XRD)

The samples were prepared for XRD analysis using a back loading preparation method. The samples were analysed using a PANalyticalX'Pert Pro powder diffractometer with X'Celerator detector and variable divergence- and receiving slits with Fe filtered Co-K α radiation. The phases obtained were identified using X'PertHighscore plus software. Graphical representations of the qualitative result will then follow. The relative phase amounts in weight % were estimated using the Rietveld method (Autoquan Program). Amorphous phases, present were not taken into consideration in the quantification.

Scanning electron microscopy

Morphology and microanalysis of the clay and composite samples were determined using ultrahigh resolution field emission scanning electron microscope (UHR-FEGSEM) equipped with energy dispersive spectroscopy (EDS). The pulverize clay samples were graphite coated. The sintered samples were studied using ultra-high resolution field emission scanning electron microscope (UHR-FEGSEM) equipped with energy dispersive spectroscopy (EDX). Particle images which are obtained with a secondary electron detector.

Results and Discussion

Tables 2 and 3 show the phases developed in the sintered samples and the physical with mechanical properties of the samples. Fig. 1 shows the x-ray diffraction pattern of the of the Ifon clay sample. Figs. 2, 3, 4 and 5 respectively shows the phases in the samples A, B, C and D sintered at various temperatures. Figs. 6-11 show the effects of sintering temperature and additives on the physical and mechanical properties of the various samples. While Figs. 12-15 show the secondary electron images, backscattered with energy dispersive spectroscopy (SEM/EDS) of the samples sintered at various temperature.

Table 2 with Figs. 2-5 show the phases developed in the various sample with increase in sintering temperature. While Fig. 9 shows the scanning electron microscope (SEM) images of the various sample.

Table 2, Figs. 2 and 9 show the effects of Sintering temperature on the phases developed in the samples without additive (Sample A). From the figure and table it is observed that at 1400 °C the phases present in sample A are 22.1% graphite, 7.6% mullite, 12.8% anorthite and 57.4% microcline. From Table 1, the raw materials from which the sample is constituted composed of 40% kaolin, 30% Ifon clay and 30% graphite. It is a well-established fact that when kaolinite (a constituent of kaolin) subjected to firing at high temperature, it

Table 2. Showing the various phases (in percentage) developed in the samples.

Sample	Graphite	TiO ₂	SiC	Mullite	Anorthite	TiC	Quartz	Microcline	Cristobalite
AT1	22,1	0,0	0,0	7,6	12,8	0,0	0,0	57,4	0,0
AT2	16,8	0,0	0,0	39,9	0,0	0,0	0,0	43,3	0,0
AT3	9,4	0,0	0,0	70,4	8,1	0,0	0,0	12,2	0,0

Table 3.	Showing	the values	of investigated	physical	and	mechanical	properties.
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Sample designation	Sintering Temp. (°C)	Engineering Stress (MPa)	Engineering strain (mm/mm)	Absorbed Energy (J)	Young's Modulus E (MPa)	Bulk (g/cm ³)	Water absorption (%)	Apparent Porosity (%)
А	1400	10.71866	0.07414	4.29543	144.5732	1.571	17.42	26.92
(40% Kao,	1500	6.59916	0.04228	1.74660	156.0823	1.646	18.26	29.92
30% Ifon, 30% C)	1600	3.28946	0.03345	0.82168	98.3396	1.555	23.08	35.96



Fig. 1. XRD pattern (phase analysis) of Ifon clay sample.



Fig. 2. Phases in Sample A sintered at various temperatures.

undergoes phase transformation through dehydroxylation to metakaolinite at around 420 ° to 660 °C [21]. At around 900 °C the metakaolinite undergoes decomposition to amorphous silica and α -Al₂O₃ type spinel. However, as the sintering temperature increases, the spinel and silica recrystallizes to mullite at above 1100 °C. Although some other researchers have reported that mullite begins to form with excess silica at a temperature above 900 °C and continues till around 1000 °C [22, 23]. This explains the origin of the mullite in the samples. However, the presence of anorthite and microcline could be attributed to the Ifon clay which has been reported to contain feldspar [3]. Furthermore, Aramide [2] reported his findings when he fired samples made from Ifon clay at temperatures between 800 ° and 1200 °C. He discovered that the major phases formed in the samples were quartz, microcline and anorthite. It is also observed that the amount of mullite formed in the sample sintered at 1400 °C is the least (7.6%) this could be attributed to presence of Ifon clay which favours the formation of microcline and anorthite [24, 25] in preference to mullite up to around 1400 °C.

Moreover, as the sintering temperature is increased to $1500 \,^{\circ}$ C the graphite content is observed to reduce from 22.1% (1400 $^{\circ}$ C) to 16.8%. Further increase in the sintering temperature to 1600 $^{\circ}$ C resulted in further

reduction in the amount of graphite content to 9.4%. This reduction in the graphite content of the sample with increased sintering temperature could be attributed to high temperature oxidation of the graphite. Although, graphite is refractory but it is prone to oxidation at temperature above 900 °C. This necessitates the incorporation of antioxidants as additives in carbon (graphite) based ceramics intended for high temperature applications [26-28]. Furthermore, it is observed that the sample contains 12.8% anorthite and 57.4% microcline respectively at 1400 °C. When the sintering temperature was increased to 1500 °C the anorthite content of the sample reduced to 0% while that of the microcline reduced to 43.3%. Further increase in the sintering temperature of the sample to 1600 °C resulted into the anorthite content of the sample being increased to 8.1% while that of the microcline reduced to 12.2%. The overall reduction in the contents of both anorthite and microcline cannot be unconnected with the increase in the mullite content with increased sintering temperature. It could be inferred that the microcline and anorthite began to transform to mullite at temperature above 1400 °C and it proceeds beyond 1600 °C. Comparing the phases observed in these samples with the earlier report [18], it is discovered that the introduction of Ifon clay to the samples lead to the development of cristobalite and anorthite in the samples AT1, AT2 and AT3. In the earlier report by the author only traces of microcline was discovered. Moreover, neither quartz nor cristobalite was observed in these samples under consideration, whereas between 2 to 7% cristobalite was observed in the earlier report. It can be inferred that both anorthite and microcline were the matrix in the samples, while mullite acted as reinforcement.

Figs. 6-8 and Table 3 show the effects of sintering temperature on the physical properties of the sintered ceramic samples. The effects of sintering temperature on the apparent porosity of the sintered ceramic samples is depicted in Fig. 3. From the figure can be



Fig. 3. Effects of sintering temperature of the apparent porosity of the ceramic samples.

observed that the apparent porosity increased with increase in the sintering temperature for the sample This behaviour of the sample in which the apparent porosity increased with increase in sintering temperature is contrary to the expectation when ceramic is being sintered [2, 29].

It is expected that most of the voids between the grain particles of the ceramic are closed up during sintering therefore reducing the porosity with increased sintering temperature. The reason for this anomalous behaviour is due to the presence of graphite in the samples. It is established in the literatures that although graphite is a refractory material, it does have a set-back of high temperature oxidation at a temperature beyond 900 °C [30, 31]. Some of the graphite particles got oxidized with increased sintering temperature, leaving voids within the ceramic material, thereby increasing the apparent porosity of the samples with increased sintering temperature. It can be observed from Fig. 2 that the graphite content of the sample reduced with increased sintering temperature. The increased porosity increased sintering temperature could also be explained with the evolution of the phases as the temperature increased. From Fig. 2 it is observed that both the graphite, and the microcline contents reduced with sintering temperature. As these matrix were been reduced they leave more pores, this leads increased porosity.

Fig. 4 shows the effects of sintering temperature on the bulk density of the sintered ceramic samples. It can be observed that the bulk density of sample A at 1400 °C was initially 1.571 g/cm^3 , it slightly increased to 1.646 g/cm^3 as the sintering temperature was raised to 1500 °C, further increase in the sintering temperature to 1600 °C resulted in the slight reduction in the bulk density of the sample to 1.555 g/cm^3 . It is also observed that the samples attained maximum density at 1500 °C. This is similar to what others have reported [18, 23, 32].

Fig. 5 shows the effects of sintering temperature on the water absorption of the sintered graphite based ceramics samples. From the figure, it was 17.42% when the sintering temperature was 1400 °C, it then



Fig. 4. Effects of sintering temperature of the bulk density of the ceramic samples.



Fig. 5. Effects of sintering temperature on the water absorption of the ceramic samples.



Fig. 6. Effects of sintering temperature on the engineering compression stress of the ceramic samples.

slightly increased to 18.26% when the sintering temperature was raised to 1500 °. The water absorption of the sample is observed to further increase when the sintering temperature was further raised to 1600 °C. It will be observed that the water absorption of the samples follow the same trend as the apparent porosity of the sample with increased sintering temperature. This is because the water absorption is directly influenced by the open pores, the structure and size [33]. Summarily, it could be generalized that the water absorption of all the samples increased with increase sintering temperature.

Figs. 6-8 and Table 3 shows the effects of sintering temperature and additives on the various mechanical properties of the ceramic samples under consideration.

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Fig. 7. Effects of sintering temperature on the absorbed energy of the ceramic samples.

Fig. 6 shows the effects of sintering temperature and additives on the engineering stress of the samples being considered. From the figure it is observed that the engineering stress of sample A at 1400 °C was about



Fig. 8. Effects of sintering temperature on the Young's modulus of the ceramic samples.

10.719 MPa. Increasing the sintering temperature to 1500 °C the engineering stress of the sample is seen to reduce to 6.599 MPa, further increase in the sintering



Sample AT1



Sample AT2



Sample AT3

Fig. 9. SEM images showing the secondary electron and backscattered images of samples.

temperature of the sample to 1600 °C is seen to result into further reduction in the engineering stress of the sample to 3.289 MPa. Comparing this with the result already discussed in Fig. 3, it will be seen that increased porosity of the sample with increase in the sintering temperature reduces the load bearing ability of the sample. This agrees with the reports of other researchers [18, 23, 32]. The load bearing ability of the sample A reduced with increase in the sintering temperature due to increase in the apparent porosity of the sample.

Moreover, comparing the result here with the earlier report [18] it will the observed that there is an improvement in the engineering stress of the ceramic composite samples under consideration here. Sample AT1 was able to attain 10.718 MPa whereas the highest cold crushing strength attained earlier was 8.05 MPa.

Fig. 7 shows the effects of sintering temperature and additives on the absorbed energy of the sintered ceramic samples. From the figure it is observed that the initial absorbed energy for sample A at 1400 °C was about 4.295 J, it however reduced to about 1.747 J when the sintering temperature was raised to 1500 °C. Further reduction in the absorbed energy was observed as the sintering temperature was further raised to 1600 °C. It however followed the same trend engineering stress followed with increased sintering temperature as discussed in Fig. 6 above.

Young's Modulus of Elasticity

Fig. 8 shows the effects of sintering temperature and additives on the Young's modulus of elasticity of the samples. From the figure it is observed that sample A has the highest value at 1400 °C as it has been the case in all other mechanical properties earlier discussed. It has a value about 144.573 MPa at 1400 °C, this however increased to 156.082 MPa as the sintering temperature was raised to 1500 °C. The value was observed to reduce to about 98.34 MPa when the sintering temperature was further raised to 1600 °C. Comparing the result here with the former report [18], there is also an improvement in the Young Modulus of the sample here. Sample AT2 was able to attain 156 MPa whereas the highest attained before was 141 MPa.

Conclusions

Ifon clay addition to the sample favours the formation of microcline in preference to mullite at temperature between 1400 °C and 1500 °C. As the sintering temperature increases to 1600 °C there is the formation of mullite phase and pores. These pores allowed the graphite content of the samples to be oxidized leading to reduction in the graphite content of the samples. The addition of Ifon clay improved on some of the mechanical properties of the samples like

the cold crushing strength and Young's Modulus compared to the earlier reported samples without Ifon clay. The sample with optimum physico-mechanical is considered to be the sample sintered at 1400 °C.

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