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The Densification and Diametral Compression Strength of Isi-Ogwuta Clay

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Abstract: - The diametral compression strength of clay was investigated. The clay sample was analyzed to ascertain its chemical composition and mineralogical constituent. The diametral clay discs were produced using a uniaxial cold pressing hydraulic press and sintered at a predetermined temperature and time (1200℃ and 15 minutes). Two diameters (D) of discs of 23mm and 29mm were used with varying thicknesses (3mm-10mm). The effect of disc thickness with intent to qualitatively define the plane stress and the plane strain fracture conditions of the clay was undertaken. The plane stress condition was obtained by discs with thickness $\leq 1/4$ D for 23mm and 29mm whilst the plane strain condition was obtained by testing discs of thickness $> 1/4$ D. The diametral compression strength of discs of thicknesses $3 - 10$mm gave a range of $14.6 - 5.5$MPa for samples of 23mm diameter whilst a range of $5.8 - 2.2$MPa was obtained for samples of 29mm diameter. Greater numbers of 29mm diameter samples failed in the normal tensile fracture mode whilst more samples of 23mm diameter failed in the triple-cleft fracture mode. 23mm diameter discs gave higher values of Weibull moduli in comparison with the values obtained for discs of 29mm diameter indicating the flaws sampled in the 23mm diameter were of the same severity. Pores were observed to be singularly effective as initiation sites for failure as shown by the negative slope of the effect of porosity on the strength of clay.

Keywords: - *Diametral Compression Strength, Plane Stress, Plane Strain, Fracture mode, Discs, Weibull moduli.*

I. INTRODUCTION

It is frequently inconvenient to measure the tensile strength of brittle materials by employing the direct conventional method of pulling apart a suitably shaped specimen owing to the difficulty of preparing such specimens from the materials concerned [1]. The use of briquettes, bobbins and cylinders or prisms with embedded studs, tested in direct tension have all suffered from local stress concentrations [2]. Therefore to overcome these problems, the strength of a brittle material is measured indirectly. Some of the indirect methods are the equi-biaxial tension test, the diametral compression disc test and the flexure bending test.

In the diametral compression disc test according to [3], a right circular cylindrical specimen is compressed diametrally between two flat platens as shown in Fig 1. The diametral compression disc test is a convenient method for determining the tensile strength of brittle materials because of the fact that the specimen is simple to prepare and to load.

Previous studies have been carried out using the diametral compression disc test and the test method appears to give a reasonable result for the tensile strength of brittle materials. In their work[3]on diametral compression of silicon nitride, stated that the volume effect was mainly responsible for the difference observed between the strength obtained using diametral compression disc test and that obtained using bending flexure test for sintered silicon nitride. on the other hand, [4] investigated the tensile strength of disc and annuli by diametral compression test and pointed out that the diametral compression test was capable of giving a good measure of uniaxial tensile test for *Griffith's* type materials.

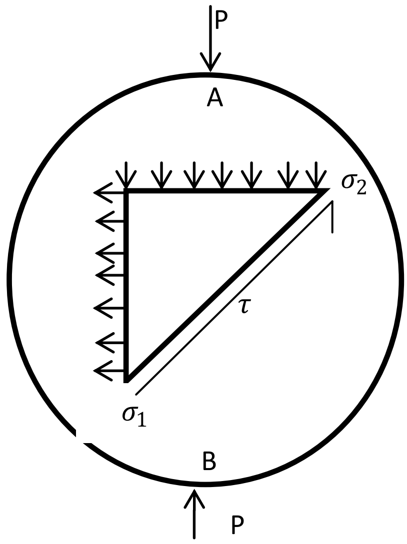


Fig.1: Stress distribution across the loaded diameter for a disc compressed between two uniaxial loading points[3].

In addition, [5] in their research on the tensile measurements of frangible bullets using the diametral compression test at quasi-static $1\mu m/s$ and high $12.5m/s$ displacement rates discovered that the tensile strength was not strongly sensitive to the displacement rate.

The thickness of a brittle material is critical when investigating the material's response to mechanical forces. Plane stress and plane strain conditions are phenomena which describe the stress states for thin and thick discs during fracture. It is believed that the plane stress condition is valid when the disc thickness is less than or equal to one quarter of the disc diameter but if the disc thickness is greater than one quarter of the disc diameter, a condition which deviates substantially from the ideal plane stress exists (plane strain).

The diametral compression test has some stringent requirements which include[6]: (i) material must be ideally linearly elastic,(ii) elastically isotropic,(iii)elastically homogeneous, and (iv) have shear and compression strengths appreciably higher than its tensile strengths.Not many materials will satisfy these conditions and consequently the application of the test result has limited applicability. As a matter of fact the first and last requirements made the test inapplicable to ductile materials in which the specimens would simply flatten out under the influence of the high shear and compression stresses at the loading points.

The fundamental aims for which this work was embarked upon were to:

(i) ascertain the applicability of the diametral compression disc test for the clay, and to

(ii) define more closely the plane stress and the plane strain fracture conditions of the clay.

II. EXPERIMENTAL PROCEDURE

2.1 Materials Characterisation and sample Production

The particle size analysis commenced with the sieving operation. Wet sieving was first carried out on about $60g$ of the clay using a sieve of $200\mu m$ mesh size. Both the filtrate and the residue were collected. Dry sieving was conducted on the residue whilst the filtrate was poured into a glass cylinder where the hydrometer reading was taken. The essence the hydrometer analysis was to obtain additional information on the fine particle nature of the clay.

The examined clay was obtained from Unwana in Afikpo-North Local Government Area of Ebonyi State, Nigeria. "The clay is popularly known as Isi-Ogwuta clay".

The clay lumps, as sourced, were crushed to smaller sizes, dried and finally ground into fine clay particles. All clay particles passed through a $-425\mu m$ mesh and were retained on a $212\mu m$ mesh. No water was added to the clay before cold forming. Several weights of the powdered clay i.e., 4g, 6g, 9g, 10g, 11g, 12g, 13.5g, 15g and 16.5g were measured out using the electronic precision balance (ConTECH model CA223) with 220g maximum and 0.1g minimum capacity. Using a hand operated hydraulic press at a pressure of 8MPa with tungsten carbide-lined interior steel dies, the powdered clay samples were compressed to discs of various thicknesses.

The cold pressed samples were fired in a muffle furnace at approximately $120°C$ per hour up to $600°C$ in order to burn off combustible materials. Finally, the samples were fired at approximately $150°C$ per hour up to $1200°C$ and held at this temperature for 15 $minutes$. The fired samples included discs of nominally 23mm and 29mm diameters with each diameter disc having different thicknesses of 3mm, 5mm, 7mm, 8mm, 9mm and 10mm.

2.2 Mechanical Test Procedure

All mechanical strength tests were carried out at room temperature on the sintered clay discs using TQ Sm1000 Universal Material Testing Machine of maximum capacity 100KN at a cross-head speed of $0.5mm/min$ in the Materials and Metallurgical Engineering(MME) laboratory, Federal University of Technology, Owerri. Prior to the mechanical strength test, the platens were tightly screwed into the two internally threaded hollows at the central points of the upper and lower sections of the mechanical tester. Subsequently, the test pieces were diametrally compressed between the two platens. In all cases, the frictional effects were assumed to be negligible.

III. RESULTS

3.1 Clay Characterisation Result

The result of the particle size analysis of the clay are given in Tables 3.1.1 and 3.1.2

Table 3.1.1: Sieve Analysis of the Clay

Sieve size (μm)	*Mass retained* (g)	*Mass passing* (g)	% passing (%)
75	0.1	59.2	98.7
150	0.1	59.3	98.8
300	0.2	59.4	99.0
425	0.3	59.6	99.3
600	0.1	59.9	99.8
850	0.0	60.0	100.0
1180	0.0	60.0	100.0

Table 3.1.2: Hydrometer Reading of Clay

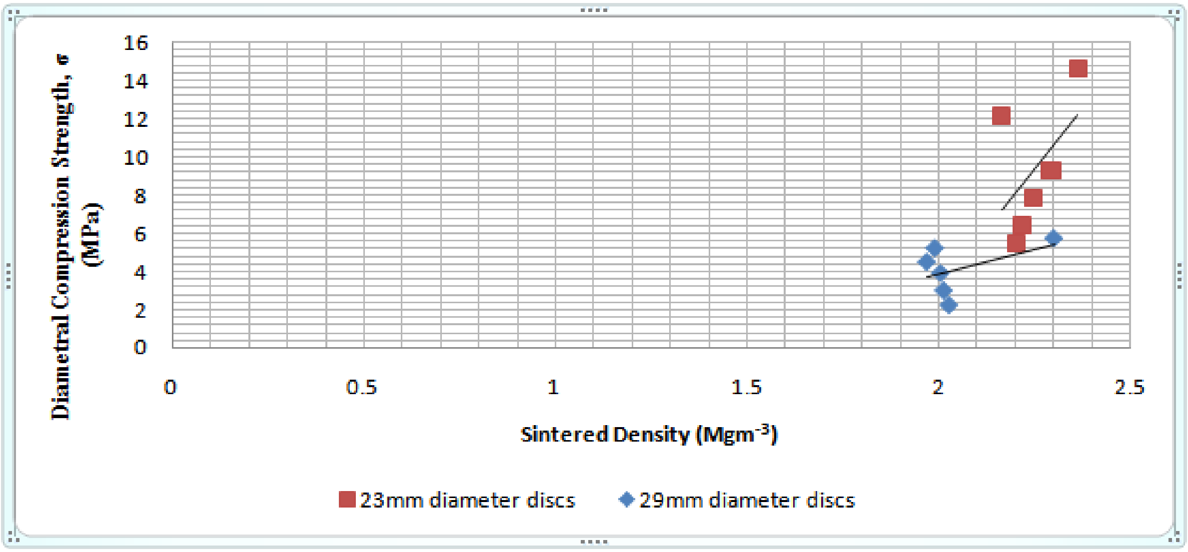
Date	*Time* (min)	*Hydro − meter reading*	*True reading*	*Effect − ive depth* (mm)	*Fully correct −ed reading*	*Particle diameter D* (mm)	% *finer than* (%)
7/4/11	0	0	0	0	0	0	0
	1	23.5	24.0	115.60	23.3	0.044	62.4
	3	21.0	21.5	125.85	20.8	0.026	55.7
	6	19.5	20.0	132.00	19.3	0.019	51.7
	10	17.5	18.0	140.20	17.3	0.015	46.3
	16	16.0	16.5	146.35	15.8	0.012	42.3
	31	13.5	14.0	156.60	13.3	0.009	35.6
	60	6.0	6.5	187.35	5.8	0.007	15.5
8/4/11	1440	1.0	1.5	207.85	0.8	0.002	2.1

3.2 Sintered Densities

The sintered densities of the clay specimen are given in Table 3.2.1 and were evaluated by direct measurements of the physical dimensions of the disc using a vernier calliper. The effect of sintered density on the diametral compression strength is shown in Fig. 3.1.

Table 3.2.1: Sintered Densities of the Clay

Diameter D (mm)	Thickness t (mm)	Average strength $\sigma(MPa)$	Sintered density SD (Mgm^{-3})
23	3	14.607	2.362
	5	12.140	2.166
	7	9.278	2.294
	8	7.876	2.246
	9	6.417	2.217
	10	5.507	2.203
29	3	5.756	2.300
	5	5.239	1.989
	7	4.515	1.968
	8	3.924	2.004
	9	3.010	2.012
	10	2.239	2.026



4.1: Effect of Sintered Density on the Diametral Compression Strength of the Clay.

3.3 Diametral Compression Test Result of the Clay

Equation (3.1) [3] was used to compute the diametral compression strengths and the results including the average, standard deviations, *(S)* and the coefficient of variations, *(cv)* are given in Table 3.3.1 and displayed in Fig. 3.2.

$$\sigma_t = \frac{2P}{\pi D t} \quad \text{(3.1)}$$

Where;

σ_t (MPa) is the maximum tensile stress,

P (N) is the applied load at fracture,

D (mm) is the disc diameter, and

t (mm) is the disc thickness.

Table 3.3.1: Effect of Disc Thickness on the Diametral Strength of the Clay.

D (mm)	t (mm)	σ_t (MPa)	S (MPa)	P (%)	cv (%)	Weibull modulus, m	
23	3(30)	14.607	±2.453	11.60	16.8	6.27*	6.61**
	5(30)	12.140	±1.585	13.89	13.1	8.44*	8.47**
	7(30)	9.278	±1.632	12.42	17.6	5.85*	6.31**
	8(30)	7.876	±1.550	16.91	19.7	5.60*	5.63**
	9(30)	6.417	±0.961	20.55	15.5	7.00*	7.16**
	10(30)	5.507	±0.991	18.47	24.2	3.25*	4.59**
29	3(30)	5.756	±2.058	14.78	35.8	2.28*	3.10**
	5(30)	5.239	±1.123	15.50	21.4	5.52*	5.19**
	7(30)	4.515	±1.620	23.23	35.9	2.56*	3.09**
	8(30)	3.924	±0.839	21.55	21.4	5.00*	5.19**
	9(30)	3.008	±0.716	25.15	23.4	5.22*	4.66**
	10(30)	2.239	±0.664	28.64	29.7	3.90*	3.74**

The number in parenthesis represents the number of specimen that was tested.

* Indicates the Weibull modulus obtained using the graphical method proposed by [6]

** Indicates the Weibull modulus calculated using the relation, m = $\frac{1.11}{cv}$ proposed by [7]

$d(mm) \; = \; Disc\ diameter,$
$t(mm) \; = \; Disc\ thickness,$
$\sigma_t(MPa) = \; Average\ diametral\ Strength,$
$S(MPa) \; = \; Standard\ deviation,$
$P(\%) \; = \; Apparent\ Porosity$
$cv(\%) \; = \; Coefficient\ of\ variation$

Table 3.3.2: Plane Stress and Plane Strain Fracture Conditions for the Clay

Clay discs of nominally 23mm diameter						
	Plane stress condition		*Plane strain condition*			
Disc thickness, t(mm)	3(30)	5(30)	7(30)	8(30)	9(30)	10(30)
Average strength, σ(MPa)	14.607	12.140	9.278	7.876	6.417	5.507
Standard deviation, S	±2.453	±1.585	±1.633	±1.549	±0.961	±0.991
Clay discs of nominally 29mm diameter						
	Plane stress condition				*Plane strain condition*	
Disc thickness, t(mm)	3(30)	5(30)	7(30)	8(30)	9(30)	10(30)
Average strength, σ(MPa)	5.756	5.239	4.515	3.924	3.010	2.239
Standard deviation, S	±2.058	±1.123	±1.620	±0.839	±0.716	±0.664

The numbers in parentheses represent the numbers of specimen tested.

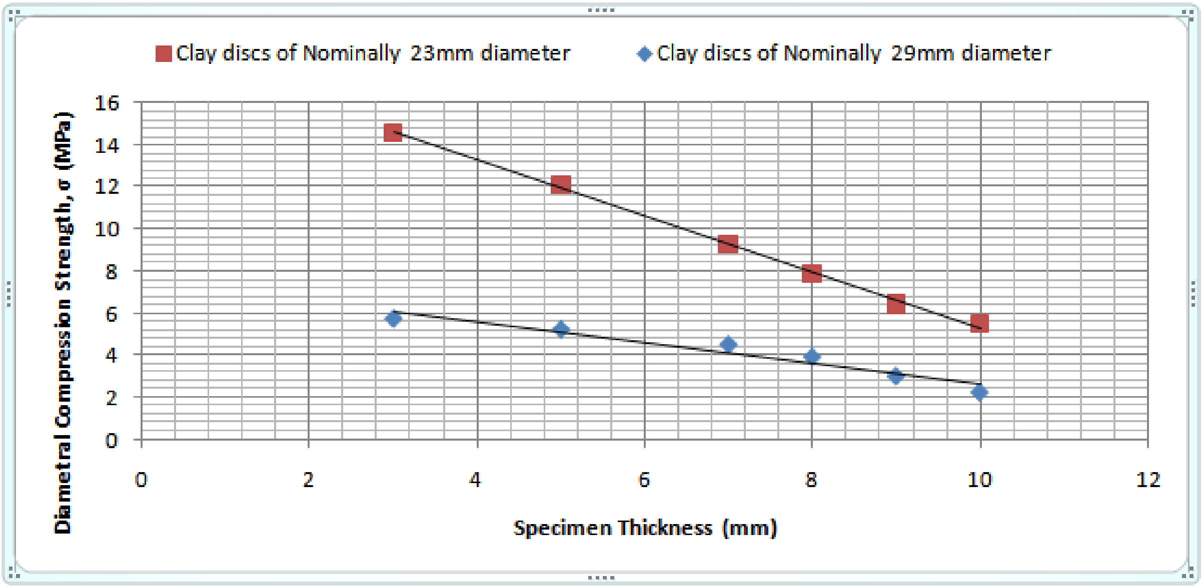


Fig. 3.2: Effect of Disc Thickness on the Diametral Compression Strength of the Clay

3.3.3 The Fracture Mode

The fracture modes (normal tensile and triple-cleft) are displayed in plate 3.1.

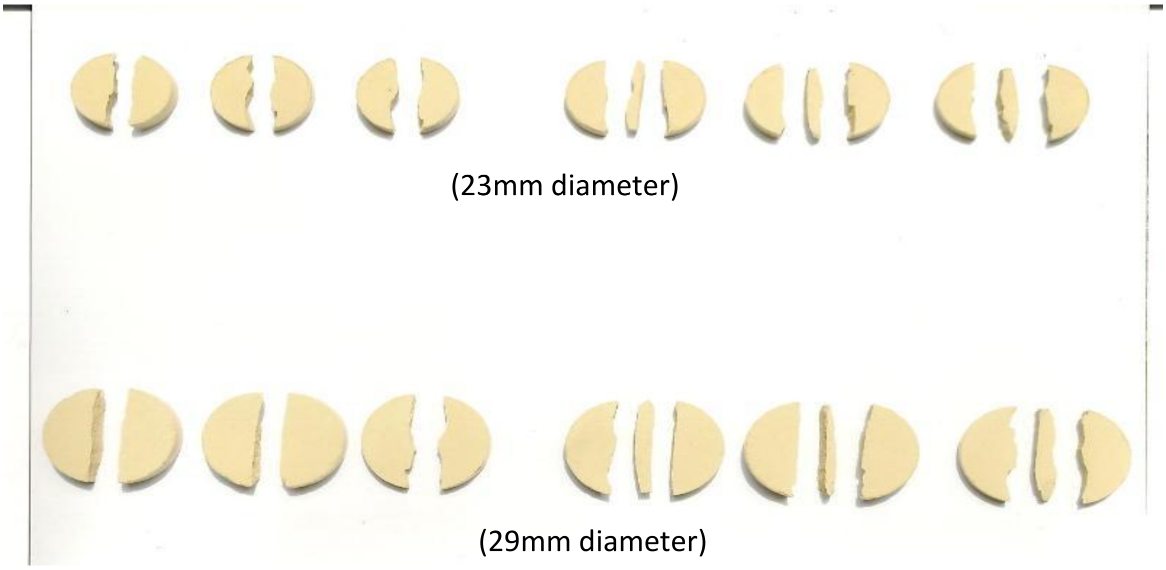


Plate 3.1: Room Temperature Fracture Modes for the Clay.

3.3.3 Effect of Porosity on the Diametral Compression Strength of the Clay

Table 3.4: Effect of Porosity on the Diametral Strength of the Clay.

Symbol	*Gradient*	*Disc diameter (mm)*
	-0.042	23
	-0.061	29

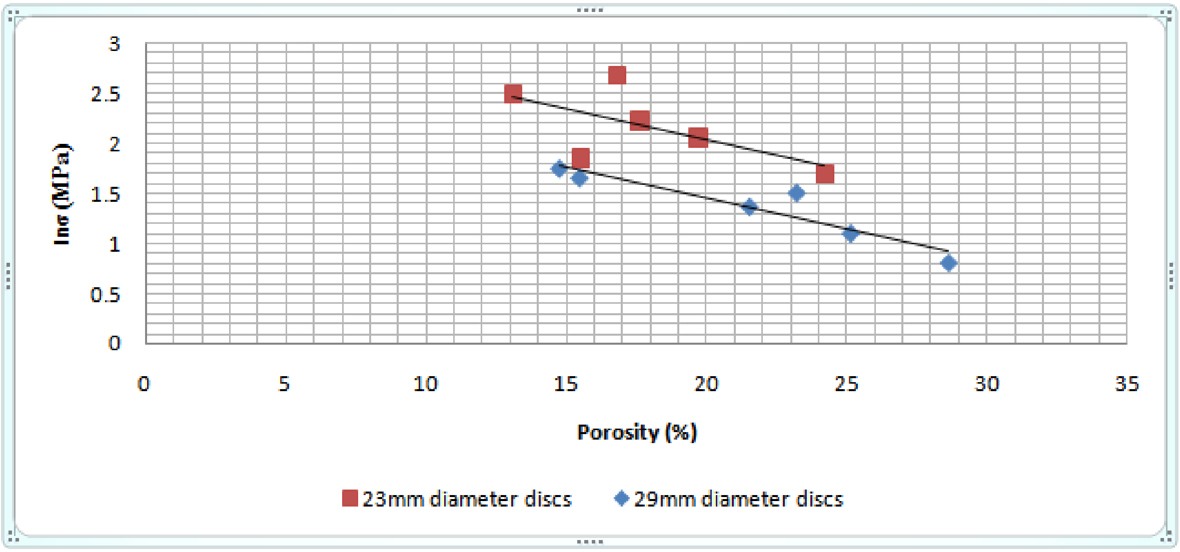


Fig. 3.3: Effect of Porosity on the Diametral Compression Strength of the Clay.

IV. DISCUSSION OF RESULTS

4.1 Clay Characterization Result

The sieve analysis showed that 98.7% of the clay particles passed through $-75\mu m$ mesh allowing about $0.1g$ of the clay to be retained in the mesh. Up to 99.0% of the clay particle passed through the $-300\mu m$ mesh whilst about $0.2g$ of the clay was retained in the mesh. A higher weight of about $0.3g$ was retained in the $425\mu m$ mesh whilst 99.3% of the clay particle was able to pass through the mesh. All clay particles were observed to pass through $-850\mu m$ and $-1180\mu m$ mesh sizes. The result of the hydrometer analysis of the clay revealed that the particle size diameter, D of the clay was about $0.04mm$ at 1 $minute$, $0.019mm$ at 6 $minute$, $0.009mm$ at 31 $minutes$ and about $0.002mm$ after 1440 $minutes$. The result of the particle size analysis of the clay was in agreement with the previous report on the particle size of clay by [9,10]. However, the essence the hydrometer analysis was to obtain additional information on the fine particle nature of the clay.

4.2 Sintered Densities

Clay discs of 23mm and 29mm diameters attained end-point densities of $2.36Mgm^{-3}$ and $2.30Mgm^{-3}$ respectively. A sintered density range of $2.17Mgm^{-3}$ to $2.36Mgm^{-3}$ was obtained for samples of nominally 23mm diameter whilst for samples of nominally 29mm diameter, a sintered density range of $1.97Mgm^{-3}$ to $2.30Mgm^{-3}$ was obtained. In general, the densification trend for the clay was observed to slightly differ in incremental order as the disc thickness decreased for the 23mm diameter specimen whilst the 29mm diameter specimen revealed an erratic densification trend with decreasing disc thickness. This observation may be due to the presence of different severity of flaws in discs of 29mm diameter.

4.3 Diametral Compression Test Result of the Clay

4.3.1 Effect of Thickness on the Diametral Strength of Clay

For both clay discs i.e., 23mm and 29mm diameters, a decrease in the average diametral compression strength was observed with increase in specimen thickness with clay discs of nominally 23mm diameter having the highest average strength values. This observation was in agreement with the statistical theory of brittle fracture which predicted lower strength value for larger specimen [11]. The reason for the observation may be due to a number of factors such as (1) the stress state, (2) porosity, (3) the fracture mode and (4) sintered density. The stress state contributed a reduction in the measured diametral compression strength such that the plane stress condition aided a decrease of *9-12%* in the diametral compression strength whilst the plane strain condition contributed a reduction of *25.4-27%* in the diametral strength for discs of 23mm and 29mm diameters. Pores were found to be effective as initiation sites for failure. The discs that failed in the triple-cleft fracture mode recorded higher strength values in comparison with the strength values obtained for the normal tensile fracture mode. An increase in diametral compression strength was observed with increase in sintered density.

The coefficient of variation, *cv* (which provides a normalized measure of the dispersion of test data) was observed to be larger for discs of nominally 29mm diameter having a range of $21-36\%$ whilst a range of $13-24\%$ was obtained for discs of nominally 23mm diameter. This observation revealed that discs of 29mm diameter indicated more scatter in the measured strength data.

The investigation of the plane stress and the plane strain fracture conditions of the clay showed that for the specimen of nominally 23mm diameter, the trend of fracture strength for the disc thickness below one quarter of the disc diameter (i.e., < *5.75mm*) was obtained as follows: for *3-5mm* disc thickness, a range of average fracture strength of *14.61±2.45MPa* to *12.14±1.59MPa* (about *9.2%* decrease in the diametral strength) was obtained whilst the mean fracture strength for the disc thickness above one quarter of the disc diameter (i.e., >*5.75mm*) for *7-10mm* disc thickness gave a range of *9.28±1.63* to *5.51±0.99MPa* (i.e., about *25.4%* decrease in the diametral strength). With regard to the specimen of nominally *29mm* diameter, the trend of fracture strength for the disc thickness below one quarter of the disc diameter (i.e., < *7.25mm*) was obtained as follows: for *3-7mm* disc thickness, a range of average fracture strength of *5.76±2.06 to 4.52±1.62MPa* (i.e., about *12%* decrease in the diametral strength) was observed whilst the average fracture strength for the disc thickness above one quarter of the disc diameter (i.e., >*7.25mm*) for *8-10mm* disc thickness gave a range of *3.92±0.84 to 2.24±0.66MPa* (i.e., about *27%* decrease in the diametral strength).

It is reasonable to conclude that within limits of experimental errors, the plane stress condition aided a decrease of *9-12%* in the diametral compression strength whilst the plane strain condition contributed a reduction of *25.4-27%* in the diametral compression strength for clay discs of 23mm and 29mm diameters.

To further establish the variability in the measured diametral compression strength, statistical tests ($F\ test\ and\ t\ test$) were carried out. The F distribution test was employed to test the null hypothesis that the data under consideration were drawn from populations that have the same standard deviation. The t distribution test was used to evaluate sample differences by using means and the distribution of sample scores around the mean. The t distribution test with a significant level of *0.01* revealed a significant statistical difference in the average diametral compression strengths between discs of nominally; *23mm* diameter by *5mm* thick and *23mm* diameter by *7mm* thick. The same statistical disparity in the diametral compression strength was obtained between discs of nominally; *29mm* diameter by *7mm* thick and *29mm* diameter by *9mm* thick. This implied that the plane stress condition was obtained for *3mm (14.61MPa)* and *5mm (12.14MPa)* disc-thicknesses for samples of *23mm* diameter whilst for discs of *29mm* diameter, the plane stress condition was obtained for *3mm (5.76MPa), 5mm (5.24MPa), 7mm (4.52MPa),* and *8mm (3.92MPa)* disc-thicknesses. On the other hand, the plane strain condition was obtained for *7mm (9.28MPa), 8mm (7.88MPa), 9mm (6.42MPa), and 10mm (5.51MPa)* disc-thicknesses for samples of *23mm* diameter whilst for samples of *29mm* diameter, the plane strain condition was obtained for 9mm (*3.01MPa), and 10mm (2.24MPa)* disc-thicknesses.

The result of the statistical test enabled the assertion that the plane stress condition was obtained for clay of *3-8mm* disc thickness for samples of *29mm* diameter. However, this condition did not extend to the thickness of *7mm (9.28MPa)* for samples of nominally *23mm* diameter.

4.3.2 The Fracture Mode

The fracture modes observed for the test were the normal tensile in which the specimen fractured into two almost equal pieces along the loaded diameter and the triple-cleft fracture which involved the splitting of the specimen into three or more pieces with fracture consisting of a central normal tensile and two nominally collinear fractures on either side of the central fracture. The observed fracture modes were similar to those previously reported by [3] for silicon nitride and [12] for sintered mullite samples.

Clay discs of 23mm diameter by 3mm thick produced the highest number of specimens (8 discs) that failed in the triple-cleft fracture mode. In general, greater numbers of the tested discs failed in the normal tensile mode with discs of 29mm diameter by 8mm thick and 29mm diameter by 10mm thick having the highest number of samples (29 discs) that failed in the normal tensile fracture mode. This observation was due to the high strength recorded for 23mm diameter samples. The reason wherein more discs of 23mm diameter failed in the triple-cleft fracture mode may be as a result of stronger particle contact areas throughout the specimen.

4.3.3 Weibull Statistical Treatment of the Strength of Clay

To test the variability in the fracture strengths for clay, the Weibull moduli, m of all the tested samples were calculated using the formula, $m = \frac{1.11}{cv}$ ($cv = coefficient$ of variation obtained from the strength data) proposed by [8] and the graphical method proposed by [7]**.** The relationship proposed by [8] gave more consistent values of Weibull moduli for both disc-diameters in comparison with the values of Weibull moduli obtained using the graphical method proposed by [7]**.**

Clay discs of nominally 23mm diameter generally gave higher values of Weibull moduli vis a' vis the values obtained for discs of nominally 29mm diameter regardless of the fact that both test-diameter discs indicated scatter in the strength value, "which is common for brittle materials". The reason for the high values of Weibull moduli obtained for samples of nominally 23mm diameter may be due to the presence of minimal clustering of flaws throughout the sample. On the other hand, inconsistent clustering of flaws which engender weak and variable strength may be responsible for the low values of Weibull moduli obtained for samples of nominally 29mm diameter. Flaws of similar severity were perhaps sampled in the 23mm diameter discs hence the higher Weibull moduli obtained.

4.3.4 Effect of Porosity on the Diametral Strength of Clay

A slope of -0.042 was obtained for samples of 23mm diameter whilst samples of 29mm diameter gave a slope of -0.061 for the plot of $In\sigma_t$ against $porosity$ (%) as shown in Fig. 3.3. The negative slope obtained for the plot of $In\sigma_t$ against $porosity$ (%) showed that pores were singularly effective as initiation sites for failure.

The negative gradient obtained for the plot of $In\sigma_t$ against $porosity$ (%) shows that the clay obeys the relationship proposed by [13, 8]**.** It indicated that pores were singularly effective as initiation sites for failure (i.e., pores were non-uniformly distributed); in contrast to the result previously reported by [3] for silicon nitride.

V. CONCLUSIONS

1. A decrease in the diametral compression strength of clay was observed during this investigation and this was found to be thickness dependent.
2. Plane stress condition was obtained for clay of 3-8mm disc thickness for samples of nominally 29mm diameter. However, this condition did not extend to the thickness of 7mm (9.28MPa) for samples of nominally 23mm diameter.
3. Greater numbers of the 29mm diameter samples failed in the normal tensile fracture mode while more samples of the 23mm diameter failed in the triple-cleft fracture mode.
4. In general, 23mm diameter samples gave higher values of Weibull moduli vis-à-vis the values obtained for discs of 29mm diameter, which indicated that flaws of similar severity were sampled in the 23mm diameter discs.
5. Pores were found to be effective as initiation sites for failure.
6. It has been shown that the diametral compression disc test is a simple test method for providing tensile strength for clay economically.

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