

Mechanical behaviour of a low-clay translucent whiteware

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Abstract

A novel low-clay translucent whiteware body, using mostly non-plastic prefired materials and only a small amount of clay, was fabricated by slip casting and the effect of slip's solid content and sintering temperature on the mechanical behaviour was investigated. The degree of densification in the sintered specimens was determined by measuring the bulk density. The mechanical behaviour was determined by measuring the flexural strength and fracture toughness. Young's modulus and hardness were also measured. X-ray diffraction (XRD) and scanning electron microscopy (SEM) studies were carried out to analyse the microstructure.

The flexural strength and fracture toughness increase with both increasing the slip's solid content and the sintering temperature up to a certain level, but further increase in solid content and sintering temperature had an adverse effect on the properties. The maximum flexural strength (~ 135 MPa) and fracture toughness (~ 1.85 MPa m^{1/2}) values were attained with specimens produced from a slip having 45 vol.% solid content at a sintering temperature of 1350 °C. It was found that the amount and distribution of closed pores, their size and possible link with each other control the flexural strength and fracture toughness of the low-clay translucent whiteware.

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1. Introduction

Two high quality whitewares, bone china and hard porcelain, are currently in production and they dominate the top end of the tableware market. Both bone china and hard porcelain are white and translucent. Bone china is highly crystalline material¹ (about 70% crystalline and 30% glass) which is resistant to edge chipping.² Strengths of ~ 100 MPa^{1,3} and a fracture toughness of ~ 2 MPa m^{1/2} (Ref. 4) have been measured. Unfortunately, the glazes used on bone china tend to be more easily scratched than those on hard porcelains. Hard porcelain is highly glassy (approximately 70% glass and 30% crystalline material) and its edges tend to chip easily.⁵ It has lower toughness value and strength than bone china: a K_{IC} value of ~ 1.2 – 1.6 MPa m^{1/2} (Refs. 4 and 6) has been reported; strengths are typically in the range of 40–80 MPa.^{3,6–9} However, the glazes on hard porcelain, which are rich in silica, are both chemically highly durable and abrasion resistant.

A novel translucent whiteware which has an appearance comparable with bone china or hard porcelain, to be chip resistant like bone china, and to be coated with a glaze similar to those applied to hard porcelain was developed recently.^{10,11} The novel whiteware differs from conventional whitewares in a way that is made by using mostly (88% of the whiteware) non-plastic prefired materials. A further feature of this whiteware was the use of low clay content of 12% rather than the usual 30–60%. This has been done to overcome the deleterious effects that arise from anisotropic firing shrinkage, which results from clay particle alignment. However, low clay usage has lowered the green strength which was measured and found to be ~ 0.7 MPa.¹² This material is going to be referred as the low-clay whiteware from onwards. The low-clay whiteware was to be composed of $\sim 65\%$ anorthite ($\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) as the major phase with $\sim 10\%$ mullite ($3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) and $\sim 25\%$ glass as minor phases. A high crystalline to glass ratio is required to maximise the strength.¹³

Mechanical behaviour of bone china and hard porcelain has long been studied and those studies were reviewed in detail in the literature.^{4,6} Since the low-clay whiteware was

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developed to combine the best features of bone china and hard porcelain its mechanical behaviour as well as the other technological properties should be established. Therefore, this study was carried out aiming to assess the mechanical behaviour of the low-clay whiteware.

Flexural strength is the most widely used method to characterize the mechanical behaviour of ceramic materials because of its simplicity. On the other hand, even tough fracture toughness is a very important mechanical property; the data is very limited on whitewares.⁴ Strength (σ_f) and fracture toughness (K_{IC}) can be related for a ceramic material which fails in a brittle manner using the basic equation:

$$\sigma_f = \frac{K_{IC}}{Yc^{1/2}} \quad (1)$$

where c is the flaw size and Y is the geometry factor. Hence, the strength of a ceramic material is a function of its toughness and flaw size. Therefore, the material should satisfy two objectives in order to obtain better strength behaviour: maximize the fracture toughness and minimize the flaw size.⁹

An increase in fracture toughness of whiteware can be achieved by increasing the crystalline/glassy phase ratio. Besides an increase in crystallinity, crack deflection, i.e., propagation of crack front around grains and second phase formation is the main mechanism that contributes to an increase in fracture toughness.¹⁴ Therefore, an increase in crystal size and second phase formation are desirable. It was reported by several authors that alumina,¹⁵ cristobalite,¹⁶ silica fume,¹⁷ rice husk ash^{18,19} and mullite⁷ in whitewares each strengthen the fired body.

Flaw size, which may be assumed to be a function of porosity,²⁰ varies considerably for whiteware material in which porosity may be as high as 15%. Flaw size may be reduced by decreasing the porosity of the material and by making the pores smaller and homogeneously distributed throughout the material. In order to reduce the pore size, a high packing density of the green body and optimization of the processing conditions are essential.^{2,21}

Vickers indentation technique is a simple and economic method for the determination of the fracture toughness of ceramic materials.²² In this method, the specimen surface is indented and immediate post-indentation crack size is measured. Cracks may form in two different ways depending on the fracture toughness of material and the indentation load: Palmqvist and median.^{23,24} A crack with $c < 3.5a$ is usually in the Palmqvist region and $c \gg a$ is assumed to be well-developed median crack.²⁵ Various equations are proposed in order to determine the fracture toughness from Vickers indentation cracks for both crack geometries.

Niihara²⁶ obtained an empirical equation for Palmqvist cracks. This equation is selected because it best describes the cracks formed in this study.

$$\left(\frac{K_{IC}\phi}{Ha^{1/2}} \right) \left(\frac{H}{E\phi} \right)^{0.4} = 0.035 \left(\frac{l}{a} \right)^{-1/2} \quad (2)$$

where H is the Vickers hardness, E is Young's modulus, $2a = d$ is the diagonal of the indentation, ϕ is the constrain factor, and l is the crack length emanating from the ends of the diagonal indentations. By arranging the above equation one can obtain the fracture toughness

$$K_{IC} = 9.052 \times 10^{-3} (H^{3/5} \cdot E^{2/5} \cdot d \cdot l^{-1/2}) \quad (3)$$

Blendell⁴ obtained an empirical equation using curve fitting method to experimental data for Palmqvist and median cracks. Blendell equation is included in this study for comparison purposes because it was employed by Batista et al.⁴ in their study on fracture toughness of bone china and hard porcelain.

$$K_{IC} = 0.0303 (Ha^{1/2}) \left(\frac{E}{H} \right)^{2/5} \log \left(8.4 \frac{a}{c} \right) \quad (4)$$

where c is the crack length from the centre of the indentation to the crack tip.

In the present study, the low-clay whiteware body slips having different solid content were prepared and specimens were produced by slip casting. The mechanical properties of the low-clay whiteware have been investigated as functions of slip's solid content and sintering temperature using sintering study, flexural strength and fracture toughness measurement, X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDX) techniques. The results obtained from the low-clay whiteware are also compared to those of hard porcelain and bone china.

2. Experimental procedure

Test specimens were prepared by slip casting with slips having different solid content (40, 42.5, 45, 47.5 and 50 vol.%). The low-clay whiteware slip has been formulated as 50 wt.% coarse prefired material, 38 wt.% fine prefired material, 6 wt.% China clay (Standard Porcelain English China Clay), and 6 wt.% Ukrainian ball clay (WBB). Compositions of the clays are given in Table 1. The composition and preparation method of the prefired materials were explained in detail elsewhere.^{10,11} The particle size distributions of coarse and fine prefired materials are given in Fig. 1. The particle size distributions of the milled powders were analysed by a

Table 1
Chemical composition of clays

	Al ₂ O ₃	SiO ₂	CaO	MgO	Na ₂ O	K ₂ O	Fe ₂ O ₃	TiO ₂	L.O.I.
Standard Porcelain English China Clay	37.0	48.0	0.07	0.3	0.1	1.6	0.65	0.02	12.5
Ukrainian ball clay	34.0	49.0	0.3	0.3	0.2	1.2	1.6	1.3	12.0

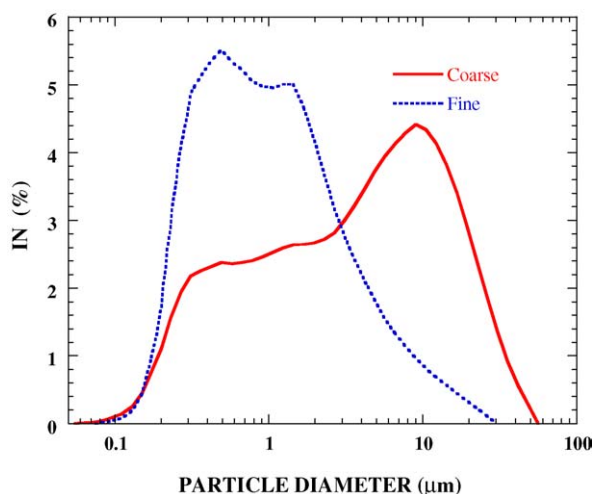


Fig. 1. The particle size distributions of coarse and fine prefired materials.

Coulter Multisizer Accu Comp 1.19 particle size analyser. For the slip preparation first, fine prefired material, China clay and Ukrainian ball clay were mixed with distilled water in a porcelain pot half filled with alumina milling balls for about 1 h. Then, the coarse prefired material was added and milled together for further 15 min. In order to produce a castable slip the viscosity should be adjusted. This is done by adding appropriate amounts of deflocculant while the slip was being mechanically stirred. On the completion of viscosity adjustment the slip was transferred to plastic container and aged for at least a week before any casting carried out.

Cylindrical rod specimens were cast to quantify the changes in fired densities, flexural strength, and fracture toughness with solid content and sintering temperatures. To do that a particular slip was poured in a cylindrical plaster mould and the amount of water absorbed by the mould walls was progressively replaced with slip until the whole mould was filled with the green cast specimen and a solid rod was obtained. The green cylindrical rods were 10 mm in diameter and approximately 80 mm in length. After withdrawal of the mould, specimens were dried at 110 °C for an overnight. Cylindrical rods produced with various solid content were then fired at temperatures from 1330 to 1410 °C with soaking times of 3 h, in the Nabertherm chamber kiln.

The densities after firing were determined from the volume of rods and their masses. The flexural strength of sintered cylindrical rods were measured with an electronic universal tester (Model 5569, Instron Ltd.) by a three-point bending test with a lower span of 60 mm and crosshead speed of 1 mm/min, based on ASTM standard C1161-90. The surface conditions of tested specimens were as-sintered. The number of samples used varied between 8 and 10 for each solid content and sintering temperature.

Vickers microhardness values were determined on the polished surface of the fired samples using a microhardness tester with a Vickers indenter (Instron Wolpert® Testor 2100®). The samples were submitted to 10 loads of 9.8 N for 13 s

on each indentation. The cracks were measured using the microscope attachment on the microhardness tester immediately after indentation. Crack measurements were only made on indents that were well defined without chipping and for which the cracks did not terminate at pores. In addition it was ensured that crack dimensions met the requirement of $c < 3.5a$ for the validity of Eqs. (3) and (4). After indentation, the surface layers of a few randomly chosen samples were polished away to confirm that the assumed Palmqvist crack system was created. For Palmqvist cracks the crack will become detached from the inverted pyramid of the indent, whilst median cracks the crack will always remain connected.

In order to use the dedicated equations for the calculation of fracture toughness it is necessary to have a value of Young's modulus which was measured by the three-point bending test, using a jig for accurately measuring the displacement of the center of each test bar with a differential transformer (LVDT, RDP Electronics, Model E25). Young's modulus measurements were carried out on rectangular bars which were polished to have dimensions of approximately 4 mm × 5 mm × 65 mm with a lower span distance of 50 mm and crosshead speed of 1 mm/min.

The crystalline phases present in the microstructure of sintered samples were identified by XRD and SEM techniques. For XRD, powdered form of sintered samples were scanned from $2\theta = 10\text{--}70^\circ$, at a scanning speed of $1^\circ/\text{min}$, using a RIGAKU 2200 DMAX diffractometer (with Cu K α radiation) at 40 kV and 40 mA. The diffractometer was calibrated with a silicon standard before use. For SEM observations, specimens were polished using 6, 3, 1 μm diamond pastes after grinding with silicon carbide powders as abrasive and lubricated with water. The polished surfaces were chemically etched in 5% HF solution for 2 min. A Phillips XL30 SFEG scanning electron microscope equipped with EDAX detector (operating at 20 kV) was used for microstructural examination of samples with secondary electron images (SEI) used predominantly. Microanalysis was performed using the embedded EDX digital controller and control software.

3. Results and discussion

Two processing parameters that would influence the mechanical behaviour of the novel whiteware investigated during this study were the solid content of the slip and the sintering temperature.

In order to maximise the solid volume fraction of a casting slip it is necessary to optimise the particle size distribution of the starting material. As it was shown by Ferreira and Diz²⁷ and Taruta et al.,²⁸ that it is possible to obtain high density slip cast bodies by using bimodal particle size distributions in which fine and coarse particles are combined in an appropriate proportion and size ratio. The par-

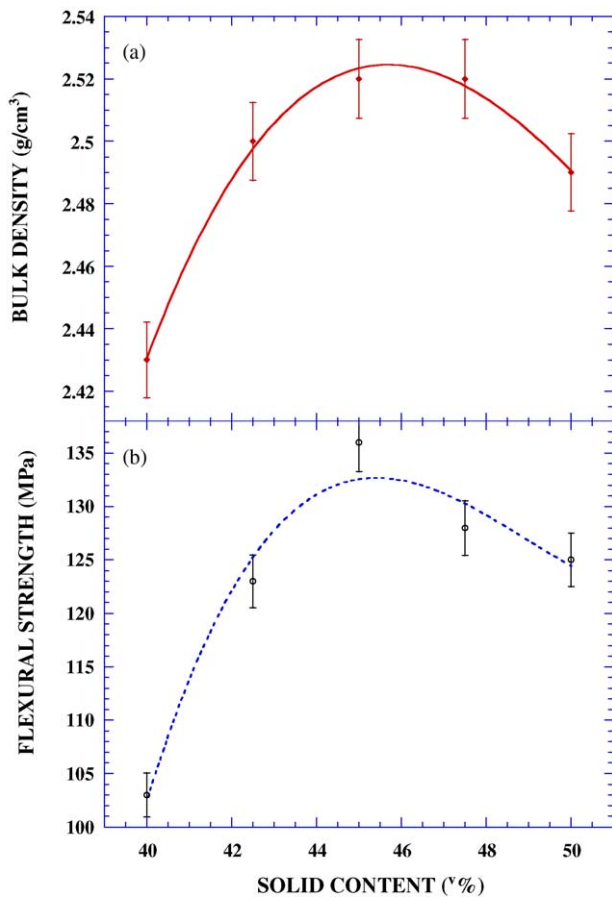


Fig. 2. (a) Densification and (b) flexural strength behaviour of the low-clay whiteware as a function of slip's solid content.

ticle size distributions of coarse and fine prefired materials used in this study are given in Fig. 1. The analysis show a bimodal distribution presenting two maximum points, which are centred at around $1\ \mu\text{m}$ (fine prefired) and $10\ \mu\text{m}$ (coarse prefired). The ratio between coarse and fine particle sizes is around 10, which is large enough for efficient particle packing.²¹

The effect of solid content on the densification and flexural strength behaviour of slip cast specimens is given in Fig. 2a and b where the bulk densities and flexural strength of the specimens after firing at $1350\ ^\circ\text{C}$ for 3 h are plotted against the solid content of the slip. As the solid content increases, bulk density and flexural strength of the body continued to increase, reached a maximum and then decreased. It appears that there is a limit on solid content of the slip to achieve optimum results. Up to 45 vol.% solid content the results show gradual improvements however, further increase in slip's solid content has an adverse effect on the properties. This behaviour could be explained in terms of changes in packing arrangement with the solid content. Slips having solid content up to 45 vol.% would allow the particles to rearrange themselves in a more closely packed structure. However, the

coarse and fine particles in slips having higher solid content than 45 vol.% may form flocs which creates more open structure which in turn produces lower bulk density during sintering.^{29,30}

The amount of closed porosity was calculated by subtracting the ratio between bulk density and powder density from unity. Powder density of a sintered specimen of the low-clay whiteware was measured by He pycnometer and found to be $2.86\ \text{g/cm}^3$. The maximum bulk density attained with specimens produced from slips having 45 vol.% solid content at a sintering temperature of $1350\ ^\circ\text{C}$ was $2.52\ \text{g/cm}^3$. The calculated volume percent of closed porosity is found to be 12. This value represents the minimum porosity level attained in this study and showed some variations both with solid content of the slip and sintering temperature reaching up to 15%. The difference between minimum and maximum density values would produce approximately 20% change in the amount of porosity. Fig. 3a–d is the SEM micrographs of the sintered samples produced from slips having solid contents of 40, 42.5, 45 and 50 vol.%, respectively, showing the effect of solid content on morphology of porosities. The sample produced from 40 vol.% solid containing slip (Fig. 3a) contained small and generally interconnected large pores both in large numbers as a result of incomplete densification. It was observed that the size and the number of large pores appeared to decrease as the solid content was increased up to 45 vol.%. The 45 vol.% solid content sample contained essentially isolated small round pores in rather small numbers (Fig. 3c). However, when the samples were produced from a slip above 45 vol.% solid content, it was observed that the size of pores appeared to increase again (Fig. 3d).

Fig. 4 shows the X-ray diffraction traces of samples sintered at $1350\ ^\circ\text{C}$ as a function of solid content. The crystalline phases identified in all the specimens are anorthite ($\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) being the major crystalline phase and mullite ($3\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$) as minor crystalline phase labelled as (a) and (m), respectively. Some glassy phases are also present. The peak intensities of crystalline phases did not significantly alter with the change of solid content indicating that the degree of crystallinity was similar. The SEM image given in Fig. 5a shows the highly crystalline microstructure of the low-clay whiteware. Fig. 5 b and c shows anorthite and mullite crystals, respectively, in higher magnification. The EDX analysis in Fig. 5d and e confirms the formation of anorthite (marked as a) and mullite crystals (marked as m). The glassy phase (marked as g), which is etched away to reveal the crystalline phases, are distributed between the crystals.

To observe the effect of sintering temperature on densification and flexural strength behaviour specimens with 45 vol.% solid content were sintered from 1330 to $1410\ ^\circ\text{C}$ with $20\ ^\circ\text{C}$ intervals. The results are shown in Fig. 6a and b. Upon heating, bulk density of the whiteware increased, reached a maximum, and then decreased. The decrease in density after reaching maximum is attributed to "bloating" (i.e., pore volume

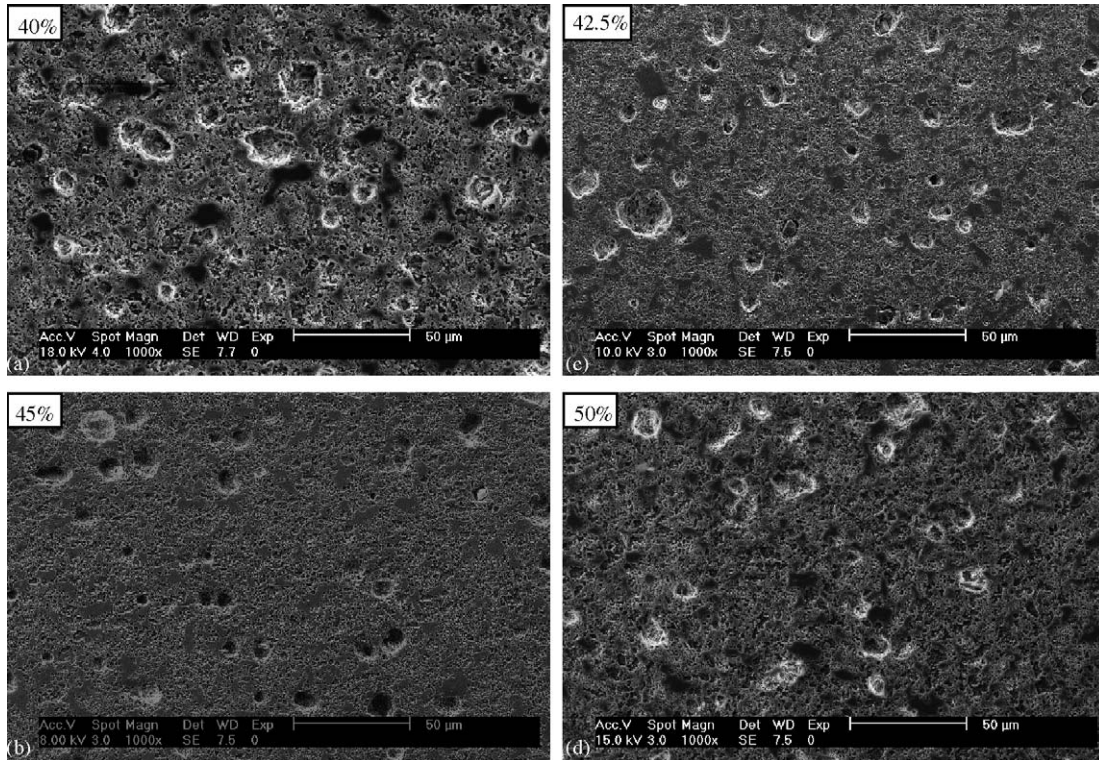


Fig. 3. SEM micrographs of the sintered samples of the low-clay whiteware produced from slips having solid contents of 40, 42.5, 45 and 50 vol.%, respectively, showing the effect of solid content on morphology of the porosities.

expansion), which arises from higher pressures (at high temperatures) of gases entrapped within closed pores. The flexural strength of the whiteware varied proportionally to its bulk density. The flexural strength increased with an increase in sintering temperature up to 1350 °C, attained a maximum and upon further heating decreased with a corresponding decrease in density. The relationship between the flexural strength and bulk density can be clearly seen in Fig. 7 where the data from varying slip content and sintering temperature experiments are pooled. The increase in strength is approximately 30%.

The maximum flexural strength obtained from low-clay whiteware is about 135 MPa which is higher than that of

bone china and hard porcelain. The high strength value of bone china compared to hard porcelain was attributed to its high crystalline content.⁴ Previous quantitative X-ray analysis study³¹ on prefired material had shown that this material contained ~76% anorthite and ~8% mullite crystals with the remainder being glass stating highly crystalline structure. However, the specimens produced from different amounts of solid containing slip and sintered at different temperatures showed some measurable differences in their flexural strength. The high strength and the variation in strength behaviour could not be explained only by its high crystalline content. SEM results clearly support that reduction in porosity plays an important role for the increase in flexural strength of the low-clay whiteware.

The strength is a function of the fracture toughness and the characteristic defect size of the material. Hence, the increase in strength may be explained by an increase in fracture toughness, a decrease in pore size or both. In order to correlate the changes in flexural strength with the fracture toughness it was necessary to determine the fracture toughness of the low-clay whiteware. The fracture toughness values were determined by means of Vickers indentation method. It was observed for the applied load, 9.8 N, that Palmqvist (Fig. 8) rather than median cracks developed; therefore, the models given in the introduction section were chosen for calculating the fracture toughness. To be able to calculate the fracture toughness by using Eqs. (3) and (4) the values of Vickers

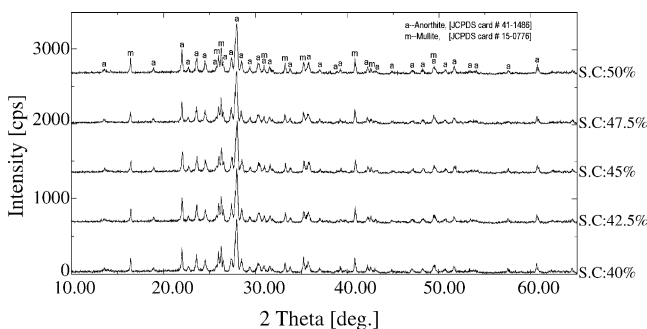


Fig. 4. XRD traces of low-clay whiteware samples as a function of slip's solid content showing anorthite and mullite formation labelled as a and m, respectively.

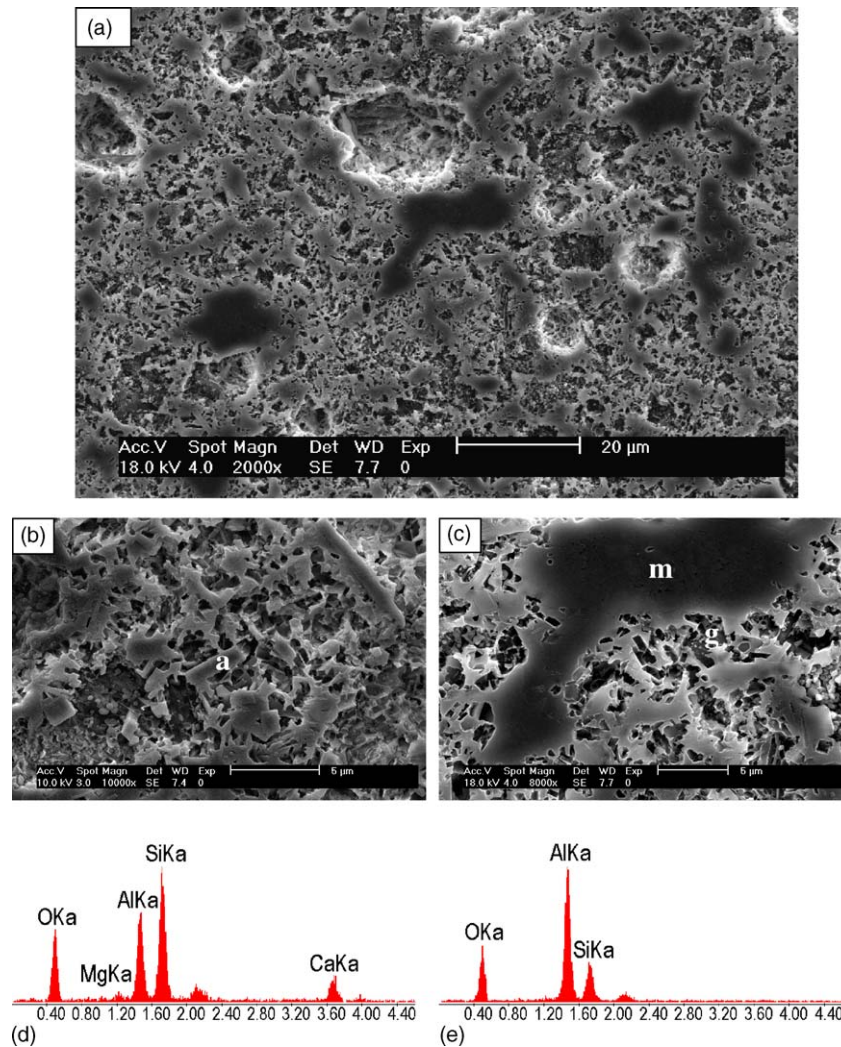


Fig. 5. (a) SEI of an etched low-clay translucent whiteware sintered at 1350 °C showing highly crystalline microstructure and (b) anorthite, (c) mullite, and glass formation labelled as a, m, and g, respectively. EDX spectra obtained from (d) anorthite and (e) mullite crystals.

hardness and Young's modulus are required. Table 2 provides Young's modulus and Vickers hardness values of the low-clay whiteware as functions of slips solid content and sintering temperature. It was observed that Young's modulus values show some variation both with solid content and sintering temperatures. This is because samples were containing different amounts of porosity, presence of which, as in general degrades Young's modulus.³² On the other hand, such a correlation was not observed in hardness values because measurements were made only at sites free of porosity.

Using Eqs. (3) and (4), the fracture toughness values given in Fig. 9a and b were obtained. Both methods produced very

similar results. The fracture toughness showed some variation both with solid content and sintering temperature. X-ray analysis showed that there were no measurable differences in the degree of crystallinity in these samples; hence, the increase in toughness cannot be attributed to increasing crystallinity or changing ratios of anorthite and mullite crystalline phases. On the other hand, there is considerable increase in the density of the material. Fig. 10 shows the relationship between the fracture toughness and bulk density. A slight increase, approximately 10%, in the fracture toughness was observed with increasing bulk density. Assuming a linear relation,³³ and extrapolating the data presented in Fig. 10,

Table 2
The Young's modulus and Vickers hardness values of low-clay whiteware as functions of slip's solid content and sintering temperature

	Solid content of slip (vol.%)				Sintering temperature (°C)		
	40	42.5	45	47.5	1330	1350	1370
Elastic modulus (GPa)	74.6	77.6	80.3	76.9	75.0	80.3	78.9
Vickers hardness (GPa)	6.5	6.7	6.5	6.7	6.6	6.5	6.6

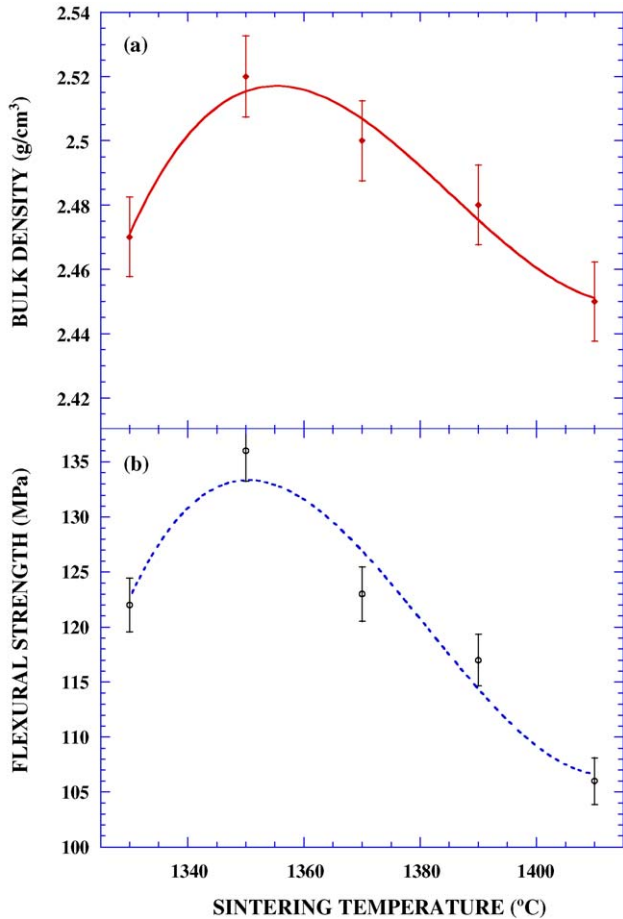


Fig. 6. (a) Densification and (b) flexural strength behaviour of the low-clay whiteware as a function of sintering temperature.

it would be calculated that if this material had been produced 100% dense, its toughness would have been $2.3 \text{ MPa m}^{1/2}$.

If the flaw size remains constant, strength–toughness relation is expected to be linear as Eq. (1) indicates. Hence, a 30% increase in strength cannot be explained by a 10% increase in toughness only. A decrease in flaw size with in-

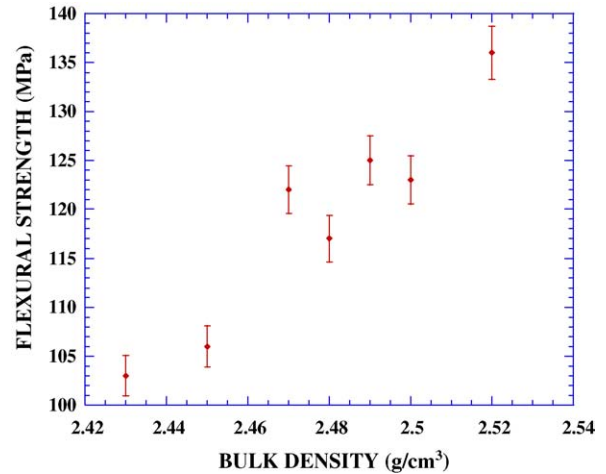


Fig. 7. The relationship between bulk density and flexural strength of the low-clay whiteware.

creasing density²⁰ (decreasing amount of porosity) may also contribute to the increase in strength.

Eq. (1) can be rearranged and for simplicity the flaw size can be assumed to be a function of porosity, $c(P)$, hence

$$Y^2 c(P) = \left(\frac{K_{IC}}{\sigma_f} \right)^2 \quad (5)$$

Eq. (5) was employed to estimate the change in flaw size with porosity using the minimum and maximum values of strength and toughness combinations assuming the geometry factor, Y , remains constant. Calculations showed that the flaw size decreased 30% with increasing density. Since pores act as the crack initiation sites, characteristic flaw size becomes smaller with the shrinkage of the pores; hence, the strength of the material increases. As explained earlier, the change in porosity with the increase of density is approximately 20% which would result in only 7% decrease in pore diameter, calculated assuming that the pores are spherical. Clearly, the decrease in flaw size (30%) is much greater than the decrease in pore size (7%). The reason behind this discrepancy may

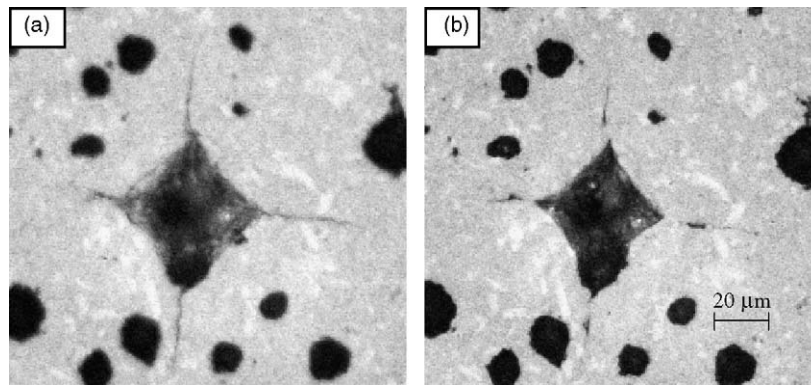


Fig. 8. Surface appearance of cracks developed from Vickers indents (a) before and (b) after polishing. The discontinuity of the cracks at the corners of the indent after polishing indicates Palmqvist cracks.

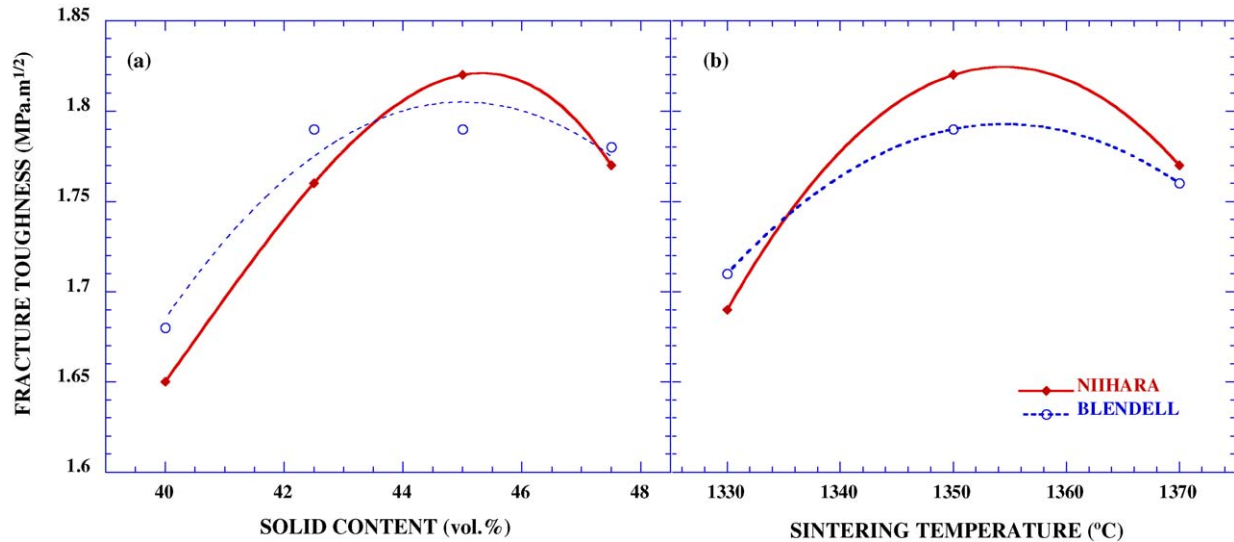


Fig. 9. The variation of fracture toughness of the low-clay whiteware with (a) slip's solid content and (b) sintering temperature.

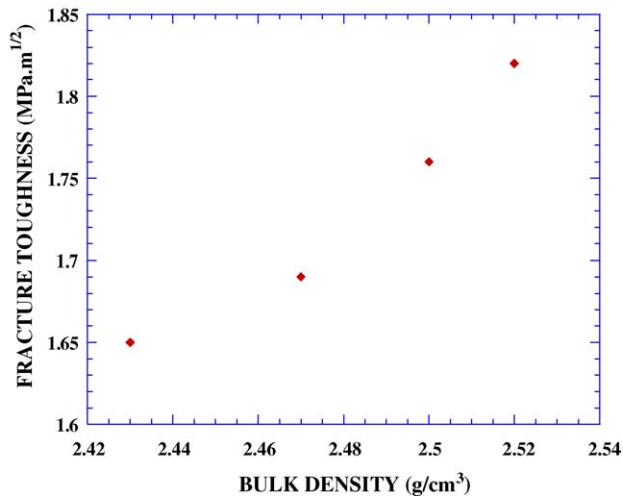


Fig. 10. The relationship between bulk density and fracture toughness of the low-clay whiteware.

be due to either larger pores shrink faster or links between adjacent pores are lost with increasing density.

4. Conclusions

Mechanical properties of a novel low-clay translucent whiteware produced by slip casting are studied. Solid content of the slip and the sintering temperature were the study parameters. XRD analysis showed that the microstructure of the material remains essentially the same for the studied process parameters while density measurements and SEM analysis showed that the porosity of the materials reaches a minimum and then increases both with increasing slip's solid content and sintering temperature. It was measured that the material's flexural strength is 135 MPa and its fracture toughness is $1.85 \text{ MPa m}^{1/2}$ for optimum processing parameters, which are

45 vol.% slip's solid content, 1350°C sintering temperature and resulting 12% porosity. Hence, the strength of the novel material is higher than both bone china's and hard porcelain's while its fracture toughness is in between the values of bone china and hard porcelain. It is found that both the strength and the fracture toughness of the material is a function of porosity of the material. As porosity decreased the flexural strength and the fracture toughness increased. The increase in fracture toughness is attributed to increasing surface area with decreasing porosity, and the increase in strength is attributed to increasing fracture toughness, but more importantly to the decreasing flaw size with decreasing porosity.

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